

# **Collapse patterns in porous materials under degradation fields**

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## **Statement of originality**

This is to certify that to the best of my knowledge, the content of this thesis is my own work. This thesis has not been submitted for any degree or other purposes.

I certify that the intellectual content of this thesis is the product of my own work and that all the assistance received in preparing this thesis and sources have been acknowledged.

Date: 31 January 2024

Leonardo Andrée Crespo Párraga



## **Authorship attribution statement**

This thesis includes materials that have been previously published or submitted for publication. Chapter 3 is currently awaiting publication and Chapter 4 presents results previously published in (Crespo-Parraga et al. 2022). The latter publication was included in this thesis as Appendix A.

In both these documents, the author of this thesis (Leonardo Andr  e Crespo P  rraga) is the main author of the manuscripts, in collaboration with his two supervisors, Dr Fran  ois Guillard and Prof Itai Einav. Leonardo performed the experiments and drafted the manuscripts with the guidance of his supervisors. All authors took part in the design of the studies, the analyses of the data, and the interpretation of the results.

In the future, the author and supervisors of this thesis intend to publish two new journal articles based on the findings of Chapters 4 and 5.

As the primary supervisor for the candidature upon which this thesis is based, I can confirm that the authorship attribution statements above are correct.

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## **Abstract**

Porous materials consist of a solid skeleton with empty spaces (pores). They are ubiquitous in nature and understanding their behaviour is an interest of various fields, including environmental science, mining, and material processing. Emerging collapse patterns in deforming porous media can significantly alter their properties. Recent findings suggest these patterns can arise from material degradation. This thesis presents an experimental study on collapse patterns in a diverse range of materials and degradation fields.

We first investigate the emergence of a novel collapse pattern, consisting of cycles of slow and sudden compaction, as a ubiquitous collapse mechanism in porous media. Three key requirements for these ‘creep-quake cycles’ are identified: a collapsible length scale in the material, sufficient compressive stress, and a non-uniform degradation field. We then develop a theoretical model that recovers the cycles based on these requirements.

After asserting the ubiquity of this mechanism, the precise conditions for its emergence in calcareous sands are investigated. This reveals that grain size distribution plays a crucial role in creep-quake collapse, outweighing the influence of grain shape. In addition, we uncover a range of stress and degradation rates in which creep-quake cycles can emerge.

Finally, these findings are related to real geological scenarios with an investigation on sand samples under compression and a transversal acid flow. Here, the collapse mechanisms of chemical degradation-induced sinkholes are examined. Remarkably, creep-quake cycles remain relevant even in these different loading conditions and are intertwined with other failure mechanisms associated with sinkhole collapse.

The findings of this thesis contribute to the understanding of collapse patterns in degrading porous media. They provide insights for future research on monitoring these patterns to forecast and prevent impending catastrophic collapse events.

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## CHAPTER 1

### **Introduction**

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From an everyday sponge to soil on the ground, pyroclastic rocks, or asteroids in outer space, porous materials are omnipresent in nature and day-to-day life. They also have broad industrial applications, such as their use as sound-absorption or heat-insulation building elements (Liu and Chen 2014) or in gas storage systems (Chen et al. 2022). Despite their diversity, these materials are unified by having a network of connected or disconnected pores, which are formed by void spaces within a solid skeleton. These voids play a major role in their behaviour, and can result in the formation of intricate deformation patterns when the materials undergo compaction or degradation processes. Instances of such patterns include field and laboratory observations on bands of compaction in porous rocks (Aydin and Johnson 1978; Rudnicki 2003) and in degrading brittle porous media (Einav and Guillard 2018).

The degradation of porous media is a common diagenetic process. It is frequently observed in materials of a geological origin (geomaterials), such as in the melting of ice formations, salt deposits dissolving in water or reactive rocks and soils exposed to acidic solutions. In turn, degradation processes in geomaterials can lead to instabilities or to their collapse, which can result in large-scale property damage, irreversible environmental impacts, threats to health and well-being, as well as human fatalities. For instance, the chemical dissolution of underground reactive soil deposits can lead to sinkhole formation (Waltham et al. 2005). Similarly, acidified tailings (mining waste), particularly sulphur-rich ones, can result in contaminant leakage and instabilities that could induce the collapse of their retaining structure (Lottermoser and Ashley 2005; Alonso and Gens 2006). Furthermore, underground dissolution processes can cause damage to subsurface CO<sub>2</sub> storage systems (Espinoza et al. 2011), and submarine landslides (Urgeles and Camerlenghi 2013). The impact of such collapse events highlights

the importance of developing an accurate understanding on collapse patterns and mechanisms in porous media, particularly geomaterials, under degradation fields.

At the forefront of the research in this topic lies the discovery of a novel collapse mechanism in a brittle porous medium, puffed rice (Einav and Guillard 2018). The mechanism consists of regular cycles of creep interluded by instantaneous collapse events coined ‘ricequakes’, which resemble compaction bands and originate from material degradation. This regular collapse pattern emerged in laboratory observations of a compressed material column under partial saturation. Since these collapse pattern observations were made solely on puffed rice, directly speaking they have few applications outside food production and handling. As a material, however, puffed rice holds the potential to be a model medium from which collapse mechanisms could be extrapolated to other brittle porous media. In fact, there is much to gain from this discovery, as the physical mechanisms driving the compaction behaviour in puffed rice can be analogous to those found in the more general realm of degrading porous media. Hence, this thesis will embark on a campaign to expand our knowledge on ‘ricequake’ compaction, and identify its role in the collapse mechanics of other porous media under various fields of degradation.

In summary, the goal of this thesis is to answer three distinct research questions:

- (1) Are repetitive compaction quakes limited to puffed rice, or are they a widespread phenomenon in brittle porous media under degradation?
- (2) If so, what variables control their emergence in geomaterials?
- (3) Finally, what are their implications and relevance in real geotechnical scenarios?

To begin addressing these research questions, Chapter 2 will present a literature review on porous materials. Throughout this chapter, we will discuss their mechanical response, as well as the effects of saturation and acidic degradation on their properties. Each of the above three research questions will then be addressed individually in the following chapters of the thesis. Chapter 3, which is prepared for publication, will present our findings on the ubiquity of ‘creep-quake’ cycles in porous media. This chapter extends the collapse mechanism identified in puffed rice to a diverse array of porous media under vastly different

conditions of degradation. Following this, Chapter 4 will shift the focus of our investigations to calcareous sands with an in-depth study on the conditions of emergence and evolution of creep-quake cycles in them. Portions of this chapter have been published in (Crespo-Parraga et al. 2022), and the full paper is included as an appendix (Appendix A). Finally, the relevance of creep-quake cycles in geotechnical scenarios will be examined in Chapter 5 with an experimental exploration on sinkhole collapse. This investigation includes experiments on sand samples under stress and a transversal flow of acid. The collapse behaviour observed in the degradation-induced sinkholes will be described and these results will be used to draw connections between sinkhole collapse and the creep-quake mechanism.

## CHAPTER 2

### Literature review

---

#### 2.1 Introduction

The emergence of intricate patterns in collapsing porous media has a long research history. In fact, the identification and use of such patterns has been the focus of diverse studies on a variety of topics, such as snow avalanches nucleation (Heierli et al. 2008; Gaume et al. 2017; Casals and Salje 2021), geophysics and earthquakes (Omori 1895; Green et al. 1990), permeability changes in petroleum extraction processes (Baxevanis et al. 2006), geological kinematic patterns (Rudnicki and Rice 1975; Du Bernard et al. 2002; Baud et al. 2004), as well as biological (Gibson et al. 1982; Eckstein et al. 2023) and structural (Papka and Kyriakides 1998; Kader et al. 2016) collapse mechanics.

The novel collapse mechanism presented in (Einav and Guillard 2018) links the emergence of cyclical compaction (ricequakes) to a saturation gradient. In turn, these observations open a new line of research in the study of degradation fields driving the formation of regular collapse patterns in a porous medium. While these findings are fairly recent, the applied conditions under which the ricequake pattern was identified have been extensively studied in the past. Establishing a well-rounded understanding of these applied conditions is an imperative first step in developing an accurate description of the mechanisms driving collapse patterns in degrading porous media. Therefore, this chapter will review the relevant available literature on the topics of porous materials, their constitutive response, and the effects that saturation and degradation processes have on their properties.

To begin, this literature review will summarise our current understanding on the response of porous and granular media to stress-strain changes, including localised deformations resulting from specific applied loading conditions. Subsequently, we will discuss the modelling of fully and partially saturated porous materials, as well as the impacts of saturation and chemical degradation on their properties. Finally, these topics will be tied together by reviewing recent findings in which their coupling results in localised degradation-induced collapse events.

## **2.2 Mechanical response**

Porous media consist of a solid matrix and voids (pores), which can be empty or filled with fluids. Granular materials are a specific kind of porous medium that consist of a collection of individual independent particles (grains). In granular materials, the body and surface forces acting on the particles, as well as their spatial arrangement, dictate their overall bulk behaviour (Lois 2020). Thus, simple grain-to-grain interactions can lead to a complex macroscopic response. While this project mainly focuses on geomaterials, such as rocks and soils, which are naturally occurring granular porous materials, most of the concepts reviewed in this chapter encompass a vast diversity of materials such as powders, grains, foams, pills, beads, bones, or even breakfast cereals. This section will begin with a review on the mechanical response of soils, followed by a summary on collapse patterns that emerge under specific stress conditions and their effects on the mechanical properties of the material itself.

### **2.2.1 Soil mechanics**

The behaviour of soils is typically modelled following variables that represent its current state and that can be used to predict its future behaviour with a set of constitutive laws. These variables generally include density, stress, particle size distribution and breakage, among others. Soil failure is commonly characterised as the point in deformation at which its grains flow as a frictional fluid, exhibiting distortions without a corresponding volume or stress change (Schofield and Wroth 1968). The strength of soil can be defined as the state at which this occurs.

Conventional plasticity models rely on the existence of a ‘critical state’ that all soils reach in order to fail, a concept first introduced in (Roscoe et al. 1958). In these models, the critical state is represented as a curve in the three dimensional  $q$ - $p'$ - $v$  space, denominated the critical state line (CSL), where  $q$  is the deviatoric stress,  $p'$  is the effective mean stress, and  $v$  is the specific volume of the soil (Schofield and Wroth 1968). Several plasticity models, such as the Cam-Clay family of models (Roscoe and Burland 1968; Schofield and Wroth 1968), make use of a ‘yield surface’ along with the CSL to model the plastic deformations of the material prior failure. Usually, these kinds of models describe soils as a continuum, overlooking the effects of soil particles interactions on the micro-scale. While this simplification of the problem is highly useful and accurate for certain soils (clays), it is incapable of accurately capturing micro-scale driven phenomena, particularly in coarse grained materials, where particle-scale interactions have a more pronounced effect on bulk behaviour. In response, modifications have been proposed in an attempt to fill this gap, in which new state parameters are added to better model coarse soils characteristics, for instance to account for the peak strength of dense/loose sand samples (McDowell and Hau 2004; Yao et al. 2004). In addition, there are now models that explicitly consider the micromechanics of soils, such as breakage mechanics (Einav 2007a; Einav 2007b), as well as its coupling with other conditions, including partial saturation (Alonso et al. 1990; Buscarnera and Einav 2012; Gao et al. 2016) and chemical reactions (Zhang and Buscarnera 2018). This inclusion of micromechanics is crucial for the understanding and modelling of coarser soils, such as sands and gravels.

### 2.2.2 Localised deformation

Porous and granular media under load can exhibit localised deformations. These arise from the micromechanics of the materials and can be triggered by changes in stress and material heterogeneity. Furthermore, these instabilities can cause local regions of material failure, which consequently generates localised changes to the physical properties of the material, including its grain size, porosity, and permeability (Olsson 1999; Du Bernard et al. 2002; Rudnicki 2002). There are three main observed localised kinematic patterns that develop in granular media as bands of dilation, compaction, and shear (Figure 2.1). Although, pure

compaction and dilation bands are not very common in nature, they frequently coexist with pure shear bands at their compressive and tensile ends.

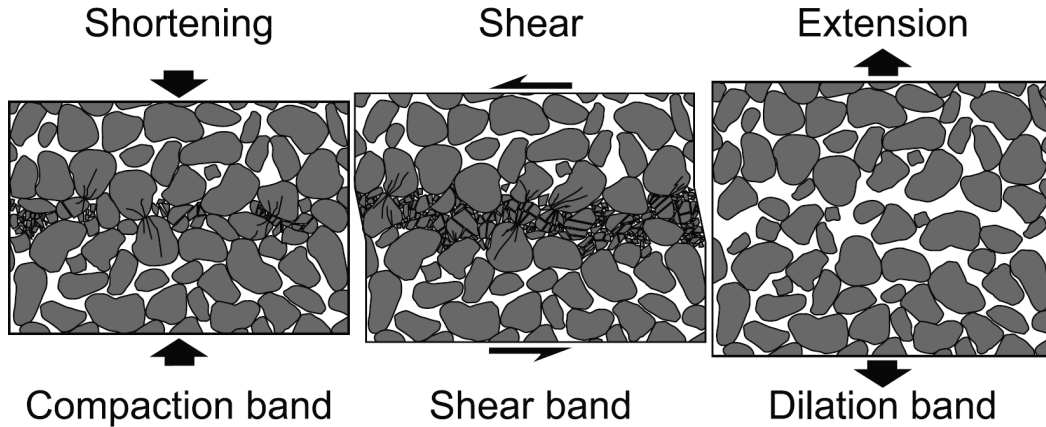


FIGURE 2.1. **Distinct localised kinematic patterns.** Figure extracted from (Du Bernard et al. 2002).

Dilation bands were first identified as a distinct localised deformation mode different from rock joints in (Du Bernard et al. 2002). They are characterised by a localised decrease in bulk density and a corresponding increase in the porosity and permeability within the band. In line with the objectives of this thesis, the following sections will mainly focus on localisation phenomena more likely to occur in confined samples under compressive stresses, such as shear and compaction bands.

### 2.2.3 Shear bands

Shear bands are the most commonly reported form of strain localisation found in nature. They appear in the form of a discontinuity in the strain rate of a deforming mass, where shear strain rates are intensified. The generation of one or multiple shear bands with or without volumetric change is one of the principal modes of failure of soil and rocks under compression (Rudnicki 2002; Tang et al. 2016). Figure 2.1 shows multiple shear band formation resulting from a plane strain tests on Ottawa sand (Alshibli et al. 2003).

The tendency of soils and rocks to develop shear bands as a failure mechanism, as well as the localised changes in material properties that this mechanism generates, have turned the study

of shear bands into a major research topic. It has been observed that shear band formation is accompanied by dilation under low confining pressures, whereas larger confining pressures result in compaction and grain cataclasis within the band (Lee 1970; Olsson 1999; Bésuelle et al. 2000). Shear band formation is also associated with changes to the porosity of the material within the band, which may increase or decrease, depending on the conditions of the shear band formation (Sulem and Ouffroukh 2006; Feia et al. 2016). However, they are known to decrease the permeability of the affected area by up to three orders of magnitude, as a result of grain crushing and reductions in pore space (in the case of compaction shear bands) or an increase in tortuosity and specific surface (in dilating shear bands). Experimental observations have linked the angle between the major principal stress and the shear band planes to numerous material characteristics, such as grain size and their distribution, density, and friction angle, as well as confining pressure (Bésuelle et al. 2000; Wolf et al. 2003; Dewers et al. 2017).

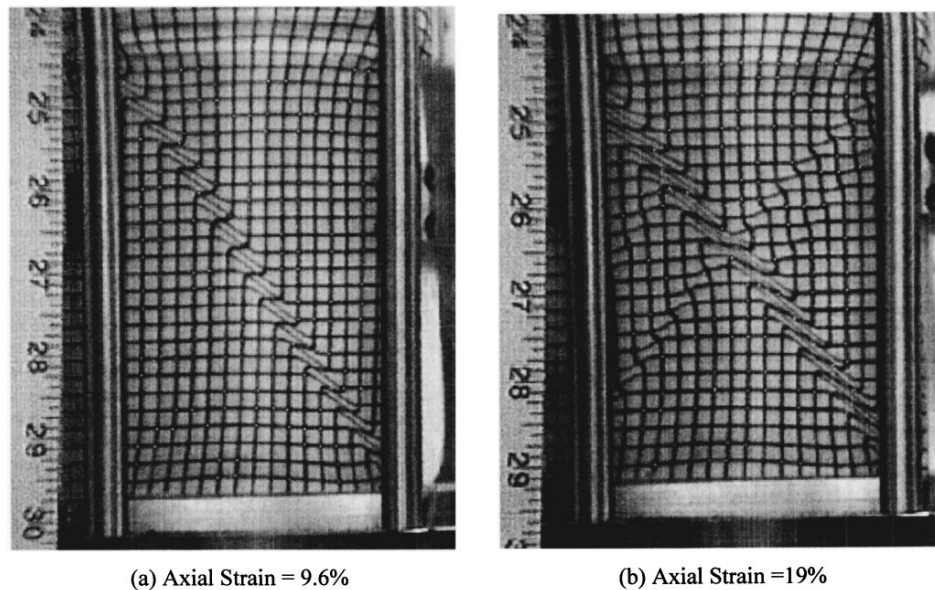


FIGURE 2.2. **Shear bands.** Photographs of plane strain test of Ottawa sand at different axial strains. Figure extracted from (Alshibli et al. 2003).



### 2.2.4 Compaction bands

Similar to a shear band, a compaction band is formed by a discontinuity in the strain rate of a deforming material where deformations are of a purely compacting nature (Rudnicki 2002; Rudnicki 2003; Arroyo et al. 2005). However, they differ from shear bands in their direction and formation mechanism. Compaction bands are localised zones of compaction that form in planes normal to the direction of highest compressive stress applied to a porous material (Rudnicki 2003). Both compaction and shear bands are commonly understood as a result of bifurcation phenomenon, where heterogeneities in the materials result in localised deformations, as modelled in (Rudnicki and Rice 1975; Sulem and Vardoulakis 1995). Some of the earliest models focused on capturing compaction band behaviour as the closing of an existing crack, in accordance the the anti-crack model proposed in (Fletcher and Pollard 1981). In more recent studies, compaction bands in geomaterials are understood as a result of stress localisations. Veveakis and Regenauer-Lieb's study on solid compaction (2015) shows that, depending on stress and strain rates, solid imperfections may result in localised regions of intensified stress, leading to the emergence of a cnoidal wave, which presents itself as compaction bands in porous geomaterials. In addition to geomaterials, compaction bands have been observed in several other porous materials under compression, including snow (Barraclough et al. 2017) and metallic foams (Park and Nutt 2001).

Some of the earliest reports on compaction bands in geomaterials are noted in porous Navajo (Aydin and Johnson 1978) and Aztec (Hill 1989) sandstone. These observations drove researchers to replicate compaction bands in laboratory conditions (Rudnicki 2003; Heap et al. 2015; Dewers et al. 2017) in order to study them in controlled environments. Experimental identifications of compaction bands have now been extensively documented in a variety of porous sandstones (Olsson 1999; Digiovanni et al. 2000; Olsson and Holcomb 2000; Klein et al. 2001; Olsson 2001; Wong et al. 2001; Vajdova and Wong 2003; Baud et al. 2004; Arroyo et al. 2005; Baud et al. 2006; Tembe et al. 2008; Heap et al. 2015; Dewers et al. 2017). Among the several findings of these publications, it is commonly reported that compaction bands were generated with large confining pressures, prior to deviatoric loading. Their initial formation has been observed near load application points in experimental samples (Klein

et al. 2001; Baud et al. 2004), and in zones of concentrated stress in notched sandstone samples (Vajdova and Wong 2003). Furthermore, compaction bands can form outside the bedding planes orientation of sedimentary porous rocks (Olsson 1999), and are commonly accompanied by localised grain breakage, pore collapse within the band, and intensified acoustic emissions (Wong et al. 1997). The ‘loud’ nature of compaction bands is a property that has been leveraged for their identification in experimental studies (Vajdova and Wong 2003; Baud et al. 2004; Baud et al. 2006; Tembe et al. 2008). Another feature used to identify compaction bands are sudden stress drops observed in compression tests with a servo-controlled loading frame (Arroyo et al. 2005; Valdes et al. 2012; Einav and Guillard 2018).

Given that compaction bands generate grain crushing, the reduction in grain size leads to a decreased porosity, and consequently to localised regions of lower permeability (Papamichos et al. 1993; Holcomb and Olsson 2003). In fact, the permeability within a compaction band may be reduced by up to two orders of magnitude compared to the material outside the band. These local reductions in rock permeability have repercussions on the flow of underground water, as well as on the engineering activities that take advantage of the porous properties of rocks, such as for extracting oil and gas from underground reservoirs (Rudnicki 2003).

### **2.2.5 Propagating compaction bands**

Typically, compaction bands appear and grow at static locations. With compaction, the material would locally stiffen and subsequent bands may appear in other locations of the medium (Baud et al. 2004). However, specific loading conditions can lead them to propagate in space. In fact, several studies (Arroyo et al. 2005; Guillard et al. 2015; Barraclough et al. 2017) have reported patterns in the propagation of compaction bands through porous media, in which a distinct moving front of compaction forms.

One of the earliest observations of travelling compaction bands were reported on strain controlled uniaxial compaction experiments in a brittle porous medium, puffed rice (Valdes et al. 2012). The deformation evolution of the puffed rice was characterised with a compaction

front travelling from the bottom to the top of the sample, then reappearing at the bottom, and travelling back to its top, see Figure 2.3.

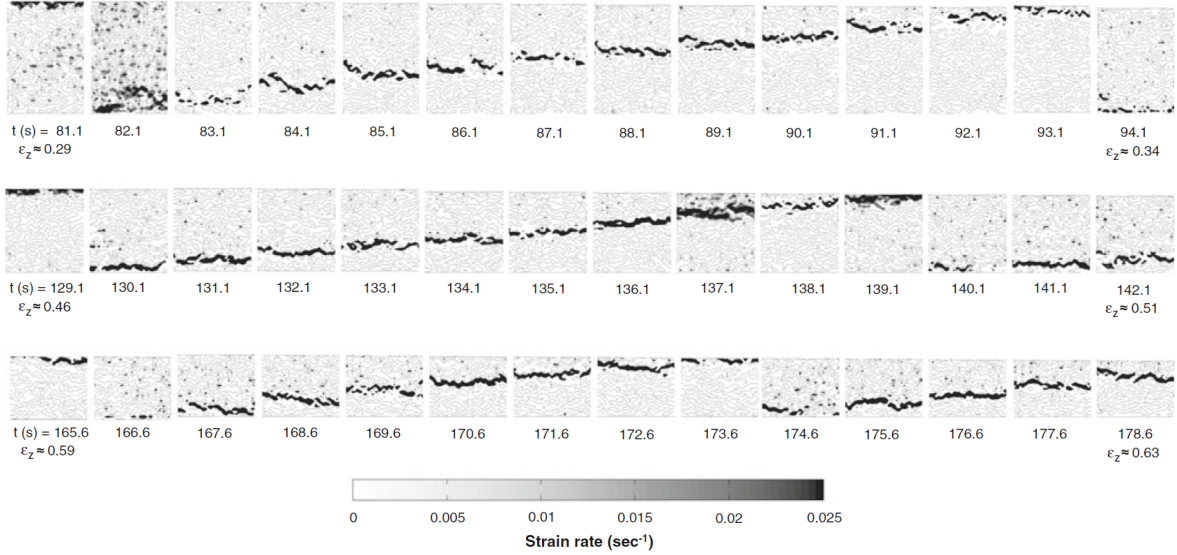


FIGURE 2.3. **Oscillating compaction band time progression.** Figure extracted from (Valdes et al. 2012).

These findings were followed by further investigations on puffed rice compaction, in which the nature of the travelling compaction band was found to be dependent on the rate of deformation applied to the material (Guillard et al. 2015). This dependency was described and explained using a spring lattice model capable of capturing localised grain breakage and reproducing the observed bulk behaviour of the material. It was concluded that low strain rates result in erratic and dispersed localised compaction events. On the contrary, increasing strain rates generate a moving compaction front. Further increases in the applied strain rate result in multiple travelling compaction fronts up to the point in which compaction is uniform throughout the sample, rather than localised. Similar observations have been made in packed snow, with the slight difference that the observed compaction band travelled back and forth between the two ends normal to the compressive force (Barraclough et al. 2017). These strain localisations were observed in both homogeneous and non-homogeneous samples of snow. Furthermore, models can now accommodate recurring stress drops in porous media at the constitutive level (Riley et al. 2023).

An additional effect studied in (Valdes et al. 2012) was the influence of boundary friction in the propagating compaction front. This was done with the use of roughened boundary walls holding the puffed rice sample. Their results indicate that boundary friction plays a major role in the compaction patterns of the sample. The influence of frictional interactions between grains and containment surfaces can therefore significantly impact the bulk response of the material.

### 2.2.6 Boundary conditions and the effect of friction

Besides laboratory considerations, boundary conditions and frictional effects are a main consideration in the design of storage facilities, such as silos to store granular materials. A particular phenomenon seen in the storage of such materials is the Janssen effect (Janssen 1895). This effect refers to the influence of particle-to-wall friction in the transfer of the weight of the granular medium onto the vessel walls. Its historical relevance in silo failure has made the Janssen effect a main point of interest in silo design (Perge et al. 2012; Lei et al. 2019; Mahajan et al. 2020). In a cylindrical silo filled with a granular material, the Janssen effect on the vertical stress caused by the self-weight of the grains  $\sigma_z$  is described with the following expressions:

$$\sigma_z(z) = \rho g \lambda (1 - e^{-z/\lambda}), \quad (2.1)$$

$$\lambda = D/4\mu K_0, \quad (2.2)$$

where  $z$  is the vertical distance from the top of the grain assembly,  $\rho$  is the bulk density of the grains,  $\lambda$  is a characteristic length,  $\mu$  is the coefficient of friction between the grains and the cylinder wall, and  $K_0$  is the coefficient of lateral pressure.

A particular study of flow dynamics in silos (Roberts and Wensrich 2002) reports on a mechanism consisting of intermittent periods of granular flow and jamming forming discontinuities in the downward velocity of the material. In this study, the gravity discharge of grains within

the silo lead to a steady downwards velocity of the material that is interrupted by sudden events in which a portion of the material would jam on the silo walls for a short period of time, ultimately slipping and continuing to flow. These events were accompanied by loud acoustic emissions and vibrations within the silo, which were used to identify their formation and evolution over time.

The past sections of this review summarised two of the main collapse patterns found in dry porous media under compressive stress and the frictional effects of boundary conditions on granular media. Building upon the existing knowledge on these two topics is indispensable for this thesis, which aims to study collapse patterns in porous materials under degradation fields. Given the majority of degradation fields used in this project are imposed via fluids, the focus of the following section will shift to the effects of saturation in porous media.

## 2.3 Saturation

The degree of saturation in a porous medium describes the proportion of its pores that is filled with liquid, typically water. This is a crucial state variable to describe the behaviour of porous media, as saturation can influence the mechanical and hydraulic properties of the material, including its strength, stiffness, effective permeability, and hydraulic conductivity (Fredlund and Rahardjo 1993; Tatsuoka et al. 2021; Lei et al. 2022). Understanding the influence of saturation is important to a variety of fields, including geotechnical engineering, environmental sciences, and underground resource extraction. Therefore, there is a vast collection of studies on flow through porous media, as well as the effects of saturation on their mechanical (Haruyama 1969; Fredlund et al. 1978; Yoshida et al. 1991; Vanapalli and Fredlund 1999; Alonso and Cardoso 2010; Zhang et al. 2017) and hydraulic properties (Richards 1931; Van Genuchten 1980; Gao et al. 2016).

Due to the porous nature of soils, particularly coarse soils, water commonly infiltrates and flows through them. Hence, a large portion of soils found in nature are fully or partially saturated by water (Bear 2013). In fully saturated granular media, the strain response to load is driven by the effective stress experienced by their grains, a concept first introduced by

Terzaghi in 1925. This change to the definition of stress accounts for the contribution of pore water pressure on the stress acting on soil particles and is defined as:

$$\sigma' = \sigma - u_w, \quad (2.3)$$

where  $\sigma'$ ,  $\sigma$ , and  $u_w$  are effective stress, total stress and pore water pressure, respectively. Similarly, the nature of the flow through saturated porous media is governed by the fundamental laws of fluid mechanics, particularly Darcy's law, which relates fluid flow to the hydraulic conductivity of the medium and the hydraulic gradient driving the flow (Bear 2013).

### 2.3.1 Capillarity and partial saturation

In contrast to full saturation, partial saturation in soils refers to a state of the material in which pores hold both water and air. Their ability to hold water, even in the presence of an air phase stems from capillary forces attracting water molecules to the soil particles and the surface tension of the water.

Capillary effects on fluids in porous media were first described as a result of the attraction of wetting liquids towards their confinement walls, pressure differences at the liquid phase boundaries, and surface tension (Richards 1931). Although these effects were initially described in fluids contained in thin capillary tubes, remarkable similarities were later observed in fluid conduction through porous media. It is now recognised that the influence of capillary effects in underground water systems is ever present, for instance in the formation of a 'capillary fringe' between the fully saturated and dry soil zones of underground water systems, as shown in Figure 2.4. Thus, the water phreatic surface in such a system does not represent a clear distinction between saturated and dry material. Instead, it is the starting point at which the degree of saturation of the soil gradually reduces from full saturation to dry (Gillham 1984; Bear 2013).

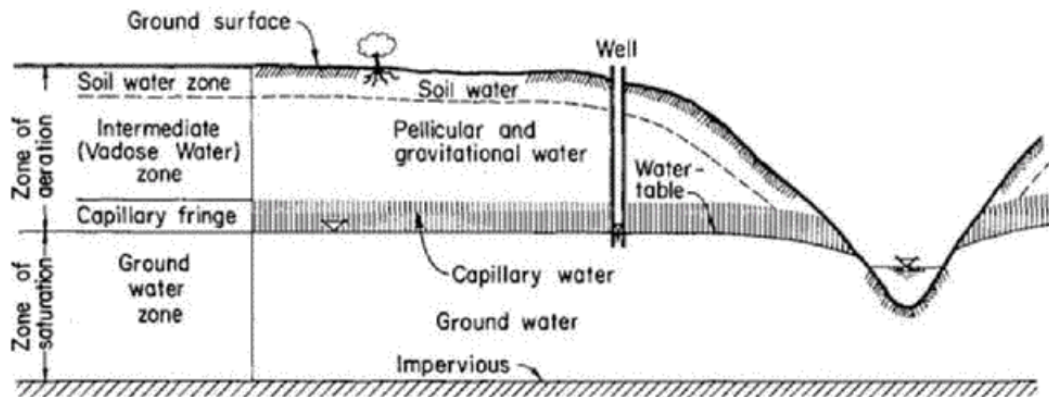


FIGURE 2.4. **Ground water system.** Figure extracted from (Bear 2013).

Owing to the interplay between fluid-to-surface attraction forces and body forces in the fluid, the magnitude of capillary effects relates directly to the ratio between fluid-grain contact area and the density of the fluid (Glover 2000). Capillary effects scale inversely to the radius of a capillary tube, and in the case of granular materials, to their grain size distribution and porosity. Consequently, capillary effects can evolve within a porous material from changes in pore and grain sizes, both of which can arise as a result of grain breakage, erosion, or dissolution (Fredlund and Rahardjo 1993; Buscarnera and Einav 2012).

Capillary effects may result in the displacement and retention of water across soil pores, even against external forces (Saig 2012). This characteristic enables fluid transport and diffusion to take place across partially saturated porous media, via interconnected pore water (Van Genuchten and Wierenga 1976). Flow through capillary channels in unsaturated porous media can be characterised by the laws of hydrodynamics and the differential equations derived from Darcy's law of flow through porous media. However, the complexity of this flow derives from the effects that surface tension and curvature have on flow pressure and the cross-sectional area available for fluid flow (Richards 1931). Looking at the hydraulic properties of the material, hydraulic conductivity under unsaturated conditions has been directly related to soil-water retention curves (SWRC) and matric suction (Van Genuchten 1980). Hydraulic conductivity in capillary flow, for instance, depends on the degree of saturation of the porous material. Figure 2.5 depicts a monotonic decrease of hydraulic conductivity with pressure head, which can be related to a local suction in the material.

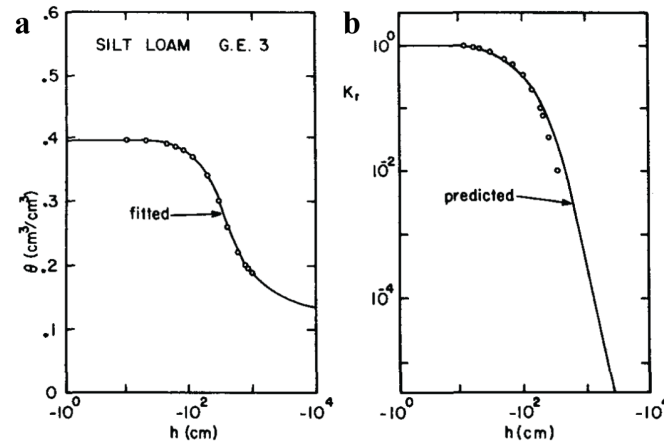


FIGURE 2.5. **Hydraulic properties varying with water pressure.** Soil-water content  $\theta$  (a) and relative hydraulic conductivity  $K_r$  (b) of silt loam as a function of pressure head  $h$  (proportional to suction) (Van Genuchten 1980). The circles are experimental observations. Figure adapted from (Reisenauer 1963).

In addition to a complex fluid flow network, the water surface tension that forms in the air-water pockets of the material can lead to a highly complex system, in which grains are no longer interacting only through contact and body forces, but can now be attracted to one another by means of water surface tension. Here, the water content within the material can act as a ‘glue’, causing the material to exhibit apparent cohesion. Numerous publications indicate that an increase in water presence (within partial saturation) is related to a reduction of soil shear strength (capped by fully dry and saturated limits) (Vanapalli and Fredlund 1999; Baud et al. 2000; Houston et al. 2008; Zhang et al. 2017; Török et al. 2019), as well as lower values of apparent cohesion (Yoshida et al. 1991) and friction angle (Yoshida et al. 1991; Houston et al. 2008). Similarly, changes in the material response with increasing degrees of saturation have been modelled via a growth or shrinkage of its yield surface (Alonso et al. 1990; Buscarnera and Einav 2012; Zhang et al. 2017).

Given the relevance of partial saturation in material properties, and its repercussions on soil behaviour, the following section will summarise the principles of unsaturated soil mechanics used to model partially saturated media.



### 2.3.2 Unsaturated soil mechanics

In a way, the modelling of fully saturated soils is similar to that of dry soils, as there is a single phase fully occupying their pore space, that being air or water. Unsaturated soils, on the other hand, present an added layer of complexity, as their pores are occupied by more than one phase, usually by both air and water. The goal of unsaturated soil mechanics is to describe the behaviour of these soils by capturing the characteristics and effects caused by both the air and water phases within their pore space (Fredlund and Rahardjo 1993; Einav and Liu 2020). In unsaturated conditions, the definition of effective stress becomes more complex than that found in Equation 2.3, since simply capturing stress contributions from water is no longer sufficient. In light of this, a single effective stress expression for unsaturated granular media was proposed (Bishop 1959):

$$\sigma' = (\sigma - u_a) + \chi(u_a - u_w), \quad (2.4)$$

where  $\sigma$  is the total stress,  $u$  is the pore pressure, the subscripts  $a$  and  $w$  stand for air and water, respectively, and  $\chi$  is the Bishop parameter, which is related to the residual soil saturation. Furthermore, the concept of suction  $s$  can be defined as the difference in air and water pressures,  $s = u_a - u_w$ . Hence, Bishop's parameter, which ranges from zero to one, acts as a weighing factor to the influence of suction on effective stress (Jiang et al. 2017). This expression is also capable of recovering the fully saturated equation established by Terzaghi when suction  $s$  reaches zero ( $u_a = u_w$ ). It is worth noting that a porous medium can maintain full saturation despite experiencing low magnitudes of suction. Starting from a fully saturated state, the presence of suction is not a sufficient requirement for air to enter the system. For this to occur, a sufficiently large (critical) pressure, denominated 'air entry suction', is required (Gillham 1984). On the other hand, in an air-water-solid system with a positive suction, we see that zero suction is also not a sufficient requirement for air to leave the system (Einav and Liu 2023). To fully saturate the system again, additional perturbations on the saturation or suction are required. To illustrate these points, Figure 2.6 shows how saturation changes under increasing and decreasing magnitudes of suction in unsaturated samples.

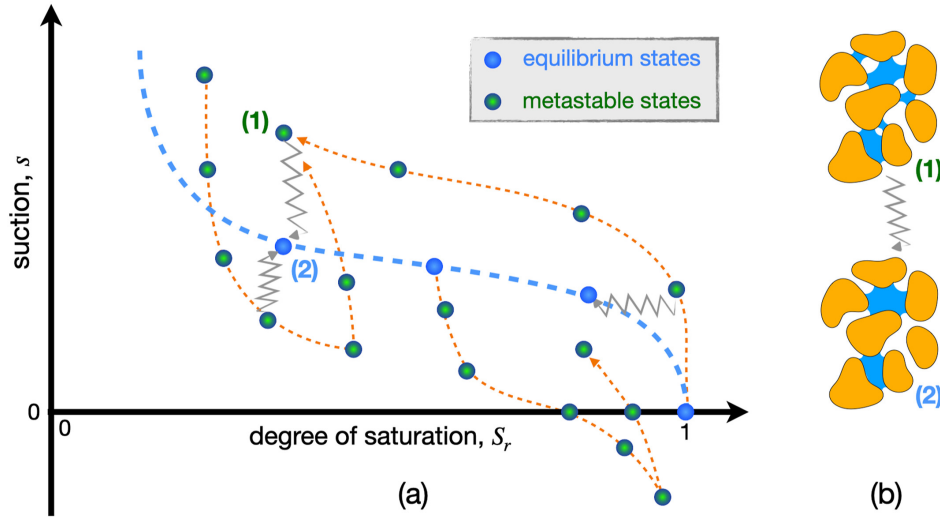


FIGURE 2.6. **Saturation states with suction.** **a:** Soil water retention curves showing metastable states visited after monotonic drying or wetting (dashed orange lines) and true equilibrium states (dashed blue line) approached after low amplitude perturbations of suction or saturation (gray zigzags), or even acoustic waves. **b:** The dynamics of fluid patches and interfaces that accompany such perturbations. Figure extracted from (Einav and Liu 2023).

For decades, the determination of the Bishop parameter  $\chi$  and its dependence on other material state variables has been a goal of a vast number of studies. To date, scientists, engineers, and soil mechanicians continue to strive for a unified description of  $\chi$ . Through this endeavor,  $\chi$  has been linked to the degree of saturation in the soil (Hassanizadeh and Gray 1980; Bear et al. 1984; Houlsby 1997), suction itself (Aitchison 1985; Khalili and Khabbaz 1998), as well as soil density (Assouline 2006; Tarantino 2009; Romero et al. 2011; Zhou et al. 2012) and grain/pore size distributions (Gao et al. 2016). However, most of these models are based on empirical observations, and are unable to capture the entirety of unsaturated soil behaviour. As a solution, (Jiang et al. 2017) formulated a thermodynamically consistent approach capable of modelling unsaturated soil behaviour through a single dependency for  $\chi$ , where  $\chi$  is defined as a function of three thermodynamic densities, one for each phase of the material: soil, water, and air. Consequently, this links  $\chi$  to the measured suction and to the soil-water retention curve, which can be determined experimentally.

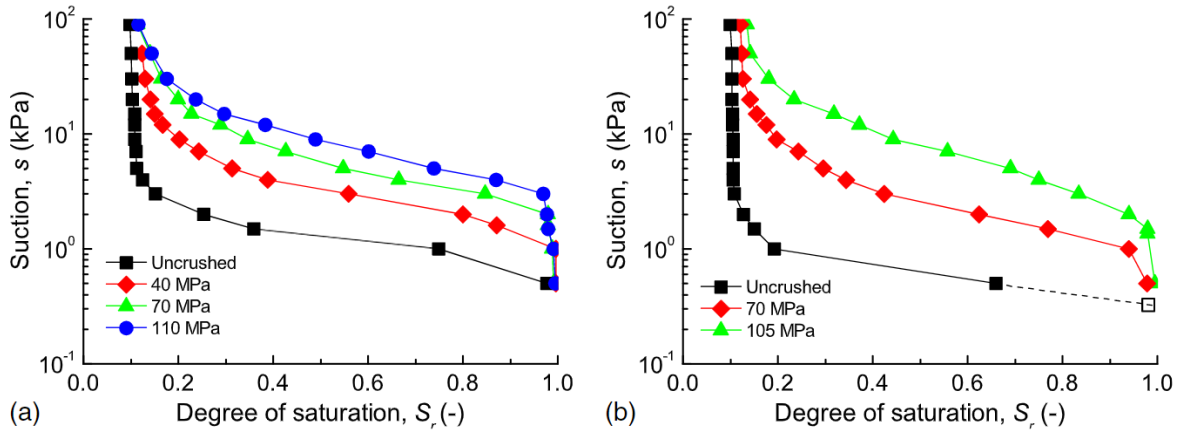


FIGURE 2.7. **Evolution of water retention curve with breakage.** Tests on **a:** Q-ROK sand and **b:** glass beads with an initially unimodal grain size distribution. Figure extracted from (Gao et al. 2016).

Given that the capacity of a porous material to retain water stems from capillary effects, its water retention curve has a direct dependency to the pores within the material, as well as the material properties and interaction with water (Gao et al. 2016). Therefore, the water retention curve for a singular material can evolve with changes in the micro-characteristics of the material, such as pore or grain size. An example of such progression is presented in (Gao et al. 2016), which investigated the influence of grain breakage on the water retention curve of granular materials, as shown in Figure 2.7.

These changes in water retention curves originated from a change in grain size caused by grain breakage. However, there are alternate paths through which a change in grain size can be imposed on a granular medium, such as chemical dissolution. While soils are most commonly saturated by water, other substances can be contained within their pores, including natural gases, oils, and contaminants. The presence of chemically active liquids may lead to the weakening of the material, as well as changes to its pore network, and consequently its behaviour under saturated and unsaturated conditions. These interactions have the potential to significantly change the porous structure of the material and in turn, cause accelerated deformations and collapse. Therefore, the following section focuses on reviewing the effects of chemical dissolution in porous media and its relevance in their collapse behaviour.

## 2.4 Chemical dissolution

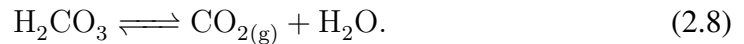
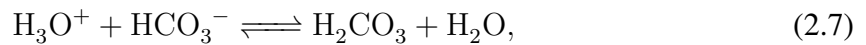
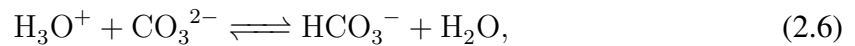
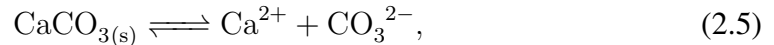
Chemical degradation is a common diagenetic process in soils and rocks that plays a significant role in their weathering and decomposition (Cha and Santamarina 2019). The characteristics of these degradation processes depend on environmental conditions, such as pressure, temperature, acidity (pH), available surface area, and the mineral composition of the material (De Boer 1977; Rutter 1983; Hellmann et al. 2002; Niemeijer et al. 2009; Espinoza et al. 2011; Cha and Santamarina 2019). Their effects are typically seen under long time scales; however, when highly reactive minerals are involved, or when acid is injected into a soil mass, these processes may have an accelerated influence on soil behaviour, and can induce structural instabilities. Calcareous geomaterials, abundantly found as sedimentary soil deposits, are well known for their reactivity in aggressive environments due to their high concentrations of calcium carbonate, which reacts strongly with acids (Panthi 2014). Apart from calcium carbonate-rich soils, silica sands have been observed to dissolve in aqueous solutions, however their dissolution rates are lower than those of calcareous sand and significant dissolution usually requires high temperatures and pressures (Rimstidt and Barnes 1980; Fournier 1983). The decomposition of silica sand occurs through the dissolution of silicon oxide ( $\text{SiO}_2$ ) in water, which forms a silicic acid ( $\text{H}_4\text{SiO}_4$ ) solution.

The reactivity of certain soils may result in significant engineering stability issues. In fact, the origin of collapse events such as sinkholes (Waltham et al. 2005), submarine landslides (Urgeles and Camerlenghi 2013), and dam instability (Gutiérrez et al. 2003; Jarvis et al. 2003; Alonso and Gens 2006) may be triggered or exacerbated by the chemical degradation of soils and rocks. Notably, these events can also occur under non-acidic weathering action, as seen in the collapse of salt mines (Stoeckl et al. 2020) and sinkhole formations caused by the dissolution of saline deposits in water (Shalev et al. 2006). In the case of chemical-dissolution-induced sinkholes and submarine landslides, their acidification is commonly attributed to dissolved carbon dioxide build up in water, which decreases its pH value by forming carbonic acid (Panthi 2014; Scavengers 2020; Shapley 2011). In tailing dams, acid mine drainage is known to occur as a results of chemical reactions between tailings and air, specially in minerals rich in sulphur (Lottermoser and Ashley 2005; Alonso and Gens 2006).

This acidification of sulfuric deposits, particularly pyrite ( $\text{FeS}_2$ ), is also seen broadly across acid sulfate soils exposed to air, as can be found in the Riverland and Murraylands of South Australia, (Fitzpatrick et al. 2009). Other engineering practices, such as  $\text{CO}_2$  storage in deep porous rock could also result in the acidification of underground water reservoirs, to pH values as low as 3 (Espinoza et al. 2011). This effect may lead to an accelerated degradation of the structural integrity of the cap rock in the sequestration system, which could result in fractures, and the eventual release of the trapped  $\text{CO}_2$ .

### 2.4.1 Dissolution process

The chemical dissolution of geomaterials is characterised by the reduction of grain mass. In the case of calcareous soils and carbonic acid in water ( $\text{H}_2\text{CO}_3$ ), mass reduction occurs due to the decomposition of solid calcium carbonate ( $\text{CaCO}_3$ ) into calcium ions ( $\text{Ca}^{2+}$ ) and carbonate ions ( $\text{CO}_3^{2-}$ ), which combine with the hydronium ions ( $\text{H}_3\text{O}^+$ ) present in the water. Depending on environmental conditions, the formed  $\text{H}_2\text{CO}_3$  can then further dissociate into  $\text{CO}_2$  gas and water. The complete chemical reaction is:



The equilibrium concentrations and prevalent carbon species varies with the pH of the solution, as shown Figure 2.8.

Depending on environmental conditions, as well as the diffusion and advection of the system, the reaction products can either precipitate onto nearby surfaces within the pores of the soil (Rutter 1983; Fowler and Yang 1999; Yasuhara et al. 2003) or be transported away (Etheridge et al. 1984; Yasuhara et al. 2003).

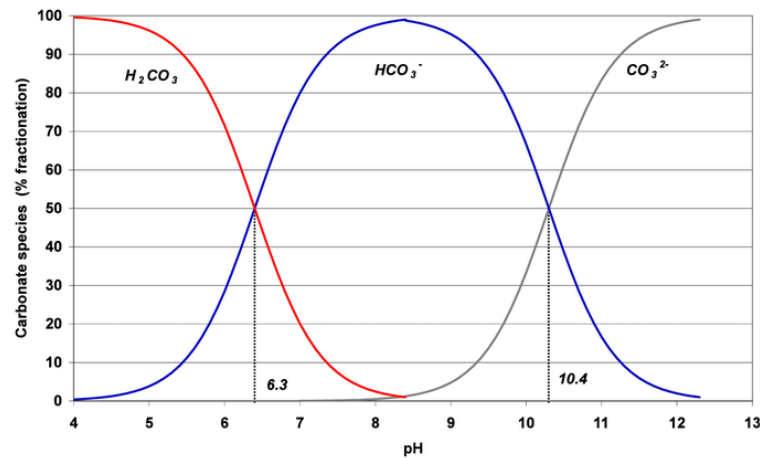


FIGURE 2.8. **Stability fields of carbon species in pure water on the basis of water pH.** Figure extracted from (Inguaggiato et al. 2011).

### 2.4.2 Chemical dissolution effects on material properties

In addition to the mass loss resulting from grain surface dissolution, chemical degradation processes have a degrading effect on the inner structure of rocks and soils, which in turn affects their macro-scale bulk material response (Hu and Hueckel 2007). Consequently, models have often considered chemical degradation as ‘damage’ on the particles and bonds of the material, which decreases their bulk elasticity and strength. For instance, (Nova et al. 2003) presented a model developed under the concept that the yield surface of bonded geomaterials decays quadratically with an increase in the duration of acid exposure. This assumption was also validated by several experimental observations (Baynes and Dearman 1978; Castellanza and Nova 2004). In bonded granular media, the acid dissolution is thought to accelerate the de-bonding process (Nova et al. 2003), introducing ‘damage’ throughout the porous structure.

Additional studies on the effects of chemical degradation on the mechanical properties rocks and soils report decreases in strength (Castellanza and Nova 2004), peak friction angle (Fam et al. 2002), stiffness (Viswanath and Das 2019), and lateral to axial stress ratio (Shin and Santamarina 2009). The latter was found to lead the soil to undergo Rankine’s failure in samples subjected to a constant uniaxial load and restrained lateral strains. On the other hand, they also report increases in porosity resulting from mass loss, and therefore an increase in permeability (Golfier et al. 2001; Hellmann et al. 2002; Cha and Santamarina 2019). At a

particle level, acidic degradation has been linked to a decrease in coordination number in grain assemblies (Cha and Santamarina 2019) and a decrease of Weibull modulus (Xiao et al. 2019). The latter was validated with crushing tests on individual grains with different durations of acid exposure.

### 2.4.3 Dissolution localisation

As with any form of strain localisation, localised zones of chemical dissolution may have significant effects on the behaviour and properties of porous materials. It is therefore important to develop an accurate understanding on the topology of the wetting fronts that can induce dissolution processes. Dissolution localisations may result from differences in material composition within the porous medium, from concentrations of stress in the material, and from preferential flow paths formed during acid flow.

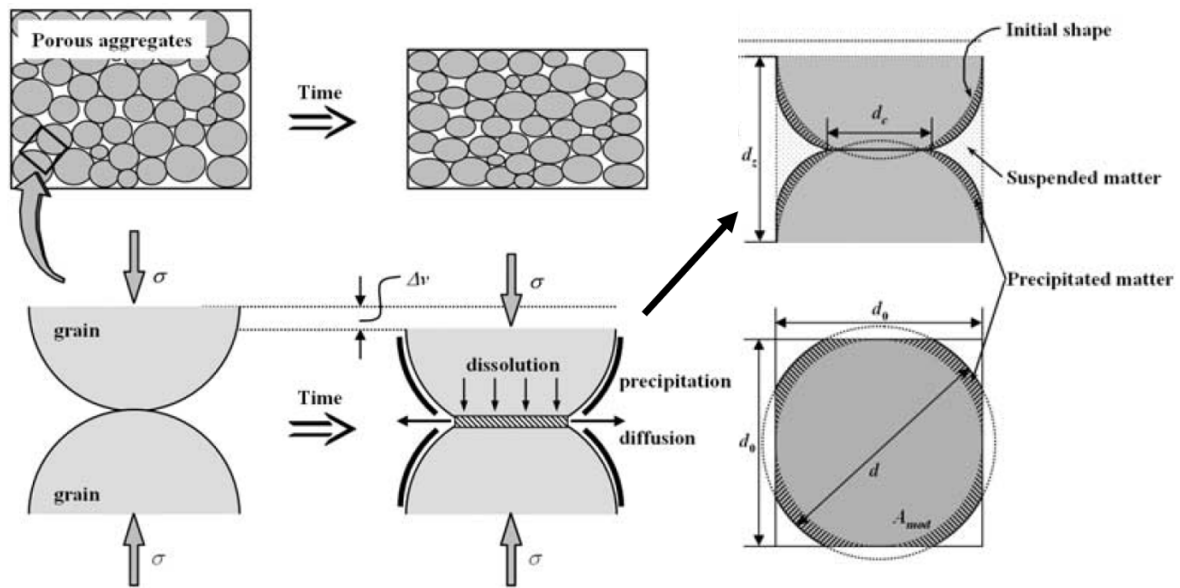


FIGURE 2.9. **Pressure dissolution.** Figure adapted from (Yasuhara et al. 2003).

High pressure environments on granular media may lead to pressure dissolution, a phenomenon in which high-stress areas in the material undergo preferential dissolution (Fowler and Yang 1999). This phenomenon is mainly relevant in materials subjected to large stresses, such as rock formations at depths of over a kilometer (Rutter 1983; Fowler and Yang 1999;

Hellmann et al. 2002). Pressure dissolution is the result of the difference in surface chemical potential between the minerals under high stress at the grain contact surfaces and those under low stress, away from the contact surfaces (Shimizu 1992; Lehner 1995; Shimizu 1995). This type of dissolution is characterised by a change of grain size towards a more unimodal distribution (Cha and Santamarina 2019). Additionally, pressure dissolution may contribute to the cementation of soils and the bonding of their grain contacts (Rutter 1983; Mitchell and Solymar 1984). Different models have considered the effects of pressure dissolution with a linear (De Boer 1977; Rutter 1983) or quadratic (Dewers and Hajash 1995; Gratier et al. 2009) relationship between the effective stress and dissolution rates of the grains. Figure 2.9 illustrates the process of pressure dissolution, as well as precipitation of decomposed mass onto the stress-free areas within the pores of the material. The precipitation of the material would then seal pores spaces form bonds, which would in turn strengthen the granular medium. This is a relatively slow process, which occurs over the timescale of several years to decades (Geng et al. 2021).

Pressure dissolution can further lead to the development of compaction patters in the fabric of a rock mass. These patterns, denominated stylolites, are typically observed as a serrated line of insoluble material deposited within the rock pores (Park and Schot 1968; Toussaint et al. 2018). As localised regions of high stress have a higher chemical potential, the minerals experiencing higher stresses dissolve faster than the surrounding material, and deposit within the rock pores. This reduces the size of the rock pores within this region, and fills the void space by mineral deposition. This process results in a reduction of porosity within the stylolite.

Other forms of localised dissolution can occur from preferential flow paths within a porous medium. Preferential flow paths can be formed by pre-existing areas of low flow resistance or from localised areas of highly soluble material (Hoefner and Fogler 1988; Daccord et al. 1993; Fredd and Fogler 1998; Jang et al. 2011). Additionally, the wetting front topology is controlled by the interactions between reaction, diffusion, and advection rates of the acid flow (Hoefner and Fogler 1988; Daccord et al. 1993; Fredd and Fogler 1998; Golfier et al. 2001; Jang et al. 2011). Chemical dissolution observations under various conditions have resulted in the identification of two non-dimensional parameters capable of describing dissolution



patterns and wormhole formation, namely the Péclet and Damköhler numbers. The Péclet number, named after Claude Eugène Péclet, is the ratio of advection rate to diffusion rate. The Damköhler number, named after Gerhard Damköhler, is the ratio of reaction rate to advection rate. The patterns of dissolution under varying Péclet and Damköhler numbers can be classified under three main regimes: uniform, compact (also referred to as ‘face’), and localised dissolution (Golfier et al. 2001). Figure 2.10 shows a visualisation of the dissolution patterns emerging from each regime.

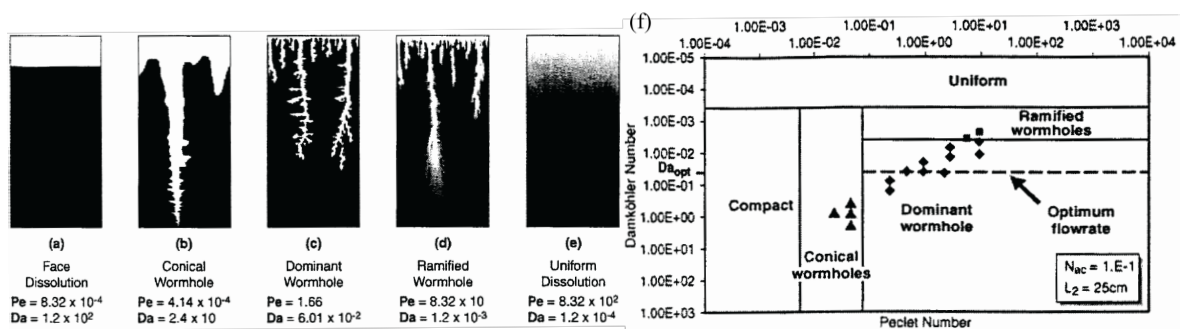


FIGURE 2.10. **Dissolution patterns.** a-e: Patterns formed in acid flow through a porous medium under a range of Péclet (Pe) and Damköhler (Da) numbers. f: Phase diagram of dissolution fronts in the Pe-Da space. Figure adapted from (Golfier et al. 2001).

As shown in the figure, uniform dissolution develops at low Damköhler and high Péclet numbers, i.e. low reaction rates or high advection rates, whereas face dissolution is achieved at a high Damköhler number and a low Péclet number, indicating high reaction and diffusion rates relative to advection rates. Dissolution localisation patterns, or ‘wormholes’, emerge at high values of both the Damköhler and Péclet numbers, as shown in the phase diagram in Figure 2.10 (f).

#### 2.4.4 Degradation induced compaction bands

As discussed earlier, compaction bands are the result of stress localisations, which may stem from heterogeneity and local instabilities in granular materials (Vajdova and Wong 2003; Stefanou and Sulem 2014; Veveakis and Regenauer-Lieb 2015). Hence, instabilities generated by the chemical degradation of a granular material can potentially induce compaction bands.

A particular study on this topic probed at the possibility of degradation induced compaction bands with a constitutive model approach (Stefanou and Sulem 2014). The model was built to capture the evolution of instabilities due to material degradation and led to the development of compaction bands as a result. The study further investigated the periodicity of degradation induced compaction bands in geological formations. Due to the slow reaction rates in natural chemical degradation processes, the model predicted that the time needed for compaction bands to form in underground acid-exposed soil deposits can be relatively long, in the order of five months. However, it is noted that this time would reduce drastically with an increase in reaction rates.

Recent experiments on the compaction and wetting of puffed rice resulted in the identification of a novel collapse phenomenon (Einav and Guillard 2018). The experiments were performed on partially saturated puffed rice under constant uniaxial loading. This study identified cyclical formations of degradation induced compaction bands. The decay of mechanical strength, stiffness, and ‘crispness’ of puffed rice as a product of moisture content increase and time of exposure to water is well documented (Ituen et al. 1986; Sauvageot and Blond 1991; Georget et al. 1994; Roudaut et al. 1998; Lewicki et al. 2004; Gondek and Lewicki 2006; Peleg 2015). These observations on material properties could be considered analogous to those made on chemical degradation on reactive porous media, reviewed earlier. In accordance with these experimental observations, as water degraded mechanical properties of the cereal, the partially saturated area of the material developed repetitive layered collapse accompanied by intensified acoustic emissions and drops in the servo-controlled axial load (as shown in Figure 2.11), exhibiting great similarity with compaction band formations in geomaterials. In this study, the repetitive compaction bands, denominated ‘ricequakes’, were successfully modelled under the assumptions of a steady saturation profile, and micropore driven collapses.

This collapse phenomenon presents a remarking resemblance to quakes observed in ice and snow sheets (Nikonov 2010; Battaglia and Changnon 2016), which are also characterised by a sudden compaction of a deep layer of the material and large acoustic emissions. The sound emissions of quakes in ice are of such magnitude, that they have been described as a ‘loud’, ‘ear-splitting’, and ‘explosive’ noise that can be heard near the breakage zone (Battaglia

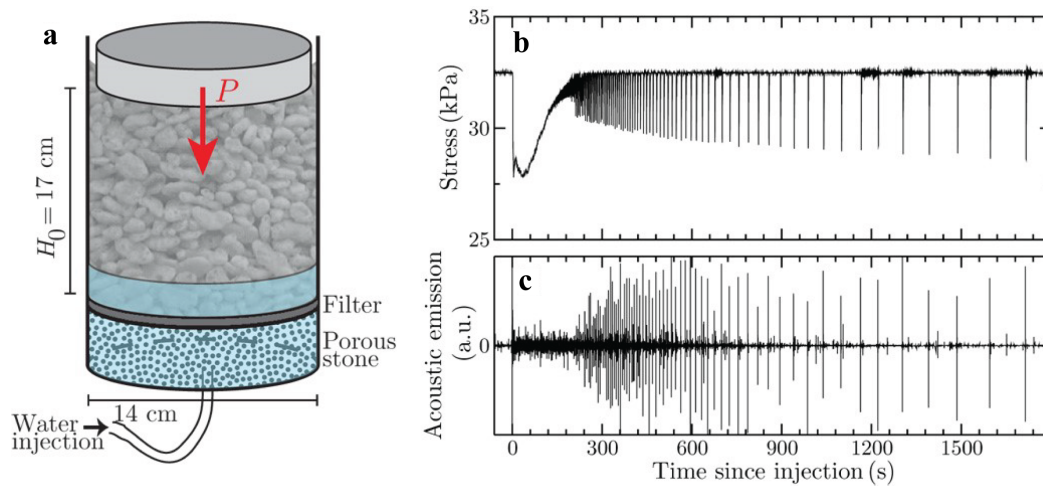


FIGURE 2.11. **Degradation induced compaction bands in puffed rice.** **a:** Experimental setup. **b:** Stress over time since water injection. **c:** Acoustic emissions over time since water injection. Figure adapted from (Einav and Guillard 2018).

and Changnon 2016). These sudden compaction collapses were explained by the pressure increase that results from the expansion of water when it freezes. Although the ‘icequake’ phenomenon is most commonly reported in the snowy mountain ranges, such as those in North America (Battaglia and Changnon 2016) and Europe (Nikonov 2010), there is evidence of their observation in lower altitude ice formations, such as volcanic glacier caps (Battaglia et al. 2019).

So far, ricequakes have been identified solely for puffed rice in laboratory conditions. This is a manufactured material with specific properties and limited relevance outside the scope of food processing and handling. However, the physical mechanisms through which ricequakes emerge in this medium are analogous to naturally occurring degradation processes in porous materials, such as the chemical weakening of geomaterials or the melting of ice assemblies. This presents an opportunity to adopt puffed rice as a model material, from which phenomenological processes in other porous media can be described. In fact, the similarities between ricequakes and ice quakes already hint towards the existence of a more global phenomenon at play.

## 2.5 Conclusion

This literature review briefly summarised the main observations on collapse mechanisms of porous materials, as well as the effects that saturation and chemical degradation have on their material properties and structural stability. The key finding is that brittle porous media under compression may exhibit localised collapse events, with or without visible propagation patterns, which commonly result in local changes in porosity, and therefore permeability. With respect to partial saturation, increasing degrees of saturation can have a significant influence on material strength and hydraulic conductivity. The main effects of chemical degradation on the properties of porous materials, specifically reactive soils, are the reduction of their mass, friction angle, stiffness, and particle Weibull modulus, as well as changes in their porosity and permeability. Lastly, this review summarised recent discoveries on the compaction mechanism of a partially saturated porous medium under degradation and stress. These findings served as a source of inspiration for the research conducted in this thesis, particularly for the work presented in the following chapter, which investigates the emergence of similar collapse patterns in different porous media under distinct fields of degradation.

## CHAPTER 3

### **A ubiquitous mechanism of creep-quake cycles in porous media**

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The formation of ‘ricequakes’ in puffed rice is a revealing and inspiring discovery, which may point at the development of this intricate collapse mechanism in other porous media. This chapter will investigate the emergence of creep-quake cycles in a diverse array of materials and applied conditions, including puffed rice partially saturated by water, calcareous sands partially and fully saturated by acid, rock salt dissolving in water, and crushed ice melting by applied heat. Most of the contents of this chapter are taken directly from a manuscript submitted for publication, titled: "A universal mechanism of creep-quake cycles in porous media". The manuscript was slightly reformatted in order to fit this thesis and its references were included in the main thesis bibliography. At the time of thesis submission for examination, the article was submitted to the Proceedings of the National Academy of Sciences of the United States of America (PNAS). At the time of submitting the thesis to The University library, the manuscript was under revision and editing to be re-submitted for publication. The author of this thesis is the primary researcher and first author of the manuscript. This work was done in collaboration with the author’s supervisors, Dr François Guillard and Prof Itai Einav, who are co-authors of the article. Sections 3.5.1 was added as additional discussions following comments received from the examiners of this thesis.

### 3.1 Introduction

We quickly learn that ice, salt, cereal, and sand do not look or taste the same. They further differ in how they crumble when pressed and how they behave when soaked in water or taken out of a freezer. One would commonly study such changes in each material separately, but there is much to gain in identifying overarching traits they might share, as these could facilitate the transfer of ideas and understanding from one material to another. Indeed, despite their differences, all these materials belong to the class of porous media, having a solid skeleton, as well as pores that can retain or transfer fluids. Most importantly, their degradation causes many problems in geosciences, for example where chemical dissolution instigates sinkhole formation (Waltham et al. 2005; Shalev et al. 2006), and global warming accelerates ice-shelf melting (Polashenski et al. 2012; MacAyeal et al. 2019). The degradation of porous media is also relevant in industry, where their properties are regularly manipulated through external thermal, hydraulic or chemical fields, for example during food and drug processing (Lewicki et al. 2004; Xie et al. 2004; Pringle et al. 2012; Gray and Rosanske 2020).

Previous work on puffed rice cereal, a seemingly inconsequential material, revealed a novel collapse pattern that develops under simultaneous load and partial water saturation (Einav and Guillard 2018). The pattern is characterised by regularly recurring compaction events, coined ‘ricequakes’, interluded by creep deformation. Ricequakes originate in a highly degraded zone within the medium and result in (Einav and Guillard 2018): (1) distinct acoustic emissions; (2) drops in applied servo-controlled load and rapid associated collapse of the sample; and (3) the formation of layers of compacted material, similar to those found in rocks (Olsson 1999; El Bied et al. 2002; Fossen et al. 2007; Stefanou and Sulem 2014; Kelka et al. 2017). This discovery of recurring ricequakes and creep, or more broadly ‘creep-quake cycles’, begs to question the possibility of similar patterns in other porous media under fields different than partial saturation.

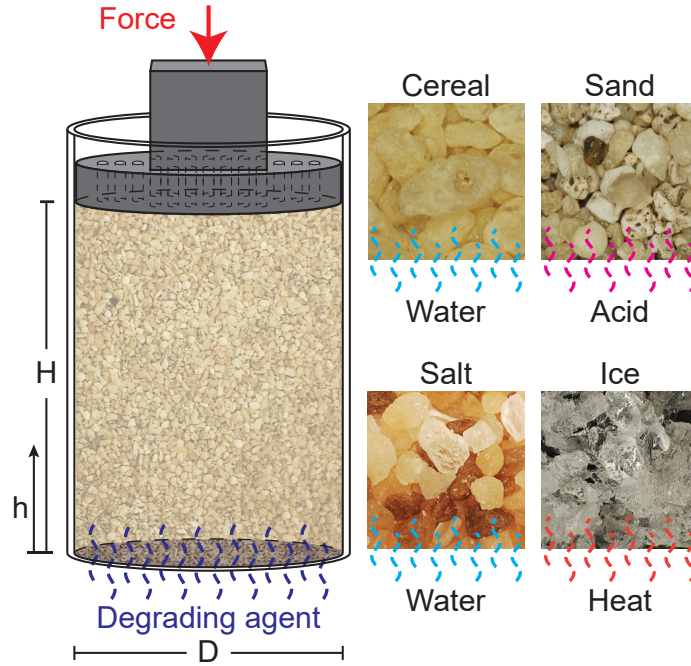


FIGURE 3.1. **Experimental setup on various uniaxially compressed materials and degrading agents.** Experiments include puffed rice degraded by water, calcareous sand by acid, rock salt by water, and crushed ice by heat.

To study this question, we developed an experimental campaign consisting of five distinct testing configurations on four different porous materials: puffed rice cereal, calcareous sand, rock salt, and crushed ice. All these materials were simultaneously subjected to compression and a degrading agent specific to their physical or chemical properties (Figure 3.1). Hence, water was used to degrade puffed rice and salt through absorption and dissolution, respectively; acetic acid was used to chemically react with calcareous sand; and crushed ice was melted by applying a heat source at the base of the sample.

As shown in Figure 3.1, the samples were placed in an acrylic tube between a porous plate and a porous loading piston to allow for liquid transfer. All the samples were carefully prepared to minimise degradation prior to testing, with different strategies depending on the material and the degrading agent, as detailed in supplementary materials. The initial height of the samples  $H_0$  was at least 1.5 times their width  $D$ .

Once prepared, the samples were slowly loaded to a nominally constant load using a servo-controller, except for the crushed ice where a dead weight was used, as its quake deformations

were significantly larger. The corresponding degrading agents were then introduced and pictures were taken through a narrow window along the central axis of the tube, from which spatiotemporal plots were generated to visualise sample deformations over time and depth (Figure 3.2).

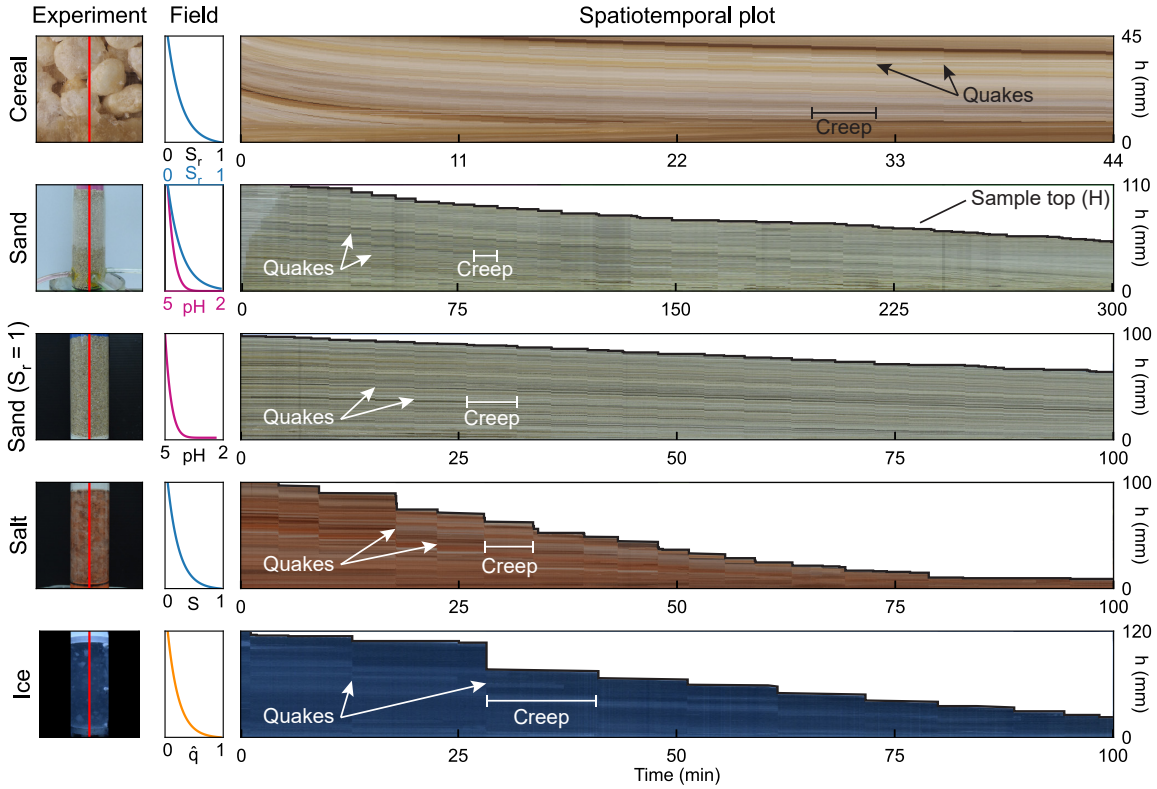


FIGURE 3.2. **Ubiquity of the creep-quake cycles.** Materials from top to bottom: puffed rice under water saturation field  $S_r$  (Einav and Guillard 2018); calcareous sand in combined fields of water saturation  $S_r$  and pH induced by an acetic acid solution, with  $\text{pH} = 2.4$  at the bottom; calcareous sand fully saturated in an acetic acid solution ( $\text{pH} = 2.4$  at the bottom); rock salt fully saturated in water under solubility field  $S$ ; and crushed ice fully saturated in brine under heat field imposed by a heat source at the bottom, where  $\hat{q}$  is the heat available for ice melting normalised by the heat input. From left to right: first column shows pictures of each experiment; second column schematically shows the corresponding degradation fields; and third column presents spatiotemporal plots of the pixels located under the red lines on the left images, which highlight the recurrence of the creep-quake behaviour. The vertical steps observed in the spatiotemporal plots are signature of quake events.



In order to accommodate for the specific properties of each tested material, the introduction of degrading agent varied slightly across the five different experiments. For the puffed rice, a known volume of water was injected from under the sample at the start and it was not renewed. The sand samples were injected with an acidic solution at a constant rate via four equally spaced inlets for the partially saturated setting and from under the bottom plate for the fully saturated one. Similar to the latter, water was injected through the bottom of the salt samples at a constant rate, and the ice was melted with a heat source placed under the bottom plate. Note that the fields shown in Figure 3.2 are indicative diagrams on the nature of the degradation fields, not experimental measurements. For further technical details on the experiments, material properties and handling, sample preparation, and the application of degrading agents, see supplementary materials (Section 3.5.2).

## 3.2 Results

Remarkably, all the spatiotemporal plots in Figure 3.2 reveal recurring sudden collapses of the porous structure with creep deformation periods in between, as detected originally in soaked puffed rice (Einav and Guillard 2018). These creep-quake cycles are confirmed in Figure 3.3 (a), which shows the nominal strain  $\varepsilon = 1 - H/H_0$  over normalised time  $\dot{\varepsilon}_0 t$  curves, where  $t$  is time,  $H$  is the current sample height, and  $\dot{\varepsilon}_0$  is the measured nominal initial strain rate in each experiment (Figure 3.3 (b)).

The deformation patterns are notably similar in all samples, with the puffed rice curve deviating from the trend, as it is the only experiment where the degrading agent is injected once initially instead of being renewed over time. This difference leads to a significant slow down of the creep-quake cycles as the degradation wears off. Further noteworthy similarities include loud bursts of sound accompanying quakes in all the experiments, as well as load drops in the servo-controlled tests, two examples of which are shown in Figure 3.3 (d).

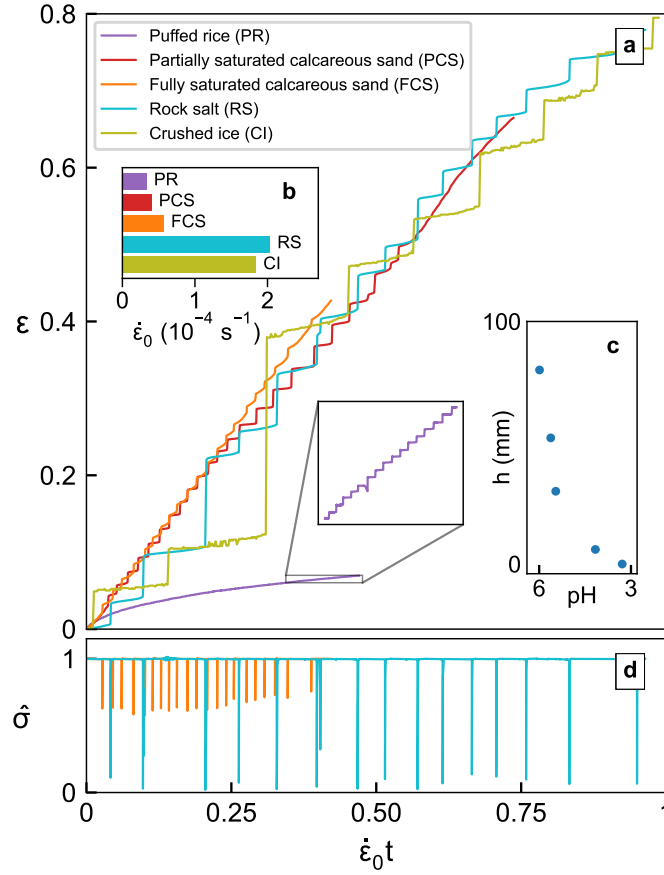


FIGURE 3.3. **Experimental results.** **a**, Strain  $\varepsilon$  versus normalised time  $\dot{\varepsilon}_0 t$ . **b**, Nominal initial strain rate  $\dot{\varepsilon}_0$  (slope of the strain-time curve from  $t = 0$  to the fifth quake of each data set). **c**, pH measurements taken from the side of the test tube during a 1 mL/min flow rate experiment on a fully saturated calcareous sand sample, averaged over 1h. **d**, Stress drops in the fully saturated sand (in orange) and rock salt (in cyan) experiments, where  $\hat{\sigma}$  is the applied stress normalised by target stress (4.35 MPa and 0.54 MPa, respectively).

### 3.3 Discussion

Despite the diversity of the studied materials, they are all porous media, and thus, carry a length-scale related to their typical pore-size. Without this length-scale, there would be no space for the pore collapse that cause quakes to occur, as is the case with a solid block. Similarly, the degrading agents are vastly different, but their presence is always non-uniform within the medium due to capillarity in the partially saturated samples, flow rate and chemical kinematics in the samples saturated in acidic or salty water, and heat transfer. In all cases, these

different physical mechanisms result in the development of a local gradient that is steepest close to the bottom of the sample, as illustrated schematically in Figure 3.2, second column. In fact, such a non-uniform field was measured directly in a calcareous sand experiment under full saturation in acidic solution, as shown by the pH profile in Figure 3.3 (c). Note that although localised collapse events were previously found in ice even under uniform melting, these occurred sporadically and anywhere within the samples (Dorbolo et al. 2012), which we further reproduced in supplementary materials (Section 3.5.3). Unlike these uniform melting experiments, the non-uniform experiments in Figure 3.2 resulted in localised, more frequent quakes, as well as accelerated creep deformations. Finally, we found that without the applied pressure no quakes develop.

These observations suggest that there are three key ingredients behind the creep-quake cycles: (1) the intrinsic length-scale of the pores, (2) the non-uniformity of the degrading agent field, and (3) the applied pressure. In hindsight, it was this understanding that originally led us to explore various experiments. Hence, to corroborate the generality of the creep-quake mechanism, we developed a simple theoretical model with these three factors at its core to explain the creep-quake behavior.

The model extends a former model for puffed rice (Einav and Guillard 2018). In both cases, a one-dimensional array of degradable elasto-brittle carriages, representative of solid-pore units, is set to sustain a uniform compressive stress  $\sigma$  under a steady, non-uniform degradation field (Figure 3.4 (a)). Unlike the previous model, here the model is not limited to porous media weakened by capillary rise, so that the ubiquity of the creep-quake phenomenon could be explained most broadly. Additionally, unlike soaked puffed rice (Einav and Guillard 2018), in the more general cases, the solid medium can recover a finite strength after localised failure due to pore closure and rearrangement. To model this, carriages are let to both degrade continuously as well as collapse multiple times. The evolution laws of the  $i^{th}$  carriage's failure strength  $\sigma_f^i$  and equilibrium length  $w_{eq}^i$  are taken as follows:

$$\dot{\sigma}_f^i = \sigma_f^i \left[ -\beta \bar{\alpha}^i + \zeta \cdot \delta(\sigma_f^i - \sigma) \right], \quad (3.1)$$

$$\dot{w}_{eq}^i = w_{eq}^i \left[ -\kappa \bar{\alpha}^i - \gamma \cdot \delta(\sigma_f^i - \sigma) \right], \quad (3.2)$$

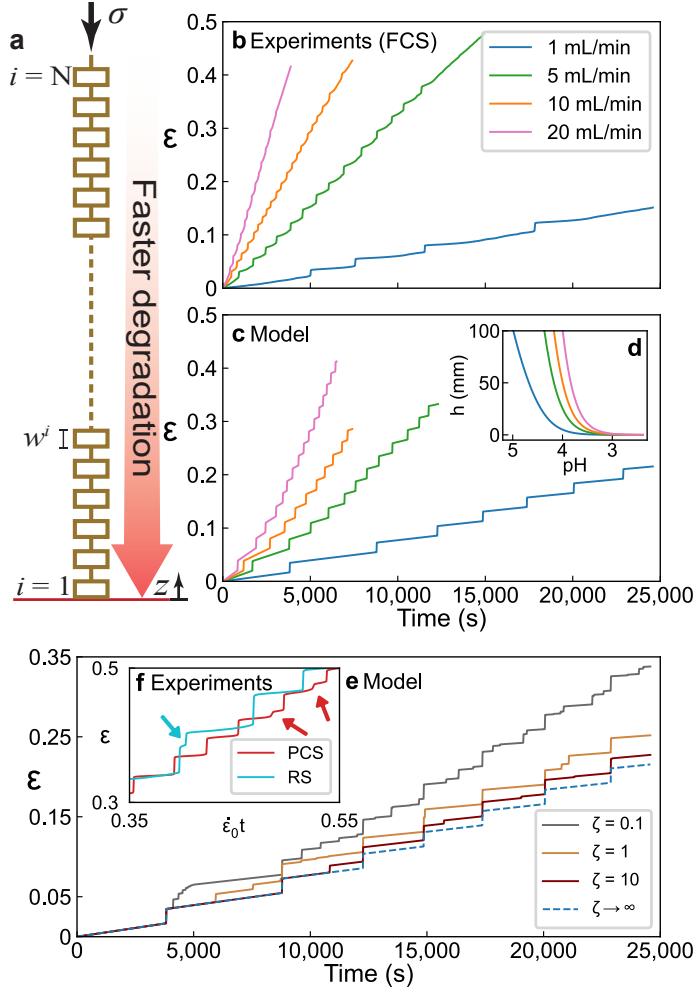


FIGURE 3.4. **Creep-quake model.** **a**, Elasto-brittle carriages under constant stress  $\sigma$  and degradation field. **b**, Strain  $\epsilon$  over time of fully saturated calcareous sand (FCS) experiments under different acid injection rates. **c**, Simulations of each experiment. **d**, pH fields recovered from chemical kinetics simulations. **e**, The effect of strength recovery post failure through  $\zeta$  on the irregularity of creep-quake cycles. **f**, Similar irregularities observed in other materials (from Figure 3.3).

where  $\bar{\alpha}^i$  is the degradation rate of that carriage, while the Dirac  $\delta$  leads to step changes in the strength and equilibrium length when the strength reaches the applied stress. To this end, the constants  $\zeta$  and  $\gamma$  control the sudden strength increase and equilibrium length decrease caused by pore collapse during quakes, respectively. The constant  $\beta$  controls the rate of reduction of failure strength due to the degradation process, while  $\kappa = 1 - \sigma/K$  depends on the elastic stiffness  $K$ , so that the actual length of the carriage is then  $w^i = \kappa w_{eq}^i$ . The overall

predicted strain is then calculated from  $\varepsilon = (H_0 - \sum w^i)/H_0$ . Constants  $\beta$ ,  $\kappa$ ,  $\gamma$ , and  $\zeta$  are dimensionless,  $\bar{\alpha}^i$  and  $\delta$  carry units of rate, and  $\sigma$  units of pressure.

The above model is simple, yet it can explain the creep-quake cycles most generally, as it can capture different degradation processes in different materials. The key to the emergence of creep-quake cycles lies in the link between the degradation of the  $i^{th}$  carriage to the local concentration of degrading agent around it, which is captured by the rate of degradation  $\bar{\alpha}^i$ . In the case of puffed rice, this rate is driven by fluid absorption in particles; for salt and calcareous sand, by dissolution; and in ice, by melting through heat. Considering the case of calcareous sand saturated in acid, one can recover  $\bar{\alpha}^i$  directly from the chemical kinetics of acetic acid and calcium carbonate (see Section 3.4 Materials and Methods). Regarding the effects of acidic dissolution on the material, previous constitutive models on bonded granular materials have adopted a similar approach of elastic strength reduction by means of a shrinking ‘yield cap’ that arises as a result of bond deterioration (Nova et al. 2003). This is comparable to the approach chosen for the evolution of  $\sigma_f^i$ , only that in one-dimension the shrinking yield surface is captured by a single point, which reduces in magnitude.

In order to evaluate the performance of the model, corresponding experiments with saturated calcareous sand were performed under various acid injection rates, which yielded different strain dynamics, showing quicker material compaction for faster injection rates (Figure 3.4 (b)). Through simple numerical integration (Section 3.4 Materials and Methods), the model successfully recovers this effect (Figure 3.4 (c)) thanks to the distinct  $\bar{\alpha}$  fields that arise from the pH profile for each flow speed (Figure 3.4 (d)). These profiles broadly agree with the experimental observations in Figure 3.3 (c). Most significantly, the model further recovers the generally consistent regular dynamics of creep-quake cycles observed experimentally. For sand, this is done by taking  $\zeta \rightarrow \infty$ , the limit where carriages could only crush once. Conversely, finite values of  $\zeta$  allow carriages to crush multiple times, thus yielding irregular creep-quake dynamics that are more consistent with some of the other materials (Figure 3.4 (e-f)).

Using an array of experiments with different materials, this chapter shows that the creep-quake failure mechanism first discovered in puffed rice can develop in any other porous media, as

long as they are subjected to simultaneous non-uniform degradation field and pressure. The ubiquity of this phenomenon was explained using a simple theoretical model that underpinned the development of both regular and irregular collapses, in light of these key factors and the length-scale associated to the pore size of the material.

Given the ubiquity of the creep-quake cycle mechanism, we anticipate that the process could be discovered in an even wider range of materials, provided the right experimental conditions. Further studies should allow to pinpoint such conditions, and probe more carefully the sensitivity of the magnitude and frequency of the collapse cycles to the pressure and the non-uniformity of the degrading agent. The model also provides a way to predict conditions that go beyond laboratory constraints. Such a comprehensive understanding of the creep-quake cycle instability is crucial in order to tackle important problems. For instance, this can support research into the stability of soils exposed to acid rain (Krug and Frink 1983), chemically attacked tailing dams (Lottermoser and Ashley 2005; Alonso and Gens 2006), CO<sub>2</sub> sequestration sites (Espinoza et al. 2011), salt deposits exposed to water filtration (Frumkin et al. 2011; Pringle et al. 2012), and icequakes in regions with extreme freeze-thaw cycles (Walter et al. 2008; Peng et al. 2014). None of these problems can be addressed without properly accounting for the instabilities of porous materials under non-uniform material degradation.

## 3.4 Materials and Methods

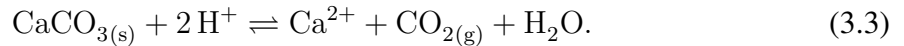
### 3.4.1 Degradation rate (FCS experiment)

According to Equations 3.1 and 3.2, the model relies on the degradation rate of the  $i^{th}$  carriage  $\bar{\alpha}^i$ . In the case of the fully saturated calcareous sand in a flowing acidic solution (FCS experiment), this rate represents the fraction of solid mass removal per unit time in that carriage. As the carriages have a finite size, the degradation rate of a given carriage could be related to a smooth field of local degradation rate  $\alpha(z)$  through spatial integration along

that carriage's length. That local degradation field  $\alpha(z)$  can be estimated from the chemical kinetics of the corresponding materials, all while assuming steady state fluid advection.

To this end, we first calculate the mass loss from chemical degradation in time for a homogeneous system. The chemical kinetic simulations were performed using the software Reaktoro (Leal and contributors 2014). The simulated sample volume was first prepared by introducing the appropriate mass of acetic acid in water to reach an initial equilibrium pH of 2.4 for the solution, corresponding to 0.9 mol/L concentration of acetic acid. These values were chosen to match the experimental pH and vinegar concentration measurements at the beginning of the FCS experiment. A set mass of calcite was then introduced to the simulated sample, corresponding to the volume occupied by the sand grains within the experimental tube at their maximum void ratio  $e_{\max}$  of 0.805 and a particle density of 2500 kg/m<sup>3</sup>.

The kinetic evolution equations are then solved, with the mass of calcite and the concentration of acid decreasing over time due to the chemical reaction. Specifically, the time evolution of the mass of calcite, the concentrations of ions ( $\text{H}^+$ ,  $\text{OH}^-$ , Acetate,  $\text{Ca}^{2+}$ ,  $\text{CO}_3^{2-}$ ), and the amount of  $\text{CO}_2$  gas released, are obtained from the chemical reaction equations for the different species. In this case, the global equilibrium of the solution under the acidic conditions is specified by:



In the physical experiments,  $\text{CO}_2$  gas escapes the system through the opening of the test tube. The overall kinetics are controlled by the slowest reaction, which is the dissolution of the solid calcite:  $\text{CaCO}_{3(\text{s})} \rightarrow \text{Ca}^{2+} + \text{CO}_3^{2-}$ . The rate of dissolution depends on the surface area of solid exposed to fluid. For simplicity, we take this surface area as a constant and approximate it by calculating the surface area of an assembly of spherical grains with a void ratio  $e_{\max}$  and diameter 1 mm, which is reasonably close to our initial calcite sample, leading to a surface area of  $\sim 24 \text{ cm}^2/\text{g}$ . In reality, there is a two-way coupling between the reaction rate and the dissolution of the grains, as specific surface area and void ratio increase and therefore speed up the rates of reaction. These effects are omitted with our simplifications to allow us to obtain a first-order estimation on the reaction rate of the sand in the acid. The chemical

kinetic and reaction constants are obtained from the compilation of water-mineral interaction parameters prepared by the U.S. Geological Survey (Palandri and Kharaka 2004). Namely, the parameters used for the reaction within the acidic regime were the rate constant  $k_r$ , where  $\log(k_r) = -0.30 \text{ mol/m}^2\text{s}$ , and activation energy  $E_a = 14.4 \text{ kJ/mol}$  for calcite. The sand samples used in our experiments contained a large proportion of aragonite, a different crystal structure to calcite for the same molecule, calcium carbonate. This structure is slightly more soluble in water than calcite. However, available literature on higher order chemical kinetics simulations (Sulpis et al. 2022) reveal that this discrepancy in solubility is unlikely to cause a major change in the degradation gradient used for the simulations shown in this chapter. Hence, to maintain a simplistic first order approach, the simulations were performed with a mass of pure calcite. Under these assumptions, the time evolution of the mass of calcite in acid was obtained.

From this time evolving mass depletion, the spatial degradation field of the mass depletion rate of calcium carbonate per unit mass  $\alpha(z)$  was obtained by assuming a constant flow rate of solution through a medium with constant void ratio. The former assumption reflects the steady advection of fluid through the sample, while the latter corresponds to the consolidation effect from the constant compressing load. The pH fields resulting from these calculations are shown in Figure 3.4 (d). Finally, the degradation rate  $\bar{\alpha}^i$  for the  $i^{th}$  carriage, with centre at the location  $z = z^i$ , was obtained by numerically averaging  $\alpha(z)$  along the carriage length  $w^i$ .

### 3.4.2 Model implementation

The model consists of a one-dimensional array of elasto-brittle carriages. Prior to commencing the simulation, each carriage (from  $i = 1$  to  $N$ ) was assigned an initial equilibrium length  $w_{eq0} = H_0/N$ , as well as an initial coordinate for its centre  $z_0^i = (i - 0.5)w_{eq0}$ , initial failure strength  $\sigma_{f0}$ , stiffness  $K$ , and applied stress  $\sigma$ .

The selected value of  $K$  was informed by available literature on calcareous sands from Bass Strait, Australia (Airey et al. 2021), which have a similar composition as the sand used in our experiments. The rest of the constants were fitted to best represent the experimental



observations. Specifically,  $\sigma_{f0}$ ,  $\beta$ , and  $\zeta$  were adjusted to capture quake frequency, and  $\gamma$  to capture the quake drops in height. Accordingly, the initial parameters chosen for the simulated results in Figure 3.4 (c) were:  $w_{eq} = 5$  mm (approximately five grain diameters),  $N = 20$  (to generate an initial 100 mm tall sample),  $\sigma_{f0} = 10$  MPa,  $\beta = 10$ ,  $\gamma = 0.4$ ,  $\zeta \rightarrow \infty$ , and  $K = 200$  MPa.

While  $K$  and  $\sigma$  were identical for all carriages and kept constant in time, carriage position  $z^i$ , length  $w_{eq}$ , and failure strength  $\sigma_f^i$  evolved following Equations 3.1 and 3.2. The two equations were integrated with a 4<sup>th</sup> order Runge-Kutta scheme (Runge 1895; Kutta 1901). As long as the stress was smaller than the failure strength, their simplified forms  $\dot{\sigma}_f^i = -\beta\bar{\alpha}^i\sigma_f^i$  and  $\dot{w}_{eq}^i = -\bar{\alpha}^iw_{eq}^i$  were integrated. During quakes, whenever the stress was larger than the failure strength, the Dirac terms were implemented as conditional statements within the numerical integration. This form of Runge-Kutta integration was compared to a simple Euler integration under a range of time steps spanning several orders of magnitude. The two methods converged to yield the same result for time steps smaller than about one second. Hence, a time step of 0.1 seconds was used for the simulations behind Figure 3.4 (c).

## 3.5 Supplementary Materials

### 3.5.1 Creep-quake cycles and the ‘Stick-slip’ model

During the formulation of the theoretical model described in this chapter, other existing models that can capture similar patterns of motion were considered. One of the best known examples is the ‘stick-slip’ model which is typically represented by a frictional slider connected via an elastic spring to a constant velocity boundary, as depicted in Figure 3.5. In this model, as the spring compresses, it accumulates elastic energy and increases the stress on the slider. Once this applied stress exceeds the frictional resisting stress from the surface the slider rests on, the slider ‘slips’, which causes the frictional resistance to drop to a dynamic frictional stress. Furthermore, the spring deforms towards its equilibrium length and its applied stress on the slider reduces. This effect causes the slider to slow down and return to rest or ‘stick’. Then,

the cycle re-starts and the ‘stick-slip’ behaviour repeats itself. This model is widely used to describe strike-slip faults and recover earthquake dynamics (Brace and Byerlee 1966; Brace 1972; Sleep and Blanpied 1992).

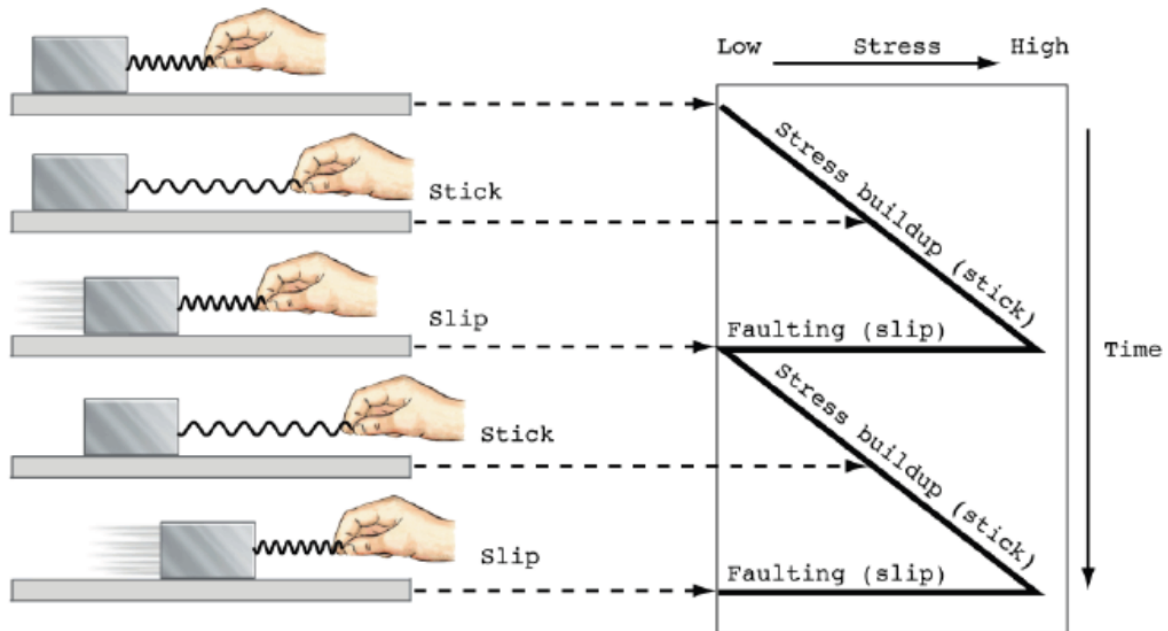


FIGURE 3.5. **Stick-slip mechanism** (Marshak 2015).

While this model is highly effective in capturing the relevant physics of fault sliding, we do not consider it appropriate for the physics that drive the creep-quake cycles in the experiments presented in this chapter. There are three main reasons for which this model was excluded from consideration. First, it requires a constant injection of mechanical work as a boundary condition. For stick-slip, there is a constant velocity which deforms the spring and drives the eventual sliding of the block. In modelling earthquakes, this velocity is given by the movement of the Earth’s crust. However, creep-quake cycles occur under constant load. In the experiments presented in this chapter, there is no injection of mechanical work, besides the initial application of the vertical stress. What drives the mechanism to emerge is the degradation processes which lead to the softening of the porous media and eventually to its collapse. If the degradation of the material is stopped, creep-quake cycles do not develop. Second, the stick-slip model relies on a frictional stress resisting the applied motion. While one could argue that there is an influence of friction from the boundary walls on the behaviour of the calcareous sand, this argument weakens when including the results from the experiments

on ice. The friction between the ice and the boundary walls is minimal and can be disregarded, as has been done for similar experiments in snow (Barracclough et al. 2017). Third, a one-dimensional array of spring sliders would not recover the solid body motion that is observed experimentally. Instead, there would be a propagation of motion as the first slider moves which would manifest in a moving compaction front from the origination of movement to the top of the sample. Therefore, even though the stick-slip model recovers similar patterns of motion, it does not appropriately capture the relevant physics that drive the creep-quake cycles in the experiments presented here.

### **3.5.2 Experimental details**

All experiments were performed in the Particles and Grains Laboratory at the School of Civil Engineering of The University of Sydney. During the experiments, photographs were taken with a Nikon D3300 camera. For the servo-controlled load experiments, the loading frame used was a Tinius Olsen H5KS; the samples were loaded at a rate of 5 mm/min until the target load was reached. Additional information related to each of the experiments is provided below.

#### **3.5.2.1 Puffed rice**

The puffed rice experiments were performed on Kellogg's® Rice Bubbles, a commercially available cereal. Before testing, the puffed rice grains were kept in sealed containers with silicon beads to avoid humidity changing their mechanical properties (Ituen et al. 1986; Peleg 2015). The tube used to test the puffed rice was made of perspex and had an inner diameter of 140 mm, while the initial height of the sample within it was 170 mm. A load of 500 N was applied to the material with the loading frame, and then that load was targeted to be closely maintained using a servo-controller. The puffed rice grains were then degraded by injecting 100 mL of water from the bottom of the sample. Here, there was no renewal of degrading agent; water was only injected once, at the beginning of the experiment. Around 10 mm of the cereal column became fully saturated and collapsed before creep-quake cycles could be

observed. Similar experiments were previously reported (Einav and Guillard 2018), where further details can be found.

### 3.5.2.2 Calcareous Sand

The calcareous sand experiments were performed on Classica® Marine Aragonite, a commercially available aquarium sand sold by Yi Hu Fish Farm Trading, a subsidiary of Qian Hu Corporation Limited. The sand was carefully sieved so that the tested samples would only contain grains between 0.6 mm and 1.18 mm. The sieved sand had a minimum void ratio  $e_{\min}$  of 0.575 and a maximum void ratio  $e_{\max}$  of 0.805; these were obtained following the standard testing procedures in ASTM D2454-16 and ASTM D4253-16, respectively. The acetic acid solution used was a commercially available product by Coles called 'Double Strength White Vinegar'. The vinegar was an aqueous solution with 8% acetic acid by weight, and it was tested to have an initial pH of 2.4. The solution was mixed with a pH indicating dye (Methyl Orange) in order to observe the pH changes as it travelled through the material. The color change from red-orange to yellow can be seen in the supplementary videos included in the supplementary materials.

The test tube used was also made of perspex and had an inner diameter of 34.2 mm. Similar to the puffed rice experiments, a nominal load was applied to the material with the loading frame, and this load was then targeted to be closely maintained using a servo-controller, here at 4 kN. Both fully and partially saturated sand samples were prepared to have an initial height of 100 mm prior compression.

In the partially saturated experiments, samples were prepared by slowly pouring the sand into the test tube. The acetic acid was injected into the lower part of the sample from four equally spaced inlets on the sides of the tube, 10 mm above the bottom. The solution was allowed to drain through the perforated bottom of the sample. Similar to the experiments on puffed rice, the liquid rose within the medium due to capillary forces, forming a saturation field which is linked to the degradation field, see Figure 3.2 (middle column, second row). In contrast, the fully saturated samples were prepared by pluviating sand into a test tube already full with water. After the samples were prepared and loaded with the loading frame, acid was injected

through a porous plate at the bottom of the sand column. The injected fluid was allowed to flow through the column and to exit the test tube through an outlet pipe located above the top surface of the sample.

The pH measurements presented in Figure 3.3 (c) were collected using five Atlas Scientific Spear Tip pH probes and a National Instruments Data Acquisition Card. The probes were staggered through the sides of the test tube to measure pH in real time throughout the duration of the experiment. Then, a set of one hour readings, taken at a rate of approximately 0.75 readings per second, was averaged to determine the data points shown in Figure 3.3 (c).

### **3.5.2.3 Rock Salt**

The rock salt experiments were performed on a commercially available product by Coles called 'Pink Himalayan Rock Salt'. The rock salt grains have a nearly unimodal grain size distribution, with a median grain size of about 1.8 mm, as shown in Figure 3.6. The material was kept in sealed glass containers with silicon beads to minimise moisture inside the container. Similar to the sand tests, the tube used in the rock salt experiments used was made of perspex and had an inner diameter of 34.2 mm. The rock salt was prepared to have an initial height of 100 mm. The grains were pluviated in salt brine (2.5% by weight), made from tap water and table salt, in order to eliminate any dissolution occurring before the start of the experiment. Once prepared, a load of 500 N was applied to the material with the loading frame and servo-controlled load system, as detailed for the previous materials. Then, fresh water was injected from the bottom of the sample, allowing the fluid to flow through the salt column, and out of the test tube column via an outlet pipe above the sample, as with the fully saturated sand experiments described earlier.

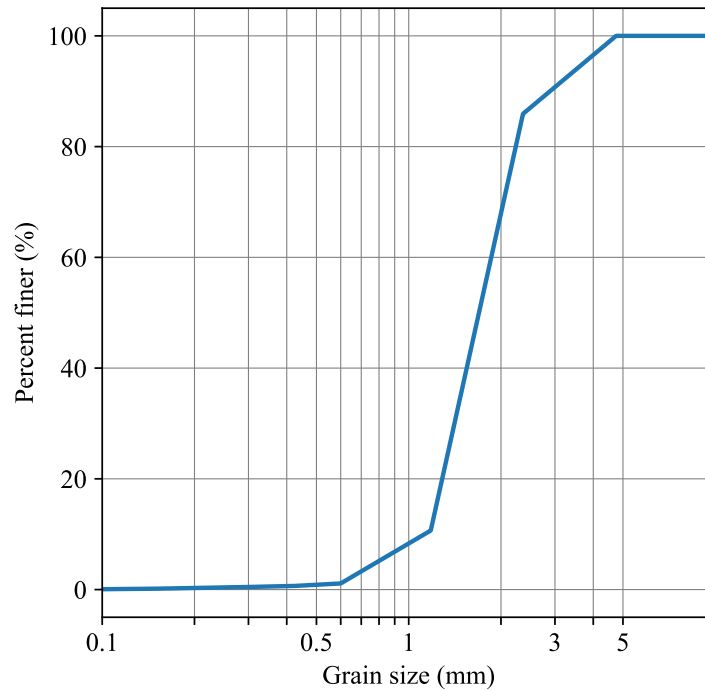


FIGURE 3.6. **Grain size distribution of rock salt samples.**

#### 3.5.2.4 Ice

For the ice experiments, ice cubes made in a home grade freezer at  $-12^{\circ}\text{C}$  were hand crushed inside a fabric bag with a rubber end hammer until all grains were smaller than 15 mm. The test tube used was perspex and had an inner diameter of 80 mm. The tube was thermally insulated with a layer of foam (50 mm thick) around its walls, leaving only a small portion of the tube not covered, to be used as an observation window through which photographs of the ice were taken. The whole setup was placed in a home grade fridge at  $2^{\circ}\text{C}$  along with the 2.5% salt brine a day prior to the experiments, so as to allow starting the degradation testing in controlled cold conditions.

The ice was poured into the test tube up until it reached a height of 120 mm. A load of 1.215 kg (11.92 N) was then applied to the samples with a set of metallic weights attached to the top piston, acting as a dead load. Notice that unlike the previously detailed tests, here the ice was not loaded with the loading frame due to the overly large quake deformations in the ice. These deformations resulted in a discontinuous application of load if applied by the

loading frame. After loading, the brine at 6°C was pumped into the system from the bottom of the tube until the water level was approximately 20 mm above the top of the ice column. This configuration was left in place for 5 minutes for the setup to be closer to thermal equilibrium with the ice and the brine.

To apply the heat flux field within the ice medium, hot water (30°C) was pumped through a coiled plastic pipe located under the bottom porous plate (30 mm away from the bottom of the ice column). The hot water ran in a closed loop so there was no fluid exchange with the brine. The pump rate of the hot water was fast enough to ensure that the initial loss of temperature of the water before and after entering the system was less than 2°C.

### **3.5.3 Comparison with ice assembly uniform degradation field**

In order to further understand the emergence of creep-quake cycles, we reproduced previous ice assembly experiments, in which a similar phenomenon is reported under a uniform degradation field (Dorbolo et al. 2012). Even though we used a differently sized tube and ice cubes (half the size), the relative scale of the current experiment and those previously reported (Dorbolo et al. 2012) was maintained. In our experiments, 362 ice cubes with dimensions of 10 mm were placed in a perspex tube with an 80 mm inner diameter. The initial temperature of the brine used was 6°C. The ice cubes were loaded with a 1.166 kg dead load stress applied on top of the sample. A layer foam was used to insulate the tube, as with our main ice experiments.

Under no added heat flux (no hot water flow below the experiment), the previously mentioned observations on ice (Dorbolo et al. 2012) were successfully reproduced with both ice cubes and crushed ice (see the latter in Figure 3.7 (a)). We then performed an identical experiment on crushed ice with the key difference that a heat flux gradient was applied using the method described in Experimental Details (Ice) (Figure 3.7 (b)), pumping hot water at 45°C through the system. The increase in quake occurrences and emergence of the creep-quake cycles is clearly observed in Figure 3.7 (b). It then becomes clear that the collapse mechanisms observed under the two melting conditions are indeed different. Notably, the presence of a

non-uniform degradation field is a stronger agent for the emergence of distinct and recurring creep-quake cycles.

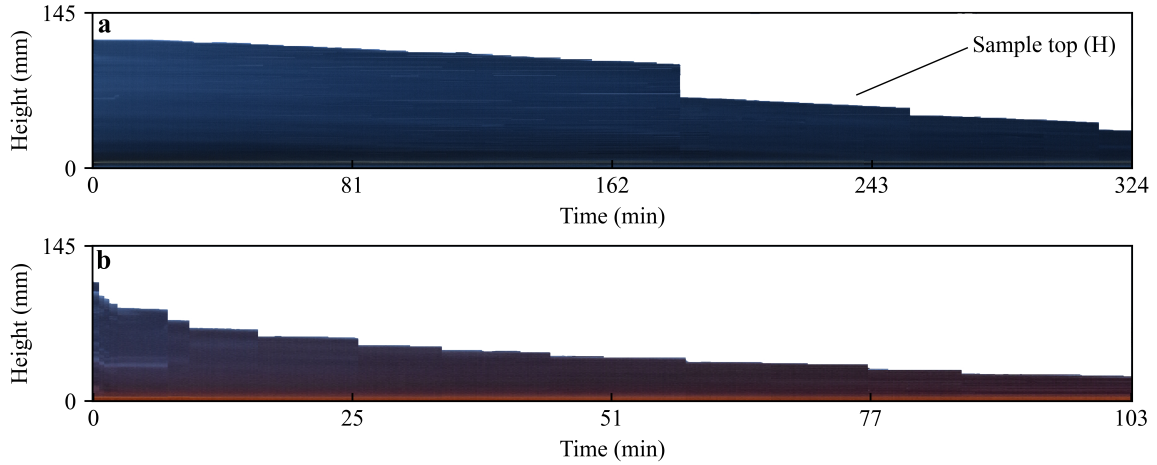


FIGURE 3.7. **Degradation gradient.** Spatiotemporal plots for **a**: crushed ice without a heat gradient, and **b**: crushed ice with a heat gradient.

### 3.5.4 Media

A collection of time-lapse videos of the experiments presented in this publication are available in an online repository ([https://www.dropbox.com/sh/rm2unm3k8kwlpve/AABfxDoStCI\\_WWqKXUtPKBaRa?dl=0](https://www.dropbox.com/sh/rm2unm3k8kwlpve/AABfxDoStCI_WWqKXUtPKBaRa?dl=0)). This repository contains the following files:

- Video\_1: Time-lapse of a 100 mm tall calcareous sand sample compressed with a servo-controlled target load of 4 kN and partially saturated in acetic acid solution with an injection flow rate of 30 mL/min via four inlets attached to the tube wall.
- Video\_2a: Time-lapse of a 100 mm tall calcareous sand sample compressed with a servo-controlled target load of 4 kN and fully saturated in acetic acid solution with an injection flow rate of 1 mL/min from the bottom.
- Video\_2b: Time-lapse of a 100 mm tall calcareous sand sample compressed with a servo-controlled target load of 4 kN and fully saturated in acetic acid solution with an injection flow rate of 5 mL/min from the bottom.



- Video\_2c: Time-lapse of a 100 mm tall calcareous sand sample compressed with a servo-controlled target load of 4 kN and fully saturated in acetic acid solution with an injection flow rate of 10 mL/min from the bottom.
- Video\_2d: Time-lapse of a 100 mm tall calcareous sand sample compressed with a servo-controlled target load of 4 kN and fully saturated in acetic acid solution with an injection flow rate of 20 mL/min from the bottom.
- Video\_3: Time-lapse of a 100 mm tall rock salt sample compressed with a servo-controlled target load of 0.5 kN and fully saturated in brine with an injection flow rate of fresh water of 5 mL/min from the bottom.
- Video\_4: Time-lapse of a 120 mm tall crushed ice sample compressed with a dead load of 11.92 N and fully saturated in brine under a heat input gradient applied by running water at 30°C at the bottom of the sample.

## CHAPTER 4

### **An in-depth study of creep-quake cycles in geomaterials**

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#### **4.1 Introduction**

The findings described in Chapter 3 were essential to the identification of creep-quake cycles as a novel collapse mechanism that could extend from puffed rice cereals to geomaterials. However, creep-quake cycles are not a commonly described occurrence in nature, despite the several reports of degradation-induced collapse, such as those resulting from sinkholes (Waltham et al. 2005; Shalev et al. 2006; Vey et al. 2021). This may point towards the existence of a specific set of conditions required for this mechanism to emerge in geomaterials. While Chapter 3 excluded in-depth investigations of individual materials to focus on the generality of the mechanism, this chapter will delve into the specific conditions necessary for creep-quake cycles to emerge in a calcareous sand.

The samples used in this chapter were from ‘Barry beach sand’, a Bass Strait carbonate sand found in south Victoria, Australia. The material and mechanical properties of this sand have been previously studied in (Al-Douri and Poulos 1992; Poulos 1986; Airey and Kelly 2010; Hull et al. 2021). We transition to the use of Barry beach sand (BBS), as it is a naturally occurring material that is more closely related to the type of sand one could encounter in real geotechnical scenarios. The Classica Marine aragonite (CMA), used in the previous chapter, is a model material, which served as a proof of concept for the emergence of creep-quake cycle in calcareous sands and is highly curated by aquarium supply manufacturers.

In line with the three requirements for creep-quake cycles to emerge (a collapsible length-scale, sufficient compressive stress, and a non-uniform degradation field), this chapter will present

an exploration on the evolution of grain-scale characteristics during chemical degradation, as well as on the effects of applied stress, injection rates, and initial sample heights on the creep-quake behaviour. The chapter will begin with a comparison of the physical characteristics and behavioural differences between Barry beach sand and Classica Marine Aragonite. Next, it will show the evolving morphology of the Barry beach sand grains during an experiment and describe the impact of micro-scale changes on the macro-scale response. Lastly, an array of experiments will be used in a parameterised study that aims to determine the individual and coupled effects of applied experimental parameters on the creep-quake response. Some of the results shown in this chapter have been previously published, with Dr François Guillard and Prof Itai Einav as co-authors (Crespo-Parraga et al. 2022). This publication is included in Appendix A for reference.

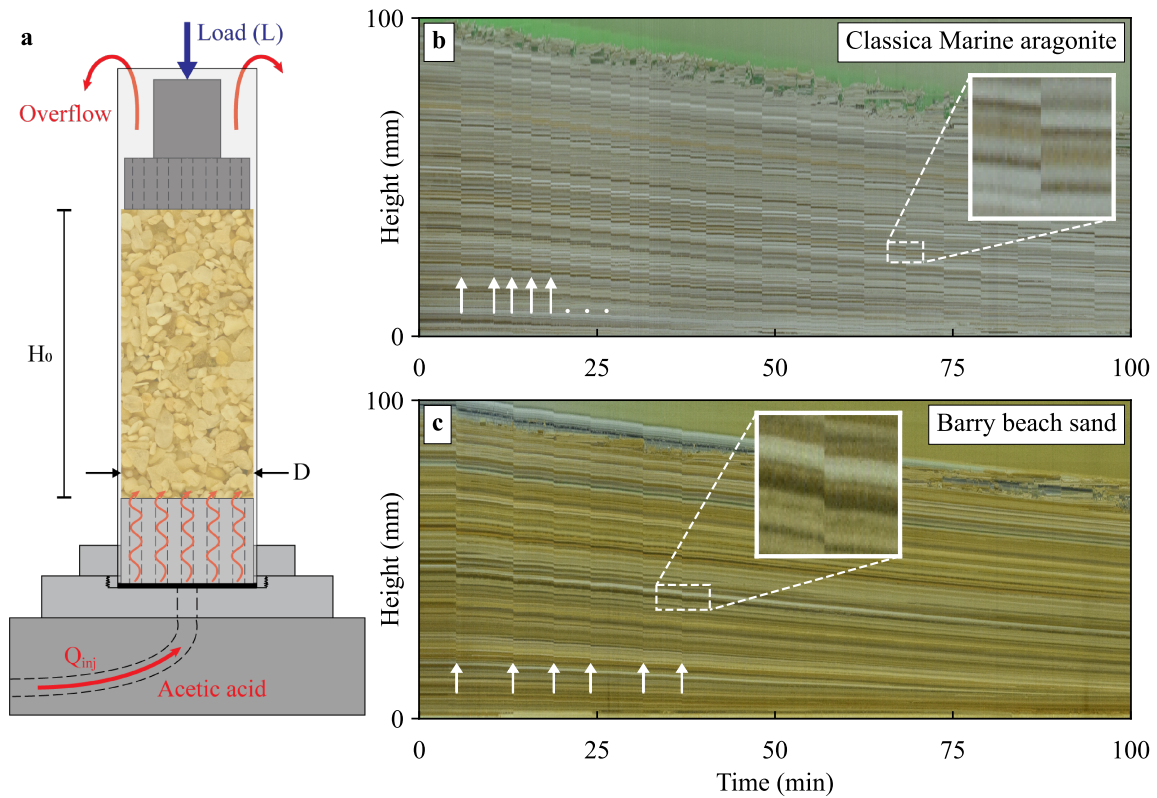
## 4.2 Methodology

### 4.2.1 Experimental setup

The experimental setup used in this chapter, as well as the preparation of the Barry beach sand samples were identical to that described in Chapter 3 for the fully saturated calcareous sand experiments. This approach was chosen to maintain consistency between the two different sand samples used. In each experiment, a sand sample with an initial height  $H_0$  was compressed to a target load  $\sigma_t$  by a servo-controller, while acid at a concentration  $C_a$  was injected into the bottom of the sample at a constant flow rate  $Q_{inj}$ . A diagram of the specific setup used in these experiments is presented in Figure 4.1.

With the application of sufficient compressive stress and a non-uniform degradation field, Barry beach sand develops creep-quake cycles similar to those observed in Classica Marine Aragonite. A comparison of the behaviour of the two sands under identical conditions is shown in Figure 4.1. However, it is immediately apparent that the response of the two sands is not identical. For instance, the Barry beach sand exhibits a lower number of creep-quake cycles before the phenomenon stops, although the dissolution continues to occur. On the other

hand, the Classica Marine aragonite continues to creep and quake throughout the duration of the experiment. This discrepancy may be due to micro-scale variations between the two sands. Namely, the high angularity of the Barry beach grains may cause a more interlocked sample, resulting in a lower void space available for grains to dislodge/break and cause a quake. In later sections of this chapter, we will further investigate this hypothesis by assessing the morphology of the Barry beach sand systematically with a series of samples from the sand at various stages of an experiment.



**FIGURE 4.1. Creep-quake cycles in two calcareous sands.** **a:** Experimental setup. A 34.2 mm inner diameter ( $D$ ) perspex tube holds the sand samples over a porous plate from which acid is injected while a loading frame compresses the sample with a porous piston head. **b, c:** Spatiotemporal plots for two nearly identical experiments on Classica Marine aragonite (b) and Barry beach sand (c). The parameters of these experiments were:  $H_0 = 100$  mm,  $\sigma_t = 4L/\pi D^2 = 4.35$  MPa,  $Q_{inj} = 10$  mL/min, with  $C_a = 8\%$  by weight for CMA and  $C_a = 4\%$  by weight for BBS.

### 4.2.2 Comparison of sand characteristics

Most of the experiments presented in this chapter were performed on Barry beach sand. Additionally, the experiments on fully saturated Classica Marine aragonite from Chapter 3 were re-used here to compare the response of the two sands under equivalent conditions.

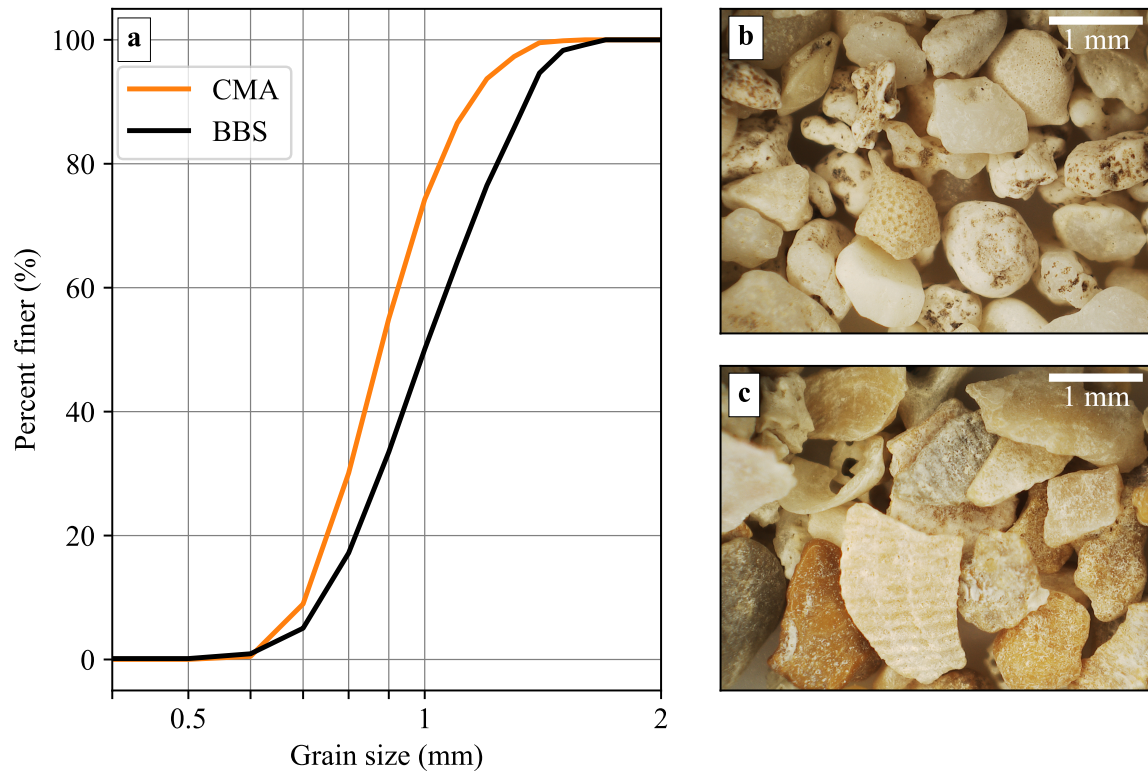
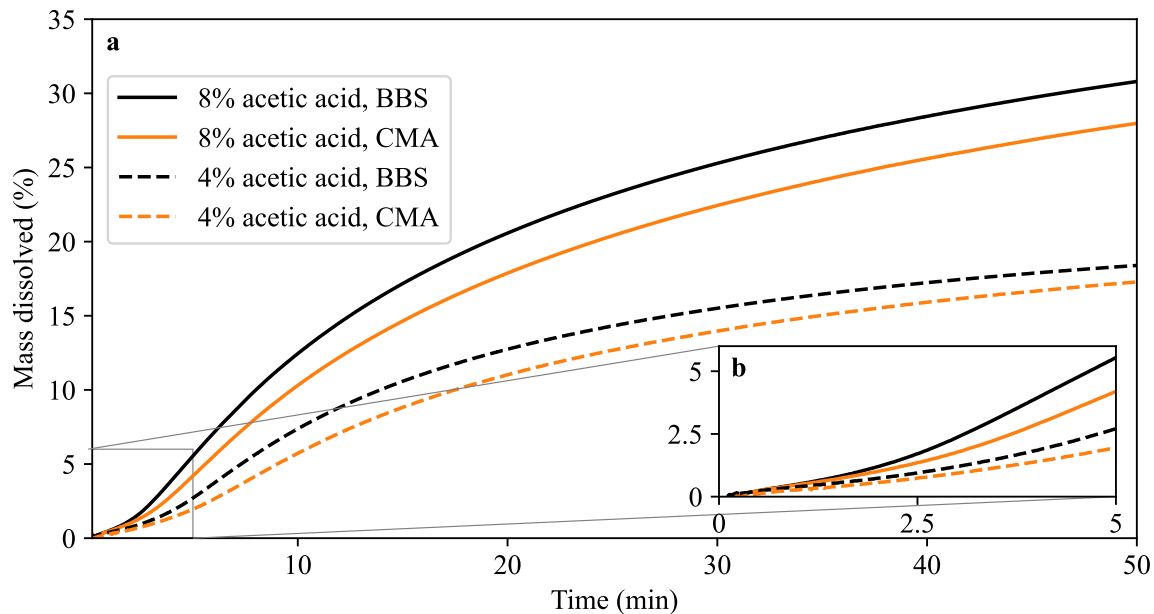


FIGURE 4.2. **Material characteristics.** **a:** Grain size distribution curves for Classica Marine aragonite (CMA) and Barry beach sand (BBS). **b,c:** Macroscopic photograph of Classica Marine aragonite (b), and Barry beach sand (c). The samples presented in this figure were sieved for grain sizes between 0.600 mm and 1.180 mm.

To maintain consistency with the experiments performed on the CMA samples, the BBS samples were sieved to keep only the grains that remained between 0.600 mm and 1.180 mm sieves. The grain size distribution curves for the two sieved sand samples can be found in Figure 4.2 (a), where it is apparent that the BBS sample has a lower percentage of fines. In addition to being coarser, Barry beach sand is also characterised by its darker brown

colouration, and by its angular and irregular grains. These characteristics are evident from the macroscopic photographs shown in Figure 4.2 (b-c).



**FIGURE 4.3. Rates of reaction.** **a:** Percentage of sand mass dissolved in time for Classica Marine aragonite (CMA) and Barry beach sand (BBS). **b:** Magnified view of the first 5 minutes of dissolution. All experiments had an initial sand mass of 10 g, which was placed in 100 g of acetic acid solution at 8% or 4% concentrations by weight. Throughout these experiments,  $\text{CO}_2$  was produced as a product of the chemical reaction and escaped the system, as they were performed at room temperature and pressure. This mass loss was monitored with continuous weight measurements of the system throughout the reaction. It was then related back to the  $\text{CaCO}_3$  mass dissolved with the stoichiometric ratios of the reaction between acetic acid and calcium carbonate.

Even though they are both calcareous sands, the difference in pigmentation between the two suggests differences in their chemical composition. A more brown colour typically indicates larger concentrations of iron oxide and/or feldspar in the sand (National Oceanic and Atmospheric Administration 2017), both of which can have an effect on the rates of reaction of the sand grains to the acetic acid used to degrade them. Additionally, their differences in shape and size have a significant influence on their surface area, which affects reaction rates. From these observations, one could expect Barry beach sand samples to have an overall larger surface area available for chemical reactions, and therefore to dissolve faster than Classica Marine aragonite. While the specifics of the chemical composition and surface area of the two

sands were not determined, their reaction rates were measured by subjecting an equal mass of each sample to equal amounts by weight of acidic solution at a given concentration. Figure 4.3 shows the rates of sand dissolution in terms of the percentage of sand mass dissolved in time. As anticipated from our qualitative observations of the two sands, the rates of reaction differ, with the Barry beach sand being more reactive to the acid. Inset (b) zooms into the first 5 minutes of the reaction, as this is the approximate span of time it takes the acid to travel through a 100 mm tall sand column of porosity  $n = 0.5$ , at an injection rate of 10 mL/min. These parameters are specific to the experiments shown in Figure 4.1. In more general terms, the time needed to flush a solution at an injection rate  $Q_{inj}$  through a material cylinder of height  $H$ , diameter  $D$ , and porosity  $n$  can be estimated as  $t_{flush} = n\pi H D^2 / (4Q_{inj})$ .

Given the faster rates of dissolution of BBS, the concentration of acid used in most experiments was reduced to 4% by weight. Later in this chapter (Figure 4.11) we see how this change led to very comparable results to those obtained in Chapter 3.

### 4.2.3 Dissolution field

In Chapter 3, we simplified the creep-quake cycle phenomenon to a one-dimensional system aiming to explain its mechanics with a simple 1-D model of breakable carriages. For this assumption to be appropriate, the degradation field within the sand should be relatively uniform horizontally. One approach to assess the uniformity of this field is to make use of the available literature on dissolution and flow through porous media. Previous studies have defined two non-dimensional numbers, the Péclet (Pe) and Damköhler (Da) numbers, to describe the behaviour of a dissolving porous medium and the nature of the dissolution front traveling through it (Fredd and Fogler 1998; Golfier et al. 2001) (see Section 2.4.3). Here, we compute these numbers as follows:

$$\text{Pe} = \frac{\text{Advection rate}}{\text{Diffusion rate}} = \frac{Q_{inj}}{D_c l} \quad (4.1)$$

$$\text{Da} = \frac{\text{Reaction rate}}{\text{Advection rate}} = \frac{R}{\rho_a Q_{inj}} \quad (4.2)$$

where  $D_c$  is the diffusion coefficient for the acetic acid,  $l$  is a characteristic length,  $R$  is the rate at which the sand dissolves in acid, and  $\rho_a$  is the density of the acid. For the CMA-03 experiment, we use:  $Q_{inj} = 166.67 \times 10^{-9} \text{ m}^3/\text{s}$ ;  $l = 17.1 \times 10^{-3} \text{ m}$ , taken as the diameter of the tube (34 mm) times the approximated porosity of the sand  $n = 0.5$ ;  $R = 15 \times 10^{-6} \text{ kg/s}$ , taken as the amount of mass dissolved from the start of the reaction to the time it takes to flush the sand sample  $t_{flush} \approx 5 \text{ min}$ , as shown in Figure 4.3 (b); and  $\rho_a = 1007 \text{ kg/m}^3$ , obtained from the relative density specified by the acid solution provider (Coles Supermarkets 2016) for a water density of  $997 \text{ kg/m}^3$ . The diffusion coefficient was not directly obtained from the acid samples; it was taken as  $D_c = 1.1085 \times 10^{-9} \text{ m}^2/\text{s}$  from (Winkelmann 2018) for an acid molarity of  $0.7572 \text{ mol/L}$  (similar to that of the vinegar used). With these parameters, the Péclet and Damköhler numbers for CMA-03 are approximated to be  $Pe = 8.79 \times 10^3$  and  $Da = 8.94 \times 10^{-2}$ . These approximations are appropriate for the acid flow within the sand at the commencement of the experiment, however, as the experiment progresses and the sample changes, so would its Péclet and Damköhler numbers.

In contrast with the uniform dissolution assumptions, these  $Pe$  and  $Da$  values indicate a ‘dominant wormhole’ dissolution regime, in accordance with (Golfier et al. 2001). This response was not observed by optical means in any of the experiments performed on calcareous sands. However, it is possible that the wormhole is simply hidden within the sample and is therefore undetectable by optical means. In order to further investigate the dissolution regime of our samples, we made use of neutron radiography techniques. These allowed us to observe what occurs inside the sand samples during an experiment with a high contrast between the sand and the acid solution. Neutron radiography, compared to X-rays, provide a clearer identification of saturated and dry parts of the samples. This technique was initially chosen to measure saturation within partially saturated CMA experiments, though this investigation was inconclusive and was ultimately omitted from the contents of this thesis. However, a small number of experiments were also performed on fully saturated CMA samples. The experiments were performed in collaboration with the Australian Nuclear Science and Technology Organisation (ANSTO) and were conducted by the author and ANSTO Instrument Scientist Dr Floriana Salvemini in the ‘Dingo’ neutron beam, located in Lucas Heights (Sydney, Australia). The setup used was identical to that described in Section 4.2.1, with the only exception being that



the tube, piston head, and porous bottom plates were made primarily out of aluminium. This was a necessary change that minimised the radioactive activation of the elements in the setup and the neutron attenuation caused by it.

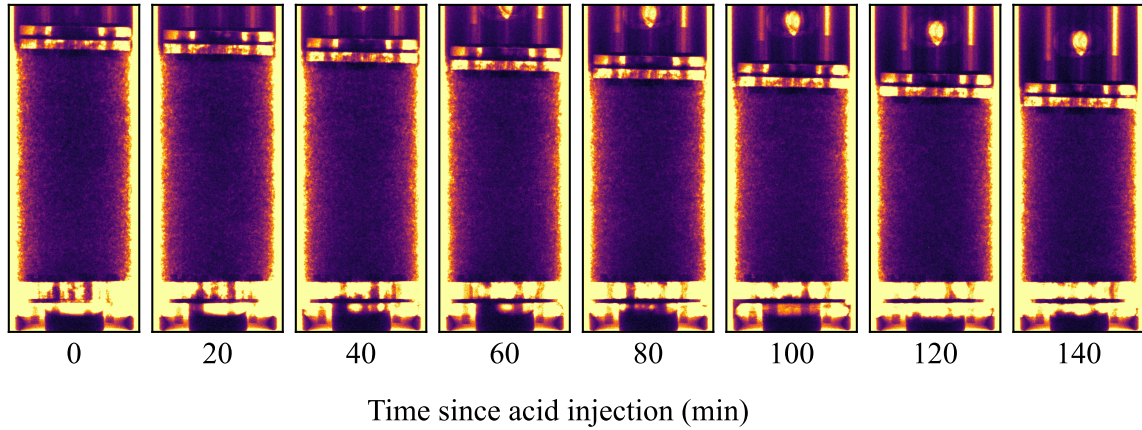


FIGURE 4.4. **Neutron radiography of experiment.** Various radiographs of an experiment similar to CMA-03 (with  $H_0 = 70$  mm) at several times since acid injection.

Figure 4.4 shows numerous neutron radiographs of an experiment on Classica marine aragonite with parameters identical to those used in CMA-03, though with a slightly smaller initial height  $H_0$  of 70 mm. Contrary to what would be expected from the analysis with the Péclet and Damköhler numbers, each of these radiographs shows a very uniform sand sample. We observe no formation of any discernible wormholes throughout the duration of the experiment. Hence, it is reasonable to conclude that the field of dissolution within our samples is generally uniform in the directions perpendicular to the loading axis of the samples, validating the assumption made in Chapter 3. The discrepancy between the expected dissolution regime and that observed experimentally could be due to the difference in material used, as well as the generation of gas bubbles, and the re-arranging behaviour of the sand grains. The experiments performed in (Golfier et al. 2001) were done in a salt medium, from which there was no gas generation. Furthermore, their experimental samples and numerical model did not include sample strains and collapse despite the opening of cavities due to the acid dissolution. It is noted the piston shows slight tilting during the experiment, however, we do not believe this has a significant impact on our analysis.

### 4.3 Grain morphology

Aiming to investigate the micro-scale characteristics of the two sands, as well as their evolution throughout an experiment, a series of morphological measurements of their grains were taken with a Malvern Morphologi G3 machine at the Particles and Grains Laboratory in The University of Sydney. This involved taking numerous sample sand grains, spreading them to minimise overlap, and making use of the Morphologi G3 software to photograph and measure them. An explanatory diagram of this process is shown in Figure 4.5, where we see an example grain and its corresponding relevant physical measurements.

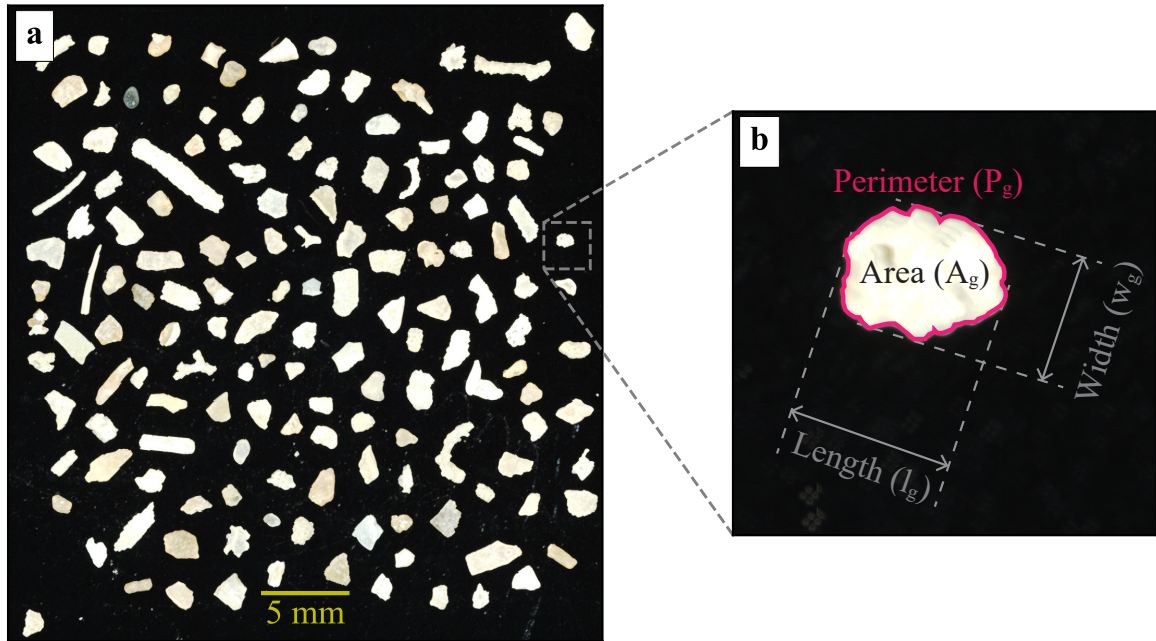


FIGURE 4.5. **Grain morphology.** **a:** Photograph of spread out BBS grains. **b:** Isolated grain with markings of its physical measurements.

#### 4.3.1 Initial grain morphology

In order to collect statistically meaningful measurements of the grain morphology of the samples, the process shown in Figure 4.5 was performed on over hundreds of individual grains. It was estimated that a minimum sample size of 400 grains was needed for meaningful

results. This was done using a Cochran sample size estimation approach (Ahmad and Halim 2017). The specific number of grains analysed for each sample can be found in Table 4.1.

As discussed with the spatiotemporal plots in Figure 4.1 (b-c), the key behind the difference in behaviour between the two sands may lie in the shape (angularity and size) of their grains. Therefore, we take particular interest in the grains size and roundness with measurements of their area, circularity, and aspect ratio. The size of the grains is taken as the area of the grains visible when photographing them,  $A_g$ . For the shape of the grains, circularity is defined as the ratio of the perimeter of a circle with the same area as that of the observed grain to the perimeter of the observed grain  $P_g$ , i.e.  $2\sqrt{\pi A_g}/P_g$ , while the aspect ratio is the ratio of width to length of the observed grain ( $w_g/l_g$ ) (Malvern Instruments 2008). These measurements were used to generate the sieve curves in Figure 4.2, as well as Table 4.1, where we can see a comparison of these shape characteristics for the two sands.

TABLE 4.1. Average morphological characteristics

|                           | Sample grain<br>count | Grain area<br>(mm <sup>2</sup> ) | Circularity | Aspect ratio |
|---------------------------|-----------------------|----------------------------------|-------------|--------------|
| Classica Marine aragonite | 1658                  | 0.718                            | 0.894       | 0.751        |
| Barry beach sand          | 672                   | 0.910                            | 0.856       | 0.720        |

Table 4.1 confirms the visual descriptions of the two sands. When evaluating the average size and shape characteristics of their grains, we can quantitatively confirm that Barry beach sand is on average coarser and has a somewhat lower circularity than Classica Marine aragonite. In contrast, the average aspect ratio of Classica Marine aragonite appears to be slightly larger than that of Barry beach sand. One caveat worth noting is that these are two-dimensional measurements of three-dimensional objects. In each grain photograph, there is a third dimension that is omitted, which is typically the smallest grain axis, since the grains tend to lie on the face that is parallel to their intermediate and long principal axes.

### 4.3.2 Grain morphology evolution in time

As the intrinsic characteristics of Barry beach sand may have a significant effect on its creep-quake cycle response, there is much to gain from an examination of the micro-scale evolution

of the sand throughout the duration of the experiment. Here, this evolution is described with a series of morphological measurements made on grain samples taken from experiments identical to that shown in the BBS spatiotemporal plot in Figure 4.1 (c). These grain samples were collected from the top, middle and bottom of the sand column for samples subjected to 10 minutes of fresh water flushing, followed by 0, 30, and 60 minutes of acetic acid injection at a concentration  $C_a$  of 4% by weight, see Table 4.2.

TABLE 4.2. Experiments for the study of the evolution of grain morphology

| Material | $C_a$<br>(%wt.) | $H_0$<br>(mm) | Target<br>force<br>(kN) | $\sigma_t$<br>(MPa) | $Q_{inj}$<br>(mL/min) | Duration of acid<br>injection |
|----------|-----------------|---------------|-------------------------|---------------------|-----------------------|-------------------------------|
| BBS      | 0.0             | 100.0         | 4.0                     | 4.35                | 10                    | No acid injection             |
|          | 4.0             |               |                         |                     |                       | 30 minutes                    |
|          | 60 minutes      |               |                         |                     |                       |                               |

Figure 4.6 depicts the grain size distributions (GSD) obtained at the three locations of the sand column after the three different durations of acid injection. From panel (a), we see that there is an initial disparity in fine particle content along the height of the sample. Since there is no particle dissolution prior to injecting acid, this is likely caused by (i) the flushing of fine particles from the bottom to the top of the tube caused by the initial injection of water, and (ii) localised grain crushing at the top of the tube resulting from the application of the target compressive stress  $\sigma_t$ . Note that the initial 10 minutes of water flow were not sufficient to flush out all the fines from the sand. While the smaller fines would have travelled at the speed of the water flow, some of the larger fine particles would have travelled slower and could have been trapped amongst other grains while moving upwards. Eventually, the initial fine content disparity reduces and disappears due to the continuous fluid flow and grain dissolution, see panels (b-c). Its transition in each location can be seen on the right side of the figure, which shows the fine content reducing with time at the top of the sand column while increasing at the bottom. The former is likely occurring due to the flushing of fines outside of the column caused by the constant fluid flow, while the latter is a result of the localised dissolution reducing the size of the grains located in this region. After 60 minutes, this process results in a consistent GSD curve for the three locations within the sand column, suggesting the

formation of a homogenised sample. In fact, this homogenisation of the sample is also visible in the measured average grain size, as shown in Figure 4.7 (a).

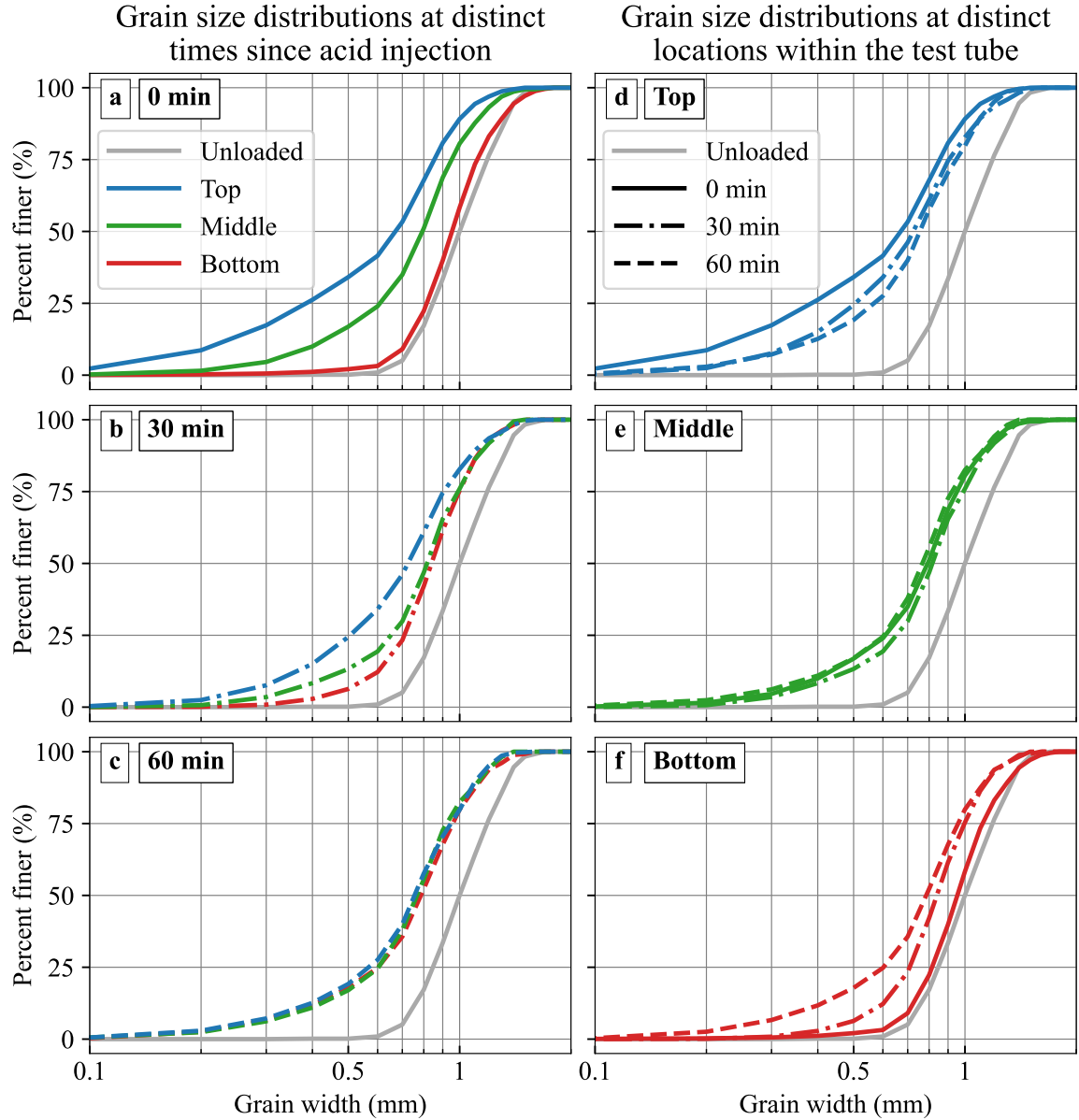
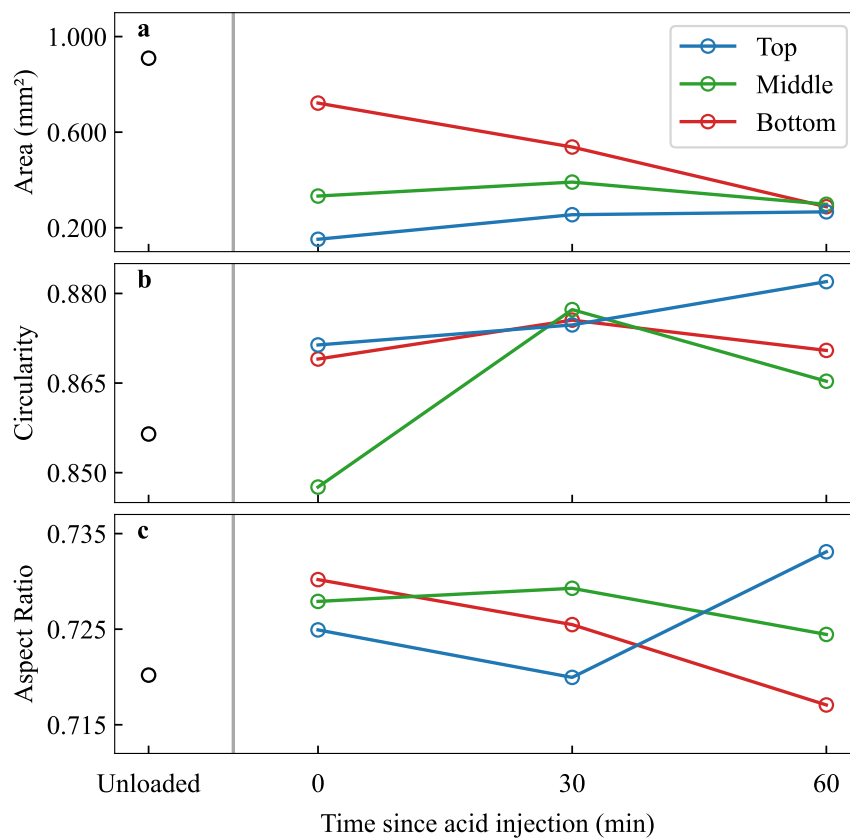


FIGURE 4.6. **Evolution of grain size distribution (GSD) curves.** Left column (a-c): Grain size distributions by location at different times since acid injection. Right column (d-f): Evolution of GSD curves in time at specific locations within the test tube. The grey curve present in all panels corresponds to an unaltered BBS sample, from Figure 4.2 (a).

It is worth noting that in these experiments, quakes would usually come to a halt within the first 40 to 50 minutes since acid injection, as seen in Figure 4.1. Therefore, this change in

GSD throughout the sample could be contributing to the ceasing of the creep-quake cycles in Barry beach sand. As the acid is injected from the bottom of the sample, the lower parts of the sand column are commonly where quakes are produced. When the fine content increases in this area, it increases the polydispersity of the sand, which results in a reduction of available pore space for quakes to occur and eventually brings this mechanism to an end. In addition, the increase of fines in between the larger grains leads to a cushioning effect of the larger grains, preventing grain breakage (Hooke and Iverson 1995).



**FIGURE 4.7. Time and spatial evolution of grain morphology.** **a:** Grain average area over time. **b:** Average grain circularity over time. **c:** Average grain aspect ratio over time. The black circle markers correspond to unloaded fresh sand grains (as seen in Table 4.1). Results obtained from experiments with the experimental parameters of BBS-01.

Similar to the grain size distribution change, the evolution of grain shape parameters at the three different times since acid injection and locations within the sample is shown in Figure 4.7. In this Figure, the circularity, and aspect ratio were taken as weighted averages

(based on the grain area). Throughout the experiment, there is a decrease in grain area (consistent with the findings of Figure 4.6), a slight increase in their circularity, and no clear trend in their aspect ratio. The increase in circularity may be explained by the breakage of sand grains. Existing ‘prongs’ or elongated parts of non-circular grains tend to break off more easily, as their connection to the main body of the particle is generally a weak zone within the grain. Furthermore, these geometrical changes can result in zones of stress concentration and bending forces that eventually lead to grain breakage. However, on close inspection the change in circularity is actually quite minute. In fact, the largest circularity reported in Figure 4.7 (b) remains lower than that of an unaltered *Classica* Marine aragonite sample. Hence, this slight increase in circularity is unlikely to have a major role in the change in behaviour of the sand column, which points towards grain size distributions being more relevant than their shape in the occurrence of quakes.

Although comprehensive, the measurements presented here have similar limitations to those discussed in Section 4.3.1, which arise from the simplified (two-dimensional) measurement of three-dimensional grains. This study could be complemented with a set of similar readings performed with non-intrusive three-dimensional imaging techniques, such as X-ray or neutron tomography. Future studies could capture the third dimension that is omitted here in order to validate our findings. The use of these imaging techniques was considered for this thesis. However, the available technology limits the sample size, spatial resolution, and time-scale at which changes can occur when injecting acid into the sand column, and therefore was not investigated further.

## 4.4 Parametric study on the influence of applied conditions

As shown in Chapter 3, the three distinct requirements needed for creep-quake cycles to occur are: A collapsible length scale (satisfied in brittle porous media by its pores), sufficient compressive stress, and a non-homogeneous degradation field. In that Chapter, we identified the need for a ‘sufficient’ compressive stress, though this was never quantified. Similarly, the effects of different ‘non-homogeneous’ degradation fields may have on the creep-quake behaviour and its coupling with different stress magnitudes were not described. So far, this chapter has investigated the role of grain morphology in creep-quake cycles, which is linked to the intrinsic collapsible length-scale of the material. Now, the remaining two requirements, will be investigated further by exploring the effects of injection rate, target applied load, and initial height with a series of parameterised experiments where each parameter was changed in isolation and later changed simultaneously.

Every experiment discussed in this study is listed in Table 4.3, along with a unique ID that facilitates their naming in text. CMA-03 and BSS-01 are the experiments from which the spatiotemporal plots in Figure 4.1 (b-c) were obtained.

Initially, experiment BBS-00 was performed in an attempt to replicate creep-quake cycles on BBS with experimental parameters identical to CMA-01. However, the faster reaction rates of this sand caused it to deform quite rapidly, which limited the span of time through which it would develop quakes. Hence, it was decided to half the acid concentration for all subsequent experiments. This started with BBS-01, which can be regarded as the ‘base’ experiment, as most BBS experiments are variations of it.



TABLE 4.3. List of experiments

| Material | $C_a$<br>(%wt.) | $H_0$<br>(mm) | Target<br>force<br>(kN) | $\sigma_t$<br>(MPa) | $Q_{inj}$<br>(mL/min) | ID     | Remarks                                    |
|----------|-----------------|---------------|-------------------------|---------------------|-----------------------|--------|--------------------------------------------|
| CMA      | 8.0             | 100.0         | 4.0                     | 4.35                | 1.0                   | CMA-01 | From Chapter 3                             |
|          |                 |               |                         |                     | 5.0                   | CMA-02 |                                            |
|          |                 |               |                         |                     | 10.0                  | CMA-03 |                                            |
|          |                 |               |                         |                     | 20.0                  | CMA-04 |                                            |
| BBS      | 8.0             | 100.0         | 4.0                     | 4.35                | 10.0                  | BSS-00 | First BBS test                             |
|          | 4.0             | 100.0         | 4.0                     | 4.35                | 10.0                  | BSS-01 | Base test                                  |
|          |                 | 100.0         | 4.0                     | 4.35                | 1.0                   | BSS-02 | Injection rate effects                     |
|          |                 |               |                         |                     | 2.5                   | BSS-03 |                                            |
|          |                 |               |                         |                     | 5.0                   | BSS-04 |                                            |
|          |                 |               |                         |                     | 15.0                  | BSS-05 |                                            |
|          |                 |               |                         |                     | 20.0                  | BSS-06 |                                            |
|          |                 | 100.0         | 0.5                     | 0.54                | 10.0                  | BSS-07 | Load magnitude effects                     |
|          |                 |               | 1.0                     | 1.09                |                       | BSS-08 |                                            |
|          |                 |               | 1.5                     | 1.63                |                       | BSS-09 |                                            |
|          |                 |               | 2.0                     | 2.18                |                       | BSS-10 |                                            |
|          |                 |               | 3.0                     | 3.27                |                       | BSS-11 |                                            |
|          |                 | 50.0          | 4.0                     | 4.35                | 10.0                  | BSS-12 | Initial height effects                     |
|          |                 | 75.0          |                         |                     |                       | BSS-13 |                                            |
|          |                 | 125.0         |                         |                     |                       | BSS-14 |                                            |
|          |                 | 150.0         |                         |                     |                       | BSS-15 |                                            |
|          |                 | 50.0          | 1.0                     | 1.09                | 10.0                  | BSS-16 | Multiple simultaneous<br>parameter changes |
|          |                 | 100.0         | 1.5                     | 1.63                | 2.5                   | BSS-17 |                                            |
|          |                 |               |                         |                     | 15.0                  | BSS-18 |                                            |
|          |                 | 150.0         | 1.0                     | 1.09                | 10.0                  | BSS-19 |                                            |

#### 4.4.1 Sufficient compressive stress

This study begins by exploring the effects of applied target stress  $\sigma_t$  on creep-quake cycles and by quantifying the stress magnitude after which these cycles emerge. This is achieved with a set of experiments with different values of  $\sigma_t$ , ranging from 0.54 to 4.35 MPa (BSS-06 to BSS-10). Apart from their target force and stress, these experiments are identical to BSS-01. Their applied stress  $\sigma_a$  over time is shown in Figure 4.8, where quake events can be identified by the drops in  $\sigma_a$ . These drops, signature of quake events, are triggered by the sudden drop

in height of the sand column, and the time needed by the servo-controller to re-establish the target load on the sample.

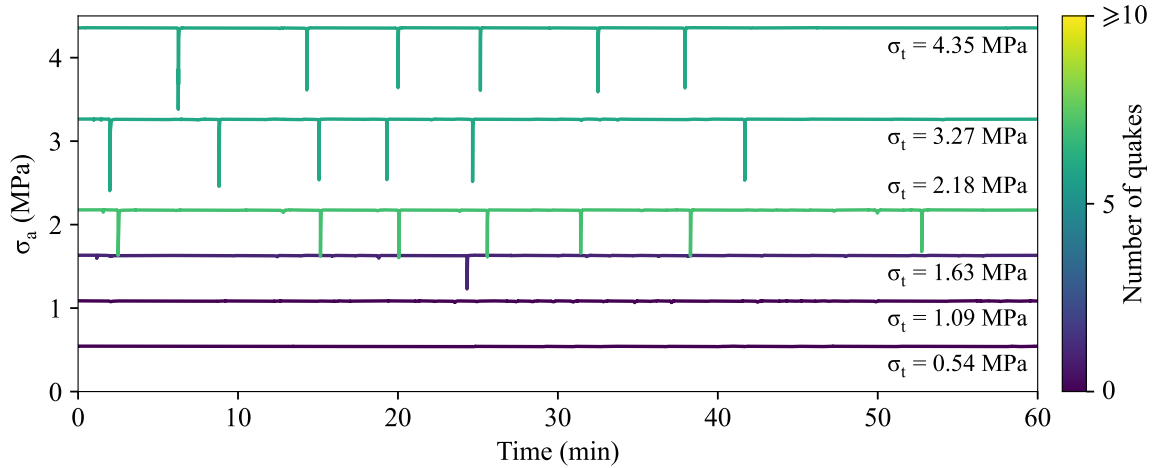


FIGURE 4.8. **Stress drops.** Drops in the applied stress  $\sigma_a$  for experiments with varying target stresses  $\sigma_t$ . Obtained from experiments BSS-01 and BSS-07 to BSS-11.

Figure 4.8 shows that the specific compressive stress needed to generate quakes in Barry beach sand lies between 1 and 2 MPa. In fact, the first recorded quake event occurs when the sand is subjected to 1.63 MPa (BSS-09).

Increasing the applied compressive stress is expected to lead to an increase of dissolution rates through two mechanisms. Namely, an increase in the surface area available for reactions caused by grain breakage, and an increase in pressure dissolution rates, as reviewed in Chapter 2. However, the magnitude of the increase in dissolution rates would remain relatively low, and its effects on creep-quake cycles would be limited, as seen in Figure 4.8. In fact, studies on the breakage of similar sized sand grains (Xiao et al. 2017) as well as on Barry beach sand grains larger than 1.18 mm (Miao and Airey 2013) reveal that the change in grain size distribution that the sand would attain at the higher stresses used in this chapter is lower than that caused by the chemical degradation of the sand grains seen in Figure 4.6 (f). Similarly, the effect of pressure dissolution was investigated by comparing the strain-time curves of these experiments, see Figure 4.9. This figure shows that while there is a change in strain rate from pressure dissolution, this change is minimal within the time-scales of these experiments.

This becomes more evident when comparing these strain rate changes with those resulting from an increase in acid injection rate. The limited effects of these two mechanisms are consistent with the trend seen in Figure 4.8. Here, it is apparent that although a minimum stress required to activate the mechanism, there is no clear relationship between the number or frequency of quakes and the applied stress once the applied stress is above that threshold.

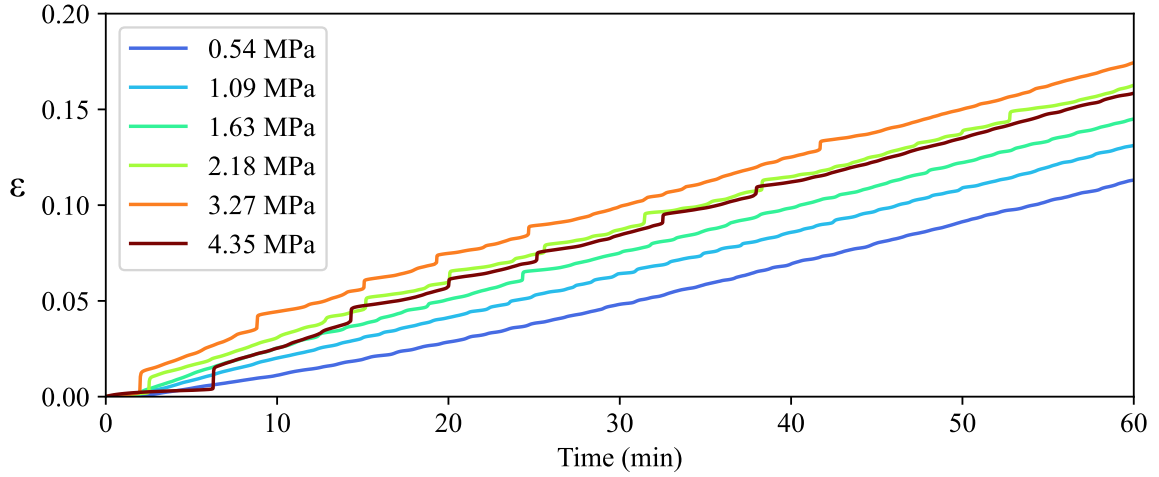


FIGURE 4.9. **Strain  $\varepsilon$  over time at different target stresses  $\sigma_t$ .** Obtained from experiments BSS-01 and BSS-07 to BSS-11.

Similarly, an investigation on the effect of  $\sigma_t$  on the drops in height and stress is shown in Figure 4.10. This figure indicates that  $\sigma_t$  has no significant effect on the average sand column height drops caused by quakes, see panel (a). Although the purple circle in this panel may suggest an increasing trend, this data point was obtained from a single quake, and it lies within the range of the error bars in the other three data points. The independence of  $H_{drop}$  from  $\sigma_t$  is an expected result, as the distance of a quake collapse is dictated by the available pore-space of the medium, and not by the stress applied to it. There is, however a noticeable decrease in the average normalised stress drops with  $\sigma_t$  shown in panel (b). The stress drops in this panel are defined as  $\hat{\sigma}_{drop} = \bar{\sigma}_{drop} / \sigma_t$ , where  $\bar{\sigma}_{drop}$  is the average stress drop. This reduction stems from the trend seen in Figure 4.10 (c). As seen in this inset, the magnitude of the average stress drops in our tests is initially proportional to the applied stress and its growth stagnates past a certain  $\sigma_t$ , which causes the value of  $\hat{\sigma}_{drop}$  to reduce. It is worth noting that the stress drops examined throughout this chapter depend greatly on the dynamics

of the loading frame used. With an ideal loading frame capable of ensuring a constant load despite sample deformations, there would be no stress drops. Hence, the magnitude of the stress drops mentioned here is specific to the loading frame and the parameters assigned to the servo-controller.

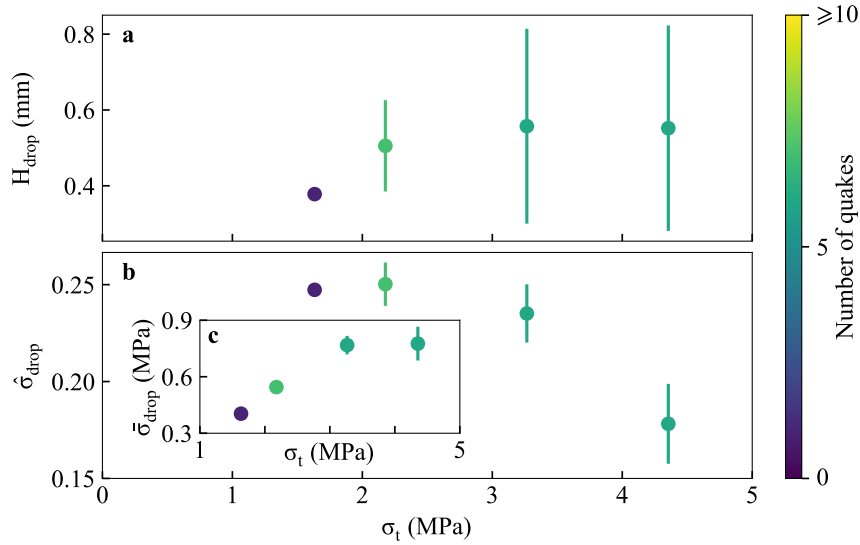


FIGURE 4.10. **Quake characteristics variations with applied stress.** Average height drops  $H_{drop}$  (a), normalised stress drops  $\hat{\sigma}_{drop}$  (b), and stress drops  $\bar{\sigma}_{drop}$  (c) for different target stresses  $\sigma_t$ . The error bars are the standard deviation of the data sets.

#### 4.4.2 Injection rate

Having discussed the parameters linked to the first two requirements for creep-quake cycles (collapsible length scale and sufficient stress), we now delve into the remaining requirement: the presence of a non-homogeneous degradation field. In calcareous sands, we apply this degradation field by injecting acetic acid from the bottom of the sand column. Hence, one of the simplest methods to alter this field and its gradient is to change the rate at which the acid is injected,  $Q_{inj}$ . This was previously done in Classica Marine aragonite, where we investigated the effects of increasing injection rates on the mechanical response of the sand. Figure 4.11 presents these results once more, as well as analogous results found on Barry beach sand.

This figure reiterates the difference in creep-quake response between the two sands, with Barry beach sand being a less favorable medium for the cycles to emerge. Additionally, it is apparent that the main effect of an increasing injection rate is the faster dissolution (and deformation) of the sand column. Again, this results directly from the availability of larger quantities of acid within the sandy medium. Increasing the concentration of acid yields similar results. In fact, an experiment performed outside the scope of this chapter with  $Q_{inj} = 5 \text{ mL/min}$  and  $C_a = 8\% \text{ wt.}$  results in a nearly identical response to that observed in BSS-01 ( $Q_{inj} = 10 \text{ mL/min}$  and  $C_a = 4\% \text{ wt.}$ ). Here, however we focus solely on manipulating the degradation gradient by changing  $Q_{inj}$  across experiments.

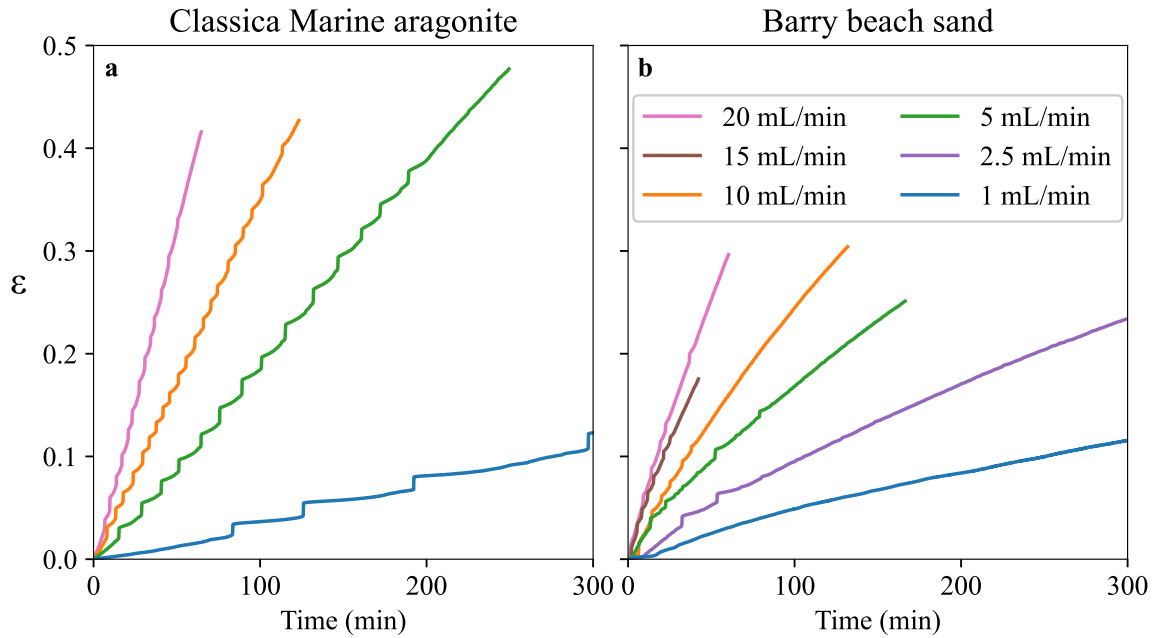


FIGURE 4.11. **Strain  $\varepsilon$  over time at different injection rates  $Q_{inj}$ .** **a:** Classica Marine aragonite injected with a  $C_a = 8\%$  solution, obtained from experiments CMA-01 to CMA-04. **b:** Barry beach sand injected with a  $C_a = 4\%$  solution, obtained from experiments BSS-01 to BSS-06.

In addition to the reduced strain rates, lower injection rates can reduce the frequency of creep-quake cycles. This reduction results in no quakes developing in the Barry beach sand experiment with the lowest injection rate (BSS-02). It is possible that this experiment could have developed quakes if it had continued indefinitely, however, we did not observe any quakes throughout the experimental durations considered in this study at this injection rate.

Similar to our observations on a minimum required compressive stress, these findings point towards a minimum injection rate required for creep-quake cycles to emerge. Although this effect was not seen in Classica Marine aragonite, it is likely that the limit for these samples is below the lowest injection rate tested ( $< 1 \text{ mL/min}$ ). It is expected that CMA samples would experience no quakes had they been tested at lower injection rates. The effect of low injection rates may be explained by the relationship between particle rearrangement and reaction rates. A recent DEM study on calcite granules dissolving in acetic acid (Alam et al. 2022) demonstrates how low reaction rates lead to higher earth pressure coefficients (larger lateral redistribution of stress), which in our experiments would result in larger frictional effects between the test tube walls and the sand samples. These frictional effects will be discussed in more detail in Section 4.4.3.

Conversely, at high injection rates the deformation caused by quakes and the accelerated creep begin to merge together, producing hardly distinguishable quake events. This points towards an upper limit on the injection rate for creep-quake cycles to be discerned. These upper and lower bounds on the injection rates that generate creep-quake cycles are a direct result of the interaction between the degradation field and the degradable medium. Close to the upper bound, the degradation field becomes much more uniform throughout the length of the sample, and can no longer trigger the localised collapse. On the other hand, slow injection rates cause the majority of acid consumption to occur within a very limited space at the bottom of the sand column, resulting in a step change between high degradation and no degradation in the sample. Hence, there is a range in which the injection rate promotes creep-quake cycles by inducing a non-homogeneous degradation field with a gradual transition between high and low degradation rates. This is consistent with the fields presented in Chapter 3.

We found no discernible correlation between the stress or height drops and the rate of injection in either Classica Marine aragonite or Barry beach sand, as seen in Figure 4.12. However, it is clear that the stress drops in Barry beach sand are lower than those in Classica Marine aragonite. This is consistent with their slightly lower drops in height, as shorter piston displacements result in lower stress drops. In fact, this difference can be further seen in the change in volumetric strain caused by every quake. Figure 4.13 shows the strain rate fields

for a number of quakes in each sand sample. From this figure, it is clear that the compaction ‘band’ or ‘area’ forms at the bottom of the sample and that the width of the band is larger in the CMA samples than in the BBS ones.

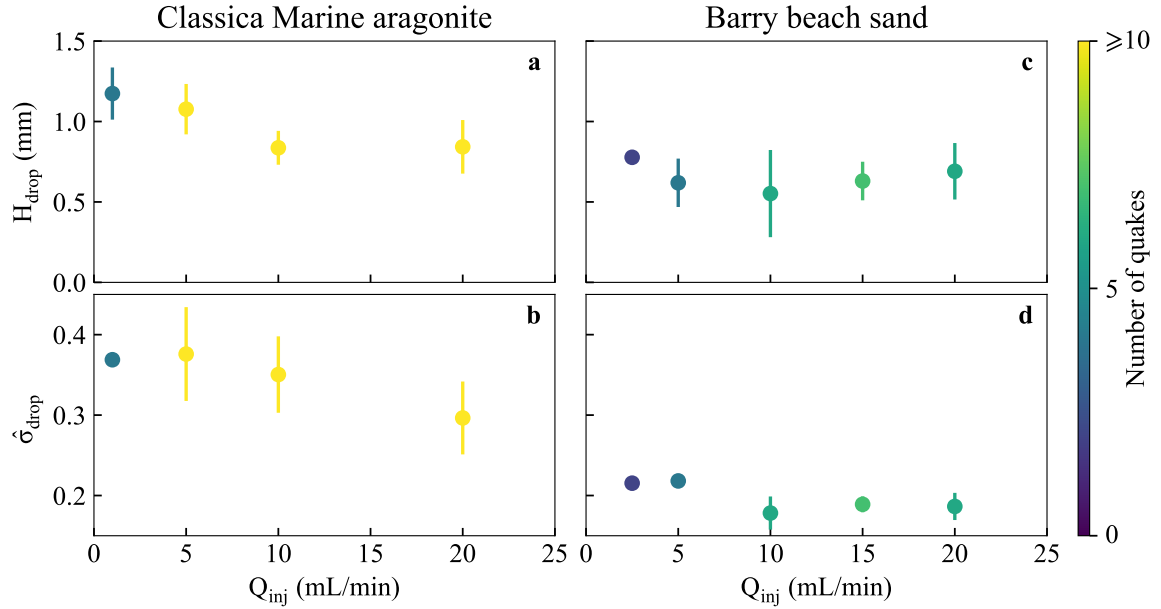


FIGURE 4.12. **Quake characteristics variations with injection rate.** Average height drops  $H_{drop}$  and normalised stress drops  $\hat{\sigma}_{drop}$  for different injection rates  $Q_{inj}$  in **a,b**: Classica Marine aragonite, and **c,d**: Barry beach sand. The error bars are the standard deviation of the data sets.

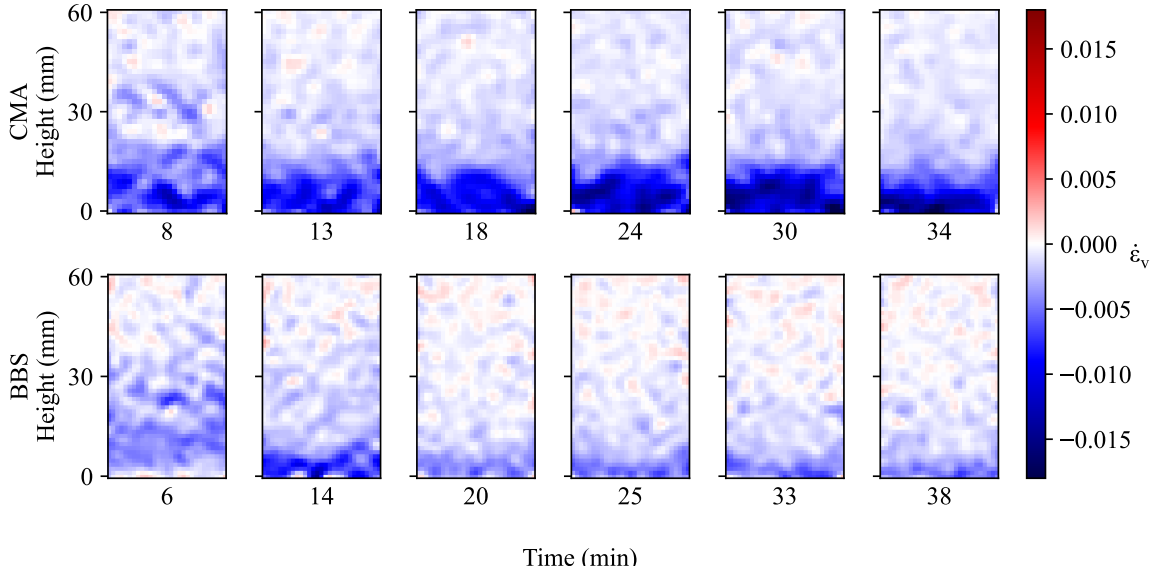


FIGURE 4.13. **Volumetric strain rate  $\dot{\epsilon}_v$  during quakes.**  $\dot{\epsilon}_v$  fields for the first six quakes of experiments CMA-03 (top row) and BSS-01 (bottom row).

Having discussed the independent influence of stress and injection rates on the creep-quake response, we can now discuss the emergence of creep-quake cycles as a function of the two. In order to do so, the total number of quakes for each experiment is plotted on a  $\sigma_t - Q_{inj}$  plane, as seen in Figure 4.14. This figure shows the interplay between  $\sigma_t$  and  $Q_{inj}$  in the formation of creep-quake cycles and the existence of a ‘Goldilocks zone’ within which they emerge. Note that even though higher values of  $Q_{inj}$  are not included, we expect these to result in experiments with no quakes. This figure further indicates that at a given point in the  $\sigma_t - Q_{inj}$  space where no quakes develop, it is possible to generate quakes by moving towards this ‘Goldilocks zone’. Take the BSS-17 experiment ( $\sigma_t = 1.63$  MPa,  $Q_{inj} = 2.5$  mL/min), for instance. While its combination of applied stress and injection rate does not generate any quake, changing either of these conditions independently towards more favourable values would induce creep-quake cycles.



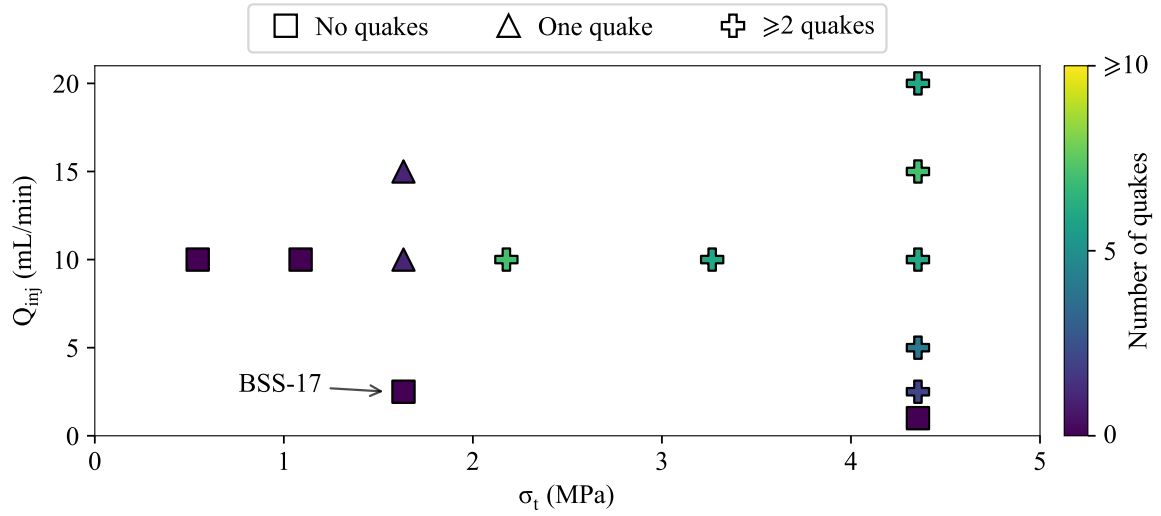


FIGURE 4.14. **Number of quakes in experiments with varying target stress  $\sigma_t$  and injection rates  $Q_{inj}$ .** Obtained from BSS-01 to BSS-11, BSS-17, and BSS-18.

### 4.4.3 Initial height

So far, this chapter has focused on the effects of two parameters that relate directly to the required conditions for creep-quake cycles. To conclude, we present an investigation on the effects of different sand column aspect ratios by changing the initial sand column height  $H_0$ . In a similar manner to how changing  $Q_{inj}$  influences the degradation field within the sand column, changing  $H_0$  can shorten or lengthen the vertical reach of the degradation field along its height. While the rate of acid depletion remains unchanged, changing the height of the sand column would place a larger or smaller portion of the sand sample within the high degradation zones, therefore changing the relative field of degradation that the sand column is subjected to. In retrospect, this effect is present throughout the experiments described thus far. As the sand column dissolves and its height shortens, the top of the column progressively moves downward within the degradation field. Hence, the following investigation into the effects of different initial heights on the creep-quake response can rationalise the time evolution of the creep-quake behaviour, especially during the later stages of an experiment, when BSS samples stop developing quakes.

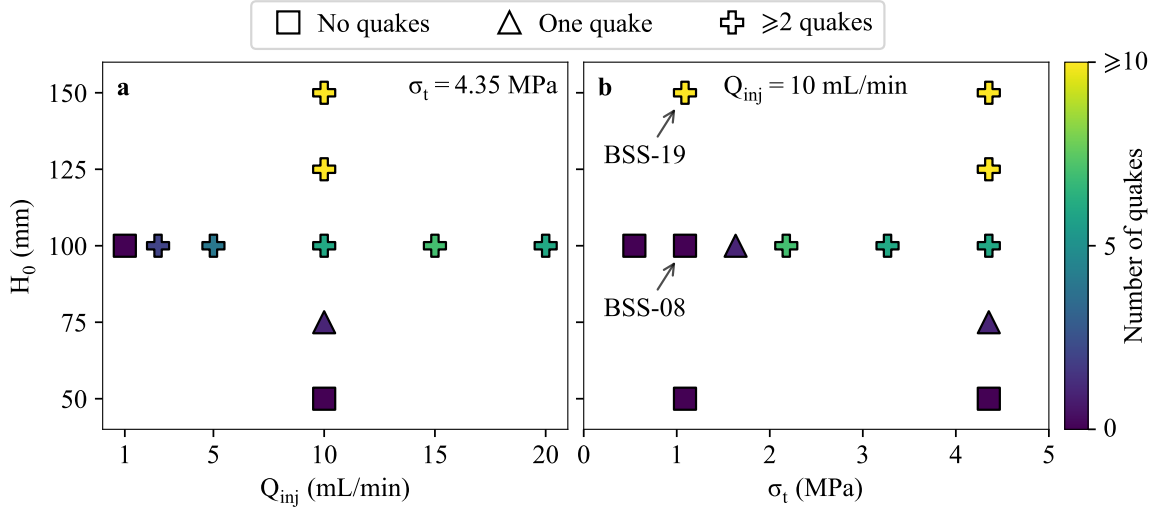


FIGURE 4.15. **Number of quakes in experiments with varying initial height  $H_0$ .** **a:**  $H_0$  against injection rate  $Q_{inj}$  with  $\sigma_t = 4.35$  MPa. **b:**  $H_0$  against target stress  $\sigma_t$  with  $Q_{inj} = 10$  mL/min. Obtained from BSS-01 to BSS-19.

Figure 4.15 indicates there is a link between initial sand column height and the emergence of creep-quake cycles. At high values of  $H_0$ , higher number of creep-quake cycles are observed. Furthermore, increasing the height can result in creep-quake cycles developing in previously unfavourable combinations of stress and injection rate. This is observed in panel (b), when comparing BSS-08 ( $\sigma_t = 1.09$  MPa and  $Q_{inj} = 10$  mL/min) at  $H_0 = 100$  mm to an equivalent experiment at  $H_0 = 150$  mm (BSS-19). Contrastingly, experiments with lower initial heights have the opposite response; where previously favourable values of stress and injection rates do not produce quakes at lower sand column heights. Note that this is consistent with the late stages of the Barry beach sand experiments. For instance, the BBS strain curves in Figure 4.11 show an absence of quakes beyond strain values of  $\varepsilon \gtrsim 0.25$ , which is equivalent to  $H \lesssim 75$  mm for  $H_0 = 100$  mm. Correspondingly, Figure 4.15 indicates that multiple creep-quake cycles do not develop on experiments whose initial heights lie in a similar range. Although both Classica Marine aragonite and Barry beach sand exhibit this behaviour, it is particularly noticeable in the Barry beach sand samples. Furthermore, when comparing all the granular materials described in Chapter 3 (puffed rice, calcareous sand, rock salt, and crushed ice), this response is observed solely in calcareous sands.

We do not have a conclusive explanation of the reason behind the influence of initial sand column height on creep-quake cycles. However, we can identify two potential factors that may lead to the results seen in Figure 4.15. The first one is the interplay between the height of the sand column and the degradation field applied to the medium. As the height of the column reduces, a constant injection rate of acid would correspond to a more uniform dissolution field within the sample, which may be responsible for the lack of creep-quake cycles in experiments with a low  $H_0$ . Yet, this cannot explain the relationship between increasing values of  $H_0$  and the more frequent triggering of quakes. In order to do so, we shift our focus to the interaction between the sand grains and the tube walls. The ‘Janssen effect’ (Lei et al. 2019), which describes arching in granular materials due to grain to wall friction (see section 2.2.6), may play a significant role in the development of creep-quake cycles in the Barry beach sand. Similar studies on grain-filled silos have identified quake-like phenomena occurring during silo discharge (Roberts and Wensrich 2002). While not identical, these can relate to our experiments, as they involve a cylinder filled with granular material, which experiences a loss of mass originating at the bottom of the cylinder and an overall downward movement of the granular medium. In these studies, the formation of localised discontinuities in the downward velocity of the grains was attributed ‘stick-slip’ motion, which are triggered by the friction between the granular material and the silo walls (Roberts and Wensrich 2002). In a similar manner, the boundary conditions applied to our calcareous sand samples may have a significant influence on their behaviour, by modifying the distribution of the stress along the sample height.

In addition to its effect on the number of quakes developed, initial height may have an effect on the displacement and stress drop associated with each quake. Figure 4.16 shows a slight increase in  $H_{drop}$  with initial height, as well as an increasing trend in  $\hat{\sigma}_{drop}$ . Taller sand columns may experience a higher magnitude of stress redistribution through wall friction, which could in turn support the sample through more dissolution and result in larger height drops. Similarly, the added mass results in an increase in the inertia of the sand column, which would in turn result in the larger stress drops observed in panel (b).

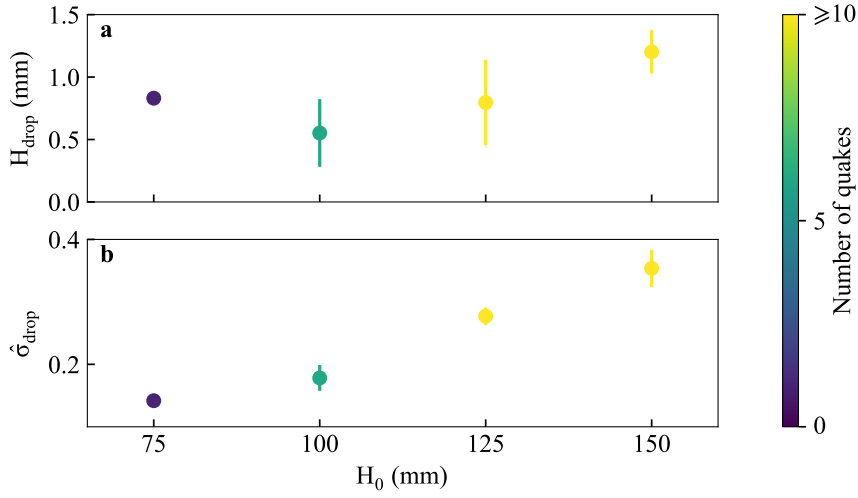


FIGURE 4.16. **Quake characteristics variations with initial height.** Average height drops  $H_{drop}$  (a) and normalised stress drops  $\hat{\sigma}_{drop}$  (b) for different initial heights  $H_0$  with  $\sigma_t = 4.35$  MPa and  $Q_{inj} = 10$  mL/min. The error bars are the standard deviation of the data sets.

Possible approaches to mitigate this effect in future research include increasing the sand column diameter or reducing the friction between the walls and the material by changing either the material or the surface roughness of the test tube. In the time frame in which the work for this thesis was performed, we did not delve into such an investigation. However, we did perform a number of experiments on Classica Marine aragonite in an aluminium test tube for the purpose of taking the neutron radiographies shown in Section 4.2.3. This change caused the sand samples to develop no quakes throughout the length of those experiments, which further points to boundary effects playing a significant role in the emergence of creep-quake cycles in calcareous sands. From post-experiment observations on the test tubes, it is clear that the inner wall of the two different tube materials interact very differently with the sand grains, see Figure 4.17. The bottom of the acrylic tube shows scratching in its inner wall (Figure 4.17 (b)), while the aluminium one shows more indentations than scratches (Figure 4.17 (d-f)).

The numerous indentations shown in Figure 4.17 (d-f) are due to the aluminium being softer than the acrylic. This property of the aluminium tube allows the sand grains to indent its inner wall and form a layer of grains that remain semi-static throughout the duration of the

experiment. Consequently, this static grain layer would increase the roughness of the tube and increase the redistribution of stress onto the tube walls. It is likely for this stress redistribution to cause the stress at the bottom of the sand column (where quakes originate) to drop below the ‘sufficient stress’ threshold, which would explain the lack of quakes in these experiments. In a similar manner, the lack of quakes in the low  $Q_{inj}$  experiments in Figure 4.11 (b) could be the result of higher friction at the boundaries, given the higher lateral pressure redistribution caused by the low dissolution rates (Alam et al. 2022).

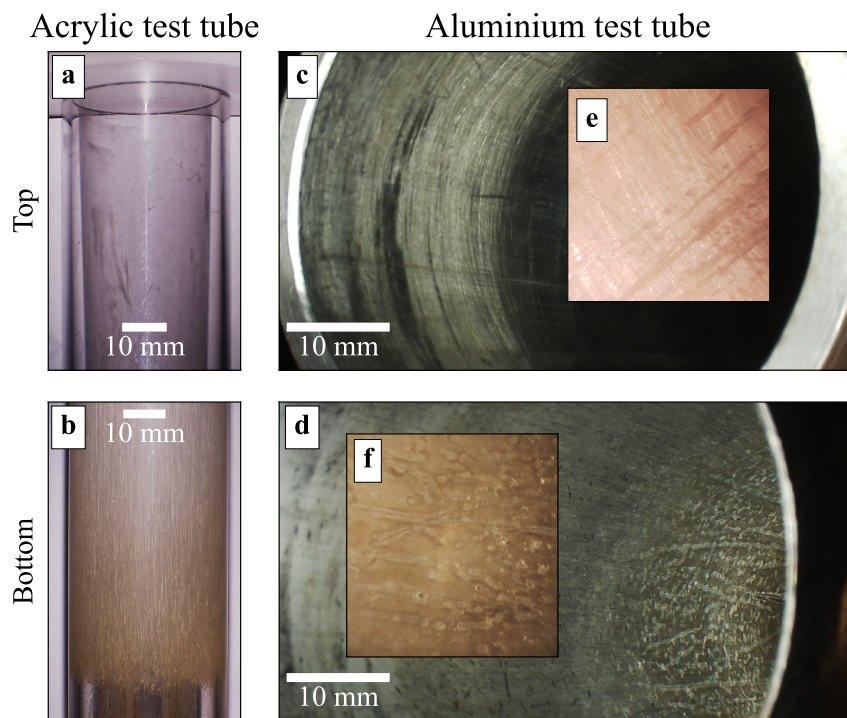


FIGURE 4.17. **Post-experiment photographs of test tubes.** **a,b:** Top and bottom of acrylic test tube, as seen from outside. **c,d:** Top and bottom of aluminium test tube, as seen from a diagonal view into the tube. **e,f:** Close-up photographs of the inner wall surface of the top and bottom of the aluminium tube test, as seen through a borescope inside the tube.

## 4.5 Conclusion

This chapter presented an in-depth investigation into the emergence of creep-quake cycles in calcareous sands, which reiterated that their creep-quake cycle response is tightly linked to a sufficient compressive stress, a non-uniform degradation field, and the collapsible length-scale associated with the material. Through this investigation, it was found that this length scale evolves constantly with grain dissolution, and that its evolution can eventually lead to the end of creep-quake cycles. Similarly, it was observed that these requirements depend on each other, as well as on the confining conditions of the samples. While this study further developed our understanding on the formation of creep-quake cycles in calcareous sands, it also opened a window for further questions on the phenomenon. The complexity of coupled effects of stress and acidic degradation, as well as experimental boundary condition limitations, provide a vast field of research to be addressed in order to fully understand the creep-quake cycle generation process. Future research can focus on observing the evolution of grains through non-intrusive tomographic techniques that can capture a complete sample configuration, as well as on the role of confining boundary conditions in the formation of these cycles. The latter can be approached experimentally, as well as through numerical simulations, where one is not confined by experimental constraints and can explore the role of side-wall friction in isolation.

The findings of this chapter were obtained primarily from one-dimensional experimental systems within a laboratory test tube. In order to explore the significance of this collapse mechanism in more general conditions, the natural step forward is to move onto model geotechnical scenarios, such as flow through a degradable soil structure. These conditions can be found in tailing dams (Lottermoser and Ashley 2005), as well as underground water systems (Gillham 1984). Hence, a study on the effects of degradation processes occurring as a result of transversal flow through a soil medium can be of great relevance to numerous industries, including those focused on mining, groundwater systems, and CO<sub>2</sub> sequestration. This is the topic of Chapter 5, in which we make use of a two-dimensional experimental setup filled with Barry beach sand and observe the effects of a transversal flow of acetic acid on its behaviour.

## **Transversal acid flow through a calcareous sand deposit**

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### **5.1 Introduction**

Throughout the preceding chapters, it has been demonstrated that imposing a degradation field in a porous medium can result in structural instabilities and the subsequent collapse of its structure. In geomaterials, such degradation-induced collapse events can have severe consequences, including environmental pollution and damage to infrastructure. Consequently, the flow of acid through geomaterials has become a topic of great interest in a variety of fields, such as environmental engineering, CO<sub>2</sub> sequestration, and mine waste storage.

So far, we have focused solely on a simplified one-dimensional experimental approach to identify creep-quake cycles in porous materials. Although there certainly are scenarios for which this simplification is appropriate, such as the humidity-driven consolidation happening within silos in between discharges or the geological formations of transversely anisotropic soils and rocks, most geotechnical scenarios involve systems with boundary conditions and degradation processes for which this simplification is no longer representative. This begs to question the relevance of creep-quake cycles as a collapse mechanism in geometries more closely related to the geological systems observed outside a laboratory.

Therefore, we now change our approach to a two-dimensional test environment, in which material degradation is caused by the transversal flow of an acidic solution. By utilising a two-dimensional flow environment, this chapter aims to establish a closer connection to real geotechnical scenarios, such as acid infiltration in a degradable porous rock or the flow of acid through a tailing dam. The effects of such flow conditions are explored through a series of

experiments conducted on Barry beach sand samples, building upon the knowledge acquired in Chapters 3 and 4. Throughout this study, a particular interest is taken in the formation of degradation-induced instabilities and arising collapse patterns, including creep-quake cycles.

The experimental setup employed in this chapter makes use of a simplified sample geometry in the form of a rectangular prism. The flow of acid is controlled by an imposed hydraulic head difference from one side of the sample to the other. This approach has been previously employed in the study of transversal fluid transport through porous media (Klenk and Grathwohl 2002; Silliman et al. 2002), as well as in investigations on slope stability and collapse in two-dimensional water flow settings (Germer and Braun 2011). In addition to visual observations made through the transparent sample tank walls, X-ray radiography techniques are implemented to monitor sinkhole formation during the experiments.

This chapter begins by describing the experimental setup and methodology. It then characterises the evolution of degradation-induced sinkholes in one particular experiment using visual and X-ray radiography techniques. The latter proved to be invaluable in accurately assessing the extent of acidic degradation within the samples. Based on these observations, collapse patterns in the sand are identified and the connection between sinkhole collapse, and creep-quake cycles is explored. Finally, this chapter offers insights into future research on creep-quake cycles, as well as on the influence of cyclical acidic flow on sinkhole collapse.

## **5.2 Methodology**

### **5.2.1 Experimental setup**

The experiments were performed in a rectangular acrylic container. The sand samples sat within the container, which was connected to acid reservoirs and was placed within a frame with spring loaded pistons, as shown in Figure 5.1. The duration of these experiments ranged from a week to a month depending on the initial density and grain size distribution of the sand samples. Hence, photographs were used to continuously monitor the samples and generate



time-lapses of the experiments. The photographs were taken in 30 second intervals with a Nikon D3300 camera.

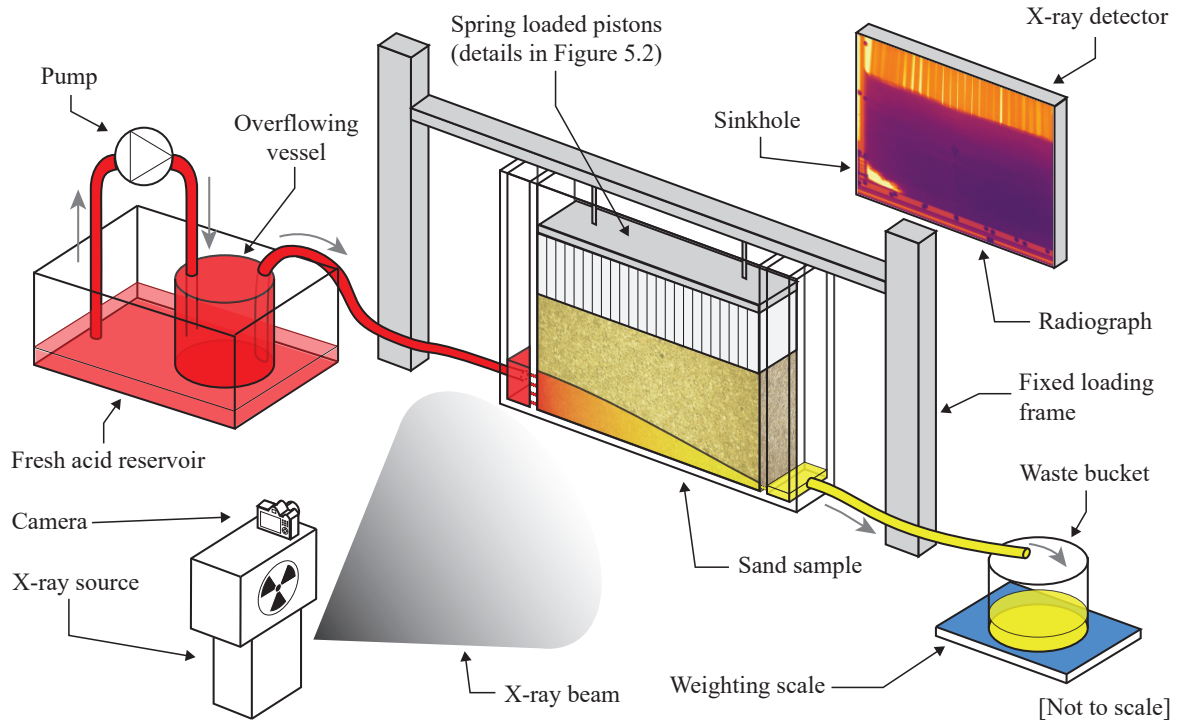


FIGURE 5.1. **Experimental setup.**

In addition to the photographs, X-ray radiographs were recorded to observe mass depletion inside the samples caused by chemical degradation, which in turn formed a sinkhole in the material. The detector used was a Dexela 2923 NDT, with a 290.8 x 229.8 mm pixel area, a 74.8  $\mu\text{m}$  pixel size, and a 14-bit pixel intensity output. The X-ray beam was set to 180 kV and 2 mA, which gave sufficient brightness and contrast for clear observations. The experimental sample sat at a distance of 1280 mm from the X-ray source and 90 mm from the X-ray detector (measured with respect to the centre of the sand prism). Due to experimental constraints, the interval between the X-ray measurements varied throughout the duration of the experiments. For the most part, the radiographs were taken at a frame rate of 0.02 frames per second for 8 hours during working days (Monday to Friday). To avoid excessive data generation, this frequency was reduced to approximately one frame every 12 hours when the dissolution processes in the sand appeared to be slow and inconsequential in short time scales.

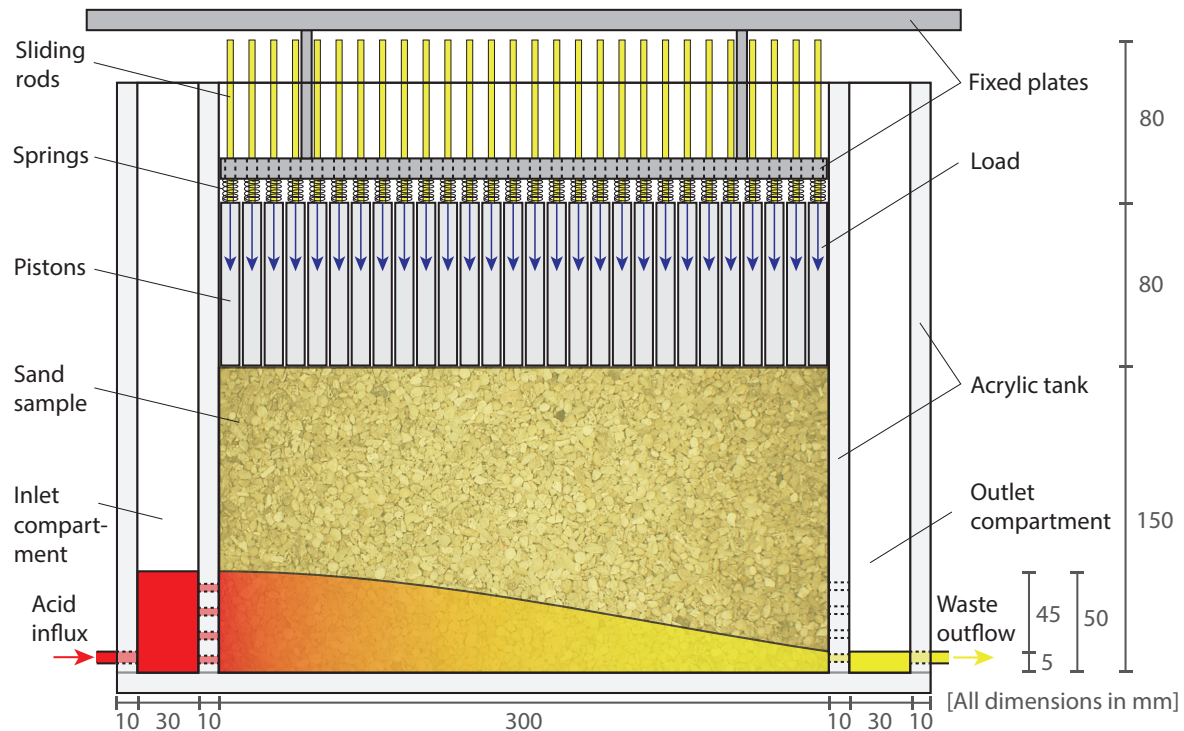


FIGURE 5.2. **Experimental apparatus.** Diagram of acrylic sample tank, acid flow, and spring-loaded piston system.

The compartmentalised acrylic tank consists of a sample compartment and two liquid compartments on each side of the sample, separated from the sand by porous walls. The tank walls were attached with stainless steel bolts and an impermeable rubber seal between the walls to avoid water leakage. The inside width of the tank was 75 mm and its inside walls were porous only up to a height of 50 mm. The two liquid compartments were used to induce the hydraulic head difference that drove the flow of acid through the sample. The inlet (upstream) compartment was connected to an overflowing vessel, which ensured a constant water head inside the compartment. The overflowing vessel sat inside a larger fresh acid reservoir, from which the liquid was constantly pumped into the vessel at a sufficiently rapid rate to constantly replenish the acid flowing into the sample. On the other side, the outlet (downstream) compartment was connected to a waste bucket, as shown in Figure 5.1. This outlet compartment had a fixed-height outlet pipe, which controlled its hydraulic head. The waste bucket sat on a weighting scale, which was used to monitor the acid mass outflow throughout the experiment. Figure 5.2 illustrates further quantitative details on the water

levels kept in the inlet (50 mm) and outlet (5 mm) chambers in order to ensure a constant hydraulic head difference (45 mm).

The spring loading system used consisted of 28 acrylic pistons with metallic guide rods. Each piston had a cross-sectional area (width  $w_p \times$  breadth  $b_p$ ) of 10 mm  $\times$  70 mm and a mass  $m_p$  of 125 g. They were each loaded by two RS 751-512 springs with a free length  $l_{sf}$  of 80.5 mm, a spring rate  $k_s$  of 0.17 N/mm, a minimum working length of 20.6 mm, and therefore a maximum spring force of 10.18 N per spring. The springs were compressed and guided downwards by the fixed plates shown in Figure 5.2. In contrast with how load was applied in previous chapters, this loading mechanism does not ensure a constant load with sample surface deformation. When these pistons move downwards, their springs elongate, reducing the load applied by the pistons onto the sand. This change was implemented to allow for differential settlement in the sand. The use of springs allowed us to minimise experimental difficulties and facilitate the imaging of the samples, as the alternative to this system would have been to install individual servo-controllers or individual dead weights on each piston.

### 5.2.2 Protocol

Each experimental sand sample was prepared by pouring the sand grains into the sample compartment following either a wet pluviation or a dry packing method (adapted from (Oliviera et al. 1996)) in order to achieve a loose or dense sand configuration, respectively. The wet pluviation method involved filling the setup with water, then pouring sand at a constant rate with a funnel from the top of the setup until reaching the desired sample height. In contrast, the dry packing method involved pouring small layers of sand ( $\sim 1$  cm thick), then tapping the layer by hand with a metallic plate (150 mm  $\times$  49 mm area) until flat, and repeating this process until the sample reached the desired height.

Once the sample was prepared, the setup was put in place for imaging, as shown in Figure 5.1. It was then loaded by compressing the 28 spring loaded pistons. Initially, every piston exerted a downward force of 21.59 N, consisting of both its self weight (1.23 N) and the force of two fully compressed springs (20.36 N), applying an initial stress of 30.84 kPa. After compressing

the springs, the experiment was started by imposing the hydraulic gradient on the sample. Initially, pure water was flushed for one hour in order to establish a steady-state flow profile and saturation field. Then, the water was switched to acetic acid at a concentration of 2% by weight. As the acid flowed through the sample, the dissolution process would change the hydraulic properties of the sand sample, altering the flow of acid through it. This was tracked via the weight measurements of the waste bucket.

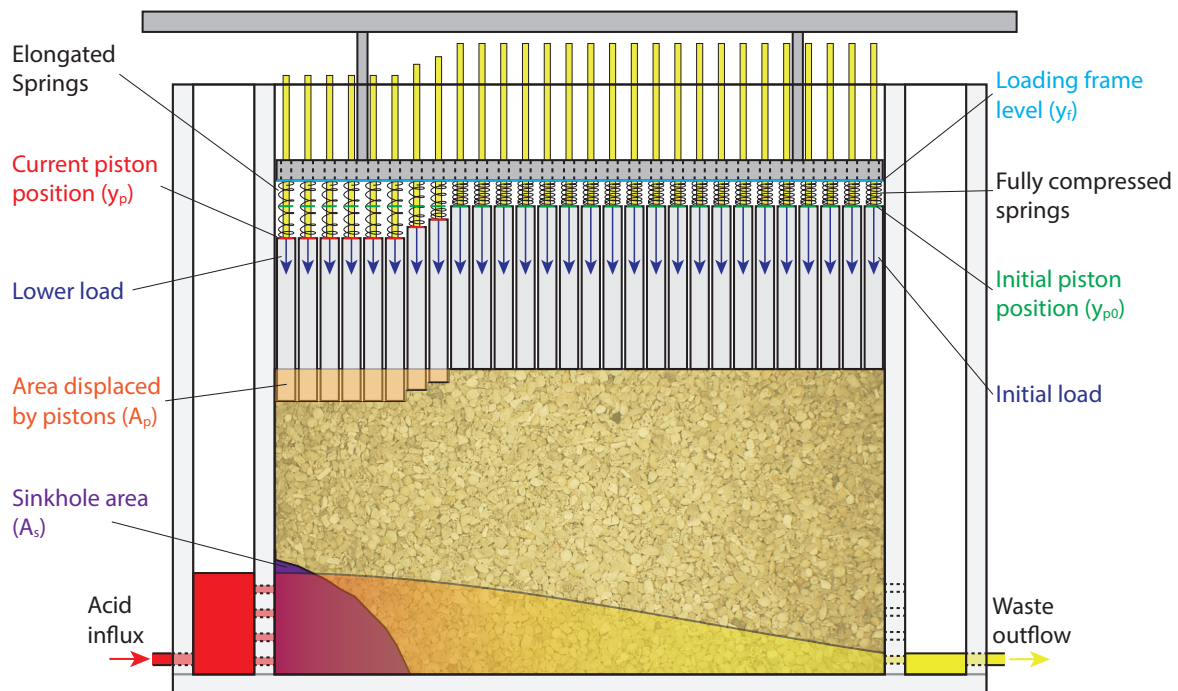


FIGURE 5.3. **Diagram of deformed sample.** This diagram shows the experimental setup after the opening of a sinkhole and surface deformation events. It depicts how the spring-loaded system works, as well as the observable areas of sample surface displacement caused by the pistons  $A_p$  and sinkhole size  $A_s$ .

Over the course of the experiment, sand would dissolve, which would cause the top surface of the sample to deform, and consequently change the stress applied on it. The sand dissolution was tracked via the X-ray radiographs, which were used to observe the density of the samples and its evolution over time. On the other hand, the photographs enabled us to monitor the experiment, as well as track the position of the pistons compressing the sand. Hence, the time-lapse photographs were used to determine the stress applied on the sample over time. A

diagram of a deformed sample is shown in Figure 5.3, which depicts the main measurements obtained with the photographs and radiographs of a given experiment.

The stress applied by each piston was defined as:

$$\sigma_p = \frac{(2k_s \langle l_{sf} - |y_f - y_p| \rangle + m_p g)}{w_p b_p}, \quad (5.1)$$

where:  $k_s$  and  $l_{sf}$  are a spring's rate and free length, respectively;  $y_f$  and  $y_p$  are the vertical positions of the loading frame face that compresses the springs and the top a given piston  $p$ , respectively (see Figure 5.3);  $m_p$ ,  $w_p$ , and  $b_p$  are the mass, width, and breath of piston  $p$ ;  $g$  is gravity; and the angle brackets are Macaulay brackets, defined for a variable  $x$  as:

$$\langle x \rangle = \begin{cases} x, & \text{if } x \geq 0 \\ 0, & \text{otherwise.} \end{cases} \quad (5.2)$$

At the start of the experiments, the springs on the pistons are fully compressed between the loading frame  $y_f$  and the initial position of the top piston surface  $y_{p0}$ . Hence, each piston applies an initial stress  $\sigma_{p0}$ , defined by Equation 5.1 for  $y_p = y_{p0}$ .

### 5.2.3 Materials

The material used in this chapter was Barry beach sand, as introduced in Chapter 4. However, the samples presented here were prepared to have a different grain size distribution to those presented earlier (BBS). This change was implemented to arrive at a more realistic sand sample, with a grain size distribution much closer to that found in naturally occurring calcareous sands. Here, we used two different grain size distributions: (1) the full distribution of the sand, as found in Barry beach (BBS-F) and (2) a distribution broader than the one used in Chapter 4, where the sand was sieved to keep grains that remain in a 0.3 mm sieve and that pass through a 2.36 mm one (BBS-B). The grain size distribution curves (GSD) for the three different distributions can be seen in Figure 5.4 (a). The data in this panel was obtained from a sieve analysis of the sand grains, unlike the GSD curves shown in Chapter 4, which were obtained from photographs. Additionally, the minimum and maximum densities ( $\rho_{\min}$

and  $\rho_{\max}$ ) for each distribution can be found in Table 5.1. These measurements were obtained following the standardised procedures described in ASMT-D2454-16 and ASMT-D4253-16 for  $\rho_{\min}$  and  $\rho_{\max}$ , respectively.

TABLE 5.1. Density range of each grain size distribution of Barry beach sand

|       | Sieve size (mm)   | $\rho_{\min}$ (kg/m <sup>3</sup> ) | $\rho_{\max}$ (kg/m <sup>3</sup> ) |
|-------|-------------------|------------------------------------|------------------------------------|
| BBS-F | Full distribution | 1328                               | 1466                               |
| BBS-B | > 0.3, < 2.36     | 1238                               | 1390                               |
| BBS   | > 0.6, < 1.18     | 1182                               | 1285                               |

This chapter will discuss three distinct experiments. One experiment was performed on a loose BBS-B sand sample (Experiment BL), while the other two used dense packings of BBS-B and BBS-F sand (Experiments BD and FD, respectively). Table 5.2 summarises these three experiments, along with their ID, bulk density  $\rho$ , and relative density  $DR = (\frac{1}{\rho_{\min}} - \frac{1}{\rho}) / (\frac{1}{\rho_{\min}} - \frac{1}{\rho_{\max}})$ .

TABLE 5.2. Experimental parameters

| ID | Material | Packing | Bulk density $\rho$ (kg/m <sup>3</sup> ) | Relative density $DR$ |
|----|----------|---------|------------------------------------------|-----------------------|
| BL | BBS-B    | Loose   | 1251                                     | 0.095                 |
| BD | BBS-B    | Dense   | 1377                                     | 0.923                 |
| FD | BBS-F    | Dense   | 1460                                     | 0.960                 |

The concentration of acid used (2% by weight) was half of that from the previous chapter (4%) to decrease the rate of sand dissolution and gas generation. Lower rates of reaction were preferred for these experiments in order to obtain quasi-static states that facilitate the acquisition of X-ray radiographs. The percentage of mass dissolved during the reaction between each grain size distribution of Barry beach sand and 2% acetic acid is included in Figure 5.4 (c). Similar to previous chapters, the acid used in these experiments was dyed with methyl orange to ease the visualisation of acid consumption. The dye tinted the solution red for pH values under 3.1 and transitioned to a yellow tint for higher pH values.

The following sections on sinkhole collapse will focus primarily on experiment BL. While all experiments in Table 5.2 will be used for comparison and discussions (Section 5.6), experiment BL presents a complete set of results from which the discussions in this chapter stem.

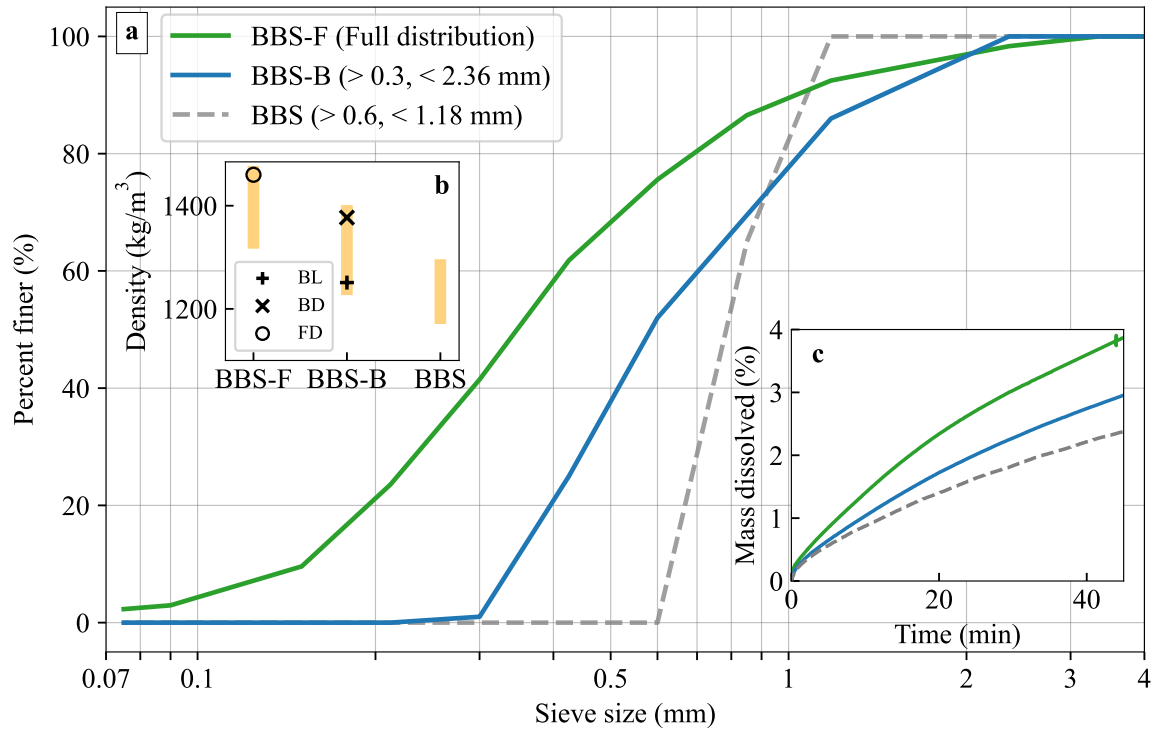


FIGURE 5.4. **Material parameters.** **a:** Grain size distribution curves for Barry beach sand sieved through: no sieve (BBS-F), 0.3 mm - 2.36 mm sieves (BBS-B), and 0.6 mm - 1.18 mm sieves (BBS). **b:** Density ranges for each Barry beach sand grain size distribution with markers for the densities recorded on experiments BL, BD, and FD. **c:** Percentage of sand mass dissolved in time for each size distribution. These curves were obtained following the process described in Chapter 4, from experiments on 10 g of sand placed in 100 g of 2% acetic acid solution.

## 5.3 Observations

At the start of the experiment, when pure water was flushed through the samples, a front of saturation began to form. This front was observed to start at the inlet and evolve as the water traveled through the sample. During this time, we observed the wetting of the whole sample, indicating that all of it experienced some degree of saturation. We envision the flow profile through the material to resemble that shown in Figure 2.4 (Chapter 2), where there is a capillary fringe above the phreatic surface, in which the material is partially saturated. In our experiments, this capillary fringe is likely capable of reaching lengths larger than our sample heights, causing all of the sample to experience partial saturation above the phreatic surface of

the flow. The exact height of the capillary fringe was not determined. Approximations of its reach using well-established methods (Lu and Likos 2004) suggest that for the characteristics of our sand sample the capillary fringe may reach heights of up to 200 mm above the water table. This would explain why the wetting front can be seen reaching the surface of the sample. Nevertheless, this approximation of the capillary fringe is only indicative of the behaviour of the water in an undisturbed sample. Once the sand begins to dissolve in the acid, its porosity and grain size distribution change, which in turn affect the capillary fringe inside the sample.

Experiments BD and FD both developed areas of high chemical degradation and sinkholes, however, neither of these experiment showed any deformations on the surface of the sample during acid injection. In contrast, several instances of surface deformation were observed during acid injection in experiment BL. This experiment had an approximate duration of 200 hours. Due to its long duration and time restrictions on when the X-ray radiographs could be obtained, the experiment was paused for approximately two and a half days past its first 50 hours. The pausing of the experiment entailed stopping the flow of fresh acid into the inlet compartment, and leaving the experimental sample open to drain into the waste bucket. With this pause, acid flowed through the sand sample only over 130 hours, out of the  $\sim 200$ . It is noted that this pause changed the state of the sample, as it was allowed to air dry during this period, until the hydraulic head difference was reinstated when the experiment was resumed. The deformation of the sample surface was monitored throughout the pause and was found to be minor. However, the re-introduction of acid into the sample caused an immediate collapse event, which will be elaborated on later in this chapter.

### 5.3.1 Acid flow

The flow rate through the sand sample was constantly monitored via the waste bucket mass readings, as detailed in Section 5.2.1. The mass of the waste bucket for experiment BL, as well as the flow rate over the duration of the experiment are shown in Figure 5.5. Additionally, this figure presents a direct visual illustration of the pause during the experiment. As shown in panel (a), the flow of acid into the sample was stopped from hour 52 to hour 120 since acid injection, resulting in a constant cumulative outflow mass and a zero outflow rate. In



panel (b), this pause was removed for ease of visualisation and the ‘stitched up’ time axis in which the data is plotted will be referred to as ‘acidification time’  $t_a$ . This time accounts only for the time in which acid in the inlet compartment was renewed. In all subsequent figures of experiment BD, the results will be presented in terms of  $t_a$ , and the time portion removed from the data where the flow was stopped will be represented by a dashed gray line.

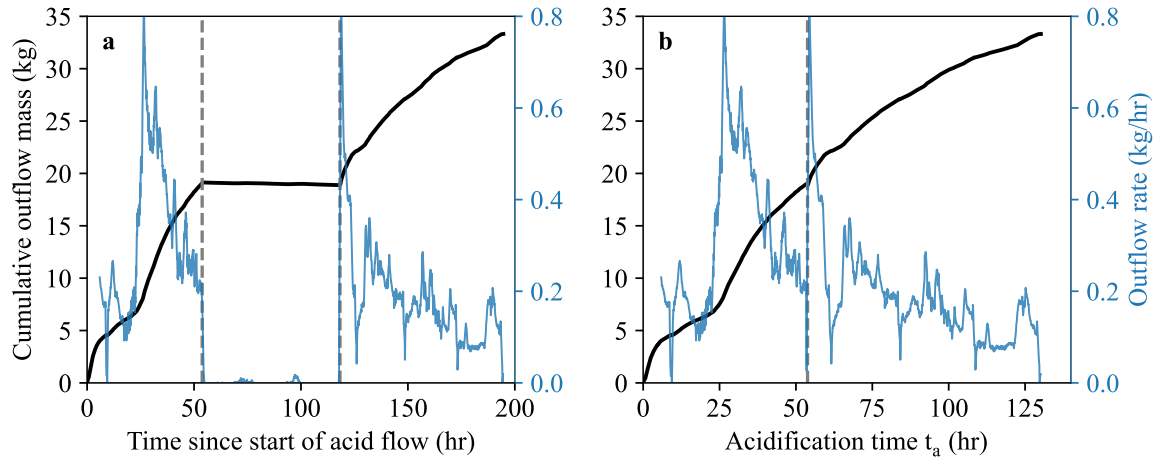


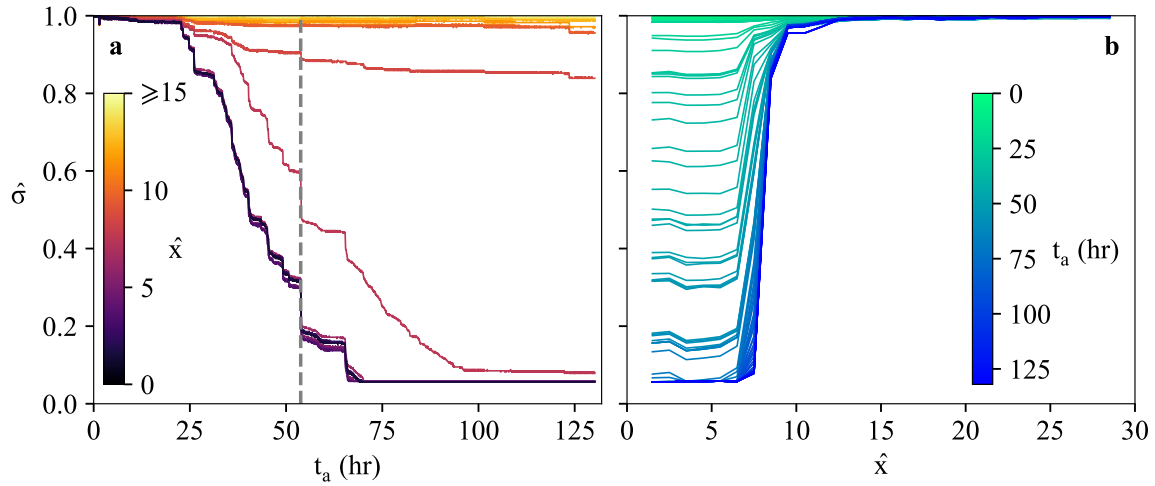
FIGURE 5.5. **Acid flow.** Cumulative outflow mass (black) and mass outflow rate (blue) over **a**: time since the start of acid flow into the sample, and **b**: time during which acid was renewed (acidification time  $t_a$ ).

As seen in Figure 5.5 (b), the flow of acid initially increases with the beginning of chemical dissolution in the sample. This initial increase was followed by a steady decrease in outflow rate over the whole duration of the experiment, which could be due to the generation of finer particles produced by the very same chemical reaction process.

### 5.3.2 Applied stress

Initially, the plates on the loading frame were lowered into the pistons to fully compress the springs on them. Then, with sample surface deformation, the springs extended and applied lower stresses on the sample. This deformation, measured as detailed in Section 5.2.2, and the consequent changes in stress applied onto the sample by each piston are shown in Figure 5.6. This figure shows the distribution of normalised stress applied over acidification time and normalised distance from the inlet  $\hat{x}$  (defined in figure).

From this figure, we gather that there were multiple collapse events that caused surface deformations throughout the duration of experiment BL. The first event engaged approximately five pistons (those closest to the inlet), as seen by the proximity of the darker lines in Figure 5.6 (a). Subsequent deformations gradually engaged more pistons and eventually led to the full extension of the springs on the pistons closest to the inlet. The gradual engaging of more pistons is further seen in Figure 5.6 (b) as the gradual increase in deformation further from the inlet. Panel (b) further shows that most of the deformation seen at the surface of the samples occurs within  $\sim 10$  cm from the inlet, with virtually no surface deformation observed further along the sample.



**FIGURE 5.6. Stress evolution over time and space. a:** Normalised piston applied stress  $\hat{\sigma} = \sigma_p / \sigma_{p0}$  over acidification time  $\hat{t}_a$ . **b:** Normalised piston applied stress  $\hat{\sigma}$  over normalised distance from inlet  $\hat{x} = x / w_p$ , where  $x$  is the horizontal distance from the inlet and  $w_p$  is piston width (10 mm).  $\hat{x}$  can be thought of as the number of piston widths away from the inlet, which is conveniently equivalent to the distance from the inlet measured in centimeters.

### 5.3.3 Sample deformation

The flow of acid through the sand sample causes a reduction in the mass of the sand grains due to the dissolution of calcium carbonate molecules into the acetic acid solution. Simultaneously, the concentration of acetic acid in the solution depletes, turning it less acidic and reducing its interaction with the sand. This generates an acid gradient within the sample, with the lowest pH located closest to the inlet. The sand within this zone reacts with the acid, which results in a density decrease and an increase of its porosity. As the dissolution process continues, a hole begins to form within the highly-acidic zone of the flow, see Figure 5.7 (a).

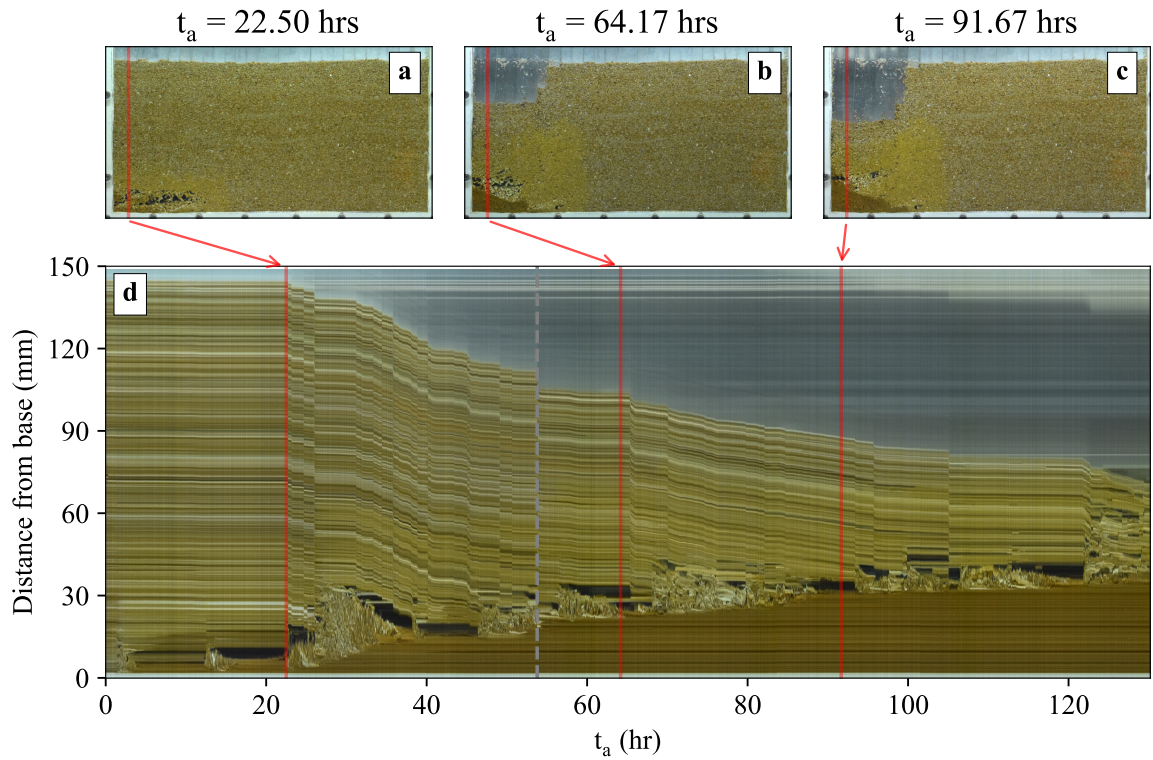


FIGURE 5.7. **Sinkhole opening and collapse progression.** **a-c:** Photographs of the experiment at different acidification times. **d:** Spatiotemporal plot of the experiment over its acidification time  $t_a$ , taken approximately 1 cm away from the inlet.

Initially, the growth of the sinkhole over time was not accompanied by any deformations on the surface of the sample, a characteristic evident in the first 20 hours of the spatiotemporal plot in Figure 5.7 (d). Past this time, numerous collapse events caused surface deformations

in the sand throughout the rest of the experiment. In addition to the surface deformation, the spatiotemporal graph also provides an indication on the sinkhole size observable on the boundary wall. At the lower end of the vertical axis, a black band begins to form from the start of the experiment. In time, the black band grows and collapses with major sinkhole collapse events, a feature that continues to occur throughout the experiment. Caution must be taken when studying this black band, as this plot is generated from a single pixel column; the presence of any dark coloured material in its field of view could result in a misguided perception of the size of the sinkhole.

The spatiotemporal plot presents other distinct features in addition to the black band. For instance, the darker brown pixel area that grows over time from the bottom of the plot is the insoluble part of the minerals that remain after the chemical reaction, which deposit as sediments at the bottom of the sample. These sediments may be the reason for the overall decrease in outflow rate seen in Figure 5.5, as they are much finer than the initial sand sample and therefore decrease the permeability of the sand from the inlet. Contrastingly, the lighter brown colour away from the sinkhole and the sediments is the ‘acid-affected zone’, visible in the photographs. Looking at Figure 5.7 (b-c), it is clear that this acid-affected zone reaches heights larger than that of the solution in the inlet compartment. Hence, capillary flow must be taking part in transporting the acetic acid above the phreatic surface of the flow. A similar mass transport in the capillary fringe of a transversal flow profile in sand is reported in (Silliman et al. 2002).

## 5.4 Sinkhole collapse characterisation

As a result of grain dissolution and sinkhole opening, grains and grain fragments within the sample may move and re-arrange under the confining stress applied from the top. This occurs continuously during sinkhole growth, ultimately leading to an instability in the granular structure of the sand and a resulting sinkhole collapse. During collapse, the mass above the sinkhole is no longer supported by the granular material and is therefore pushed downwards

under the stress applied by the pistons. The first sinkhole collapse event in experiment BL was captured by photographs and X-ray radiographs, both of which can be seen in Figure 5.8.

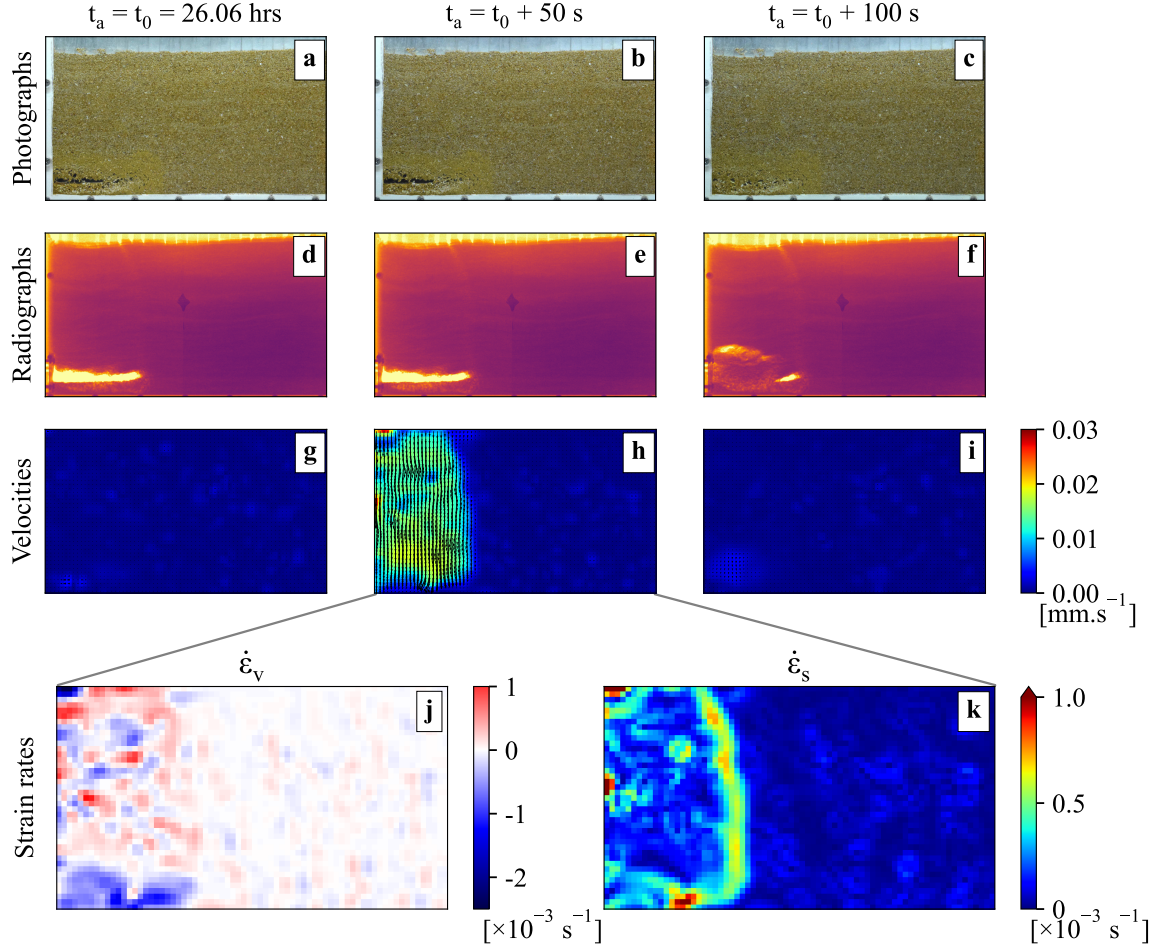


FIGURE 5.8. **Sinkhole collapse.** **a-c:** Photographs of the sand before, during and after collapse. **d-f:** X-ray radiographs of the sand corresponding to the times of the photographs above. **g-i:** Velocity fields corresponding to the radiographs above, obtained with particle image velocimetry (PIV). **j, k:** Volumetric  $\dot{\epsilon}_v$  and shear  $\dot{\epsilon}_s$  strain rates corresponding to the velocity field shown in panel (h). Here,  $\dot{\epsilon}_v = \dot{\epsilon}_{kk}$  and  $\dot{\epsilon}_s = \sqrt{\dot{\epsilon}'_{ij}\dot{\epsilon}'_{ij}/2}$ , where  $\dot{\epsilon}$  and  $\dot{\epsilon}'$  are the strain rate and deviatoric strain rate tensors, respectively.

Notably in panel (c), a portion of the grains directly above the sinkhole detached and fell into the acidic solution during collapse, forming a more arched structure which held the now displaced sand mass above the sinkhole. The use of X-ray radiography provided us with an improved view of the true magnitude of the sinkhole within the sample, since it shows the density of the material across its entire thickness. In contrast, the photographs

of the experiment can only capture the material along the walls of the testing apparatus. In comparison to the photographs, the radiographs make it apparent that a large portion of the sinkhole cannot be seen when looking at the side walls. Furthermore, the X-ray images provide an unparalleled observation of the shear band, and its associated dilation, during the collapse of the sinkhole (see panels (e-f)), a characteristic barely visible in the photographs. This is thanks to the porosity increase resulting from the dilation within the band, which makes it appear brighter under the X-rays.

During this sinkhole collapse event, the block of sand above the sinkhole moved directly downwards, as seen from the velocity magnitude fields in panels (g-i). These fields were obtained through the 'PIVlab' software, using a fast Fourier transform PIV algorithm on X-ray radiographs at 0.02 frames per second, passing the images through four consecutive square interrogation windows of side lengths of approximately 45, 29, 15, and 7 mm. Applying PIV on the radiographs produced velocity fields that account for the motions in the sand through the entire breadth of the sample, rather than only those visible at the boundaries (Guillard et al. 2017). From panel (h), it is clear that only the area to the left of the shear band moved downwards, while the rest of the sample remained practically static. This differential movement is further observed by computing the velocity field's volumetric and shear strain rates (see panels (j) and (k), respectively). The shear band observed by the X-ray radiographs appears again in panel (k), with high shear strain rate values. On the other hand the volumetric strain rate field in panel (j) does not show a consistent band, even though the sample had to dilate in order for the band to be observable in the X-rays. This dilation is not depicted by the volumetric strain rate since the main motion of the sand is in shear. Furthermore, the time scale through which the bulk movement of the sand was captured is relatively short for the slow velocities that cause the dilation in the sand within the shear band. However, the collapse of the sand onto the bottom of the tank is depicted by the large blue (compression) area with a red (dilation) zone above it.

The velocity and shear strain field of this sinkhole collapse share a remarkable similarity with previous experimental studies on the failure of granular materials induced by a trapdoor (Liang et al. 2020). This trapdoor failure study was performed on a sand sample with a similar



aspect ratio as that of the BL experiment prior its first sinkhole collapse. Figure 5.9 shows a comparison of the two with photographs of the samples after failure, as well as the cumulative shear strain fields of experiment BL and the experiment in (Liang et al. 2020). The similarity lies primarily in the solid-like movement of the sand mass above the sinkhole, the shape of the sinkhole itself (resembling a rectangular prism), and the shear band developing at an orientation nearly perpendicular with the horizontal. Note that the red zone in panel (a) should be ignored, as it is the result of the fluid flow of acid and motions of the sand grains suspended in it.

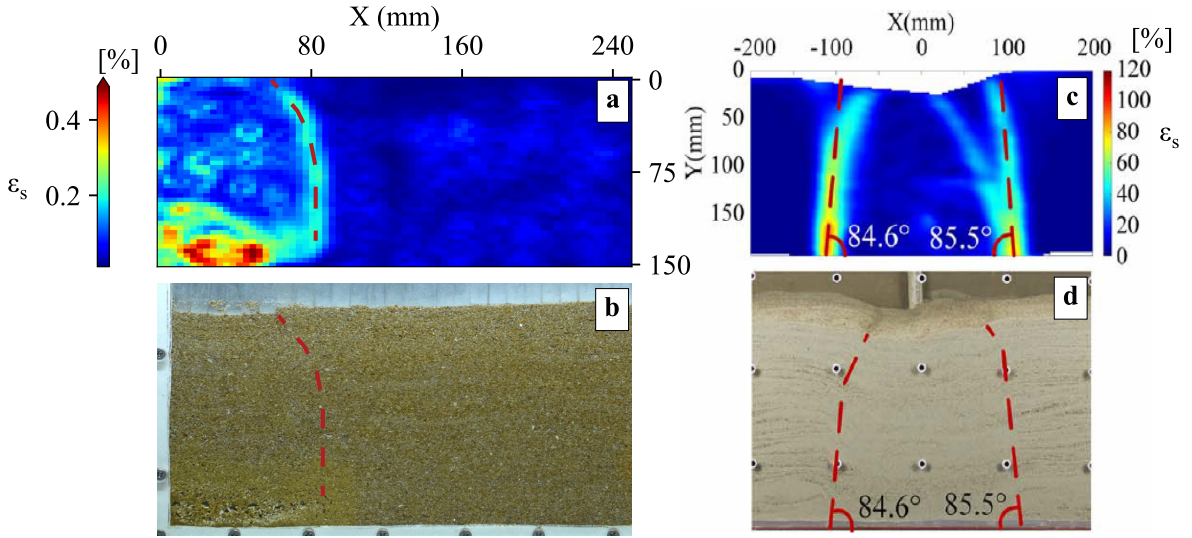
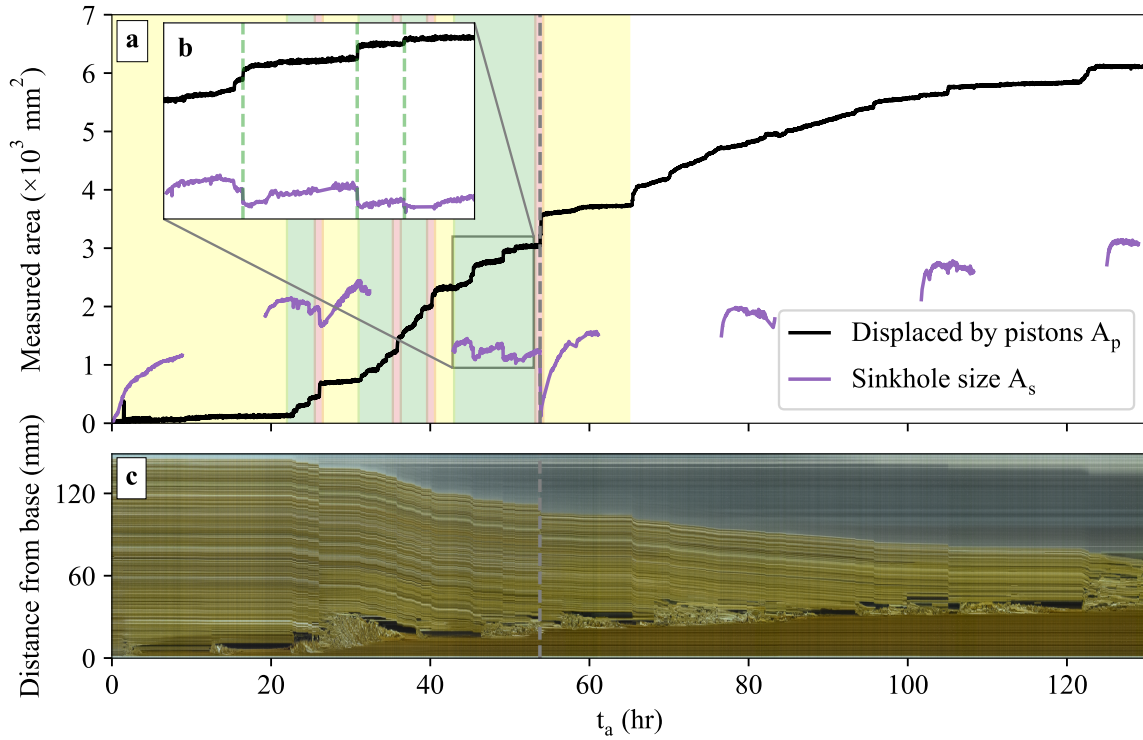


FIGURE 5.9. **Comparison between sinkhole and trapdoor collapse mechanisms.** Dashed orange lines were added to indicate shear bands. **a,b:** Shear strain field and photograph of sinkhole collapse. The shear strain field was obtained by integrating  $\dot{\epsilon}_s$  over the duration of the first sinkhole collapse (500 s) for radiographs taken at 0.02 frames per second. **c,d:** Shear strain field and photograph of trapdoor collapse, adapted from (Liang et al. 2020).

## 5.5 Creep-quake cycles

As previously mentioned, the initial growth of the sinkhole is not accompanied by corresponding surface deformations. In addition to the spatiotemporal plot, this disparity is shown by the measurements of total surface displacement and sinkhole size shown in Figure 5.10.



**FIGURE 5.10. Creep-quake cycles and sinkhole collapse.** **a:** Measured areas of piston displacement  $A_p$  (from photographs) and sinkhole size  $A_s$  (from radiographs) over acidification time. Shading has been added for the first portion of the experiments to distinguish between periods of no surface deformation during sinkhole growth (yellow), creep-quake cycles (green) and sinkhole collapse (red). **b:** Magnified section of the plot in panel (a), in which creep-quake cycles occurred, as marked by the dotted green lines. **c:** Spatiotemporal plot of the experiment over its acidification time.

The former is the observable sample area displaced by the pistons  $A_p$ , calculated as the sum of all piston displacements times their width:  $A_p = \sum w_p |y_p - y_{p0}|$ , where  $w_p$  is the piston width (10 mm),  $y_p$  is the vertical coordinate of the top of a piston  $p$ , and  $y_{p0}$  is its value at  $t_a = 0$ . On the other hand, the sinkhole size area  $A_s$  is taken as the area of the hole visible in the material density field obtained with the X-ray radiographs. This was computed with a pixel value count on binary images obtained after applying a threshold on the radiographs pixel intensity, i.e.  $A_s = s^2 \sum B_i$ , where  $s$  is the scaling ratio of length to pixels in the image and  $B_i$  is the Boolean value (0 or 1) of pixel  $i$  in the binary image. The threshold was chosen to be 2000 pixel intensity, a value that typically sat at around 25% of the total range of pixel intensities observed in the radiographs, within the tail of the pixel value histogram, and at



the transition point between a dark and a bright pixel. This choice ensured an accurate and consistent representation of the sinkhole size throughout all the radiographs.

While sinkhole growth does not necessarily cause piston movement, piston movement is often correlated to the closing of the sinkhole, as it displaces the sand mass above the sinkhole downwards. Figure 5.10 reiterates that there is close to zero surface deformation within the first 20 hours of the experiment, despite the constant growth of the sinkhole. After this time, there were a number of small deformation events in close succession prior to the first main sinkhole collapse event (the collapse event described in the previous section). This progression can be seen repeatedly throughout the experiment, as shown by the shading added to Figure 5.10 (a). This panel shows the periods of no surface deformation with sinkhole growth highlighted in yellow, followed by the green periods of small successive deformations, which lead to major sinkhole collapse events, shaded in red. The shading of the different periods was added up until the first instance of piston disengagement, which is the point in which piston springs are fully extended ( $|y_{p0} - y_p| \geq l_{sf}$ ) and no more stress is applied onto the sand besides that on the self weight of the pistons ( $m_p g$ ). This occurred at approximately 65 hours, as shown in Figure 5.6 (a).

The smaller collapse events can be identified from the small surface deformations and the small reductions in sinkhole area that occur right before the larger change in both, an example of which is shown in inset (b). This series of events is remarkably similar to the observations made in Chapter 4 on the formation of creep-quake cycles in calcareous sands. The creep on the top of the sample is accompanied with sinkhole growth up until an unstable state is reached, where quakes interrupt the constant creep period and the sinkhole size suddenly reduces. Here, we see how creep-quake cycles remain relevant and survive as a collapse pattern despite being interrelated with other collapse mechanisms in a much more complex geometry; in this case, a trapdoor-like failure mechanism triggered when the sinkhole becomes too large to sustain the applied load.

## 5.6 Discussion

### 5.6.1 Creep-quake cycles as a signature signal for major collapse

The link between the emergence of creep-quake cycles and major sinkhole collapse seen in experiment BL is further supported by the results in the dense sand experiments: FD and BD.

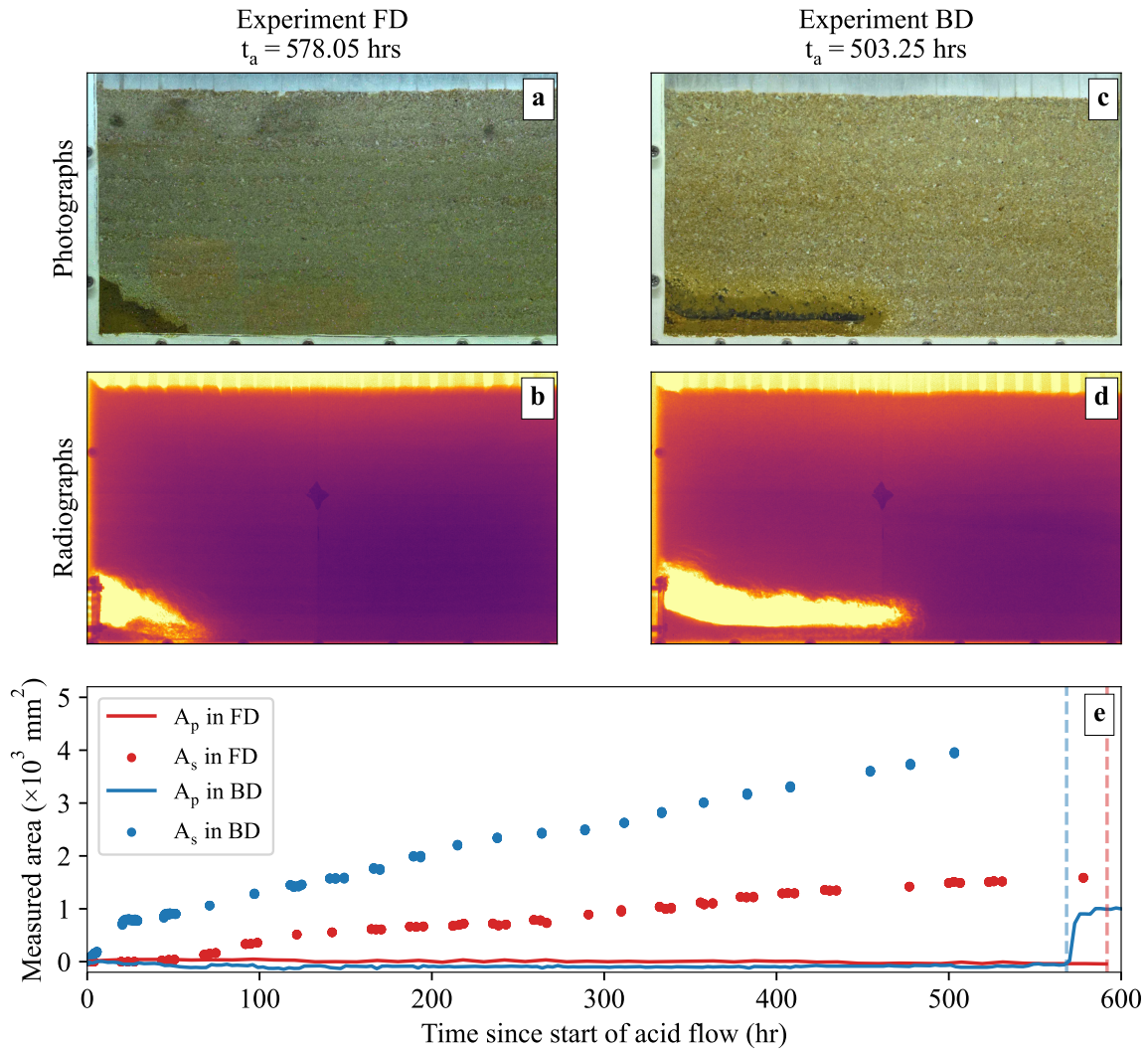


FIGURE 5.11. **Sinkhole growth without collapse.** a-d: Photographs and X-ray radiographs taken by the end of experiments FD (a-b) and BD (c-d). e: Measured areas of piston displacement  $A_p$  (from photographs) and sinkhole size  $A_s$  (from radiographs) of experiments FD and BD over their duration. The times at which acid renewal was stopped are marked by dotted red and blue lines for experiments FD and BD, respectively.

Figure 5.11 shows a set of photographs and radiographs taken by the end of these two experiments, as well as their measured areas of surface deformation and sinkhole size. Note that the vertical dashed lines in panel (e) denote the end of acid renewal for each experiment. It is clear from this figure that creep-quake cycles did not appear throughout the experiment. Accordingly, no major collapse events occurred during the steady flow of acid through the samples, despite sinkholes opening and growing within them. The sand samples in both experiment FD and BD had an overall lower permeability than the sample in experiment BL. This resulted in lower flow rates of acid for an identical hydraulic head difference, and in much longer experiment durations. The cumulative flow and flow rates throughout both of these experiments is shown in Figure 5.12. On the two extremes, the total cumulative mass of acid flow for experiment FD ( $\sim 11$  kg in  $\sim 600$  hours) was approximately one third of that in experiment BL ( $\sim 34$  kg in  $\sim 130$  hours).

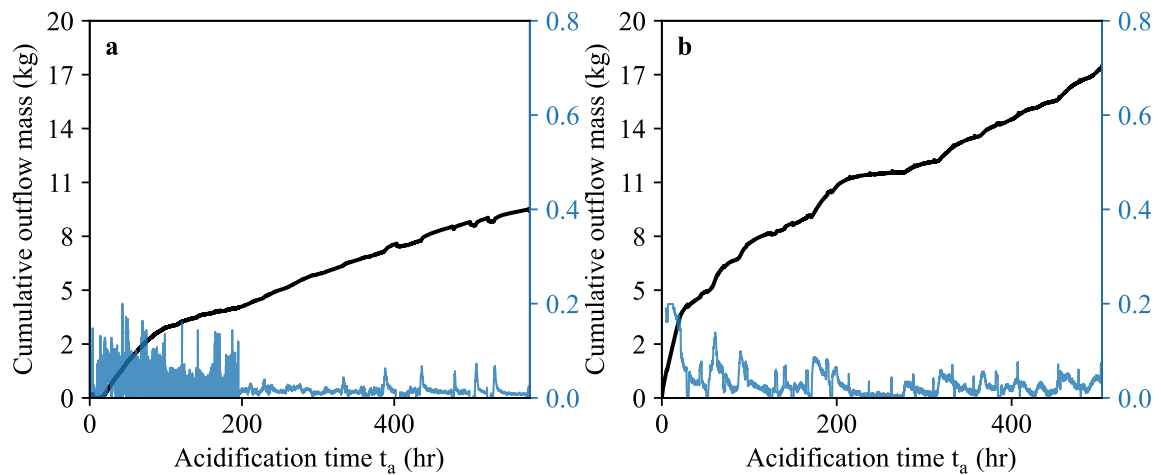


FIGURE 5.12. **Acid flow for experiments FD and BD.** Cumulative outflow mass (black) and mass outflow rate (blue) in time during which acid was renewed (acidification time  $t_a$ ).

Our identification of creep-quake cycles preceding different collapse mechanisms in sands may aid the assessment and forecast of sinkhole collapse. Currently, in-field sinkhole monitoring techniques include subsidence monitoring (post sinkhole collapse), radar images (GB-InSAR), tiltmeters to measure deflections on the ground, as well as seismic wave propagation for sinkhole collapse identification and their mapping (Hardy Jr and Belesky 1987; Gutiérrez et al. 2019). Seismic wave tomography is a technique with which one can reconstruct an

underground (hidden) sinkhole, in order to determine its size and stability. In our experiments we accomplish a similar goal via the use of X-ray radiographies, which allowed us to make a direct link between the evolution of the sinkhole and creep-quake cycles. From our findings, we can envision future ground monitoring systems that make use of these patterns to compliment their monitoring and predictions of major ground collapse events. Throughout this chapter, we see that sinkhole formation and growth is not accompanied by surface-level deformations, making it difficult to assess the conditions of a sinkhole from visual observations of the ground surface alone. Despite creep-quake cycles presenting only small surface deformations, they are known to produce acoustic emissions during quakes. Future monitoring systems could capitalise on these acoustic emissions to seek the signature patterns of creep-quake cycles and link them to the likelihood of a major collapse event. Such an advancement would lay the basis to further improve predictions of imminent underground sinkhole collapse.

Our findings are indicative of a possible connection between creep-quake cycles and major sinkhole collapse. However, further research is needed to validate their correlation and to investigate the possibility of capitalising on our knowledge of creep-quake cycles to forecast sinkhole collapse. In order to do so, future experimental investigations should aim to minimise the influence of boundary conditions on the test samples. The experiments presented here were performed in a fairly narrow tank, which could have influenced the behaviour of the material through frictional effects at the tank walls. Additionally, the sinkholes formed in the ‘highly-acidic’ area of the sample, which is close to the inlet, and therefore close to three boundary walls. In retrospect, these frictional effects may have had a significant impact on the collapse behaviour (or lack thereof) of the dense sand samples. This is in addition to the fact that the denser and finer sands used would experience larger magnitudes of capillary effects, since their pores are smaller than those of the loose BBS-B. Given that the sand samples in experiments FD and BD were both densely packed, they would need to expand prior to reaching their critical state and failing. This phenomenon, combined with the narrow tank walls preventing material dilation, increased the stress re-distribution onto the tank walls, resulting in sand samples that could hold themselves up despite the presence of large sinkholes. Future studies should therefore aim to track creep-quake cycle patterns in sinkholes

forming well within the sandy medium. This could be achieved by placing the acid inlet in the centre of the sample, far from the boundary walls. To further minimise the influence of boundary walls, future research is encouraged to move away from a rectangular test sample onto different boundary conditions, such as a wide cylindrical setup, where the acid inlet is placed in the centre of the sample. This change would result in different flow conditions, however, the proposed cylindrical setting remains relevant in real-geotechnical scenarios, such as in the leakage of underground pipes, which have previously resulted in sinkhole formation (Hadjmeliani 2015).

### 5.6.2 Earthquake statistical patterns in sinkhole collapse

With surface deformations, caused by either creep-quake cycles or the trapdoor-like sinkhole collapse mechanism, the loading pistons displace downwards and their springs elongate. This movement dissipates the elastic energy stored in the springs relative to when they were initially compressed. An examination of the cumulative dissipation of elastic energy, shown in Figure 5.13 (a) by the solid blue line, reveals a striking resemblance to the cumulative moment curves typically used in the study of earthquakes (Davis 2016; Yeck et al. 2017). Similar to Figure 5.10 (a), Figure 5.13 focuses on the experimental data acquired from  $t_a = 0$  up until the first instance of piston disengagement ( $t_a = 65$  hr). Furthermore, Figure 5.13 (a-c) include the same shading used previously, where yellow indicates periods of no surface deformation, green indicates creep-quake cycles, and red indicates a major sinkhole collapse event.

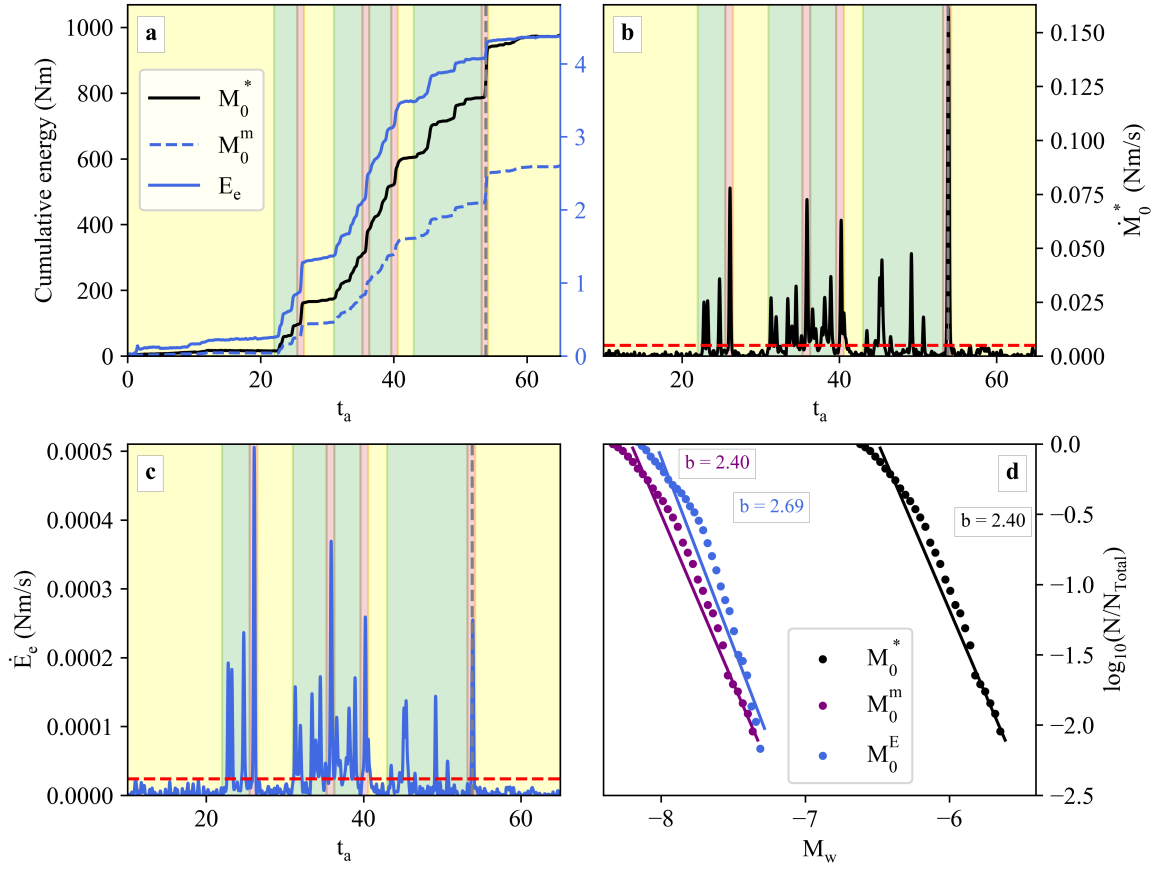
In order to qualitatively compare our results to earthquake statistics, we computed the equivalent earthquake magnitude  $M_w$  for the collapse events observed in our sinkhole experiments based on definitions established in (Kanamori and Brodsky 2004). A similar approach has been previously used to study earthquake mitigation using analogous experiments on a model material, absorbent porous paper (Tzortzopoulos et al. 2021). This comparison required us to obtain an estimation of the energy released during a quake event. In (Kanamori and Brodsky 2004),  $M_w$  is defined as a function of a seismic moment  $M_0$  with the following relation:

$$M_w = \frac{2}{3} \log_{10}(M_0) - 6.07. \quad (5.3)$$

For this conversion,  $M_0$  must be computed in Nm and is defined as:

$$M_0 = DSG, \quad (5.4)$$

where  $D$  is the shear displacement offset over a surface with area  $S$  within a medium of a given rigidity, which is taken as the shear modulus  $G$  of the material.



**FIGURE 5.13. Energy release and statistical similarities between collapse events in experiment BL and earthquake laws.** **a:** Cumulative energy dissipation in terms of seismic moment  $M_0^*$ , modified seismic moment  $M_0^m$  (defined in text), and elastic energy  $E_e$  over acidification time  $t_a$ . **b:** Rate of seismic moment  $\dot{M}_0^*$  over  $t_a$ . **c:** Rate of elastic energy dissipation  $\dot{E}_e$  over  $t_a$ . **d:** Gutenberg-Richter law in terms of  $M_0^*$ ,  $M_0^m$ , and  $M_0^E$ . The dots are experimental measurements and the lines are their corresponding lines of best fit, with slope  $-b$ .

We propose three distinct methods to estimate  $M_0$  in experiment BL and input it in Equation 5.3. The first approach is similar to that used in (Kanamori and Brodsky 2004). For this, we computed  $M_0^*$  following the conventions used in earthquake statistics. Here, we define Equation 5.4 as follows:

$$M_0 = M_0^* = GS \sum_{p=1}^{n-1} (\Delta_t y_{p+1} - \Delta_t y_p), \quad (5.5)$$

where the number of pistons  $n = 28$ , the shear modulus  $G = 1.59$  MPa, which obtained from standard tests on Barry beach sand (Al-Douri and Poulos 1992),  $S$  is an idealised surface area in which the sample is sheared, which we defined as a rectangle whose sides are the breadth and initial height of the sand sample (75 mm  $\times$  150 mm),  $\Delta_t$  denotes a change in time  $t$ , and  $y_p$  is the vertical coordinate of piston  $p$ , as defined in Figure 5.3.

In our system, however, energy is more likely to be dissipated through friction in the material than elastic shear. Therefore, we propose replacing  $M_0$  in Equation 5.4 with a modified seismic moment  $M_0^m$ , in which we define the rigidity of the material as the shear stress resisting the differential displacement of the pistons,  $\tau$ . We implement this modification to focus on the energy in the system that is dissipated through friction on the failure plane, instead of elastic dissipation on the failure plane, which  $G$  would suggest.  $M_0^m$  is defined as:

$$M_0 = M_0^m = \tau S \sum_{p=1}^{n-1} (\Delta_t y_{p+1} - \Delta_t y_p), \text{ and} \quad (5.6)$$

$$\tau = \mu K_0 \bar{\sigma}_v, \quad (5.7)$$

where the friction coefficient  $\mu = \tan(\phi)$  with  $\phi = 45.7^\circ$  for Barry beach sand (Al-Douri and Poulos 1992), the coefficient of lateral earth pressure  $K_0 = 1 - \sin(\phi)$  (Jaky 1944), and the typical vertical stress applied by the pistons  $\bar{\sigma}_v = (30.843 - 1.757)/2 \approx 15$  kPa. For simplicity,  $\bar{\sigma}_v$  was taken as the midpoint between the minimum and maximum stress a piston

can apply through the experiment, given by Equation 5.1 for  $y_p = y_f - l_{sf}$  and  $y_p = y_0$ , respectively. With these parameters, we obtain  $\tau = 4.24$  kPa.

Lastly, we can compare this frictional dissipation to the dissipated elastic energy during a given time span,  $\Delta_t E_e$ . This method was devised to minimise any assumptions on material parameters or failure geometries and instead focus on the more objective measurement of changes in the elastic energy stored in the springs pushing the pistons. With this definition:

$$M_0 = M_0^E = \Delta_t E_e, \quad (5.8)$$

$$\text{where } E_e = k_s \sum_{p=1}^n ((l_{sf} - |y_f - y_{p0}|)^2 - \langle l_{sf} - |y_f - y_p| \rangle^2). \quad (5.9)$$

Note that the elastic energy of piston  $p$  is stored in two springs. Hence, there is a factor of 2 that gets cancelled from the 0.5 factor in the spring elastic energy equation. Furthermore,  $E_e \geq 0$ , as the springs start fully compressed ( $y_p = y_{p0}$ ), release all their stored elastic energy when fully elongated ( $y_f - y_p = l_{sf}$ ), and do not dissipate any further elastic energy when  $y_f - y_p > l_{sf}$ , hence the Macaulay brackets. In addition to the elastic energy of the springs, the gravitational potential energy stored in the pistons and the soil would also change as the sinkhole collapses. However, this change is negligible compared to the elastic energy change. In fact, the maximum change in potential energy is two orders of magnitude smaller than that of the elastic spring energy.

Figure 5.13 (a-c) reiterate the three behavioural patterns observed in the sand sample: no surface deformation, creep-quake cycles, and major sinkhole collapse. Notably, Figure 5.13 (a) shows a large disparity between the cumulative energy calculated with  $M_0^*$  (black line plotted for the left vertical axis) and those calculated with our modified approaches (blue lines plotted for the right vertical axis). The proximity between the  $M_0^m$  and  $E_e$  lines is consistent with the frictional dissipation mechanism, and reasonable given the numerous assumptions made to calculate the frictional energy dissipation.



The curves in Figure 5.13 (b) and (c) were obtained as the discrete derivatives of  $M_0^*$  and  $E_e$ , respectively. In all approaches used for Figure 5.13, the data used to determine  $M_w$  was smoothened with a Gaussian running average and trimmed to exclude the pixel-dependent image analysis noise in the  $\dot{M}_0^*$ ,  $\dot{M}_0^m$ , and  $\dot{E}_e$  (the upper dot notation denotes rates in time). The thresholds used for  $\dot{M}_0^*$  and  $\dot{E}_e$  are shown as red dotted horizontal lines in panels (b) and (c), respectively. The smoothening of the data was done in order to reduce the influence of noise in our readings. The selection on the degree of smoothening was kept at a point that was sufficient to minimise noise, yet not enough to have significant losses in resolution.

Figure 5.13 (b-c) emphasise the difference in the characteristics of the two collapse mechanisms, as the energy released by creep-quake cycles is generally lower than the larger energy bursts that occur after them (caused by major sinkhole collapse events). Furthermore, these panels show the clearest indications on how the smaller signals of creep-quake cycles precede major collapse events. This particular characteristic differs from the typical observations in earthquake monitoring, which are described by the broadly-used Omori's law (Omori 1895). Opposite to our observations, large magnitude earthquakes trigger subsequent smaller events (aftershocks) that reduce in frequency as time since the earthquake increases.

In contrast, a great degree of similarity with earthquake statics is seen when examining the Gutenberg-Richter (GR) law, a concept first introduced in (Gutenberg and Richter 1944). This law states that the number of earthquakes  $N$  of magnitude greater than or equal to a magnitude  $M_w$  is given by:

$$\log_{10}(N/N_{\text{Total}}) = a - b \log_{10}(M_w), \quad (5.10)$$

where  $a$  and  $b$  are constants, and  $N_{\text{Total}}$  is the total number of earthquakes. In other words, the frequency of events of magnitude greater than a magnitude  $M_w$  decays exponentially (to the power of  $b$ ) with  $M_w$ . This relation can be seen in Figure 5.13 (d) for our three definitions of  $M_w$ . As shown in the panel, the statistics on the collapse of the sinkhole in experiment BL can be reasonably described by the GR law. Note that the small  $M_w$  values are a result of the conversion to earthquake magnitudes, since the energy released in these collapse events are many orders of magnitude smaller than those seen in real earthquakes.

While there is a significant difference in the scales between the results obtained with  $M_0^*$  and those obtained from our modified approaches with  $M_0^m$  and  $M_0^E$ , the  $b$  coefficients collected from our analyses are consistent with each other, and with other  $b$  coefficient values reported on the statistic of earthquakes (Godano et al. 2014). The discrepancy between the curves of  $M_0^*$  and  $M_0^m$  may be partly due to our selection of  $G$ . In the tests performed in (Al-Douri and Poulos 1992), the stress  $\sigma$  exerted on the Barry beach sand samples was close to 100 kPa. Considering a scaling of  $G \propto \sigma^{1/2}$ , a stress of  $\bar{\sigma}_v \sim 15$  kPa would reduce  $G$  by a factor of 2.6, which would place the  $M_0^*$  curve closer to the other two, yet this value of  $G$  remains two orders of magnitude larger than  $\tau$ . It is also noted that the choice made on the smoothening of the energy curves in Figure 5.13 (a) affects the thresholds used in the calculation of  $M_w$ , which in turn would have an effect on the  $b$  parameter, as stated in (Godano et al. 2014). Higher thresholds, needed with low smoothening, led to significantly higher  $b$  coefficients, a trend that is consistent with the results shown in (Godano et al. 2014).

In summary, we define two distinct categories of collapse in our experiments, those caused by creep-quake cycles and those resulting from major sinkhole collapse. Creep-quake cycles cause lower bursts of energy dissipation than (and commonly precede) major sinkhole collapse. Lastly, we find that the statistics of all combined collapse events is well described by the Gutenberg-Richter law.

### 5.6.3 The influence of cyclical acidic degradation

In experiment BL, the inclusion of a period drying in between the flow of acid resulted in a major sinkhole collapse event upon the re-introduction of acid. Similarly, in experiment BD, a major collapse event occurred some time after the renewal of acid was stopped, even though there were no collapse events during the steady flow of acid. The collapse in experiment BD can be seen by the jump in the blue line in Figure 5.11 (e). These observations may indicate that a cyclical application of acidic degradation could accelerate the sinkhole collapse process. However, the cause of the collapse events cannot yet be singled out to cyclical acidic degradation. The two observations of this phenomenon have been made on sand samples whose

saturation was changing, which can change the mechanical response of the material and lead to collapse. Moreover, the collapse in experiment BL happened during wetting, while the one in experiment BD happened during drying.

Although a study of cyclical chemical degradation was not the focus of the experiments presented in this chapter, our findings present a great opportunity for future studies to explore the correlation between cyclical chemical degradation and the triggering of sinkhole collapse. To begin, future research on this topic is encouraged to conduct experiments in which the effects of saturation and chemical degradation are individually assessed. Such experiments could have cycles of acidic degradation that are interluded with fresh water flow through the sample or air drying. These cycles could be arranged in a water-acid-water succession or an drying-water-acid-water-drying one. The impact of these potential findings is wide-reaching and can spark interest in several research communities, including those focused on geotechnical stability, mining waste management, and underground resource extraction.

## 5.7 Conclusion

This chapter has provided an investigation into the formation of collapse patterns in a Barry beach sand sample under transversal acid flow. The behaviour of the samples was monitored through the use of photographs, X-ray radiographs and measurements of the solution outflow. These observations captured numerous collapse events caused by the formation of a sinkhole in the sample. The collapse behaviour of the soil during major sinkhole collapse events exhibited similarities to previous studies on trapdoor failure experiments. Furthermore, the statistics on the collapse patterns in our experiments were found to be consistent with the Gutenberg-Richter law, which is broadly used to describe earthquakes.

Most significantly, creep-quake cycles were observed intertwined with the much larger sinkhole collapse events. The emergence of these cycles can have significant implications in ground stability monitoring, as it can aid in the anticipation of major degradation-induced collapse events. However, further investigations on the precedence and relation of creep-quake

cycles with major sinkhole collapse are needed in order to establish a reliable correlation between them.

Additionally, the observations of collapse events triggered by the drying and subsequent re-introduction of acidic flow into the sample suggest the possibility of cycles of degradation or water saturation being able to trigger sinkhole collapse. These findings open up new avenues for future research, such as exploring the applications of cyclical chemical degradation in the control of flow through porous rocks and soils. This research direction also presents its own added complexities, as slow drying cycles might concentrate acidic degradation at the particle contacts and potentially create bonds in the material via cementation. Hence, there could be a competing mechanism with stabilising effects which may play a role in certain wetting-drying regimes.

## CHAPTER 6

### **Conclusion and perspectives**

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Inspired by the finding of ‘ricequakes’ in a seemingly inconsequential model material (puffed rice), this thesis has presented a comprehensive study on the formation of previously unidentified collapse patterns in porous media, creep-quake cycles. These cycles consist of periods of slow material deformation (creep) interluded by sudden collapse events (quakes). We demonstrated the emergence of this pattern as a collapse mechanism that is inherent to all porous media, the conditions for its occurrence in calcareous sands, and its consequence and concomitant presence with other collapse mechanisms, such as sinkhole collapse. The results detailed in this thesis also present numerous avenues for further research to continue exploring the significance of these collapse patterns in degrading porous media.

Creep-quake cycles were identified experimentally on a diverse array of porous materials, including puffed rice, sand, rock salt, and ice. These experiments served as the basis from which we established our claim on the ubiquity of this collapse mechanism across all porous media. Three conditions were defined as necessary requirements for this phenomenon to emerge. First, the physical medium must have a collapsible length-scale for quakes to occur, a requirement inherently satisfied in porous media by the existence of pores. Second, the medium must be subjected to a sufficient compressive stress. Third, it must be simultaneously exposed to a non-uniform field of degradation. These requirements were identified thanks to the wide range of porous media studied, as well as the substantially different degradation fields, which included various saturation, dissolution rate, and available heat gradients. To corroborate and build upon these findings, future studies could investigate the emergence of creep-quake cycles in porous materials with different properties to those presented in this thesis. For instance, the materials studied here all exhibited brittle collapse, yet there are

numerous ductile porous media with high industrial applicability, such as metallic foams or lattices. One could study the emergence of creep-quake cycles in such ductile porous media by performing direct tests on metallic lattices under heat gradients, or by examining model ductile materials, such as melting wax, low melting point metals, or even aerated chocolate. Furthermore, future studies could better characterise the compaction dynamics of creep-quake cycles with the use of highly controlled designed materials, which could be fabricated using additive manufacturing technologies such as 3D printing. On a different direction of inquiry, one could investigate the potential for a reversed mechanism of creep-quake cycles with the use of granular media made of expanding materials, such as hydrogels. Placing such a granular assembly in a non-homogeneous field of saturation could result in a gradient of rates of grain growth, which could lead to continuous slow increases in the height of the material column interluded by sudden rapid growth events, much like creep-quake cycles, but caused by swelling rather than degradation. Similar to the work presented in this thesis, investigating the potential ‘reversed quake’ effect can have implications in real-geotechnical scenarios. These include characterising patterns developed in expansive geomaterials interacting with water, such as clays (Smalley 2006). In fact, swelling materials are a significant challenge in tunnelling, and can lead to collapse of underground infrastructure (Selmer and Palmstrom 1989).

Our conceptual model of the creep-quake mechanism were validated with a simple theoretical model capable of recovering the cycles based on the three key requirements presented earlier. The model was implemented with a set of one-dimensional elastic breakable carriages within a non-uniform field of degradation that resembled those observed experimentally in chemical degradation fields. This model laid the basis for a theoretical framework that can be used to predict the creep-quake response of a material from descriptions of its material parameters and their response to a degrading agent. Future work on this model and its implementation could incorporate laws of material degradation caused by melting to capture the behaviour of the ice samples in Chapter 3. Furthermore, the reach of our model could be extended with its implementation in multi-dimensional systems. A first step would be to expand the current model onto two-dimensional spring lattices. In fact, a similar approach has been previously used to model dynamic compaction patterns and propagating compaction bands in dry puffed

rice (Guillard et al. 2015). Implementing the model in a two- or three-dimensional space and accounting for capillarity would further allow to capture the impact of partial saturation, rather than simplifying it with a one-dimensional degradation gradient. Ultimately, the goal would be to develop a continuum constitutive description of the stress-strain response of porous media under degradation fields. In this line of research, a recent study by Riley et al. (2023) has successfully modelled recurrent stress drops in dry porous media (without degradation) at a constitutive level.

The different properties of the materials studied in Chapter 3 result in different conditions for the emergence of creep-quake cycles. The second part of this thesis was focused on characterising these conditions for a particular material, calcareous sand. This was done with the aim of expanding our knowledge on the collapse mechanics and patterns seen in naturally occurring geological formations. Therefore, we devised an in-depth experimental study on the formation and evolution of creep-quake cycles in calcareous sands. As a result, we found that acid dissolution causes changes in grain size distribution, which relates to the collapsible length scale of the medium, and can lead to a decrease in available pore space and the eventual cessation of creep-quake cycles. Additionally, a parametric study on the influence of applied loading and degradation conditions on the sand unveiled a ‘Goldilocks zone’ for quake formation in terms of compressive stress and rates of acid injection. These two parameters relate directly to the requirements for a sufficient stress and the formation of a non-uniform field of degradation. Moreover, an inquiry on the effects of initial sample height suggested that quakes are more frequent in taller sand columns.

Although Chapter 4 presented a comprehensive study on the conditions for creep-quake cycles in calcareous sands, the experimental nature of this investigation imposed various limitations that can be addressed in the future. These include the use of rapid X-ray or neutron tomography techniques to discern the full extent of grain shape and size evolution during creep-quake collapse, as well additional experiments on the parametric study of creep-quake cycles in terms of injection rate and initial sample height, and wider test tubes to minimise the influence of boundary conditions. Beyond experimental investigations, a different approach would be to analyse the emergence of creep-quake cycles in simulated environments. The

use of simulation techniques, such as the Discrete Element Method (DEM), could prove immensely valuable in corroborating the key requirements for creep-quake cycles to emerge, as well as facilitating data gathering for parametric studies. For instance, a recent study makes use of the DEM to simulate calcite carbonate granules under compression and dissolution in acetic acid in order to examine the microstructural behaviour of the material and the effects of dissolution rates on the lateral redistribution of stress (Alam et al. 2022). Unlike experiments, this method is not limited by physical constraints, a characteristic that can be useful in determining the influence of boundary conditions on the emergence and frequency of creep-quake cycles. However, accurately simulating particle breakage, water saturation, and chemical reactions in DEM would require further investigations.

Given that most geotechnical collapse scenarios involving soil-acid interactions occur within boundaries that cannot be captured with a one-dimensional system, we moved onto an exploration of two dimensional collapse in sand. In Chapter 5, we examined the collapse mechanics of a sinkhole caused by the transversal flow of acid through a calcareous sand. This shift allowed us to explore conditions with a much closer resemblance to those found in nature and in real geotechnical scenarios, such as underground acid flow or flow through a tailing dam. The two-dimensional experiments in this study show how creep-quake cycles can survive as a collapse mechanism of the medium despite the more complex geometry and the co-existence of other different collapse mechanisms. In fact, these cycles were observed preceding major sinkhole collapse events in numerous instances.

This final observation presents significant potential in the monitoring of ground stability, as it may hold the key to improved forecasting degradation-induced collapse events. Such investigations can leverage the knowledge gathered in this thesis to mitigate the impacts of disastrous collapse events. Future research is therefore encouraged to investigate the connection between creep-quake cycles signals and the likelihood of impending collapse events in geomaterials. Given that sinkholes are not commonly accompanied by significant surface deformations prior to their collapse, the monitoring of underground acoustic signals emanating from repetitive quakes would be of great use. Previous studies have made use of similar acoustic emissions to monitor underground developments, such as coal fracturing



during underground coal gasification (Su et al. 2017) and leakage in underground pipes (Van Hieu et al. 2011). In a similar manner, the signature acoustic emissions generated by quakes could be analysed to obtain information on the location and likelihood of a major collapse event. This could prove immensely useful in areas at high-risk of sinkhole subsidence, such as those found in the vicinity of the Dead Sea, Israel (Shalev et al. 2006; Frumkin et al. 2011). In fact, the monitoring of acoustic emissions is a well-established technique used to study the onset of collapse in porous media. Its applications extend to avalanches and landslides, as well as the stability of embankments, foundations, and mines (Koerner et al. 1981). Although further investigation is required to establish a definitive link between creep-quake cycles and sinkhole collapse, the observations made in Chapter 5 present great potential for future research to fill this gap and make ground-breaking advances on ground stability monitoring techniques.

To conclude, this thesis presented a ubiquitous mechanism of creep-quake collapse in porous media, as well as the conditions for its emergence and its relevance in a degrading porous geomaterial. The findings and conclusions of this work offer valuable insights that build upon our collective knowledge on the formation of collapse patterns in degrading porous media.

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## APPENDIX A

### **Paper publication: Identification of Creep-Quake Cycles in Calcareous Sands**

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The following paper was published in: IWBDG 2022: Multiscale Processes of Instability, Deformation and Fracturing in Geomaterials, as part of the Springer Series in Geomechanics and Geoengineering book series (SSGG), in December 2022. I was the primary researcher and lead author of this work, with the collaboration of Dr François Guillard and Prof Itai Einav.



# Identification of Creep-Quake Cycles in Calcareous Sands

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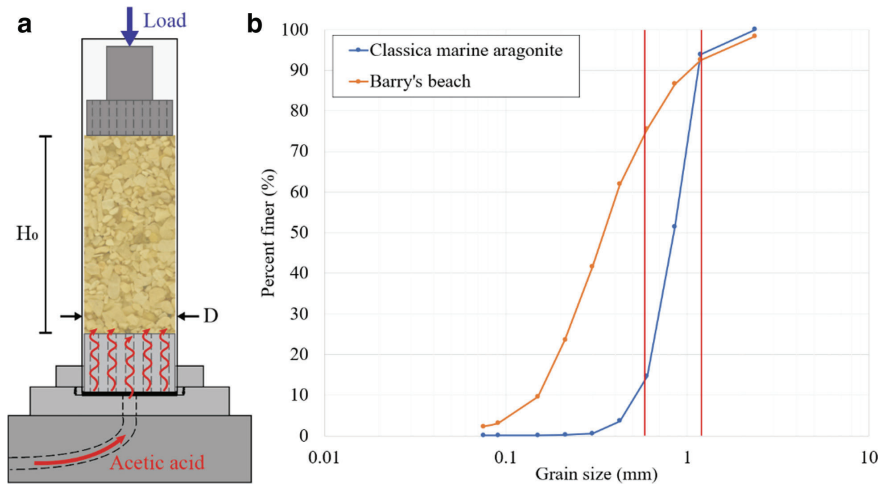
**Abstract.** Degradation processes in porous media concern numerous industries. In geomaterials, these processes are capable of triggering localised instabilities which may result in the collapse events, as has been observed with the formation of sinkholes and the collapse of rock salt mines. Previous studies have identified periodic creep-quake collapse patterns in porous materials under simultaneous compression and degradation. However, the possibility of the development of this particular collapse pattern in geomaterials is yet to be explored. Here, we reproduce the creep-quake cycle patterns in two different calcareous sands and present the physical mechanism that underpins it. Our experiments show that this mechanism results in sudden localized collapse events, loud acoustic emissions, and drops in the servo-controlled load used to compress the sand samples. We then explore the behavioural change of the sand when subjected to different degradation rates and different applied stresses. Finally, we explore the parametric limits of applied stresses and degradation rates within which calcareous sand undergoes creep-quake cycles. Our results show that this collapse mechanism can emerge in geomaterials that are simultaneously subjected to sufficient compression and a non-uniform degradation field.

**Keywords:** Calcareous sand · Acetic acid · Creep-quake cycles

## 1 Introduction

The chemical dissolution of geomaterials is a common diagenetic process found in nature. The effects of these processes may result in the damage of man-made or natural structures, such as leakage and structural instability in tailing dams [1, 2], sinkhole formation [3], damage to underground CO<sub>2</sub> storage systems [4], and submarine landslides [5]. The consequences of such events may include environmental pollution, human injuries, and loss of lives. This highlights the importance of developing an accurate understanding of the behaviour of geomaterials under acid degradation. In recent years, a novel collapse mechanism was identified experimentally in uniaxially loaded, partially soaked puffed rice [6]. The mechanism consists of localised cyclic collapse events, named ‘ricequakes’, that initiated within the partially saturated material and were interluded by periods of accelerated creep deformation. The discovery of these ‘creep-quake’ cycles motivated our investigation of this distinct collapse pattern in other porous media. Specifically, we

consider the effect of chemical dissolution processes in calcareous sands under load, which can be closely related to the conditions under which the puffed rice exhibited creep-quake cycles. In this paper, we describe how the creep-quake collapse pattern emerges in these geomaterials using an array of experiments performed on two different calcareous sand samples. The sand samples were both loaded uniaxially and chemically degraded from the bottom, simultaneously. Additionally, we show the effects of stress and degradation rates on the behaviour of the calcareous sands, with which we discuss the creep-quake cycle mechanism and how it emerges in this material.



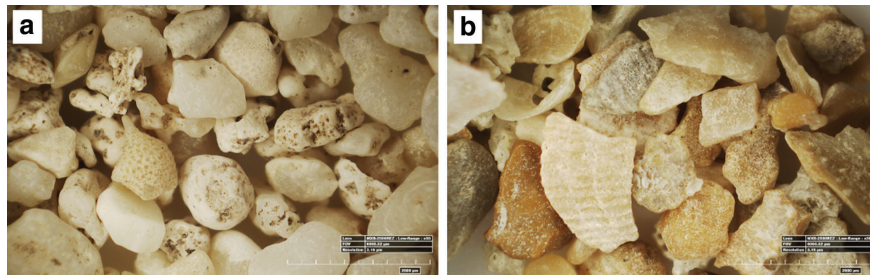
**Fig. 1.** **a** Experimental set-up. **b** Sieve analysis of two different calcareous sands. The red vertical lines show the limits at which the grain size distribution was capped to make the experimental samples

## 2 Methodology

The experimental set-up used across all the experiments is depicted in Fig. 1a. It consisted of a cylindrical sand sample with a diameter ( $D$ ) of 34.2 mm and an initial height ( $H_0$ ) of 100 mm in an acrylic tube. The samples sat between two porous plates that allowed fluid flow through them, with the bottom plate being static and the top plate acting as a loading piston connected to a Tinus Olsen H5KS loading frame. The tube wall was sealed at the bottom with an aluminium base, with an inlet tube that allowed for liquid injection.

The preparation of the samples involved initially filling the test apparatus with water, then pluviating the sand into the tube until reaching the desired initial height. This was done in order to reach a loose initial sample density. The samples were then compressed uniaxially by the loading frame at a constant strain rate of 5 mm/min until the target load was reached. At this point, the loading mode was switched to stress control, and the servo-controller maintained that target load throughout the experiment. It is noted that

due to the nature of using a servo-controller, there was a constant target stress applied on the sand samples. As will be discussed later in the paper, this resulted in drops to the stress applied whenever a quake occurred. However, the stiffness of the machine used allowed for the recovery time of those stress drops to be significantly faster than the frequency of the quake events. Therefore, its effects on our experimental results are considered to be minimal.



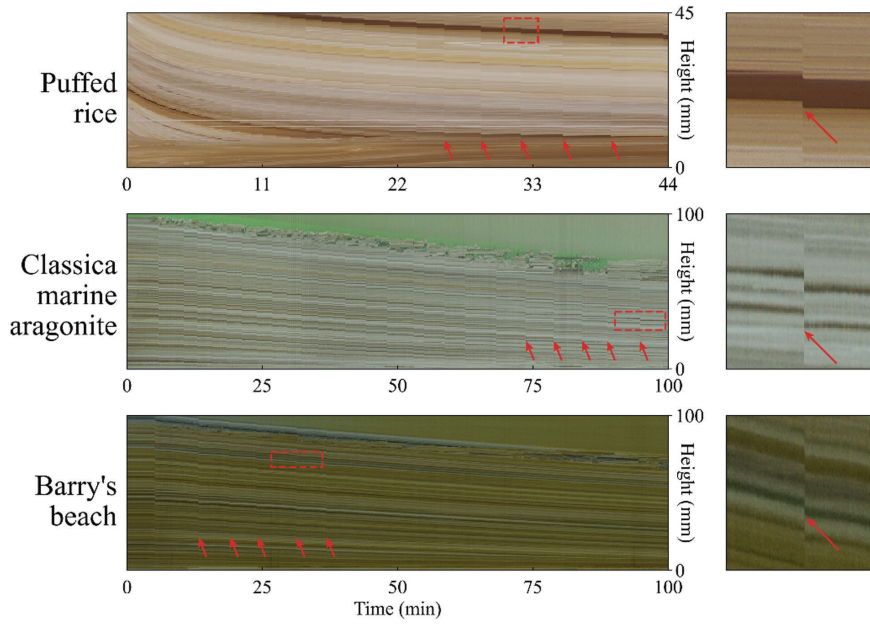
**Fig. 2.** Macroscopic photograph of the two sands **a** Classica marine aragonite **b** Barry's beach sand. The photographs were taken on sieved samples. Hence, the grain sizes are between 0.6 and 1.18 mm

Once the load was stable, the acidic solution was injected from under the bottom plate at a constant flow rate with a peristaltic pump connected to an acid reservoir. Although the injection rate of the acetic acid varied across experiments, it was kept constant for the duration of each experiment. Once the solution had travel through the height of the sample, it overflowed from the top of the tube onto a collection bucket for disposal. As the acid flowed through the samples, its hydrogen ions concentration decreased as it chemically reacted with the calcium carbonate of the sand. Consequently, the solution exiting the samples was significantly less acidic than the injected one. The macroscopic photographs in Fig. 2 were taken with a Hirox microscope, while the photographs of the experiments were acquired with a Nikon D3300 camera placed in front of the set-up. The experiment photographs were taken at a rate of one frame every five seconds and they were used to produce the sand spatiotemporal graphs shown in Fig. 3.

Two different types of calcareous sand samples were used: 'Classica marine aragonite' and 'Barry's beach' Sand. The Classica marine aragonite was purchased as an aquarium supply. This sand was an ideal material to react with acid and explore creep-quake cycles in geomaterials, since it is commonly used as a pH buffer in aquarium tanks. The sand is characterised by round coarse grains of approximately 1 mm in diameter (Fig. 1b). In contrast, the Barry's beach sand samples were obtained from south Victoria, Australia. This sand has been previously studied and its engineering properties are detailed in literature [7]. In comparison to the Classica marine aragonite, the Barry's beach sand has a larger content of fines, which is clearly visible in its sieve analysis. It is further noted that the Barry's beach sand coarse grains were significantly more angular than the typically more spherical grains of the Classica marine aragonite, as can be seen in the macroscopic photographs in Fig. 2. The two sands were stored in a cool dry place and were sieved prior to each experiment in order to remove grains coarser

than 1.18 mm or finer than 0.6 mm. The red vertical lines in Fig. 1 show the fraction of the sand samples taken to make the experimental samples.

The acid solution used to dissolve the calcareous sand samples was purchased as ‘Coles white vinegar’ and as ‘Coles double strength vinegar’ from a local supermarket. The vinegar purchased is an aqueous acetic acid solutions with a concentration of 4 and 8% acetic acid by weight, corresponding to an initial pH 2.6 and 2.4, respectively. Double strength vinegar was the used across all the experiments on the Classica marine aragonite, while single strength vinegar was used for the Barry’s beach sand experiments, as the Barry’s beach sand was found to react to the vinegar at significantly faster rates.



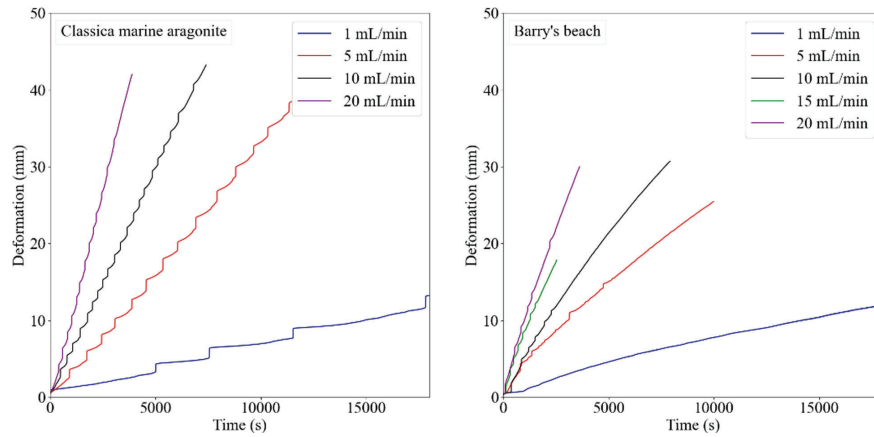
**Fig. 3.** Spatiotemporal plots for experiments on three different materials. Left column: full spatiotemporal plot, right column: zoom in of one quake event (red delimited area in spatiotemporal plots). From top to bottom: puffed rice [6], Classica marine aragonite, and Barry’s beach sand. The two sand experiments had an initial height of 100 mm, an injection flow rate of 10 mL/min, and a compressive stress of 4.35 MPa. On each spatiotemporal plot, five quake events are signaled by a red arrow to ease their observation. In these plots, quakes appear as discontinuities in the downward pixel pattern

### 3 Results

Similar to the effect of water saturation in puffed rice, we found that the chemical degradation process in the sand grains together with the axial compression may generate creep-quake cycles due to the localized degradation of the material. The similarity



between the puffed rice and the calcareous sands collapse pattern is evident in the spatiotemporal plots shown in Fig. 3. The plots were generated by taking a column of pixels in the centre of the samples from the experiment photographs and plotting it (vertical axis) over time (horizontal axis). This imaging technique allows for a static observation of the one-dimensional behaviour of the samples over time and facilitates the identification of creep-quake cycles. In these graphs, creep is seen as the gradual downward trend in the pixels, which is interrupted by a sudden larger downward displacement with every quake. The two cyclical deformation stages are further shown in Fig. 4 as deformation versus time curves. The two sand experiments in Fig. 3 had identical testing parameters, as detailed in the caption, with the exception of the concentration of acetic acid injected, as mentioned in the methodology Sect. 2. In addition to the visible similitude of the creep-quake cycles across these three materials, the quake events in the sand samples were also accompanied by drops in the servo-controlled load used to compress them, as seen in Fig. 5, and distinct acoustic emissions, two features that were highlighted in the original discovery of ricequakes [6].



**Fig. 4.** Deformation versus time curves for two sets of experiments under different acid injection rates: Classica marine aragonite (left) and Barry's beach sand (right)

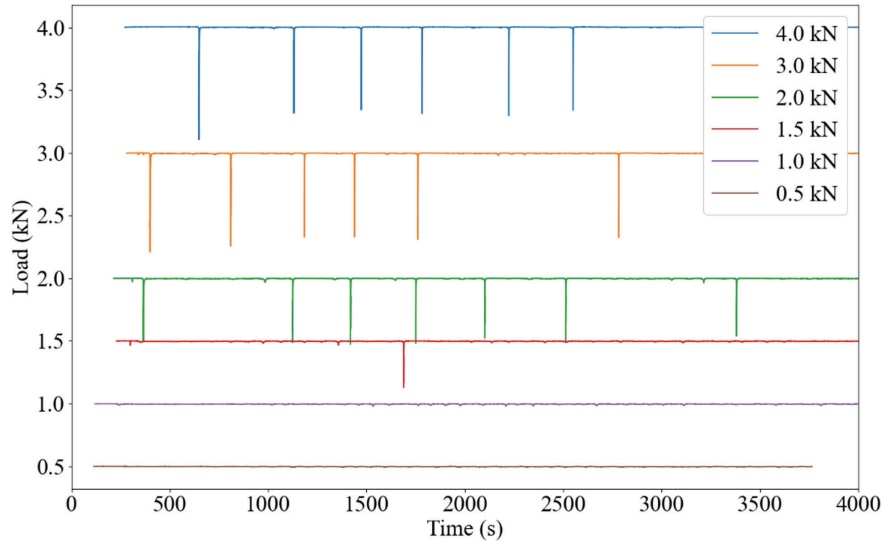
### 3.1 Injection Rate Effect

Once creep-quakes cycles were identified in the two sands, the effect of acid injection rate was explored, see Fig. 4. This was achieved with two set of identical experiments where the only parameter varied was the injection rate. The experiments in Fig. 4 consisted of a 100 mm tall sand column under an applied stress of approximately 4.35 MPa (4 kN force). As expected, a lower acid injection rate resulted in a slower rate of deformation of the sand samples. This trend also led to prolonged creep periods, separating the quake events by larger spans of time. In the case of the Classica marine aragonite, this does not prevent quakes from occurring, as they are prevalent throughout the four different experiments all the way until the experiment was stopped. However, the Barry's beach sand samples do

not exhibit creep-quake cycles at lower reaction rates, and the quakes seem to disappear early instead of being regularly present for the whole duration of the experiment. This feature is also visible in the spatiotemporal graphs in Fig. 3, as there are no more sudden displacements of pixels after approximately 80 min. Furthermore, it is observed that higher injection rates result in creep-quake cycles that are far less discernible from creep alone. This is clearly seen in the 20 mL/min injection rate experiments in both sand samples.

### 3.2 Loading Magnitude Effect

The effect of load magnitude on the development of creep-quake cycles was further studied with a set of experiments on Barry's beach sand samples. The findings of these experiments are summarised graphically in Fig. 5. These experiments were performed on 100 mm tall sand columns with a 10 mL/min acid injection rate and with different applied loads. This shows that quakes can only occur when sufficient confining load is applied. At low load magnitudes, creep-quake cycles come to a halt and degradation accelerated creep is the sole mechanism through which the sand deforms. However, there is no clear effect between load magnitude and quake frequency. A similar trend was observed in the Classica marine aragonite samples, although not presented in this figure for conciseness.



**Fig. 5.** Measured load versus time curves for a set of experiments on Barry's beach sand with varying applied target load

## 4 Discussion

In line with the effects of partial saturation in puffed rice, the acetic acid degrades the calcareous sands mostly at the bottom of the samples. As the acid travels through the

material, its concentration of hydrogen ions available to react with the calcium carbonate in the sand decays. This results in reaction rates within the material which reduce with distance from the bottom base plate. The field of degradation that the sand experiences is estimated to have significantly faster reaction rates at the bottom of the samples.

The creep periods in the samples are a result of the mass reduction caused by the acetic acid reacting with the sand grains. This reaction makes the sand grains smaller and may lead to their rearrangement, resulting in the accelerated creep deformation we observe in our experiments. Quakes, however, are the result of an instability within the granular structure. This instability arises from a localised increase in porosity and decrease in strength of the granular medium and ultimately, a quake occurs when the region can no longer withstand the compressive stress applied. When a sand grain within a highly degraded region of the sample (close to the bottom plate) breaks or dislodges into available space (pores), it may overload the granular structure, thus triggering a local collapse event (a quake). The non-homogeneity of dissolution along the loading direction is key to the development of recurring creep-quake cycles. This interpretation is supported by Fig. 4, where we see how faster injection rates result in less distinct creep-quake cycles. This is likely caused by a homogenisation of the degradation field. Faster injection rates result in a larger concentration of hydrogen ions further away from the bottom of the sample, causing a more uniform degradation of the samples in comparison to the slower injection rates. This can be seen in the similarity between the 15 and 20 mL/min injection rates experiments. At these faster rates, the sample deformation is no longer limited by the distribution of hydrogen ions within the material, but instead by the chemical kinetics of the reaction. Hence, the results of our experiments point towards two required ingredients for creep-quake cycles to emerge in sands exposed to acidic degradation. Firstly, a non-homogeneous degradation field which results in a localised zone of weakness and higher porosity due to the grains loss of mass, consequently generating quakes. Second, sufficient load is required for these cycles to occur, as demonstrated in Fig. 5. Without sufficient pressure, the breakage or rearrangement of grains within a highly degraded zone does not appear to result in sudden collapse. The lower stress allows the granular structure to rearrange and slowly re-stabilise while constantly degrading.

The difference in creep-quake cycles duration between the two sands is explained by the morphology of their grains. While the angular nature of the Barry's beach sand grains causes a faster consumption rate of the sand due to their larger surface area, it also reduces the porosity of the sand column, generating less space for grains to dislodge or break into. Therefore creep-quake cycles in Barry's beach sand come to a halt earlier than they do in Classica Marine Aragonite. When exposed to the experimental conditions for long enough, Classica Marine Aragonite also stop undergoing creep-quake cycles.

An additional distinction appears when comparing the collapse patterns of the puffed rice and the two sand samples. Ricequakes are characterised by a growth in creep duration over time [6]. Visibly (Figs. 4 and 5), this is not a characteristic of the collapse patterns of the two sand samples. We explain this difference with the accumulation of inert material at the bottom of the cereal samples and the lack of renewal of degrading agent in that case. In the puffed rice experiments, a known volume of water was injected at the start of the experiments and it was not renewed. As time progressed, the collapsed cereal mass

at the bottom of the tube increased and this widened the distance between the base of the tube and the undegraded (and therefore susceptible to collapse) grains. This results in the undegraded grains saturating at a slower rate as time progresses and causes the growing creep periods observed in [6]. In contrast, the acid solution was constantly renewed in the sand experiments and the sand grains continued to degrade even after a quake has occurred, as they continually weakened and reduced in size. This difference in how the degrading agent and the samples interact explains why growing creep periods are not observed in the sand experiments.

## 5 Conclusion

In conclusion, we have shown that the mechanism of creep-collapse cycles originally found in puffed rice [6], ‘ricequakes’, can emerge in geomaterials under acidic degradation, using two distinct calcareous sands. One-dimensional experiments were performed in which we applied compressive load and a non-uniform chemical degradation field to the sand specimens, simultaneously. We observed that changing the degrading field has a direct effect in the creep-quake behaviour of the sands, making the collapse patterns less pronounced as the high degradation front broadens. These observations ultimately point to the fact that in order for creep-quake cycles to emerge in geomaterials under chemical degradation, the materials has to experience sufficient compressive stress, as well as a non-uniform degradation field.

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