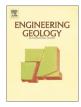
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# Effects of mineral suspension and dissolution on strength and compressibility of soft carbonate rocks



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#### ABSTRACT

Calcarenites are highly porous soft rocks formed of mainly carbonate grains bonded together by calcite bridges. The above characteristics make them prone to water-induced weathering, frequently featuring large caverns and inland natural underground cavities. This study is aimed to determine the main physical processes at the base of the short- and long-term weakening experienced by these rocks when interacting with water. We present the results of microscale experimental investigations performed on calcarenites from four different sites in Southern Italy. SEM, thin sections, X-ray CT observations and related analyses are used for both the interpretation-definition of the structure changes, and the identification-quantification of the degradation mechanisms. Two distinct types of bonding have been identified within the rock: temporary bonding (TB) and persistent bonding (PB). The diverse mechanisms linked to these two types of bonding explain both the observed fast decrease in rock strength when water fills the pores (short-term effect of water), identified with a short-term debonding (STD), and a long-term weakening of the material, when the latter is persistently kept in watersaturated conditions (long-term effect of water), identified with a long-term debonding (LTD). To highlight the micro-hydro-chemo-mechanical processes of formation and annihilation of the TB bonds and their role in the evolution of the mechanical strength of the material, mechanical tests on samples prepared by drying partially saturated calcarenite powder, or a mix of glass ballotini and calcarenite powder were conducted. The longterm debonding processes have also been investigated, using acid solutions in order to accelerate the reaction rates. This paper attempts to identify and quantify differences between the two types of bonds and the relative micro-scale debonding processes leading to the macro-scale material weakening mechanisms.

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#### 1. Introduction

Calcarenites are sedimentary soft porous rocks found in many areas around the world and common in the Mediterranean region (Apulia and Sicily in Italy, Malta, Cyprus, and Israel). Due to the highly porous carbonate structure, the exposure along coastlines, the interaction with infiltrating rainfall and/or sea water can be at the origin of various geomechanical instabilities involving carbonates. Along the Apulian coastline (Italy) large caves develop creating suggestive landscapes due to the progressive deterioration of the material induced by the combined action of waves, sea spray, and water rock infiltration. The calcarenite weathering becomes even more important when we think of these rocks as building stones used in historical buildings and monuments that undergo degradation and damage due to the centuries

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of prolonged exposure to the atmosphere. The physico-mechanical characterisation of such lithotypes has been usually carried out to verify their use as construction and ornamental stones (Andriani and Walsh, 2003, 2007a; Anania et al., 2012) or to analyse their characteristics at a local scale.

Schlumberger (2014) assesses that about 60% of all the oil and 40% of all the gas reserves are in carbonate rock reservoirs. The best examples are the Middle East fields, where 70% of oil and 90% of gas reserves are in carbonate rocks. In addition, an interest in  $CO_2$  sequestration brings in focus the issues of the enhancement of the rock dissolution in contact with  $CO_2$  (Makhnenko et al., 2014).

The microstructural changes that induce the modification of the mechanical macro-scale response and behaviour of a dry weak/soft rock when put in contact with water are distinctly different when observed immediately after the immersion and when the immersion lasts over a long time. From an engineering point of view it is noted that calcarenites (but also other rocks as chalk and loess) may lose almost instantly up to 60% of their dry uniaxial compression strength and stiffness after saturation with water (Brignoli et al, 1995; Papamichos et al, 1997; Lagioia et al, 1998; Castellanza et al, 2009; Collins and

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Sitar, 2009; Eslami et al., 2010; Ciantia et al., 2013). Short-term sequential wetting and drying cycles (a series of 5, 10 and 15 cycles) have been found to cause a significant reduction in the uniaxial compression strength (3.6, 25, 45%) compared to the dry material associated with a slight weight loss (0.12, 0.17 and 0.27%) in fine-grained calcarenites. The same effect is less marked for the coarse grained calcarenite where to a larger mass loss (1.09%) a smaller reduction of the dry uniaxial compressive strength is recorded (3.4%) (Andriani and Walsh, 2007b). As explained therein, the weight loss is certainly due to the detachment of grains that are less strongly bound to the samples and is not due to dissolution phenomena, which can occur only with difficulty in such a short time. Fig. 1a shows typical stress–strain results obtained during uniaxial stress compression tests on dry samples and on wet samples after the material becomes saturated due to a single immersion in water.

Recently, it has been suggested that over the long-term, the continuously saturated calcarenite, if the dissolved calcite is continuously washed away from the sample such for open systems, weakens due to the dissolution of grains and bonds (Castellanza, and Nova, 2004; Ghabezloo and Pouya, 2006; Ciantia and Hueckel., 2013; Ciantia et al., 2013, 2014). In the long-term chemical dissolution of calcite is the fundamental mechanism affecting the material mechanical behaviour of calcareous soft rocks. Hence, processes such as pressure solution and stress corrosion cracking become fundamental as they may lead to enhanced dissolution due to chemo-mechanical coupling mechanisms. For example, the increase of dissolution rate due to stress induced micro crack formation has been described by Ciantia and Hueckel (2013). Dissolution induced, mass loss effects on the material resistance are clearly visible in Fig. 1b which represents the results of uniaxial compression tests on calcarenites that have been subjected to acid accelerated chemical weathering (Ciantia et al., 2014). Saturation and dissolution induced strength reduction is also clearly visible in Fig. 1c where the yielding points of a Gravina calcarenite subject to different loading paths are represented in the deviatoric *q*-*p* plane. While the long-term effect has a straightforward chemo-physical explanation, which is the dissolution induced mass loss of calcium carbonate (Castellanza, 2002; Ciantia and Hueckel, 2013), the short-term interaction with water has not been systematically examined and fully understood. Possible explanations of the short-term weakening of porous calcareous rocks induced by water inundation as found in literature are:

- i) Capillary effects: a drop in suction induced by water saturation (Brignoli et al., 1995; Coop and Lee, 1995).
- ii) Rehbinder effect (Rehbinder and Lichtman, 1957): reduction of surface energy of calcite due to a polar interaction between water and grain surfaces causing a repulsive force between calcite microcrystals, and a consequent strength reduction (Malkin, 2012). The Rehbinder effect seems to support the adsorption of water weakening model proposed by Risnes et al. (2005).
- iii) Intergranular pressure solution (Rutter, 1976): is a mechanism whereby the stress concentration at grain contacts causes local dissolution, transport of the solutes out of the contact and precipitation on the less stressed faces of the grains. Although it is often invoked (Renard et al., 2005; Le Guen et al., 2007) to explain the long-term deformation and failure of porous rocks in the presence of water, a very fast dissolution ( $\approx 8$  h) of grain contacts may occur even in the short term, as noticed by Pietruszczak et al. (2006) for chalk.
- iv) Subcritical cracking: even though it is very often considered that this weakening effect of water is more significant in the long term, it has sometimes been invoked to explain the short-term brittle behaviour of silicate rocks (e.g. Jeong et al., 2007). In the case of carbonate rocks, the nature of stress corrosion reactions is not well understood, as noticed by Atkinson (1984).

In this paper a new mechanism, consistent with all the above hydrochemo microscale mechanisms, explaining the short-term weakening of different soft calcareous rocks is proposed. The paper is structured in the following manner: in Section 2 the description of the tested lithologies and their physical mechanical properties are presented. In Section 3 a complete set of micro-investigations by means of X-ray Micro-Computer-Tomography (MCT), Scanning Electron Microscope (SEM) and Mercury Intrusion Porosimetry (MIP) performed to define

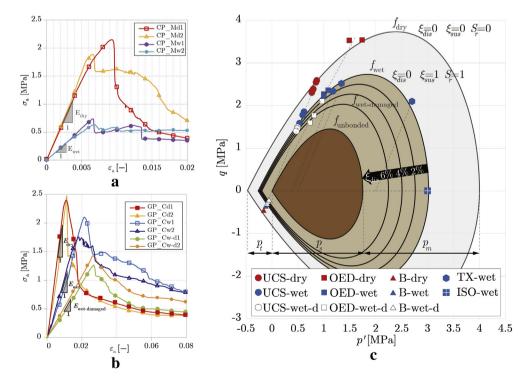


Fig. 1. UCT (Uniaxial Compression Test) stress vs. strain curves of calcarenites from (a) Canosa di Puglia (CP\_M) in dry (d) and wet (w) conditions, and (b) Gravina di Puglia (GP\_C) in dry (d), wet (w) and wet-acid-damaged (w-d) conditions. The w-d samples were submerged in a weak acetic-acid solution for about 60 min (see Ciantia et al, 2014). c) Effect of saturation and of dissolution on the size of the yield locus for a Gravina calcarenite (GP\_C).

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