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ABSTRACT

This study investigates the mechanical behavior of biogenic carbonate sands from Puerto Rico at grain-scale level. Micro-computed tomography has also been used to get insights on the internal structure of these particles before and after loading. The crushing strength of these particles are smaller comparing to the values reported for silica sands. It has also been shown that these particles have complex internal structure including a network of pores connected with channels. This study also demonstrates the effect of intragrain structure of biogenic carbonate sands and shows how internal grain structure plays a role on particle fracture.

INTRODUCTION

Carbonate sands can be found in offshore and tropical environments. They account for about 40% of ocean floors and are globally distributed, including along the coasts of Australia, Persian Gulf, Gulf of Mexico, South of China, and Ireland (Airey et al. 1988, Coop 1988, Hassanlourad et al. 2008, Salem et al. 2013, Xiao et al. 2017). Across the United States, they can be found in Hawaii, coasts of Florida and Puerto Rico (Sandoval and Pando 2012, Morales-velez et al. 2015). Unlike silica sands, that are alluvial sediments made of quartz, carbonate sands are formed as a result of the deposition of skeletal remains of marine organisms (biogenic) or by the chemical precipitation of calcium carbonate in a calcium rich environment (non-biogenic) and contain high amount of calcium carbonate (Fookes 1988).

Differences have been reported in the macro-scale behavior of carbonate and silica sands including their compression behavior, shear behavior and dynamic behavior (Poulos et al. 1982, Golightly and Hyde 1988, Coop 1990, Hyodo et al. 1996, Kwag et al. 1999, McDowell and Harireche 2002, Porcino et al. 2008, Brandes 2011, Beemer et al. 2019a, Saeidaskari et al. 2020, Rasouli et al. 2021). This can be attributed to the unique characteristics of the carbonate sand particles as the result of their deposition environment. While silica sand grains are bulky, round and practically solid with miniscule amount of pores, carbonate sand particles are usually elongated and angular with high internal porosity (Cil and Alshibli 2012, Al Mahbub and Haque 2016, Ma et al. 2019, Li et al. 2020, Mohtashami et al. 2022).

Although more studies have become available recently considering the macro-scale behavior of carbonate sands, a few studies have been focused on the mechanical behavior of carbonate sands at grain-scale level (Orense et al. 2013, Beemer et al. 2019b, Ma et al. 2019, Zhang et al. 2019, Li et al. 2020, Lv et al. 2020, Xuehui et al. 2020, Kuang et al. 2021, He et al. 2022). There is still a need for understanding the behavior of carbonate sands particularly considering the internal structure of these particles.

With the development of the X-ray micro-computed tomography (micro-CT) technique, insights are provided into the 3D internal structure of materials. Particularly, in the field of geotechnical engineering, this technique has become a popular tool in the studies at grain-scale levels. X-ray CT is a non-destructive imaging technique that produces high resolution 3D image by measuring the level of attenuation of X-ray intensity, for a material bombarded with X-rays is measured. The final product is a series of gray-scale slice images. The level of the X-rays attenuation is a function of the specimen chemical composition, its geometry, and the energy level of the X-ray source. This technique requires minimal sample preparation and has relatively quick scanning time (Druckrey and Alshibli 2016, Zhao et al. 2020).

This study took an experimental approach to investigate the mechanical behavior of carbonate sand particles using uniaxial compression tests and the internal structure of the particles using micro-computed tomography. The effect of the internal structure of the grains of the crack initiation and propagation mechanism also has been qualitatively analyzed.

PARTICLE CRUSHING STRENGTH

Uniaxial compression tests are used to study the mechanical behavior of these particles. In this test, the particle is compressed between two rigid flat plates. Crushing strength of the particles depends on the failure force and the initial dimension of the particle along loading. The method proposed by Oka and Hiramatsu is employed in this study to find out the crushing strength of each particle (Hiramatsu and Oka 1966).

$$\sigma = 0.9 \frac{F_f}{d_o^2} \tag{1}$$

The strength of some manmade materials like steel is constant because they have uniform structures that is not dominated by the presence of flaws. However, natural materials like soil have structures dominated by flaws. Therefore, instead of a single number as the strength value, a statistical distribution called Weibull distribution has been widely used to represent the statistical variability of the particle strength resulting from the presence of incipient flaws within the particles (Weibull 1939, 1951, Lobo-Guerrero and Vallejo 2006). The probability of survival (P_s) of a brittle material with volume (V_o) under stress (σ) can be described by the two-parameter Weibull distribution function shown in Equation (2).

$$P_s(V_o, \sigma) = exp\left[-\left(\frac{\sigma}{\sigma_o}\right)^m\right] \tag{2}$$

Using the uniaxial compression tests, the probability of survival at crushing stress σ is calculated in Equation (3) as:

$$P_s(V_o, \sigma) = \frac{number\ of\ particles\ crushed\ at\ (S>\sigma)}{total\ number\ of\ tested\ particles} \tag{3}$$

Taking two times the logarithm from Equation (2) results in a linear relationship between the parameters which can be derived from the uniaxial compression tests. A least square linear regression can be conducted to determine the Weibull parameters (σ_o, m), described by Equation (4).

$$\ln[\ln\left(\frac{1}{P_s(V_o,\sigma)}\right)] = m ln\sigma - m ln\sigma_o \quad (4)$$

In this expression σ_o is the scale parameter, called characteristics stress, as an indication of the mean crushing strength of the particles and m is the shape parameter, called Weibull modulus, as an indication of the variability of the particle crushing strength where a higher modulus means less variability in the crushing strength (Weibull 1951, Bolton and McDowell 1997).

MATERIAL AND EXPERIMENTAL SETUP

Materials used in this study include bryozoan particles which are one of the main constituent particles of a batch of carbonate sands from Puerto Rico. These particles are slender tubular fragments of bryozoan skeletal remains (particle size 1-2 mm) that are shown in Figure 1. SEM and XRD analysis on the particles revealed that these particles are mainly made of aragonite (>98%) which have needle-like crystals as one of the polymorphs of calcium carbonates.

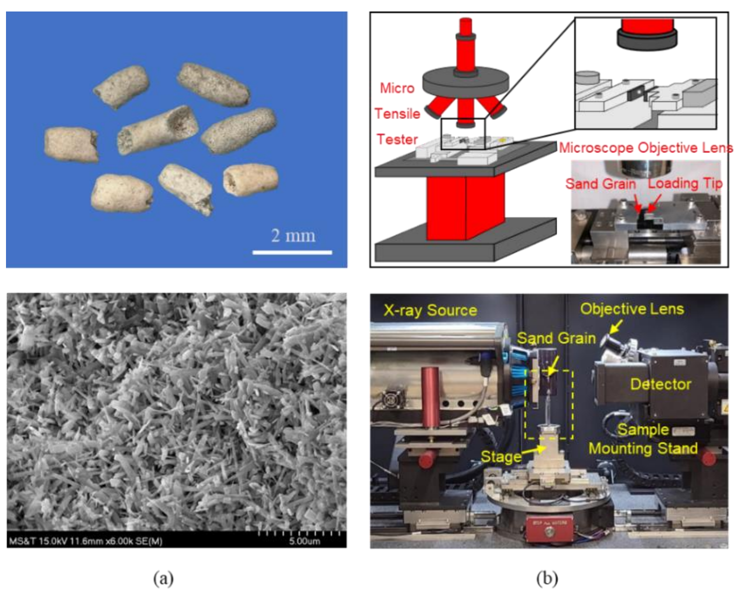

Figure 1. optical image of typical bryozoan particles used in this study (a-top), SEM image of bryozoan showing aragonite crystals (a-bottom), experimental setup for uniaxial compression test (b-top), Micro-computed tomography scanning setup (b-bottom)

37 uniaxial compression tests have been conducted on the bryozoans by a micro tensile tester device from Deben, UK. The device was used in compression mode and the uniaxial compression tests were conducted under a Hirox KH-8700 optical microscope to record the deformation of particle during compression. The experimental setup can be seen in Figure 1(b).

Displacement-controlled compression tests were conducted at a constant displacement rate of 0.1 mm/min until cracks fully developed and the particle broke and was not able to sustain any load. Failure force for each grain is obtained from the load-displacement curve as the peak force and the initial particle size between the loading plates is measured using Fiji/Image J software (Schindelin et al. 2012) from the optical images.

To investigate the internal structure, particles were scanned by Zeiss Xradia 510 Versa at 50kV, 4W, with no filter, using 4X objective and 1601 projections with 360 degrees of rotation. The scans have been processed in Dragonfly software 2020.1 (Object Research Systems, Montreal, Canada).

To quantify the internal structure, image processing techniques have been applied on the acquired micro-CT scans. Image processing techniques can be found in several studies (Kikkawa et al. 2013, Kong and Fonseca 2018, Beemer et al. 2022). The gray-scale CT scans have been segmented into solid and pore phase by using Otsu's method. This method is a global intensity thresholding method applicable to images with bimodal histograms. In this technique, the threshold is determined by maximizing the intensity variance of inter-classes which correspond to minimizing the intra-class intensity variance (Otsu 1979).

After, segmenting the scans, the internal porosity of the particle is measured by dividing the volume of pore pixels to the total volume of particle pixels. The full statistical analysis of the pore structure including several pore descriptors such as pore size, pore orientation and pore connectivity can be done by pore network modeling which is not the focus here. More details on the method and its applications on carbonate sand particles can be found in (Mohtashami et al. 2022).

RESULTS AND DISCUSSION

The internal structure of bryozoans has been extracted from the micro-CT scans. The scans revealed that the internal structure of bryozoans is mainly include large pores that have been connected through large and long channels that goes through the whole particle. Therefore, there is a network of pores in the bryozoans than isolated pores which make their fracture mechanism complicated. The scams have been segmented into pore and solid phase and porosity is measured as 33%.

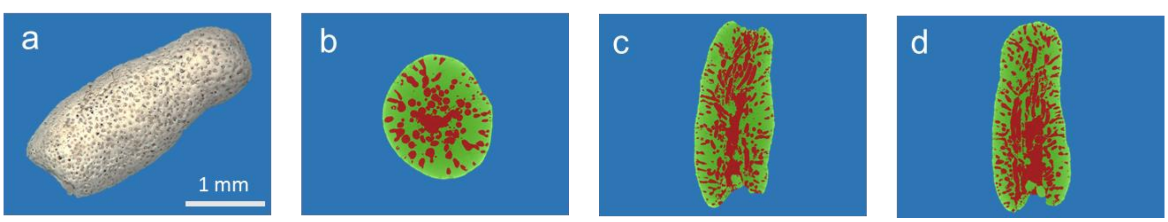

Figure 2. Bryozoan particle along with segmented micro-CT scans in three perpendicular planes; (b) Z plane, (c) Y plane, (d) X plane, red color represents pore phase and green color represents solid phase

From the load-displacement curves derived from the uniaxial compression tests, it showed that most bryozoan particles (34 out of 37) go through a progressive fracture with only 3 grains show sudden load drops with no more ability to sustain loads. While, silica sand grains have been reported to show brittle fracture, bryozoan particles showed more of a ductile behavior.

Also, for these particles fracture and formation of cracks can be followed easily under the microscope. Among 34 with the progressive fracture, 13 particles gain strength upon loading. This can be attributed to the pore collapse and deterioration that can occur inside the particle that fills up other pores as the result of loading leaving a solid matrix of material. The load-displacement corresponding to the mentioned behavior are shown in Figure 3.

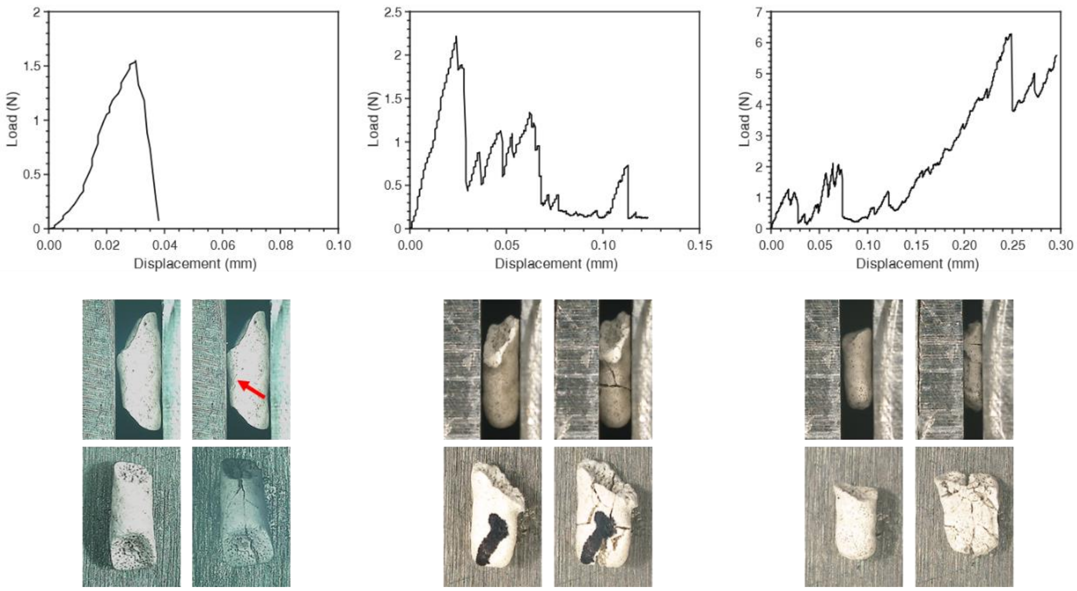

Figure 3. Three types of load-displacement curves observed for bryozoan particles

Weibull analysis has been performed on the crushing strength data and Weibull modulus and characteristics stress are derived as 2.18 and 2.95 MPa respectively. The result of fitting Weibull distribution to the crushing strength data are shown in Figure 4. The Weibull modulus for silica sand has been reported to be around 3 in the literature. The lower Weibull modulus of bryozoans can be attributed to the internal structure of these particles.

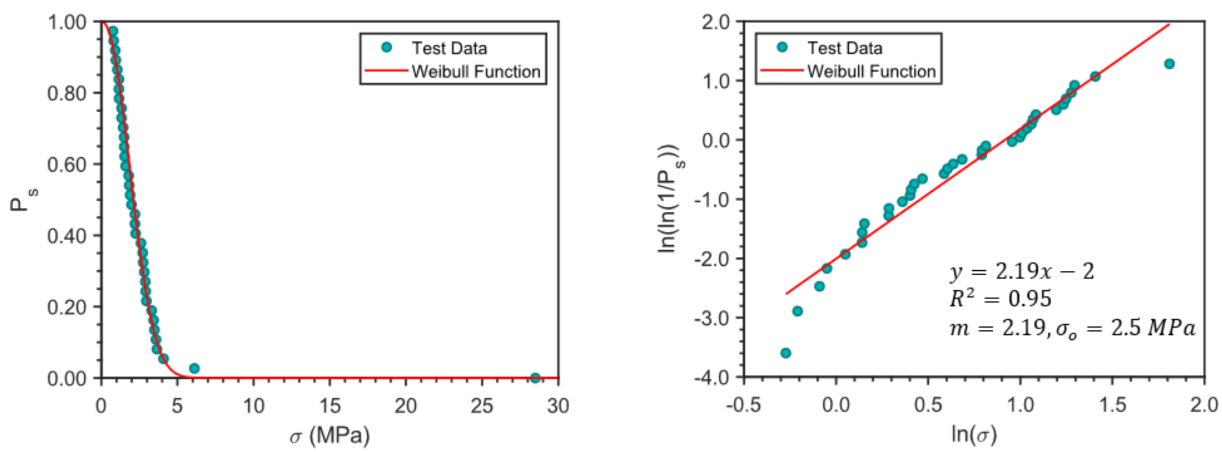

Figure 4. Probability of survival of the particles measured in uniaxial compression test data and the fitted Weibull

Bryozoans have a small characteristics strength comparing the reported values for carbonate sands. This can be attributed to the high porosity of these particles and the interconnected pores and pore channels which contributed to the easy crack propagation through the material. Some of the reported values in the literature are summarized in Table 1.

Table 1. Summary of Weibull parameters for different selected silica and carbonate sands

Material	Particle Size (mm)	Particle Shape	Weibull Modulus	Characteristic Strength (MPa)
Silica Sand (Nakata et al. 2001)	0.25-0.3 0.6-0.7 1.4-1.7	Angular	1.82 2.17 3.04	110.87 72.18 30.96
Ottawa Silica Sand (Cil and Alshibli 2012)	0.599-0.853	Rounded	3.26	137
Carbonate Sand, Australia (Beemer et al 2019)	1		3.58 5.11	1.76 26.57
Coral Sand, Philippines (Xuehui et al. 2020)	5-8.45 8.45-10.50 10.50-14.50	Relatively rounded	1.79 2.27 2.00	6.54 6.26 5.12
Carbonate Sand, South of China (He et al. 2022)	1-2 2-5 5-10		1.66 1.96 3.29	23.62 8.40 3.49
Carbonate Sand, Playa Santa, Puerto Rico (This study)	1-2	Tubular	2.18	2.95

Based on our observations on the particle after loading under optical microscope, there were main cracks either along the loading direction or along the longer direction of the particle. There were other branched cracks that were propagated along the pores. A few of the particles are shown in Figure 3. Fracture surfaces of the broken pieces were investigated using a scanning electron microscope as shown in Figure 5. (d). It was observed that the fracture surfaces cut through the intragrain pore network following zones of weakness within the grain.

To investigate the crack initiation and propagation in bryozoan particles, micro-CT scans of a loaded particle have been provided in Figure 5 (a-c). For this grain, the loading was stopped at the first load drop and the grain was scanned then to investigate the generation of the crack and its geometry. The 3D reconstruction of the loaded grain using micro-CT scans along with the optical images captured during loading showed that in this particle, in contrast to what was expected, the first crack was not initiated at the points of contacts between loading plates and the particle. It was generated inside the particle presumably at the internal pores as points of stress concentrations. Upon loading, the crack propagated along the loading tips up to a point (O) and propagated as the second crack along the loading direction from that point Figure 5 (e-g). The crack path followed the internal pores. While further studies are needed to conclusively investigate the effect of the complex internal structure of carbonate sands on their breakage behavior, our results provide evidence on contributing role of the internal structure on the fracture behavior of the particles including the crack initiation and propagation mechanism.

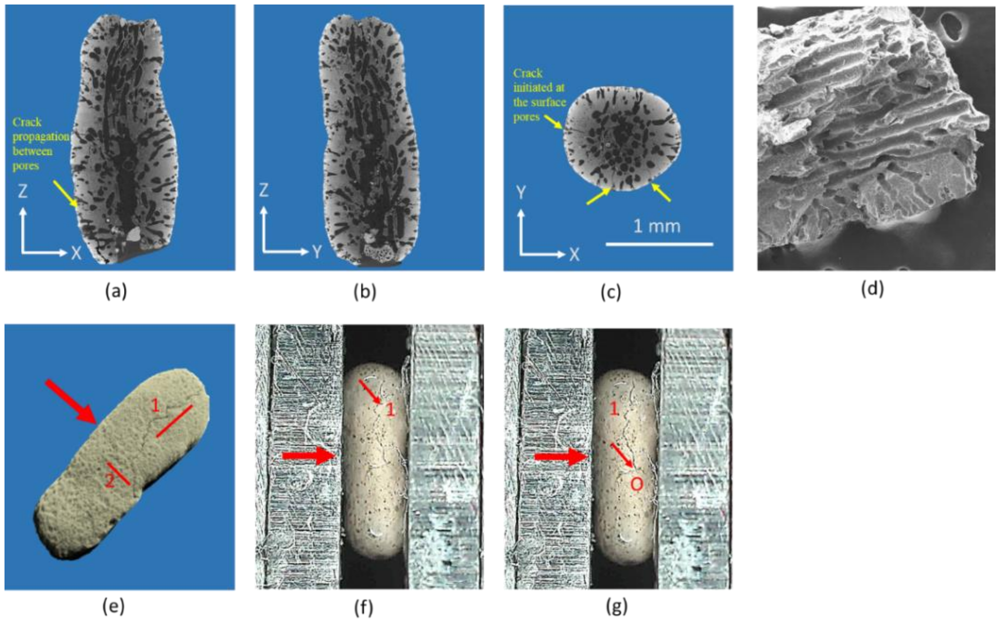

Figure 5. micro-CT scans of a bryozoan particle after loading (top row), load was applied perpendicular to ZY plane, loading direction is shown by red arrow

CONCLUSIONS

In this study we took an experimental approach to investigate the crushing strength and breakage behavior of biogenic carbonate sand particles from Puerto Rico. Uniaxial compression tests and micro computed tomography are employed to analyze the breakage behavior and internal structure of the particles. The concluding remarks of this paper can be summarized as below:

- The internal structure of bryozoans includes considerable number of pores. Porosity for a bryozoan particle is calculated as 33%. The pore structure of these grains includes a network of inter-connected pores which facilitates the crack propagation inside these particles
- The crushing strength of bryozoan particles follows Weibull distribution. The characteristic strength for these particles is measured as 2.5 MPa which is considerably lower than the values reported for silica sands. This value is considered small as well comparing the similar value reported for other carbonate sand particles. Furthermore, Weibull modulus of carbonate sand is lower indicating a higher variability of its crushing strength. This can be attributed to the complex internal structure of carbonate sand
- This study made an effort in understanding the contributing role of the internal structure of carbonate sands on their fracture behavior. Based on the acquired results, internal pores can be considered as the points of stress concentration where cracks initiated.
- The results emphasize on the important role of the internal structure of carbonate sands on their breakage behavior and the internal structure should be considered as one of the influential factors in the models and design schemes. The data also adds to the current database of the studies investigating the mechanical behavior of carbonate sands at grain-scale level.

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