Study on the unsaturated grinding of quartz sand

Mathis Thouret[1,*], *Claire* Mayer-Laigle[1,**], *Vincent* Richefeu[2], *Komlanvi* Lampoh[1], and *Jean-Yves* Delenne[1]

[1]IATE, Univ. Montpellier, INRAE, Institut Agro, Montpellier, France
[2]Univ. Grenoble Alpes, 3SR, F-38000 Grenoble, France

Abstract. While dry and saturated milling processes have been extensively studied for their applications in the food, pharmaceutical, and mining industries, the comminution of unsaturated granular materials remains poorly understood and requires further research. This short paper investigates the comminution of unsaturated *versus* dry quartz sand as a model medium by analyzing particle size distributions. Deconvoluting these distributions allows us to track both fine and coarse particles, thus improving our understanding of how water influences their grinding processes.

1 Introduction

Milling is one of the most important processeses in the industrial sector. In 2021, Weir Group noted that 1% of the world's electricity is used only for grinding in the mining industry [1]. Although the process of breaking down wet and dry granular materials, known as comminution, is commonly and widely used in industries such as food and pharmaceuticals [2, 3], the comminution of unsaturated granular materials is less well understood. In the unsaturated state, water forms bonds between the particles and can affect how the material behaves and how energy is transferred during milling.

This paper investigates how water content affects the comminution of quartz sand. Quartz sand was selected because it neither absorbs nor adsorbs water, which means that water only interacts through capillary forces. Thus, any observed effects during milling are only due to the bonding between water and particles. To understand this unsaturated grinding process, we compared it with dry milling to assess the impact of water on grinding efficiency. This comparison involved the deconvolution of the particle size distributions (PSD) using the beta law [4], separating the distribution into fine and coarse particles. This approach allows us to monitor changes in the PSD for each particle type during comminution. Through this analysis, we explain how the water content influences the grinding of different populations of particles and the unique mechanisms involved.

2 Feedstock water content

For the study, we used a precalibrated quartz sand with a size range of 400 to 800 microns, sourced from Hofer Chemie GmbH®, DE. To select a range of water content for the study, we set the maximum water content to the level that fully saturates the sample, assuming that the volume of quartz remains constant.

An apparent volume V of tapped quartz sand can be separated into the volume V_s of the sand grains and the volume V_v of the surrounding pores (voids):

$$V = V_s + V_v \longrightarrow \frac{m_s}{\rho} = \frac{m_s}{\rho^*} + V_v \tag{1}$$

where ρ and ρ^* are the apparent density of the tapped sand and the quartz-grain density, respectively. This tapped density, *i.e.*, compacted with 2500 taps (after which the volume no longer changes significantly), enables us to establish a reference volume, V_v, for the voids in the sand:

$$V_v = \frac{m_s}{\rho} - \frac{m_s}{\rho^*} = \frac{m_s(\rho^* - \rho)}{\rho\rho^*} \tag{2}$$

As a result, by calculating the void volume of the raw sand, the maximum water content corresponding to full saturation can be determined: $V_v/V_w = 1$, where V_w represents the reference maximum volume of water for the raw sand. The tapped density was measured using an Autotap densimeter (Quantachrome®, USA), yielding a value of $\rho \approx 1612\,\mathrm{kg/m^3}$. The quartz density was measured with a nitrogen pycnometer, AccuPyc II (Micromeritics®, USA), giving a value of $\rho \approx 2650\,\mathrm{kg/m^3}$. Based on the definition of water content, $W = m_\ell/m_s$, where m_ℓ is the mass of the liquid, a *relative water content* is defined as:

$$W_a = \frac{W}{W_{\max}} \tag{3}$$

where W_{max} was determined to be 26%. It is important to note that the parameter W_a does not represent the water content as defined in soil mechanics (a mass ratio). In fact, since the sample volume and particle size change during the grinding process, the classical degree of saturation (S_r) also varies in contrast to W_a, which remains constant as it does not depend on the sample volume. This water

*e-mail: mathis.thouret@inrae.fr
**e-mail: claire.mayer@inrae.fr

content (W_a) reflects a saturation ratio, which refers to the saturation the material would exhibit in a conventionally dense state (before grinding), while avoiding confusion with the saturation degree, which is more appropriated for quasistatic experiments.

3 Comminution process and PSD deconvolution

Quartz sand was milled using a vibratory ball mill (MM500, Retsch®, DE) with two grinding chambers. A total of 13.7 g dry sand was placed in each chamber. The required amount of water was added and mixed with the sample to achieve the desired W_a. Short grinding times were used for each sample and water content, specifically 0.5 min, 1 min, 2 min, 3 min and 5 min, as the fastest particle size reduction occurs at the beginning of the grinding process. After each grinding period, the particle size distributions were determined using a laser diffraction analyzer (LS 13320 XR, Beckman and Coulter®). To perform particle size measurements, the grains are suspended in water. The use of an ultrasound probe helps disperse the particles and ensures that the measured sizes correspond to individual particles, not agglomerates. This process, known as deagglomeration, uses ultrasonic cavitation to break down the agglomerates into individual particles, ensuring accurate particle size measurement.

The approach used in this study is based on the PSD deconvolution technique outlined in [4], which is used to analyze comminution processes [5]. This technique decomposes PSDs using Beta distributions. Given that PSDs often exhibit asymmetry and contain multiple peaks, each peak can be described by its own PSD, representing a distinct particle population.

The distribution of each peak of a PSD is defined by discrete classes of particle sizes s as follows:

$$D_k(s \mid \chi_k) = \begin{cases} w_k \dfrac{(s_k)^{\alpha_k - 1}(s_k - 1)^{\beta_k}}{B(\alpha_k,\ \beta_k)} & \text{if } s \in [a_k,\ b_k] \\ 0 & \text{otherwise} \end{cases} \tag{4}$$

where the reduced particle size of the particle population k is given by $s_k = (s - a_k)/(b_k - a_k)$. The parameter set χ_k consists of $\{\alpha_k,\ \beta_k,\ a_k,\ b_k,\ w_k\}$, where α_k and β_k are the shape parameters of the Beta distribution $B(\alpha_k,\ \beta_k)$, a_k and b_k define the range limits of the distribution, and w_k represents the maximum value.

The complete PSD is the sum of all population distributions:

$$D(s \mid \chi) = \sum_{k=1}^{N} D_k(s \mid \chi_k) \quad \text{where} \quad \chi = \cup_{k=1}^{N} \chi_k \tag{5}$$

where N is the number of populations in the PSD. All PSD deconvolutions were performed using a C++ code developed in-house. This code allows us to obtain the set of parameters χ_k for each distribution k, starting from the raw PSD data (volume fraction as a function of particle size classes).

The deconvolution algorithm involves optimizing the parameters χ to minimize the following error:

$$E(\chi) = \sum_{s_i} \left[P(s_i) - D(s_i \mid \chi) \right]^2 \tag{6}$$

The minimization of the least squared error is carried out using the library `cminpack` [6]. Each PSD curve is processed in its historical sequence, with the initial parameters for the current curve being the optimized parameters from the previous curve. However, due to the insufficient frequency of the PSD constructions, the initial parameters were manually adjusted for each optimization.

4 Results and analysis

Figure 1 illustrates the evolution of PSD during the grinding of dry sand and unsaturated sand ($W_a = 20\%$). These

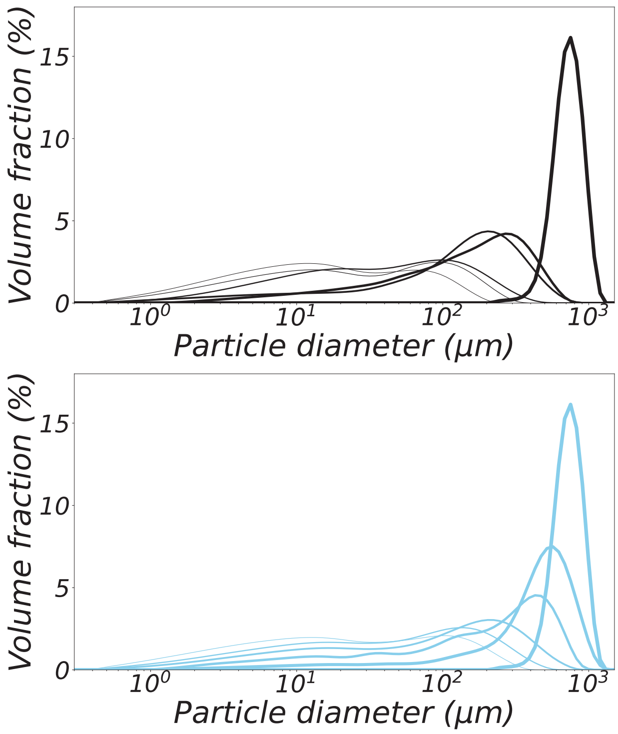

Figure 1: Evolution of PSD for dry (top) and unsaturated (bottom) sand ($W_a = 20\%$). The PSD measurements are conducted at 0 min, 0.5 min, 1 min, 2 min, 3 min and 5 min (decreasing order of line thickness).

plots highlight a difference in grinding efficiency between the two experimental conditions. For dry sand, there is a noticeable shift in the PSD. For unsaturated sand, the evolution is distinct; while there is a slight increase in fine particles over time, the milling process does not cause any shift in the PSD. Based on these measurements and the deconvolution of beta laws, the populations of fine and coarse particles were analyzed throughout the grinding process for each W_a. We consistently consider a maximum of two populations ($N = 2$), referred to as "k = fine" and "k = coarse" particles. Those populations correspond respectively to the two peaks observed in each distribution for all grinding times. These two appear to be undergoing simultaneous comminution. Based on this observation, a deconvolution was performed to track their evolution over time.

The modes of the PSDs, which correspond to the particle diameters at the deconvoluted peaks, were monitored for each grinding time and each W_a in order to compare dry and wet grinding. For a given distribution of the population k, the mode m_k is determined by setting the derivative of Eq. 4 to zero:

$$m_k = a_k + (b_k - a_k)s_k^0 \quad \text{where} \quad s_k^0 = \frac{\alpha_k - 1}{\alpha_k + \beta_k - 2} \tag{7}$$

Figures 2 and 3 illustrate the evolution of the modes (peak diameters), distinguishing between fine and coarse sizes, respectively. Each figure first presents the mode by fixing W_a and varying the grinding time, followed by its representation as a function of W_a for different grinding times.

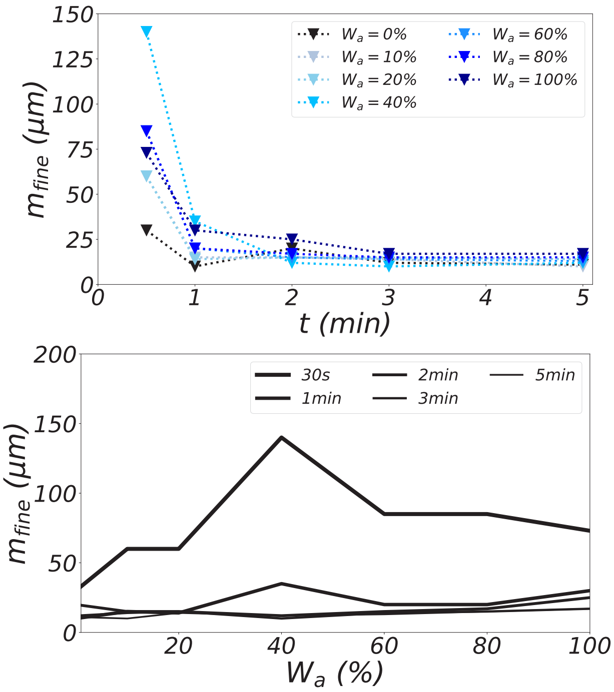

Figure 2: Evolution of m_{fine} over grinding time for various values of W_a, followed by its variation as a function of W_a for different grinding times.

These figures highlight the significant difference in the grinding behavior of the finer and coarser particle populations in the presence of water. For fine particles, variations in m_{fine} between different water contents are noticeable only during the first minute of grinding. Beyond this point, no significant differences are observed and the values of m_{fine} become nearly identical in all water contents (Figure 2). In contrast, for the coarser particle population (Figure 3), a more distinct difference emerges. The presence of water within the granular medium forms capillary bonds between the particles, influencing force transmission during the grinding process. This effect may be

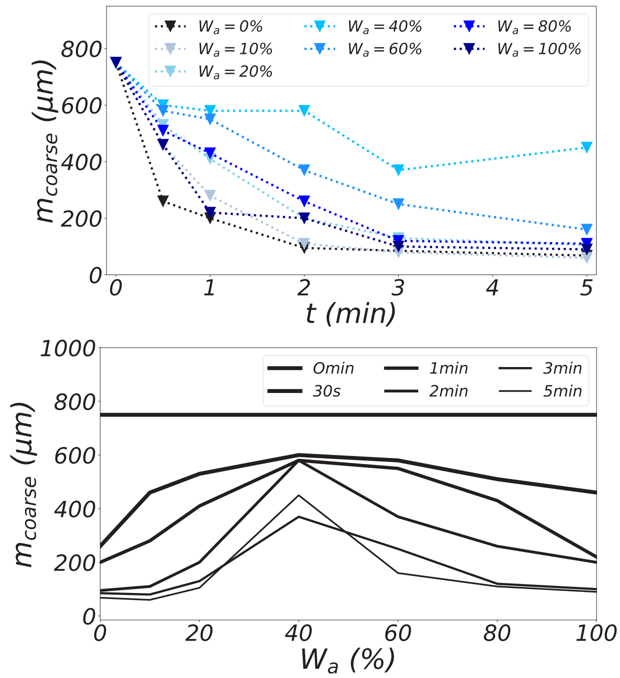

Figure 3: Evolution of m_{coarse} over grinding time for various values of W_a, followed by its variation as a function of W_a for different grinding times.

attributed to the formation of aggregates, though these aggregates were broken during the measurement of grading size curves.

These water-particle interactions appear to have a greater impact on coarser populations. A closer examination of the plots as a function of W_a reveals that the grinding process varies unevenly with W_a. Specifically, while grinding is highly effective in the dry case, its efficiency gradually decreases until $W_a \approx 40\%$. Beyond this specific water content, m_{coarse} decreases again, illustrating a recovery in grinding effectiveness up to full saturation ($W_a = 100\%$), where a size reduction comparable to that observed in the dry case is achieved. This critical value of water content appears to correspond to the maximum cohesion state for sand (see, *e.g.*, [7]). Consequently, incorporating cohesion indicators into the analysis could provide deeper insight into grinding mechanisms in unsaturated granular media.

Figure 4 illustrates the evolution of the ratio $m_{\text{fine}}/m_{\text{coarse}}$ for different grinding times and values of W_a. This representation highlights that two distinct grinding regimes can be identified. During the first minute of grinding, a significant decrease in m_{fine} is observed, leading to a reduction in the ratio, which defines regime I. Beyond this initial phase, the ratio begins to increase, as the amount of fine particles reaches a plateau (see Figure 2), while the coarser particles, which are more influenced by the presence of water, start to break down – this marks the transition to regime II. Finally, although water does not appear to have a significant impact on the comminution of fine particles, some noteworthy observations can be made. Figure 5 illustrates the evolution of the peak amplitude corresponding to m_{fine} as a function of different W_a values for the last two grind-

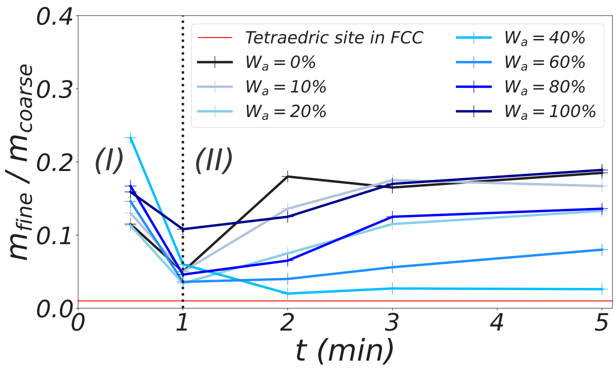

Figure 4: Evolution of the ratio $m_{\text{fine}}/m_{\text{coarse}}$ over time for different values of W_a.

ing durations. Even though m_{fine} appears to remain constant after 3 minutes of grinding, the representation of the volume fraction emphasizes that grinding is still occurring. Indeed, for each value of relative water content, the volume fraction increases over time. Furthermore, it is observed that the volume fraction decreases from $W_a = 0\%$ to approximately $W_a = 40\%$ before increasing again up to $W_a = 100\%$. This observation leads to the same conclusion as for coarse particles, while also highlighting that the volume fraction serves as a reliable indicator for assessing grinding differences between various W_a values when m_{fine} no longer exhibits significant variation.

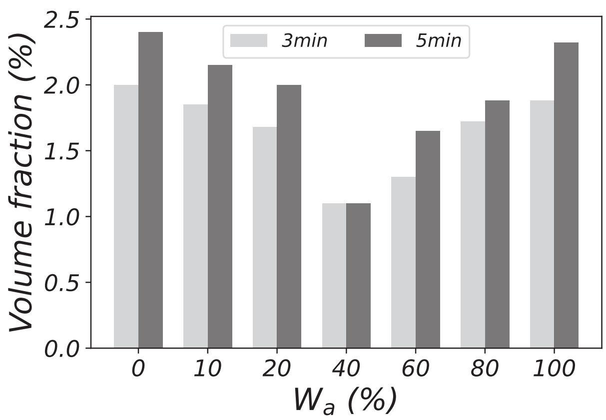

Figure 5: Evolution of the amplitude of the peak corresponding to m_{fine} as a function of different relative water content W_a for the last two grinding times.

5 Conclusion and perspectives

This study demonstrates that water can influence the comminution process of quartz because of the presence of capillary bonds between the particles. These bonds alter the transmission of energy and forces during milling, leading to distinct grinding behaviors for fine and coarse particles. Through deconvolution of the particle size distributions, we identified that water primarily affects the grinding of coarser particles. The results reveal that the grinding efficiency is highly dependent on the water content, with a noticeable decrease in the efficiency at the intermediate water content $W_a \approx 40\%$, followed by recovery under fully saturated conditions. This behavior is likely associated with the formation of aggregates and the resulting change in particle cohesion.

To better understand the role of water in comminution, future studies should focus on the relationship between the cohesion of granular media and the grinding efficiency. Specifically, it would be valuable to explore how capillary bonds between particles influence grinding energy and the creation of a specific surface area. In addition, incorporating cohesion indicators in the analysis of unsaturated grinding processes could provide a deeper insight into the mechanisms at play. By developing a more comprehensive model linking grinding energy and particle cohesion, it may be possible to optimize the processes for unsaturated granular materials, with implications for industries such as food, pharmaceuticals, and mining.

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