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Segregation of sand-rubber chips mixtures subject to vertical tapping under confinement

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Abstract

Mixtures of rigid sand particles and soft rubber particles (rubber chips) are prepared and submitted to vertical taps in a confined cell to investigate their propensity to segregate. The mixture evolution is characterized by means of image analysis of slices obtained by a gelification technique. We show that, in case of equally sized particles, the rubber particles tend to migrate towards the bottom of the system. Yet, the segregation is not complete and is reduced when the rubber fraction is increased. Also it competes with the size induced segregation if rubber chips are larger than sand particles. A tendency to form horizontal clusters is clearly observed and increases with the number of taps. This horizontal segregation is reduced if the rubber fraction is smaller and is weakly influenced by grain size difference. Both the vertical segregation and the formation of horizontal heterogeneities are prevented by adding a small amount of water.

Keywords:

sand-rubber mixtures, segregation, tapping, image analysis

1. Introduction

The number of scrap tires disposed in huge piles across the world continuously increases each year. These dumps are not innocuous since they are considered as (i) serious fire hazards, (ii) breeding grounds for pests, and (iii) obviously unpleasant sights. Used rubber tires do not easily decompose, thus it is necessary to find alternative for their disposal that are both environmentally relevant and economically feasible. Among the current applications for used tires, one can mention energy generation, tire retreading, highway crash barriers, breakwaters... Despite these recycling solutions, the amount of tires in landfills still has an increasing trend every year. Thus, a need still exists for new recycling solutions. Several possibilities consist of using shredded tires (usually referred as “tire chips”) mixed with sand, as alternative soils

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which can be used in various geotechnical applications such as backfilling for retaining structures, slope and highway embankment stabilization, road constructions, soil erosion prevention and seismic isolation of foundations (Bosscher et al., 1997; Lee et al., 1999; Kaneko et al., 2012; Elchalakani et al., 2016; Edeskär, 2006; Edil, 2005; Park et al., 2003; Reddy and Saichek, 1998; Tsang, 2008; Senetakis et al., 2011).

While their potential range of applications is wide, a full understanding of their behavior is still missing. This originates from the highly heterogeneous nature of such mixtures as a consequence of the high stiffness and deformability contrasts of the constituents. The differences in particle interactions, thus at the micro-scale, induced by the presence of rubber in sand, affects both the peak and critical state stress friction angle at the macro-scale (Li et al., 2019). Such alternative soils can be considered as granular materials made of two types of constituents, sand and rubber, differing in the mechanical properties governing particle interactions (Young modulus, Poisson ratio, restitution, solid friction...) and volume density. In addition, some sand-rubber interaction parameters such as the friction coefficient were shown to display a velocity dependence (He et al., 2012). Potentially, particles of sand and rubber can also differ by their shape, size and particle size distribution.

The mixing quality of such granular systems is crucially important. Any heterogeneity in the spatial distribution of particles can lead to the formation of weak zones within the system and thus a mechanical destabilization of the system like liquefaction (Yoshimine and Koike, 2005). Unfortunately obtaining a good mixture quality in systems consisting of particles with different properties is quite challenging. Like any other granular assemblage, sand-rubber mixtures might indeed be subjected to segregation which, due to the aforementioned deleterious effects on their mechanical properties, may reduce the effective use of this recycling solution. Segregation of granular materials is particularly difficult to prevent because it may appear at several stages of their processing. For example, transportation by road, flow in industrial facilities, deposition and compaction are well known sources of segregation. The different particle properties which can cause segregation include particle sizes and size distribution, density, shape, morphology, contact friction, elasticity, brittleness, chemical affinities, ability to absorb moisture (Williams, 1976; Rosato et al., 2002, 1991; Enstad, 2001). For dry and stiff materials, the size ratio is probably the most important parameter controlling the occurrence of segregation. As it will be presented later, our results show that in our configuration (i.e. soft and deformable particles mixed with stiff and non-deformable grains in a confined geometry) this is not the case. An important literature can be found on the segregation induced by the two former parameters. Segregation induced by the difference of mechanical properties is much less studied though some works detail out the segregation due to mechanisms such as rolling, sieving, soil crushing, etc. (Kuerbis and Vaid, 1988; Ottino and Khakhar, 2000; Lőrincz et al., 2005; Watabe et al., 2014). Yet, as mentioned above, segregation in sand-rubber mixtures can be detrimental for their effective mechanical properties. It is thus interesting to understand the mechanisms of the aforementioned segregation as well as how it can be prevented. Note that, segregation can also be provoked mainly to optimize sorting which is crucial for many applications like recycling waste or purification of powders. The aim of the present work is to study the segregation of confined sand-rubber granular mixtures undergoing vertical tapping (Philippe and Bideau, 2002; Ribière et al.,

2005c,a). Our experimental setup consists of a sand-rubber mixture placed in a vertical, cylindrical vessel. To prevent granular convection which appears easily during tapping (Philippe and Bideau, 2002; Ribière et al., 2005b) we confine the whole by adding a mass at the top of the granular material. We chose this type of mechanical solicitations because of (i) their simplicity and (ii) the similarities with the solicitations experienced by the mixtures during their transportation on roads using lorries/trucks. Also, the aforementioned added confinement is justified by the absence of granular convection during transportation.

In this paper, we will show that, with the increasing number of taps, the light grains have a tendency to migrate towards the bottom of the system leading to a vertical segregation which is not complete. This tendency is reduced if the rubber fraction is reduced and the influence of the sand-rubber size ratio is found to be complex. We will also show that our systems tend to form horizontal clusters which appear to be weakly influenced by the grain size ratio, whereas the number of clusters may increase with the rubber fraction. Finally, we will demonstrate that both horizontal and vertical segregations can be significantly weakened by cohesion to the system, for example by adding a small amount of water.

2. Materials & investigative methods

2.1. Materials

The objective of the experiments was to study the segregation in sand-rubber mixtures. The rubber grains were composed of shredded recycled tire rubber (SBR, 0.5-2.5 mm size range, from Delta Gom, France). In order to help the characterization of mixing and segregation through image analysis, a white quartz sand was produced at the laboratory by crushing commercial quartzite gravel with a jaw crusher, and then sieving to extract the desired size ranges. The particle density of sand and rubber were respectively $\rho_{sand} = 2650 \text{ kg/m}^3$ and $\rho_{rubber} = 1000 \text{ kg/m}^3$. The study focused on three size ranges for sand (1.25-1.6 mm, 2-2.5 mm and 2.5-3.15 mm) and two for rubber (1.25-1.6 mm and 2-2.5 mm), which were combined in order to understand the effect of the size difference between sand and rubber on the mixture heterogeneity.

2.2. Experimental procedure and programme of testing

The experimental procedure consisted of 4 major steps:

1. Mixing of sand and rubber
2. Tapping of mixed sand and rubber using a vibrating shaker
3. Solidification of sample using a binder
4. Capturing images of solidified samples

All these stages will be detailed out in the following sections. As regards to the tests performed, the parameters which were varied during the experiments were: number of taps, sand/rubber grain size ratio, rubber volume fraction and moisture content. The parameters were varied with respect to a base case, consisting of a sand /rubber size ratio S/R=1 (sand: 2-2.5 mm, rubber: 2-2.5 mm), for a volume fraction of rubber (RF)

of 51% (estimated from the particle intrinsic densities, from a mass fraction of 28.6%), in dry conditions. The variations with respect to the base case were:

1. two other S/R size ratios : S/R=0.63 (sand: 1.25-1.6 mm, rubber: 2-2.5 mm) and S/R= 1.98 (sand: 2.5-3.15 mm, rubber: 1.25-1.6 mm),
2. a lower rubber volume fraction (RF=26%),
3. a wet configuration, corresponding to a water addition of 4% of the mass of the sample .

The number of taps was varied only for the base case, while it was fixed to 10000 (which was verified for the base case to produce relevant segregation, as it will be discussed in the following) for the other tests.

2.3. Sand-rubber mixing process

For the initial sample preparation, a laboratory mixing set-up (see Fig. 1) was designed. In order to minimize the segregation during further handling, the material was mixed in the same container which was subsequently vibrated. A cylindrical metal container of 110 mm in height and 108 mm in diameter was chosen as the mixing container. The sample dimensions are of sufficient size at least 10 times larger than the largest particles to insure material representativeness. Mixing blades were designed by taking into account the dimensions of the container and were fabricated using a PLA polymer (Polylactic acid), in a commercial filament 3D printer (ULTIMAKER 2+). Four equally spaced mixing blades were mounted on a shaft, one orthogonal to the other. The impeller was mounted on a commonly used laboratory agitator, which in the mixing step was operated at 250 rpm for one minute. This combination of velocity and time was checked to ensure good mixing. The initial amount of material in the sample was prepared in order to obtain a bed height/diameter aspect ratio of approximately 0.6 (bed height 65 mm). The mixing process started with measuring the required mass of sand and rubber in a glass vessel and pouring it using a small funnel into the mixing container, mounted on the mixing setup. In case of mixing with cohesion (*i.e.* with moisture), a given quantity of water was added to the mixture poured in the container. After the mixing, the container was removed carefully from the mixer and transported to the vibrating shaker equipment. In some cases, in order to assess the mixing performance the sample container was directly sent to the solidification process (explained in further sections).

2.4. Tests using the vibrating shaker

Figure 1 shows the the experimental setup for the vibration tests. It is important to mention here, that the setup was not developed according to conventional standards, but was specifically developed for studying the segregation problem, starting from a similar configuration previously developed in literature on compaction due to tapping (Ribi re et al., 2005a). The sand-rubber mixture container was placed on the top of a holding arrangement connected to a vibrating shaker (LDS V406). Signal control and data acquisition were performed using the InstruNet control system and the software Dasy-Lab. In particular we chose to follow the method adopted by Ribi re et al. (2005a) to produce the taps. Consequently, a vertical displacement was created in the vibrating

shaker by producing an entire period of sine wave at a constant frequency $f= 30$ Hz at intervals of $\Delta t = 1$ s. As explained by Ribière et al. (2005a), the resulting overall response of the system is complex. For a better understanding, an accelerometer was installed on the moving element of the shaker and its signal was acquired by the InstruNet. An example of a signal monitored by the accelerometer is presented in Fig. 2.

As seen in Fig. 2, shown by the red ellipse, the system initially undergoes a positive acceleration followed by a negative peak equal to $-\gamma_{max}$. Further it again undergoes a rise shown by a positive acceleration and then the fall of the mixture to the bottom of the container has been represented by $-\gamma_{fall}$. As it is evident from this example, the acceleration created by the fall, $-\gamma_{fall}$ can be different and even larger than the first acceleration peak, $-\gamma_{max}$. For the purpose of control of the imposed movement, only the first acceleration peak is of interest. The most important parameter was the normalized intensity (Γ) of the vertical tap, defined by $\Gamma = \gamma_{max}/g$, where g is the acceleration due to gravity. Since several other parameters were to be varied in this work, it was preferred to choose one reference value of Γ : a value was chosen, $\Gamma = 3$, that preliminary investigations showed to produce segregation in a reasonable time. Note also that $\Gamma = 3$ corresponds to the maximal acceleration that can be used in our system (higher accelerations force the grains and the counter mass out of the vessel). Preliminary investigations also showed that probably due to the small size of the system, if the surface of the bed was free to move, strong convection effects may emerge, which induced remixing of the grains. This remixing had to be avoided in this study, which focuses on a method to quantify segregation in sand rubber mixtures, with potential application to large systems (*e.g.* trucks/ lorries). For this purpose, a technique suggested by Philippe (2002) was used, wherein a mass equivalent to the mass of sand-rubber mixture was placed on the top of the container. This added confinement was observed to be extremely effective in reducing this remixing effect. As regards to the duration of the tests, different sequences with a number of vertical taps ranging from 10^2 to 10^4 were performed.

2.5. Solidification of the sand-rubber mixture sample using a binder

In order to quantify the heterogeneity of the mixture before and after being subjected to tapping, a solidification technique proposed by Dal Grande et al. (2008) was found to be effective. A commercial gel for candles (Candelis, melting temperature 75°C .) was used as the binder material. The mass of gel used was approximately equal to the mass of the sand-rubber mixture in the container. At ambient temperature, the gel was solid enough to be cut and placed on the top of the bed in the container. The container was then placed in an oven at 80°C for 6 h in order to allow the gel to melt and uniformly impregnate the container. Further, in order to avoid the entrapment of air bubbles in the mixture container, several small holes (diameter 0.5 mm) were drilled at the bottom of the container before filling the container with the sample. After 6 h, it was checked that the excess gel was coming out through all the holes at the bottom of the mixture container, thus confirming the completion of the melting and percolation. The mixture container was then removed from the oven and allowed to cool down and solidify at the room temperature for over 12 h.

2.6. Image processing analysis of samples

After ensuring that the samples were completely solidified, the metal containers were torn apart and opened in order to remove the sample. The samples were cut into horizontal slices in order to quantify the heterogeneity of the mixture in the container. The number of slices was quite important as well, in order to obtain maximum possible information to better quantify the segregation and at the same time, not to disturb the structure while slicing. The best compromise consisted in having 5 horizontal slices of the entire solidified mixture sample. The slices were cut very carefully in order to minimize the loss of grains during the process.

The slices of the solidified samples were then placed inside a white container equipped with a ring of LED lights (see Fig. 1) and images were captured using a digital camera. For every slice, there were two surfaces of which the images were taken, *i.e.* bottom surface and top surface of the slice. Images of all such slices were taken and then they were used further for analyzing the segregation in the mixture. In order to minimize the possible disturbances to image processing caused by the small portions of gel being present at the slice surface due to the cut, a heat gun was used to gently allow the small portions of the gel to melt and settle down. Then, the images of the slices were captured using the process described above. The mixture quality was analysed by image processing of the pictures by means of Python scripts. The image analysis framework is exemplified in the sketch given in Figure 3, and was based on the statistics (horizontal means and variance) of locally averaged compositions. This is a classical and robust technique for estimating the amount of segregation (Rumpf, 1990).

The main steps of the image processing framework are summarized in the following:

1. the images captured were zealously cropped around the section of the slice, yielding an image size of approximately 3000 pixels x 3000 pixels (1 mm \approx 28 pixels) ;
2. the pictures were converted to grayscale and a median filter (with a circular kernel with a diameter of 21 pixels, which was chosen after careful analysis, and which was smaller than the smallest sand grains) was applied to them to minimize the glare spots produced by light reflection on gel;
3. the pictures were thresholded with a mean threshold filter to produce a binary image, and the background was masked away;
4. a local concentration of white pixels (corresponding to sand particles) was computed on sampling disks (of diameter 500 pixels, which was chosen because it is sufficiently larger than particle size and sufficiently smaller than the box size) equally distributed on each slice.
5. the distribution of local concentrations were analysed statistically.

In particular, for each cut level, z_j the mean and the standard deviation of the distribution of local concentrations, c_i , where i denotes the sampling disks, were extracted:

$$\bar{c}(z_j) = \frac{1}{N_j} \sum_{i=1}^{N_j} c_i(z_j), \sigma_c(z_j) = \sqrt{\frac{1}{N_j} \sum_{i=1}^{N_j} (c_i(z_j) - \bar{c}(z_j))^2}, \quad (1)$$

where N_j is the number of sampling disks for each cut j .

For each picture, 28 sampling points were employed, which means that for intermediate cuts $N_j = 56$, given that for each cut two pictures were taken (top and bottom). On the other hand, for the bottom-most picture, corresponding to the bottom of the sample, $N_j = 28$, while the top surface was not treated because it wasn't possible to remove the excess gel efficiently. Given the type of measurement, the colour of the two components, and the shape of the particles, the concentration c_i does not exactly correspond to the concentration of sand particles. In order to convert the concentration of white pixels to a concentration of sand particles, a stereological transformation would be needed, taking into account the size dependent mutual screening effect particles, and possibly other effects. However, our aim was to study the effect of material and process parameters with respect to a base case, therefore local white pixel concentrations can be directly used to quantify the effect of such parameters on the heterogeneity and spatial distribution of sand in the samples. The analysis was therefore made on the white pixel concentrations. In order to help interpret the results, we employ in the following a normalization for the mean and standard deviation. For the mean, we will analyse the variation with respect to the global mean :

$$\tilde{c}(z_j) = \frac{\bar{c}(z_j) - c_o}{c_o}, \text{ where } c_o = \frac{1}{N_l} \sum_j^{N_l} \bar{c}(z_j). \quad (2)$$

where c_o is the overall average of the white pixel concentrations, and $N_l = 5$ is the number of levels at which photos were taken (4 cuts plus the bottom surface of the sample). For the standard deviation, a classical normalization was used (Rumpf, 1990), dividing it by the maximum standard deviation of a completely segregated binary mixture, evaluated at each level ($\sqrt{\bar{c}(z_j)(1 - \bar{c}(z_j))}$):

$$\tilde{\sigma}_c(z_j) = \frac{\sigma_c(z_j)}{\sqrt{\bar{c}(z_j)(1 - \bar{c}(z_j))}}. \quad (3)$$

A global measure of the heterogeneity was also used, i.e. the normalized global standard deviation:

$$\bar{\sigma}_c = \frac{1}{\sqrt{c_o(1 - c_o)}} \sqrt{\frac{1}{N_l} \sum_{j=1}^{N_l} \frac{1}{N_j} \sum_{i=1}^{N_j} (c_i(z_j) - c_o)^2}. \quad (4)$$

In the following, the spatial heterogeneity is evaluated by means of such indicators, with particular respect to the effect of the sand/rubber (S/R) size ratio, rubber volume fraction, presence of water and the number of taps.

3. Results

3.1. Effect of number of vertical taps

The main process parameter which was studied was the test duration, i.e. the total number of taps to which the material was subjected. The effect of the number of taps

was characterized for the reference case (dry, sand/rubber (S/R) size ratio of 1 and rubber volume fraction of 51%). In order to show the segregation at work and help understanding the quantitative results obtained with the image analysis technique, Fig. 4 shows the images of one (intermediate) slice for each of the samples, *i.e.* for sand-rubber mixture after mixing and after 100, 1000 and 10000 taps. From the images of the slices, it can be seen that, for 100 taps, the sample looks like a mixed one and then horizontal segregation starts to develop and increases up to 10000 taps.

Quantitative results obtained by means of the technique described in the previous section are displayed in Fig. 5, which shows the evolution of the sample heterogeneity with height and in time. The points in the figure correspond to the average of three independent tests, and the error bars to the standard deviation of these repetitions. Concerning the vertical profiles of the mean concentration variation (Fig. 5a), they point at the development of a vertical heterogeneity. Quite surprisingly, this heterogeneity is characterized by the lightest component migrating towards the bottom, the opposite to what happens in free surface configurations. However, the vertical variations of composition are relatively small, and well far from a complete segregation. This heterogeneity appears between 100 and 1000 taps and seems to quite stabilize after 1000 taps.

On the other hand, the vertical profiles of the normalized standard deviation (Fig. 5b) are a witness of a horizontal heterogeneity which continuously increases with the number of taps. Combined with the mean concentration profiles, this translates the horizontal segregation pattern which was visible in Fig. 4. Also, for longer times, the heterogeneity is significantly higher at the top of the sample than at the bottom.

Before describing the effect of the other material parameters, it is important to note that, while segregation was not evident at 100 taps, this albeit small excitation of the material in the shaker contributed to a better performance of the gel percolation process, probably due to a slight compaction of the system. In fact, a large void of air was often noted if the samples were solidified immediately after the mixing process. Given that the difference in heterogeneity observed between the simply mixed samples and the samples subjected to 100 taps was negligible, in the following tests, the reference samples were always subjected to 100 vertical taps after mixing process before being solidified. On the other hand, the segregation effect was strongly visible at 10000 taps, and this was chosen as the reference for characterizing the effect of the dynamic loading. Thus, the results which will be discussed in the following sections (different size ratios, rubber volume fractions and moisture content) will be based on the comparison between samples subjected to mixing and 100 taps and those subjected to mixing and 10000 vertical taps.

3.2. Effect of sand/rubber size ratio

As discussed previously, 3 different sand/rubber (S/R) size ratio mixture samples were prepared and analyzed with an objective to quantify the effect of size difference on the segregation characteristics. As above, 3 tests for each *i.e.* mixing and vertical tap tests were conducted. Regarding the profiles of the mean concentration variation (Fig. 6a), when sand is smaller than rubber (S/R=0.63), the profiles are quite comparable to the base case (S/R=1), with a relatively higher presence of rubber at the bottom of the sample. This contrasts with what happens in free surface configurations, in which

large or light grains tend to accumulate towards the top surface. When sand particles are larger than rubber particles ($S/R=1.98$), the profiles appear to be more complex. In particular, there seems to be a partial inversion of the vertical heterogeneity in the intermediate levels with a tendency to transport sand particles to the bottom of the sample. The complex vertical structuring of the mixture after 10000 taps is directly visible from example pictures in Fig. 7.

On the other hand, as regards to the horizontal heterogeneity, the vertical profiles of the normalized standard deviation (Fig. 6b) do not display strong variation with sand-rubber size ratio. This can be explained by the fact that horizontal clustering does not probably involve the same mechanisms as vertical segregation (e.g. sifting), and therefore is not affected by size differences between the particles.

3.3. *Effect of rubber fraction*

It is important to characterize the effect of the relative concentrations of the components of sand-rubber mixtures, given that the composition influences the technological properties of the material. Rubber fraction can be thought to play an important role in the segregation characteristics of sand-rubber mixtures, since the densities are very different for the two constituents. In this work, tests were performed on the mixture samples with 2 different rubber fractions, *viz.* 21% and 56% (by volume, the second being the reference case). Greater rubber fractions are not interesting for structural applications. Initially, an effort was made to prepare and test the samples for a lower (11%) rubber fraction as well, however such samples were found to be very hard to cut after solidification and the slice cutting process also caused a huge loss of grains. Hence, only 26% and 51% rubber fractions have been discussed here.

Results for these tests are reported in Fig. 8. Concerning the vertical profiles of the mean concentration (Fig. 8a), for a 26% rubber fraction, a slight increase of vertical heterogeneity due to the vertical taps with respect to the mixing case is visible, however this heterogeneity is lower than that obtained for 51% rubber fraction. The same is true for the horizontal heterogeneity, described by the normalized standard deviation in Fig. 8b, which is generally lower for a lower rubber fraction. This can be confirmed also by visual inspection of the slice pictures, of which two examples (corresponding to the middle of the sample) are shown in Fig. 9, to be compared with Fig. 4b-d. It is evident that a development of horizontal heterogeneity is hardly visible from the pictures. Therefore, for the same number of taps, segregation of sand-rubber mixtures appears to strongly depend on the composition, with a significantly lower segregation in the test with lower rubber fraction.

3.4. *Effect of water addition*

It is well known that the addition of a small amount of liquid (*e.g.* oil, water) increases the cohesion between particles and therefore reduces their flowability and, in turn, the potential for segregation (Mosby et al., 1996; Tang and Puri, 2004). Since, the sand-rubber mixtures under study are found to segregate in dry conditions, it was interesting to study the effect of addition of water to such mixtures during the mixing process. One wet case was considered for simplicity, and the amount of water to be added was chosen considering that water induces cohesion, but above a certain amount

of liquid, lubrication effects become important, and therefore act in the opposite sense. Considering the size and shape of the particles, the optimal water amount is around a few percent of the mass of the sample (Artoni et al., 2019). Consequently, tests for mixing and vertical taps were performed by adding 4% water of the total weight of the mixture used. As it was stated above, water addition was studied with respect to the base case (size ratio $S/R = 1$, and rubber fraction $RF = 51\%$). Water was added during the mixing process itself and then the samples were subjected to vertical taps. Similar to the previous tests, 3 tests for each condition, *i.e.* mixing plus 100 taps, and mixing followed by 10000 taps were performed. In these tests, the image processing was disturbed for the bottom-most slice by the formation of rust at the contact with the iron box, which altered the colour of the white particles. Nevertheless, the analysis could be performed without problems for the other slices.

The results, displayed in Fig. 10, are evident: while for the dry case (black symbols) which highlighted the development of vertical heterogeneities due to the dynamic loading, for the wet case (red symbols) the mean concentration profile (Fig. 10a) was not affected significantly by the tapping. Concerning horizontal heterogeneities, normalized standard deviation profiles seem to suggest that water addition reduces clustering already in the mixing phase (dashed lines), and then that the tapping phase produces a very small amount of clustering. The results thus suggest that the introduction of wetting liquid such as water can reduce the segregation in sand-rubber mixtures under the conditions mentioned in this study.

3.5. Global heterogeneity index

In the previous sections, the effect of different parameters were discussed with respect to horizontal dispersion and vertical heterogeneity. Here, we summarize the results obtained for all the tests by means of a global heterogeneity estimate, *i.e.* the global standard deviation (Eq. 4). The change in $\bar{\sigma}_c$ between 100 and 10000 taps, for all the tests, is condensed in Tab. 1. It allows to sum up the results on the effect of several parameters on the potential of segregation of sand-rubber mixtures. From a global point of view, the mixture heterogeneity or segregation as measured in our confined, vertical tapping set-up:

1. increases with the number of taps,
2. decreases when capillary forces are present, due to the addition of water,
3. decreases when the rubber fraction is decreased,
4. does not seem to be affected by the size difference between sand and rubber.

If the complete analysis provided in the previous sections allows to understand the complex spatial structuring of the mixture heterogeneity, the $\bar{\sigma}_c$ data allow to summarize the effect of multiple parameters. The experimental setup and analysis techniques used in this work can therefore provide a global measure of the potential of sand-rubber mixtures to segregate, and are arguably interesting for practical applications.

4. Discussion and Perspectives

In this work, the segregation of confined sand-rubber mixtures due to vertical tapping was studied, by varying the number of taps, the sand/rubber size ratio (S/R), the

volumetric rubber fraction and the moisture content. The segregation analysis was carried out using Digital Image Analysis techniques on samples extracted by a solidification technique, allowing to obtain information about vertical and horizontal heterogeneities. In our experimental setup, the main results can be briefly presented as follows:

- A vertical heterogeneity develops when increasing the number of taps, which is characterized by a tendency of light particles to migrate to the bottom of the sample. This heterogeneity does not lead to a full vertical segregation, and is reduced if the rubber fraction is reduced, and nearly eliminated by the addition of a small amount of water. The sand-rubber size ratio has a complex influence because large grains seem to tend to migrate towards the bottom of the cell, and therefore size segregation may compete with density segregation when sand particles are larger in size.
- A tendency to form horizontal clusters was clearly observed, increasing with the number of taps. This horizontal heterogeneity appears to be reduced if the rubber fraction is smaller, and as for the vertical heterogeneity, it is nearly eliminated by the addition of a small water amount. Size differences between the grains do not appear to affect strongly their horizontal distribution.

It is not easy to give an explanation of the observed segregation patterns. With respect to vertical heterogeneity, the tendency to accumulate lighter particles at the bottom of the sample seems to point that segregation in our confined cell is not driven by density. Still referring to vertical segregation, the complex effect of the particle size also suggests that size segregation is not really relevant, at least in the standard sense, and if exists, it is strongly affected by other mechanisms. These considerations, together with the evidence on the development of horizontal heterogeneity features indicate that, in our set-up, segregation may be due to an instability coming from differences in particle interactions (contact stiffness and friction) rather than particle mass or size. To better understand the mechanisms controlling these segregation patterns it would be interesting to modify the geometry of the vessel to detect any effects. Also, inclining slightly the system with respect to the horizontal could be a good way to test the soundness of the horizontal segregation.

This work is meant to be a first step at the laboratory scale to collect evidences on the development of heterogeneous microstructure in sand-rubber mixtures subject to dynamic loading. It is of potential interest to assess the parameters influencing segregation in applications related to the transport of granular materials for geotechnical applications. The results presented here may provide a guidance for further research, which will be able to elucidate more in detail the effect of material and process parameters by focusing on the subranges of interest for potential applications. Also, this work shows that the mixtures studied may be prone to segregate strongly in some ranges of the parameter space. They prove that, with concern to the potential applications, it is important to consider the effect of the heterogeneity of the microstructure on the mechanical properties of the materials. Such segregation studies are therefore the counterpart of experimental and numerical (DEM) tests which aim to determine the

mechanical behavior of sand-rubber mixtures under typical loading conditions experienced by geotechnical systems.

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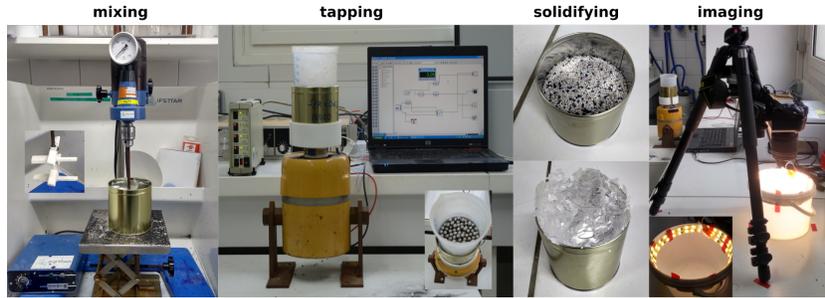


Figure 1: Experimental setup showing, from left to right, (i) the mixing device with adjustable stand and a particular of the 3D printed mixing blades, (ii) the vibrating shaker with the sand-rubber mixture container on top and with the control equipment, with a particular of the counter-mass containing steel beads used for providing confinement, (iii) pictures of samples after tapping and placement of the gel for solidification, (iv) the simple controlled lighting and imaging setup.

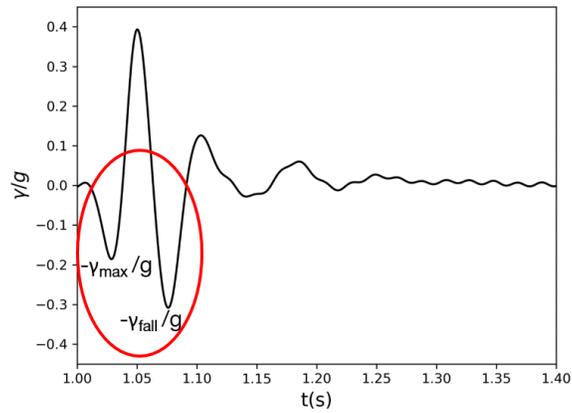


Figure 2: Example of a signal monitored by the accelerometer. The acceleration created by the fall of the grains on the bottom γ_{fall} is different from the acceleration applied to the system γ_{max} .

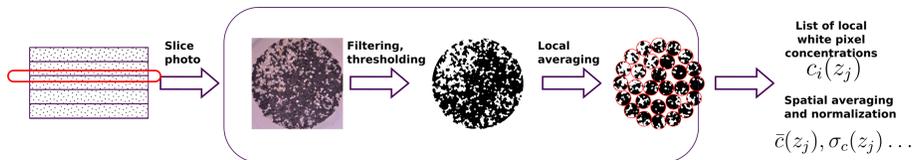


Figure 3: Sketch of the image analysis procedure.

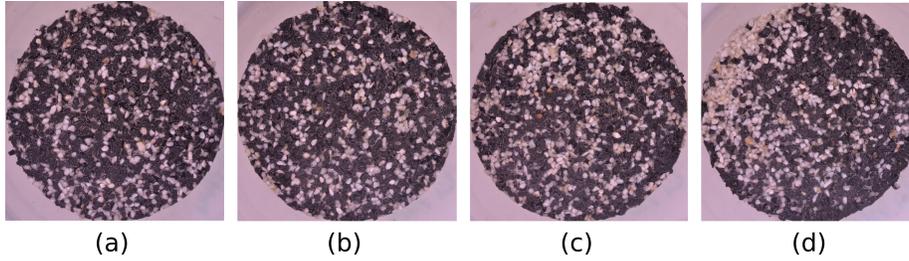


Figure 4: Sample images of sand-rubber mixture for size ratio 1: (a) after mixing, and after respectively (b) 100, (c) 1000, and (d) 10000 vertical taps.

Condition	RF %	S/R	$\bar{\sigma}_c$, 100 taps	$\bar{\sigma}_c$, 10000 taps
Dry	51	1	0.187 ± 0.009	0.344 ± 0.020
Dry	26	1	0.175 ± 0.013	0.239 ± 0.036
Dry	51	0.63	0.203 ± 0.003	0.371 ± 0.041
Dry	51	1.98	0.237 ± 0.013	0.391 ± 0.044
Wet	51	1	0.152 ± 0.004	0.191 ± 0.018

Table 1: Global heterogeneity index $\bar{\sigma}_c$ defined in Eq. 4, at the beginning (100 taps) and at the end of a test (10000 taps), for all the conditions discussed in this work.

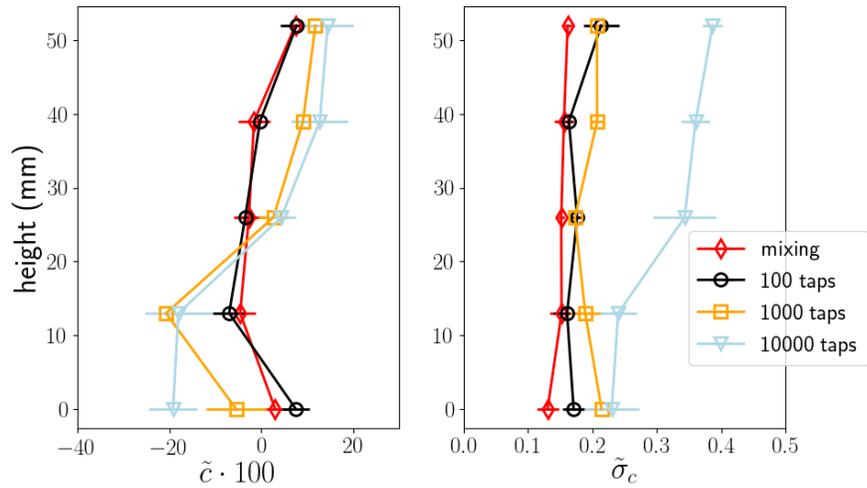


Figure 5: Evolution of vertical profiles of (a) the mean concentration variation and (b) the normalized standard deviation, for different test durations (number of taps) for the reference dry sample S/R = 1 and RF= 51 % case.

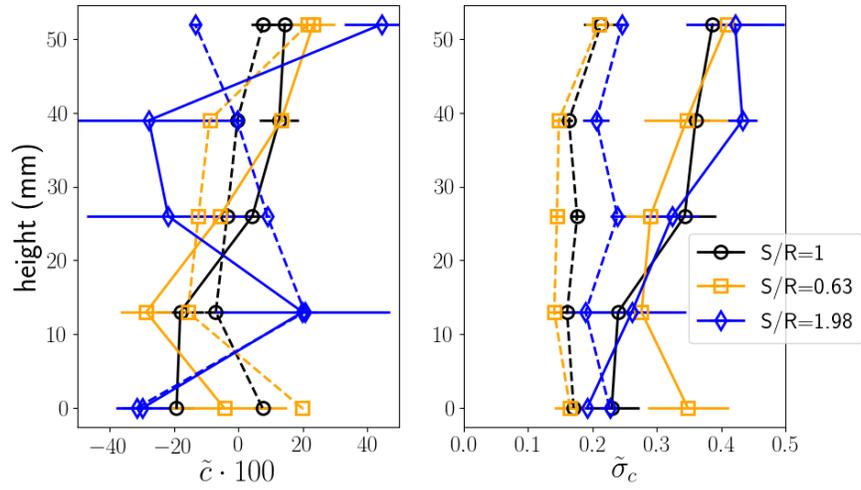


Figure 6: Evolution of vertical profiles of (a) the mean concentration variation and (b) the normalized standard deviation, for different values of the sand-rubber particle size ratio (S/R). The dashed lines represent the reference state (mixing plus 100 taps), while solid lines represent the fully vibrated samples (mixing plus 10000 taps).

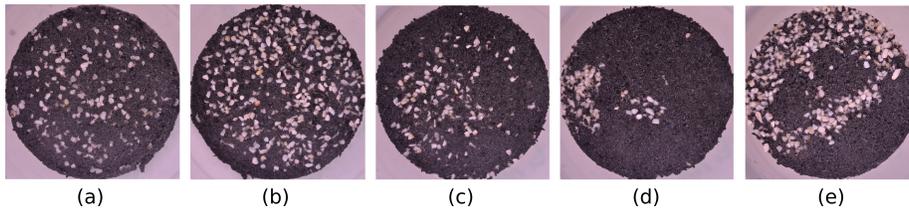


Figure 7: Sample images of sand-rubber mixture for size ratio 1.98 and a 51% volume rubber fraction after 10000 vertical taps, at different heights (height increases from left to right): complex vertical structuring is evident.

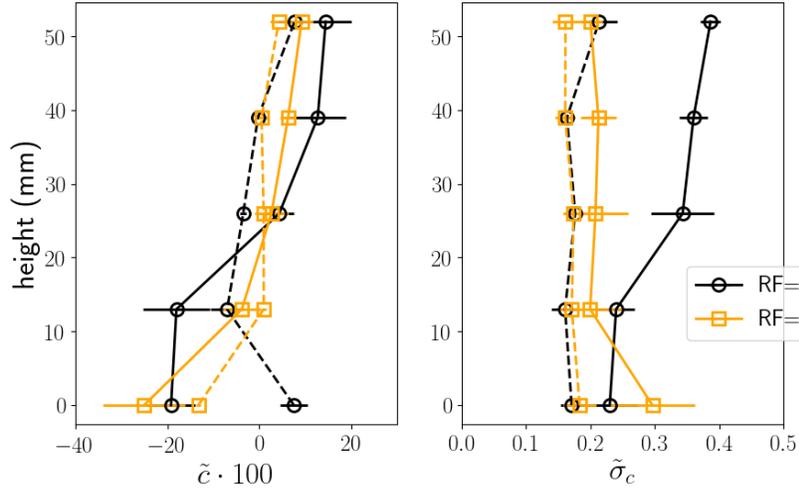


Figure 8: Evolution of vertical profiles of (a) the mean concentration variation and (b) the normalized standard deviation, for two values of the volumetric fraction of rubber in the mixture. Dashed lines represent the reference state (mixing plus 100 taps), while solid lines represent the fully vibrated samples (mixing plus 10000 taps).

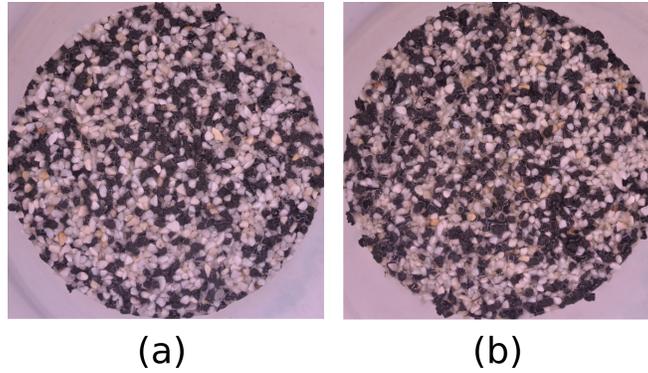


Figure 9: Sample images of sand-rubber mixture for size ratio 1 and a 26% volume rubber fraction : (a) after 100 and (b) 10000 vertical taps.

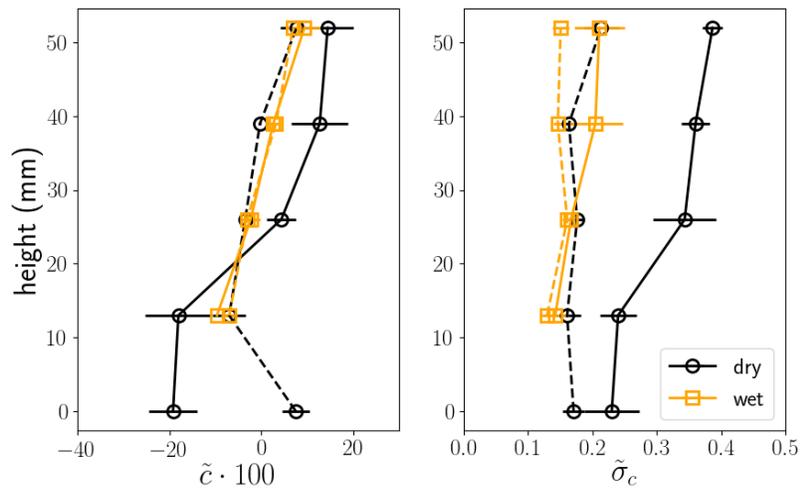


Figure 10: Evolution of vertical profiles of (a) the mean concentration variation and (b) the normalized standard deviation, for a wet mixture, in comparison to dry conditions. Dashed lines represent the reference state (mixing plus 100 taps), while solid lines represent the fully vibrated samples (mixing plus 10000 taps).