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1 **Experimental investigation on the grain-scale compression**  
2 **behavior of loose wet granular material**

3

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30 **Abstract**

31

32 The behavior of model granular materials (glass beads) wetted by a small quantity of liquid  
33 forming capillary bridges is studied by one-dimensional compression test combined with X-  
34 ray computed tomography (XRCT) observation. Special attention is paid to obtain very loose  
35 initial states (initial void ratio of about 2.30) stabilized by capillary cohesion. XRCT-based  
36 analyses involve spherical particle detection adapted to relatively low-resolution images,  
37 which enable heterogeneities to be visualized and microstructural information to be collected.  
38 This study on an ideal material provides an insight into the macroscopic compression  
39 behavior of wet granular materials based on the microstructural change, such as pore distance  
40 distribution, coordination number of contacts, coordination number of neighbors, number of  
41 contacts per grain.

42

43 *Keywords:* Wet granular material; Microstructure; One-dimensional compression; X-ray  
44 computed tomography; Grain-scale analysis

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57 **List of notation**

58

59	$a$	size of standard volume
60	$b$	size of extended volume
61	$\langle d \rangle$	average diameter
62	$d_{\min}$	minimum diameter
63	$d_{\max}$	maximum diameter
64	$e_0$	initial void ratio
65	$e$	void ratio
66	EV	extended volume
67	$f(r)$	signature curve
68	$\Phi_0$	initial solid fraction
69	$g(r)$	radial distribution function
70	$i, j, k$	voxel indices
71	$i_C, j_C, k_C$	center position of detected sphere
72	$\mathbf{I}(i, j, k)$	intensity at voxel $(i, j, k)$
73	$\nabla \mathbf{I}(i, j, k)$	gradient vector at voxel $(i, j, k)$
74	$N$	number of particles
75	$N_C$	number of pairs in contacts
76	$p(r)$	average number density of particles
77	$q(i, j, k)$	vector from $(i_C, j_C, k_C)$ to voxel $(i, j, k)$
78	$r$	radii of particles / radial distance
79	SV	standard volume
80	Si	step of scan (S1, S2, S3, S4)
81	$z$	total coordination number

82  $z(h)$  coordination number of close neighbors

## 83 **1 Introduction**

84

85 Granular materials appear in various natural and industrial processes. They are often  
86 composed of individual grains that vary in shape, size and surface texture. Such parameters  
87 can remarkably affect the packing of granulates and their contact distribution characteristics.  
88 In the case of wet granular materials, the existence of liquid menisci between particles plays a  
89 key role in the overall behavior of the assembly. Capillary cohesion bestows to these materials  
90 specific mechanical features that do not exist with dry grains, such as the ability to form stable  
91 structures with very low densities, and a strong sensitivity to external applied forces. So far,  
92 there are a lot of experimental studies on their macro- and micro-mechanical behaviors, such  
93 as: cohesive soils [1, 2], wet beads [3, 4], loess [5, 6], wet sand [7–9], among others. These  
94 works evidenced the link between the macroscopic mechanical behavior and the  
95 microstructure change. However, few works have yet focused on the grain-scale behavior.

96

97 Currently, the mechanical grain-scale behavior of a wet granular material is usually  
98 investigated by discrete element methods using 2D and/or 3D simulations [10–24]. These  
99 studies precisely characterized the change of microstructure such as coordination numbers (of  
100 contacts, of distant interactions, of compressive and tensile contacts), radial distribution  
101 function, force chains, distribution of forces between particles, *etc.* under the applied external  
102 forces. However, few works have compared numerical simulations (*e.g.* using the discrete  
103 element method) and experimental results in order to validate the numerical method [25–30].

104

105 X-ray computed tomography (XRCT) has tremendous potential due to its ability to carry out  
106 volumetric imaging inside the specimen. It has become a widely used technique and is  
107 appropriate for numerous types of materials including particulate and porous materials [31,

108 32]. Furthermore, this technique has been used in several studies to investigate the  
109 microstructure of granular materials under mechanical loading [8, 33–39]. Most of these  
110 works focus on the analysis of granular packing in dense states and with large-size  
111 particulates. Few works have experimentally investigated the mechanical grain-scale behavior  
112 of wet granular materials by using XRCT [40–44].

113

114 In this paper, we present an experimental investigation, by means of XRCT, on a loose wet  
115 granular material. A cylindrical sample constituted of thousands of glass beads with liquid  
116 bonds were subjected to an oedometric compression test, which consisted of several  
117 compression steps. At each step, a XRCT scan was performed. The sample heterogeneity is  
118 first analyzed in order to get a general understanding of the pore-scale compression behavior.  
119 The main objective of the present works is to give a further insight into the grain-scale  
120 compression behavior of loose wet granular materials.

121

## 122 **2 Experiments**

### 123 *2.1 Material and specimen preparation*

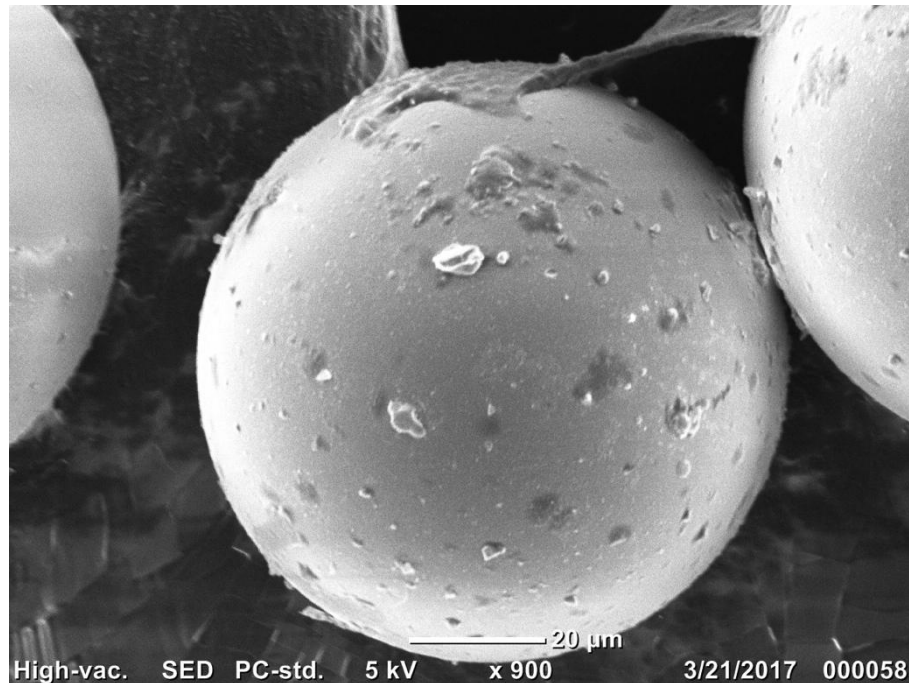
124

125 The investigated material consists of industrial spherical glass beads (diameter equals 87  $\mu\text{m}$   
126 to 110  $\mu\text{m}$  with an average value of 100  $\mu\text{m}$ ). The specific density of this material is equal to  
127 2,460  $\text{kg}\cdot\text{m}^{-3}$ . A Scanning Electronic Microscopic image of a glass bead is shown in Figure 1.  
128 The image shows that the surface of the bead is slightly damaged (beside the presence of  
129 some tiny dust grains) which makes it not perfectly smooth.

130

131 To prepare the specimen, the glass beads were first mixed with water to reach gravimetric  
132 water content (mass of water divided by mass of solid) of 3.5 %, which ensured that the  
133 specimens always remained in the pendular regime. The wetted glass beads were then placed

134 on a 200  $\mu\text{m}$  opening sieve installed 80 mm above an oedometric cell. Afterward, a machine  
135 vibrated the sieve vertically in order to gradually pass the wet glass beads through the sieve  
136 and let them fall into the cell.  
137



138  
139 Figure 1: Scanning Electronic Microscopic image of a glass bead.

140  
141 This procedure allowed the preparation of loose specimen with a porosity  $n = 0.70$   
142 (corresponding to a void ratio  $e_0 = 2.33$ , a solid fraction  $\Phi_0 = 0.30$ , and a degree of saturation  
143 of  $S_r = 3.7\%$ ). This degree of saturation, smaller than 5 %, corresponds to the pendular regime  
144 following Newitt & Conway-Jones [45].

145  
146 **2.2 Oedometric compression behavior**

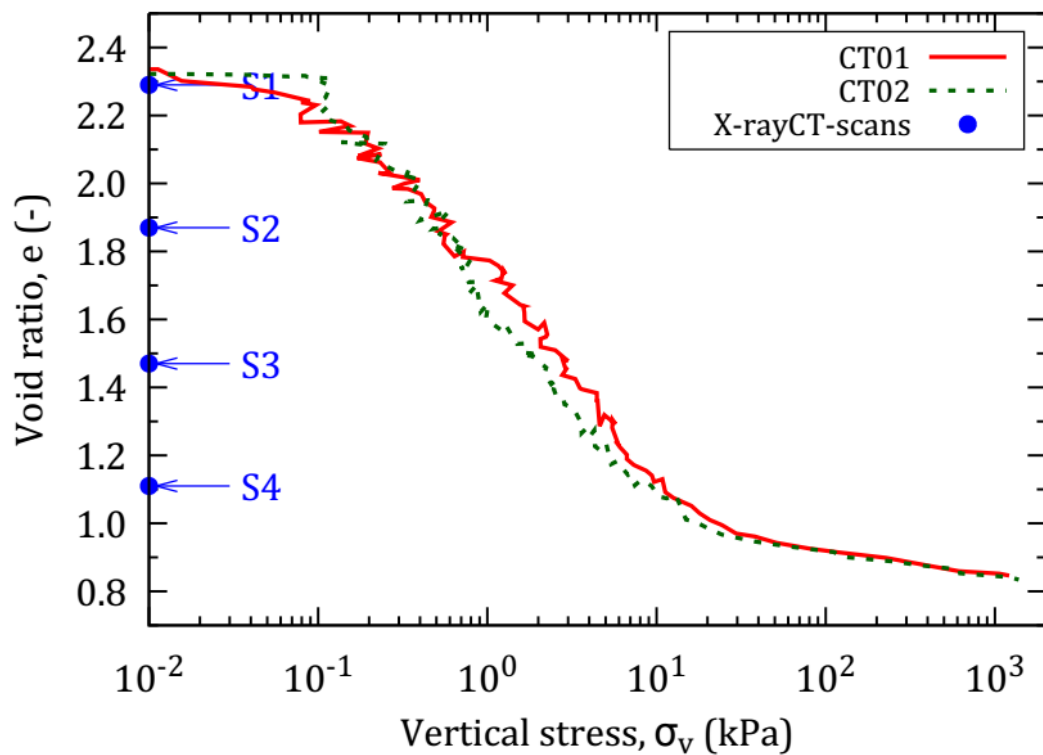
147  
148 In order to perform oedometric compression tests, the specimen (50 mm in diameter and 20  
149 mm in height), prepared by the procedure described above, was carefully placed on a loading  
150 platform. A piston was fixed to a load cell to measure the vertical load applied to the sample.

151 A LVDT sensor measured the displacement corresponding to the compression. The cell was  
152 then moved upward with a rate of 0.5 mm/min.

153

154 Figure 2 illustrates the compression curves of two specimens (CT01 and CT02). Based on the  
155 shape of these curves and the range of vertical stress, three stages could be identified. Firstly,  
156 under low stresses ( $\sigma_v < 0.1$  kPa), void ratio  $e$  is slightly decreased. Secondly, the void ratio is  
157 sharply decreased. Finally, under higher stresses ( $\sigma_v > 10$  kPa), the void ratio is gradually  
158 decreased.

159



160

161 Figure 2: Void ratio versus vertical stress during oedometric compression tests for two  
162 specimens. The circle points show the conditions of XRCT scans.

163

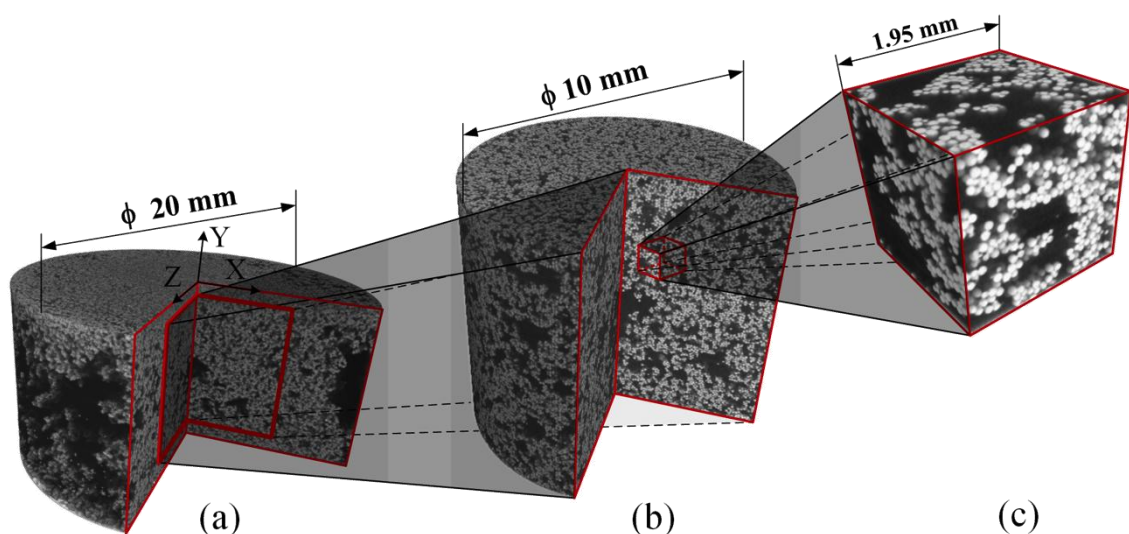
### 164 2.3 X-ray computed tomography

165



166 In the present work, XRCT scans were performed by using an UltraTom microtomography  
167 device. Prior to the observations with XRCT, the specimen (20 mm in diameter and 10 mm in  
168 height), prepared by the procedure described in the section 2.1, was compressed to reduce its  
169 void ratio to  $e = 2.29$  (position S1, see Figure 2). Once this state was reached, a XRCT scan  
170 (global scan) was performed for the whole specimen. The central zone of the sample (10 mm  
171 in diameter and 80% of the sample's height) was subsequently scanned (local scan). Figure 3  
172 shows an example of reconstructed 3D images obtained from these scans. The voxel size was  
173 13  $\mu\text{m}$  for the global scan and 6.5  $\mu\text{m}$  for the local scan. Following the scan corresponding to  
174 the position S1, the same scanning procedure was repeated three times to obtain the XRCT  
175 images corresponding to positions S2, S3 and S4, as shown in Figure 2. One piston having  
176 appropriate length was designed for each position. That allowed the correct control of the  
177 height of the sample to be scanned (there was no pressure applied to the piston during the  
178 scan). A global scan took 80 min and a local scan took four hours. During these scans, the  
179 piston was maintained above the sample to avoid evaporation of water. The small gap  
180 between the piston and the cell (0.05 mm) allows draining air during compression while  
181 avoiding evaporation during the scan. The cell and the pistons were made of Polymethyl  
182 methacrylate (PMMA) in order to optimize the quality of the XRCT images.

183



184

185 Figure 3: (a) Global scan, (b) local scan, and (c) investigated cube.

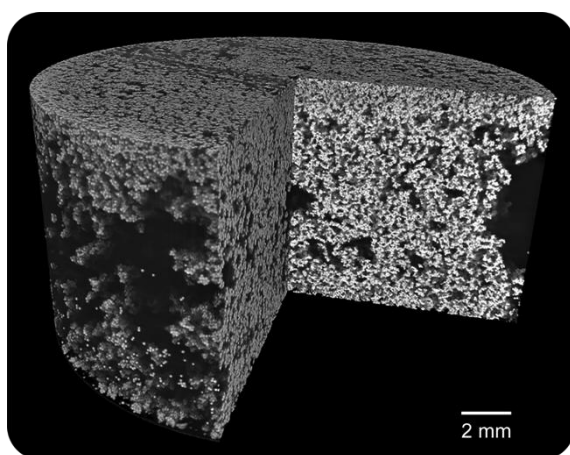
186

#### 187 **2.4 Analysis of sample heterogeneity**

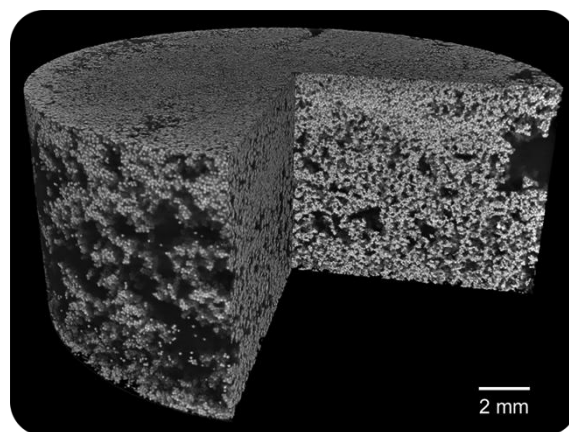
188

189 The reconstructed 3D samples, obtained by global scans at four compaction levels, are shown  
190 in Figure 4. It is well recognized that the density is not homogeneous within the cylindrical  
191 sample, especially at the periphery where more voids can be observed; higher densities and  
192 fewer voids exist at the top and the bottom of the sample. There are also several large voids in  
193 the center of the specimen in the initial stages (Figure 4*ab*). Similar results were observed  
194 previously on unsaturated loose volcanic sands [44]. This heterogeneity is due to the  
195 specimen preparation, and also to the boundary conditions imposed by the oedometer cell. In  
196 other words, during the specimen preparation, the friction between the cell and the grains  
197 limits the movement of grains. At higher compaction levels (Figure 4*cd*), the structure is  
198 strongly rearranged in the central layers because the loosest state was observed in this region  
199 (horizontal band) while the structure is slightly affected in the bottom and top layers.

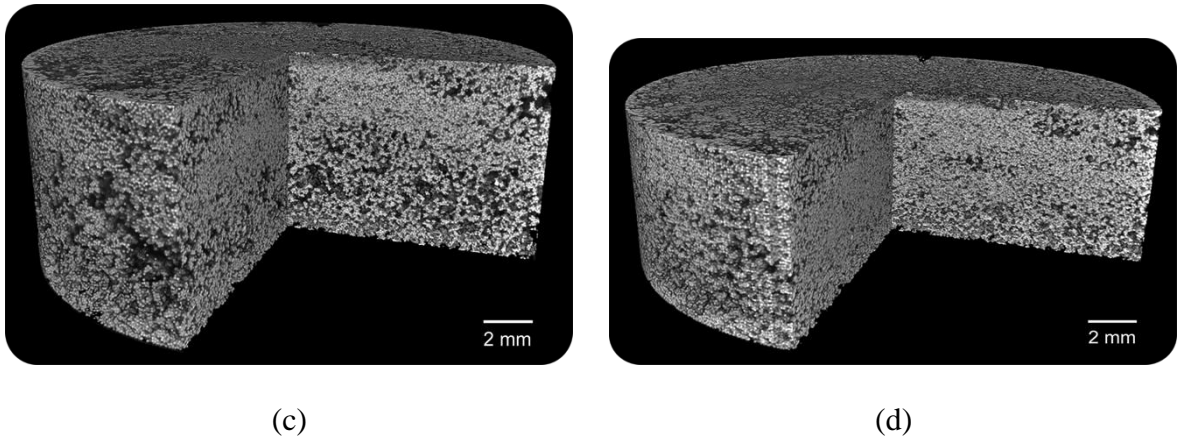
200



(a)



(b)

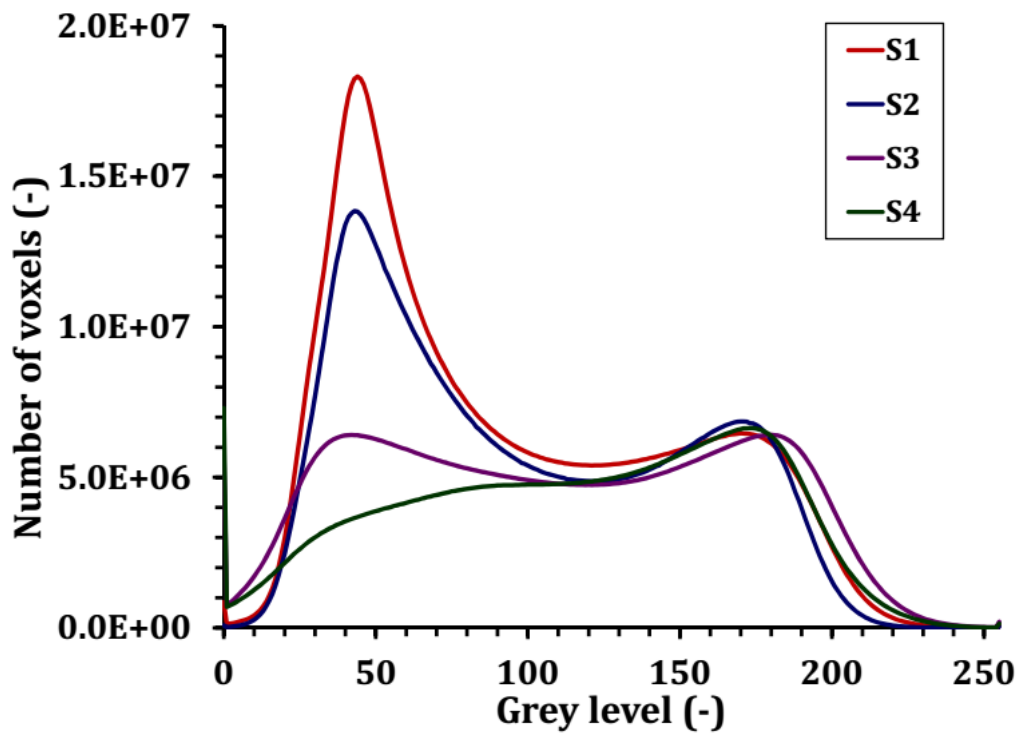


201 Figure 4: 3D reconstructed images of the sample at different compaction levels. (a) S1, (b) S2,  
 202 (c) S3, and (d) S4.

203

204 The grey level histograms are shown in Figure 5. Depending on the compaction level, two  
 205 peaks are more or less visible on each histogram. The first one on the left-hand side  
 206 corresponds to air-dominated voxels. Its number is smaller at a denser state. The peak on the  
 207 right-hand side corresponds to grain-dominated voxels and its number did not change during  
 208 compaction.

209



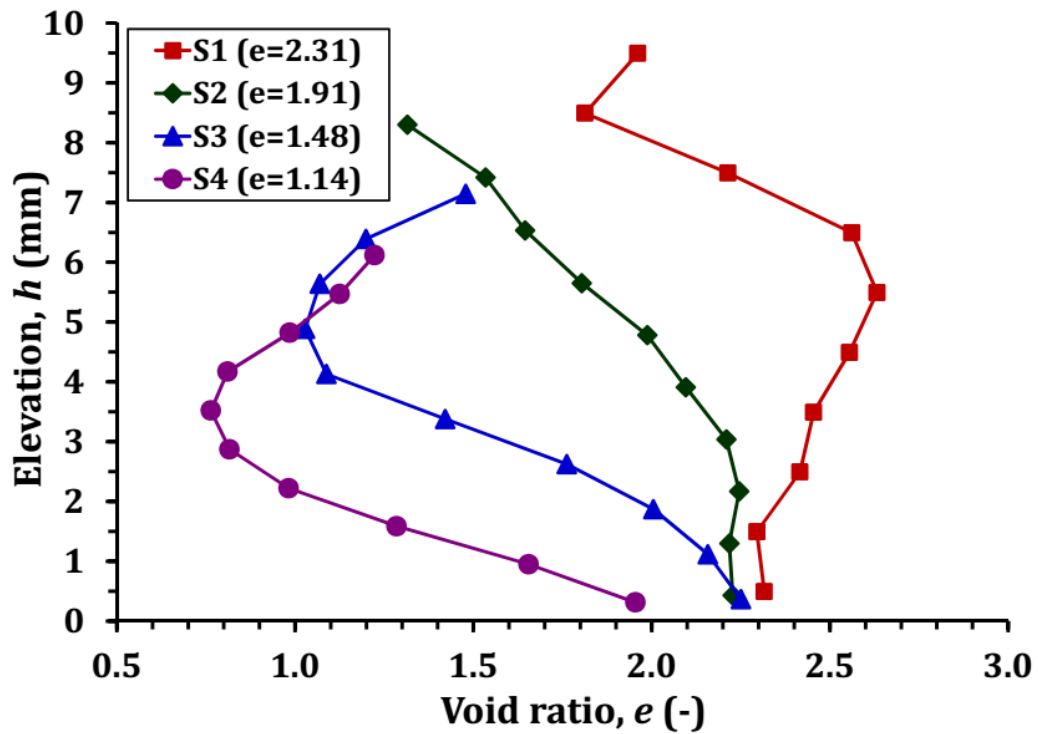
210

211 Figure 5. Grey level histogram for the four scans.

212

213 The void ratio distribution according to the elevation ( $h$ ) during the compression is illustrated  
214 in Figure 6. The void ratio is directly calculated from the tomography images. To obtain void  
215 ratio values, a grey-level threshold value was chosen for each scan, based on their grey level  
216 histogram. This value (around 130) was determined within the interval between the peaks  
217 corresponding to the air phase and the aggregates, respectively. The IsoData method [46] was  
218 chosen for the segmentation process. After thresholding, the aggregates (white) and the air-  
219 filled voids (black) are clearly distinguished. The void ratio is then recalculated by dividing  
220 the number of black voxels by the number of white voxels of the segmented image. Note that  
221 for the four global scans, the void ratios obtained by this method are similar to the imposed  
222 values; the average void ratios of the four curves (2.31, 1.91, 1.48 and 1.14, corresponding to  
223 S1, S2, S3 and S4) are very close to the macroscopic ones, which are 2.29, 1.87, 1.47, and  
224 1.11 respectively. In this study, as the volume of water is smaller than 5% of volume of voids  
225 at the beginning, the presence of water has been ignored during the whole image analysis  
226 process. The binarization and watershed approach are not used because of the low resolution  
227 of the images. The voxel size was 13  $\mu\text{m}$ , for the global scan, that means each grain has  
228 around 7 pixels diameter. In other words, the contact boundary of grains is not clear to  
229 separate. In fact, we had tried to apply the sub-voxel algorithm in order to increase the  
230 resolution of image, especially at the contact boundary but this method increases significantly  
231 the computation time.

232



233

234 Figure 6: Void ratio versus elevation at different compaction levels. Note that the value in the  
 235 parenthesis is the average void ratio.

236

237 In the initial state (S1 – red curve), the void ratio is largest at  $h = 5.5$  mm ( $e_{\max} \approx 2.65$ )  
 238 because of the existence of several large pores in the center and the periphery of the specimen  
 239 (see Figure 4a). Meanwhile the sample is denser at the bottom and densest at the top. The  
 240 higher density at the bottom could be explained by a higher stress induced by the weight of  
 241 the sample. The higher density at the top could be related to the preparation of the sample  
 242 surface. Actually, after having filled the cell with wet grains, the excess grains on the top of  
 243 the sample were removed to obtain a planar sample top surface. Even if this action was done  
 244 with care, that would induce supplementary stress that slightly compacted the top layer of the  
 245 sample. At the stage S2 (dark-green curve), the structure is notably compacted and  
 246 rearranged; void ratio in the middle and at the top is significantly reduced. The structure in the  
 247 middle is remarkably disturbed when passing the stage S2 to S3 (blue curve). At the densest  
 248 state (S4 – dark-purple curve), a strong decrease of void ratio is obtained in the middle ( $e_{\min} =$   
 249 0.7) while the structure at the bottom is slightly rearranged.

250

### 251 **3 Processing for local scan images**

#### 252 **3.1 Principles**

253

254 Based on the local scans, virtual cubical specimens (edge length = 1.95 mm) were extracted to  
255 analyze the grain-scale behavior of the sample during compression. Hereafter, these cubes are  
256 referred to as *Standard Volume*, or SV; one example is shown above in Figure 3c. Ten SVs  
257 were extracted at ten different positions (at the top, at the bottom, and in the central layers;  
258 and for each layer, at the periphery and at the center) in order to consider the sample  
259 microstructure heterogeneity. The subsets did not spatially overlap each other. It is worth to  
260 note that a much higher number of SV, extracted randomly, would be preferable but it would  
261 significantly increase the computational cost.

262

263 The algorithm used to detect spherical structures within 3D images, initially developed by Xie  
264 *et al.* [47] for 3D images, was used and modified following Peng *et al.* [48] (proposed for 2D  
265 images) along with the Adaptive Hough Transform algorithm of Illingworth & Kittler [49].  
266 Note that this algorithm is not applicable for natural materials (i.e. sand grains) where more  
267 advanced methods are required [50, 51].

268

#### 269 **3.2 Algorithm for detecting spherical grains**

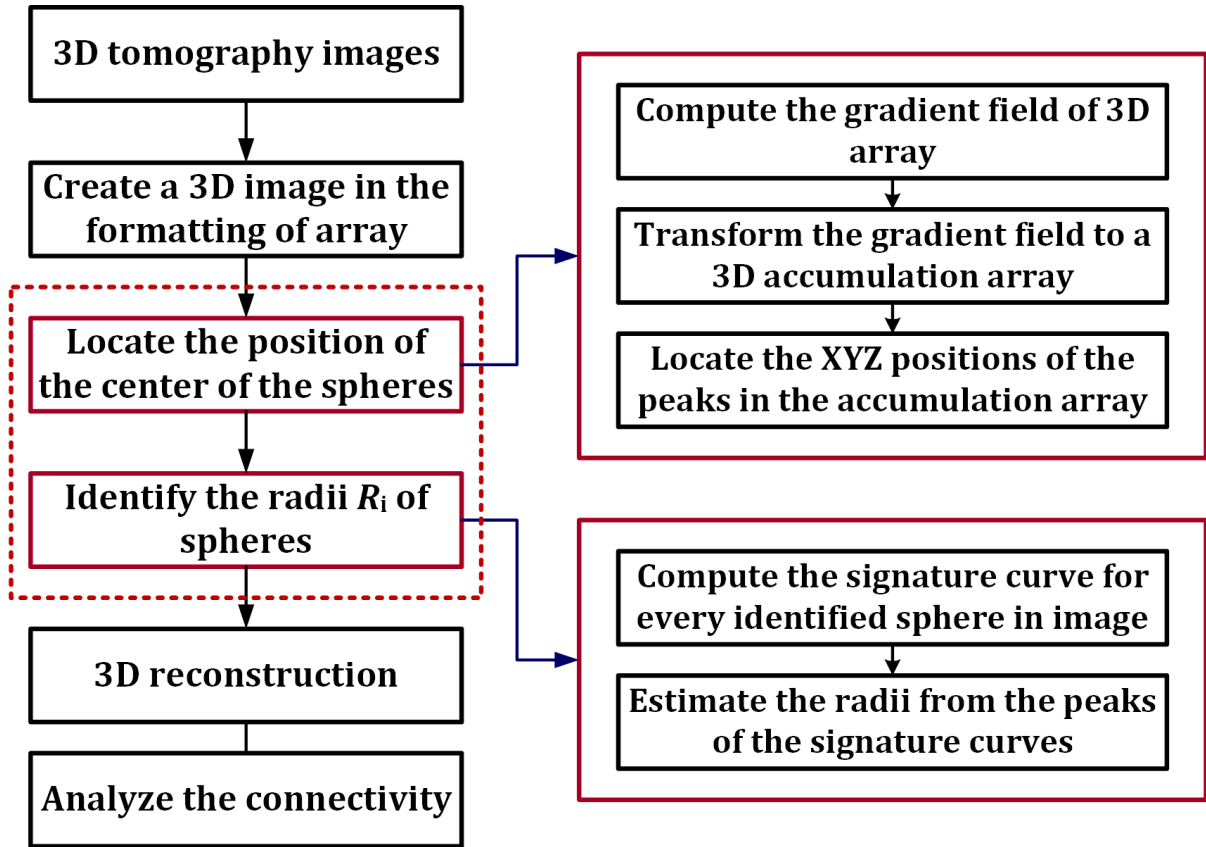
270

271 The algorithm to detect spherical structures is illustrated in Figure 7. A 3D image with a cubic  
272 shape, and formatted as a 3D array, is first extracted from the local scans. The position of the  
273 center of the spheres is then directly located within the 3D array. Afterward, the radii of the  
274 spheres (with identified central position) are identified. Finally, the 3D reconstruction process  
275 and analysis are carried out based on the detected spheres.

276

277 The detailed description of the main algorithm (see dashed-red square in Figure 7) is given in  
278 the following subsections.

279



280

281 Figure 7: Algorithm used to detect the spherical structures.

282

### 283 3.2.1 Locating the center of the spheres

284

285 To compute the center's position of a sphere in the 3D image, the voxels that belong to that  
286 sphere are first identified, and then the centroid of those voxels is computed. The gradient  
287 field of image intensity is computed using the following equation:

$$288 \nabla I(i, j, k) = (V_x, V_y, V_z)|_{(i, j, k)}$$

$$289 = (I(i, j, k) - I(i, j, k - 1), I(i, j, k) - I(i, j - 1, k), I(i, j, k) - I(i - 1, j, k)),$$

290

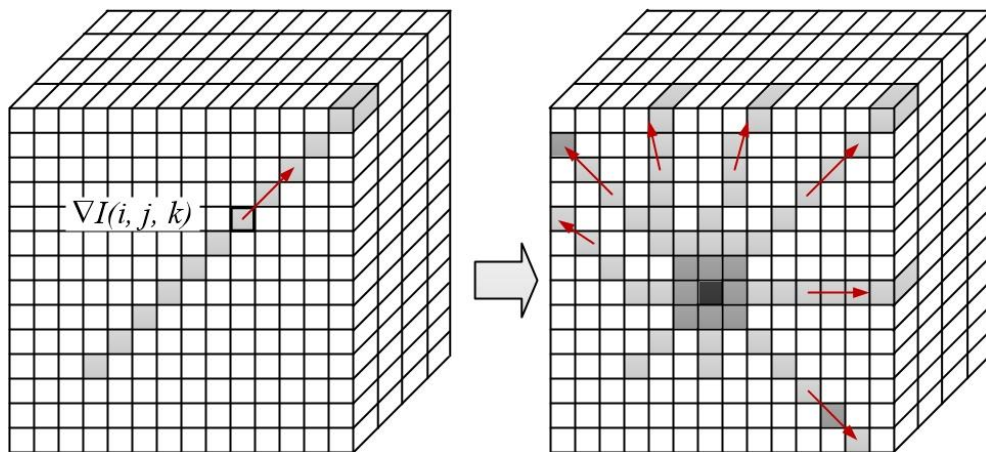
(Erreur ! Signet non défini.)

291 where  $(i, j, k)$  are the voxel indices,  $\nabla\mathbf{I}(i, j, k)$  is the gradient vector at voxel  $(i, j, k)$ , which  
292 consists of  $x, y$  and  $z$  components, and  $\mathbf{I}(i, j, k)$  is the image intensity at voxel  $(i, j, k)$ .

293

294 In a gradient field, the nonzero gradient vectors are either pointing toward the center of a  
295 sphere or away from it. So, a transform is defined to convert the gradient field to an  
296 accumulation array, in which the voxel intensity corresponds to the probability of that voxel  
297 being the center of a sphere. The accumulation array, constructed by an additive process (see  
298 Figure 8), has the same dimension of the gradient field. In the accumulation image, the  
299 maximum intensity represents the center position of a sphere. In the gradient field, a weight  
300 value is added to the voxels which have a nonzero gradient vector  $\nabla\mathbf{I}(i, j, k)$  in the  
301 accumulation array. The line segment in each direction is defined by the vector  $\nabla\mathbf{I}(i, j, k)$ . The  
302 length of the line segment is set to be the possible maximum diameter of the spheres, and the  
303 magnitude of  $\nabla\mathbf{I}(i, j, k)$  is used as the weight value. The accumulation array is built from the  
304 collecting of all nonzero gradient vectors in all directions. In Figure 8, the darkest voxel has  
305 the maximum weight.

306



307

308 Figure 8: Construction of the accumulation array from the gradient field: the nonzero gradient  
309 vector added to the accumulation array (left), and the accumulation array after adding the  
310 nonzero gradient vectors in various directions (right).

311



312 3.2.2 Estimating the spheres' radius

313

314 In order to determine the spheres' radius, a signature function  $f(r)$  is defined over the gradient  
 315 field of the sphere's image, where  $r$  is the distance to the center position of the sphere and  $f(r)$   
 316 is the averaged dot product of the gradient vector and the radial vector.

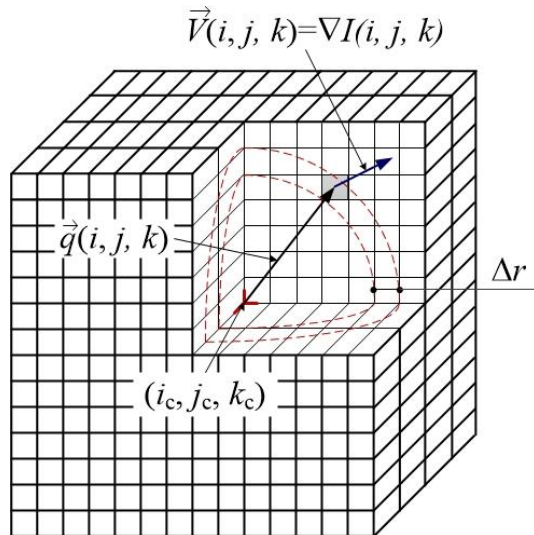
317

318 Figure 9 shows the definition of the signature function, in which  $(i_c, j_c, k_c)$  is the center  
 319 position of a detected sphere. For a voxel  $(i, j, k)$  in the neighborhood of  $(i_c, j_c, k_c)$ ,  $V(i, j, k)$  is  
 320 the gradient vector of  $(i, j, k)$  and  $\vec{q}(i, j, k)$  is the vector joining  $(i_c, j_c, k_c)$  to the center of  $(i, j,$   
 321  $k)$ . A sequence of discrete  $r$  values with fixed interval is selected. It represents the distances to  
 322 the center of the sphere  $(i_c, j_c, k_c)$ . For each  $r$  value,  $f(r)$  is defined by the following equation:

323 
$$f(r) = \frac{\sum_{\|\vec{q}(i,j,k)\|-r < \Delta r/2} [\vec{V}(i,j,k) \cdot \vec{q}(i,j,k) / \|\vec{q}(i,j,k)\|]}{\sum_{\|\vec{q}(i,j,k)\|-r < \Delta r/2} 1}, \quad \text{(Erreur ! Signet non défini.)}$$

324 where the sum is over all the voxels whose distance from  $(i_c, j_c, k_c)$  is within the range  $[r -$   
 325  $\Delta r/2, r + \Delta r/2]$ .

326

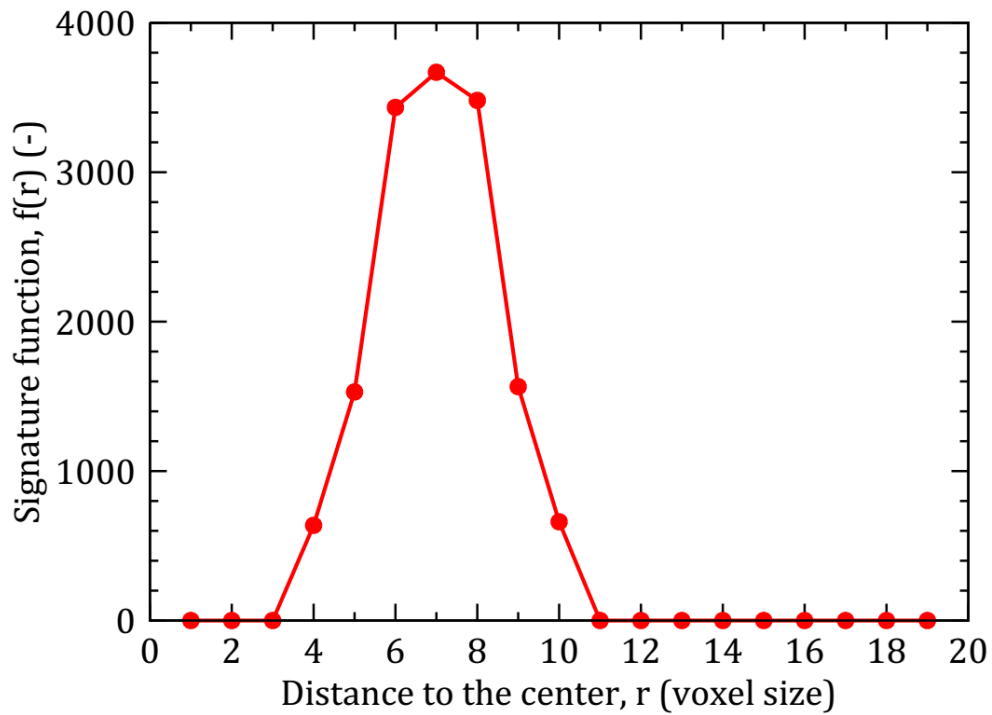


327

328 Figure 9: Definition of the signature function.

329

330 Figure 10 illustrates an example of the signature function. The radius of the sphere  
331 corresponds to the distance to the center presenting the highest value of  $f(r)$ . In the example  
332 shown, the radius equals seven times the voxel size (*i.e.* 45.5 microns). Note that, in the  
333 present work, only rounded values of radius are considered.  
334



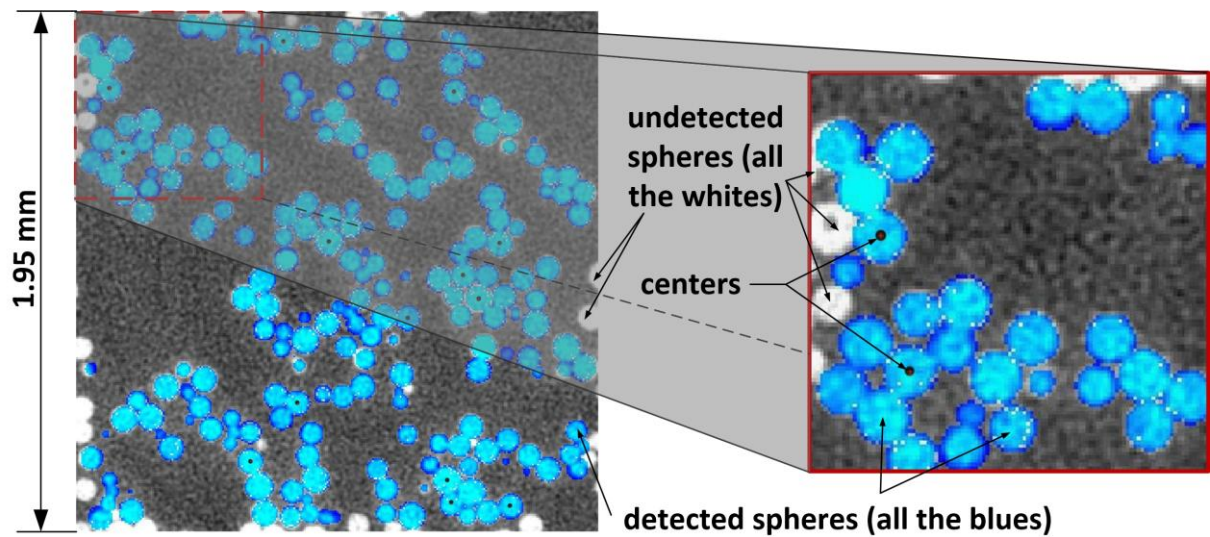
335

336 Figure 10: An example of signature function computed from the 3D image.

337

338 Figure 11 shows an example for one slice in a 3D image. The blue disks are the detected  
339 spheres, and the red points represent their centers. Incomplete white disks at the border are not  
340 used for the detection of the centers.

341



342  
343

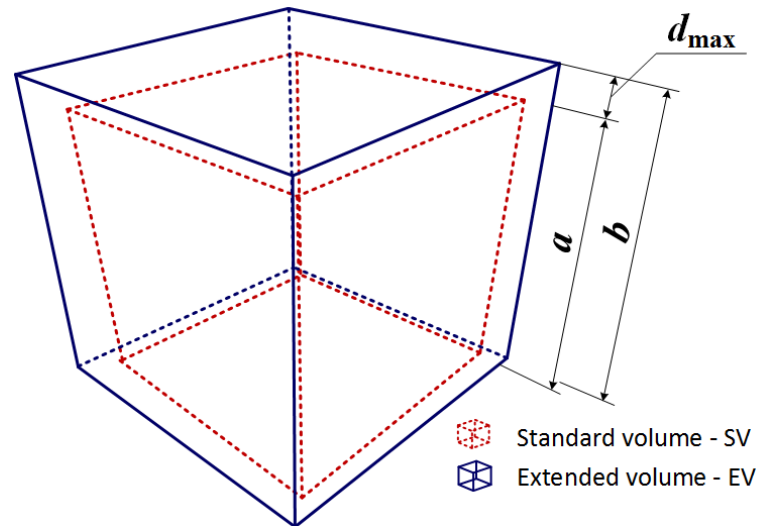
344 Figure 11: A slice in a 3D detected image.

345

### 346 3.2.3 Estimating the lost contacts after the detection process

347

348 As mentioned above, after the detection process, the spheres that intersect the border of the  
 349 cube cannot be identified (Figure 11). Hence, the contacts between these spheres and their  
 350 neighbors inside the SV cannot be considered. In order to estimate those lost contacts, a  
 351 method is proposed as follows. A new cube which is larger than the SV is created. It is called  
 352 the Extended Volume (or EV) with the edge length  $b$  (see Figure 12),  $b = a + 2d_{\max}$ , where  $a$   
 353 is the edge length of the SV,  $d_{\max}$  is the maximal diameter of the detected spheres. The EVs  
 354 are then analyzed with the same input parameters and compared with the SVs. All grains  
 355 inside the SVs and the EVs having the same coordinates are first identified. The grains, inside  
 356 the EVs (but not identified with the SVs) can be then distinguished.



357

358 Figure 12: Method to find the lost contacts; the inner cube is the standard volume (SV) and  
 359 the outer one is the extended volume (EV).

360

### 361 **3.3 3D images reconstruction**

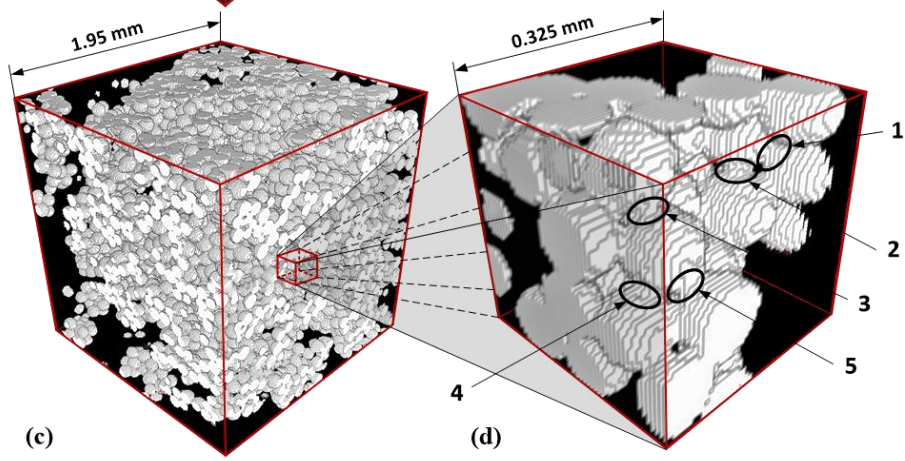
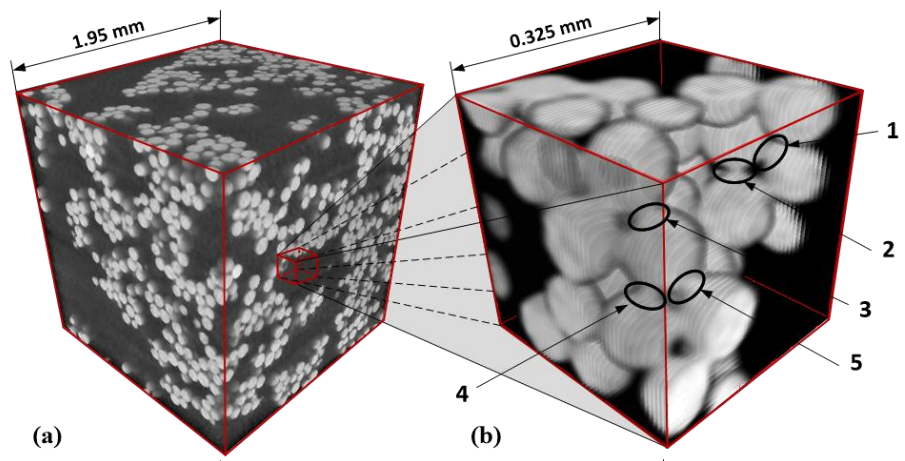
362

363 Figure 13 illustrates an example of 3D images reconstruction for the SV before and after the  
 364 detection. The specimens (a) and (b) are directly reconstructed from 3D tomography images,  
 365 the small cube (b) being extracted from the specimen (a). The specimens (c) and (d) are  
 366 reconstructed from 3D binary images. The specimen (e) is reconstructed from the exact  
 367 positions of particles after the detection process. The small cubes (f) is extracted from (e). As  
 368 the detected radius can only equal to rounded values of voxel size, a tolerance of 1 voxel size  
 369 (6.5 microns that is 7.14 % of the average grains' diameter) is then added to the grains'  
 370 diameter in order to better consider the error related to the detection process. With this  
 371 correction, the images (e) and (f) become (g) and (h), respectively.

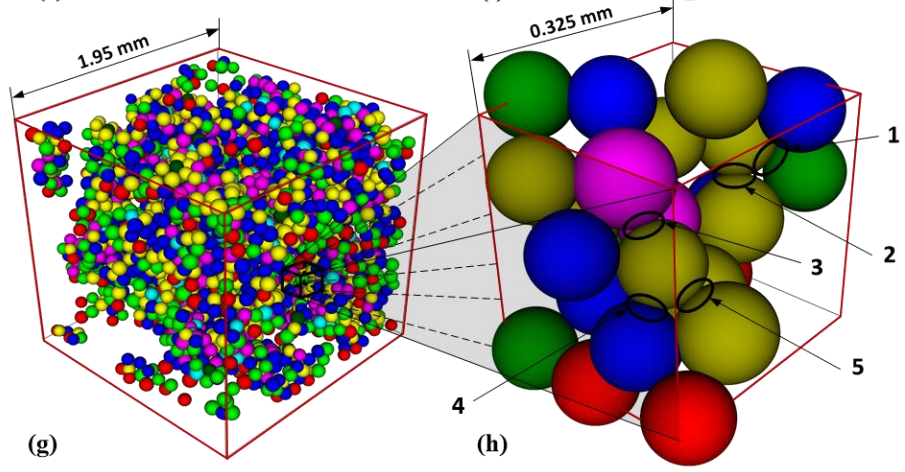
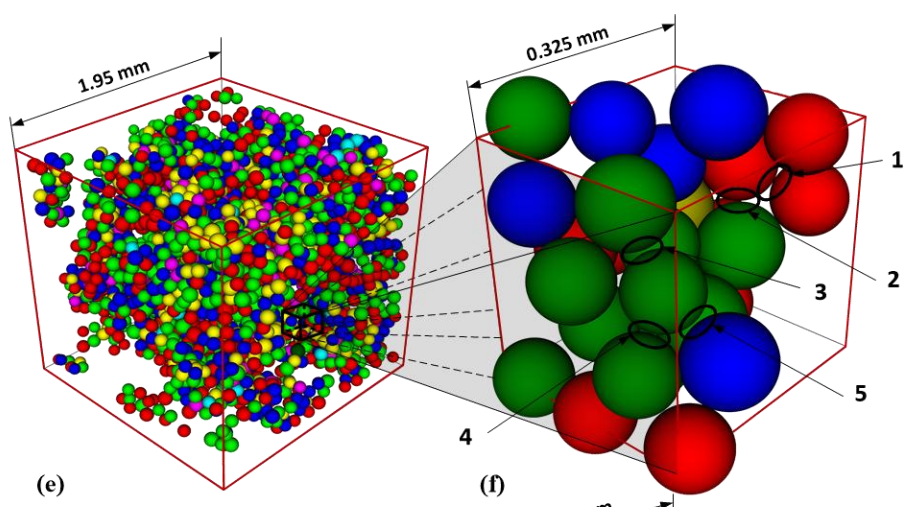
372

373 Figure 13a shows several large pores (up to 1 mm) and stacks of spheres having dimension  
 374 ranging from 0.3 to 0.5 mm. As the average diameter of sphere is 100 micrometers, around 15  
 375 layers of voxels compose each sphere (as seen clearly in Figure 13b).

376



377



378



379 Figure 13: Example of reconstructed 3D specimens: (a, b) Original image - (c, d) Binary  
380 images of the investigated cube; (e, f) Reconstructed 3D image with tolerance of 0 voxel; (g,  
381 h) Reconstructed 3D image with tolerance of 1 voxel.

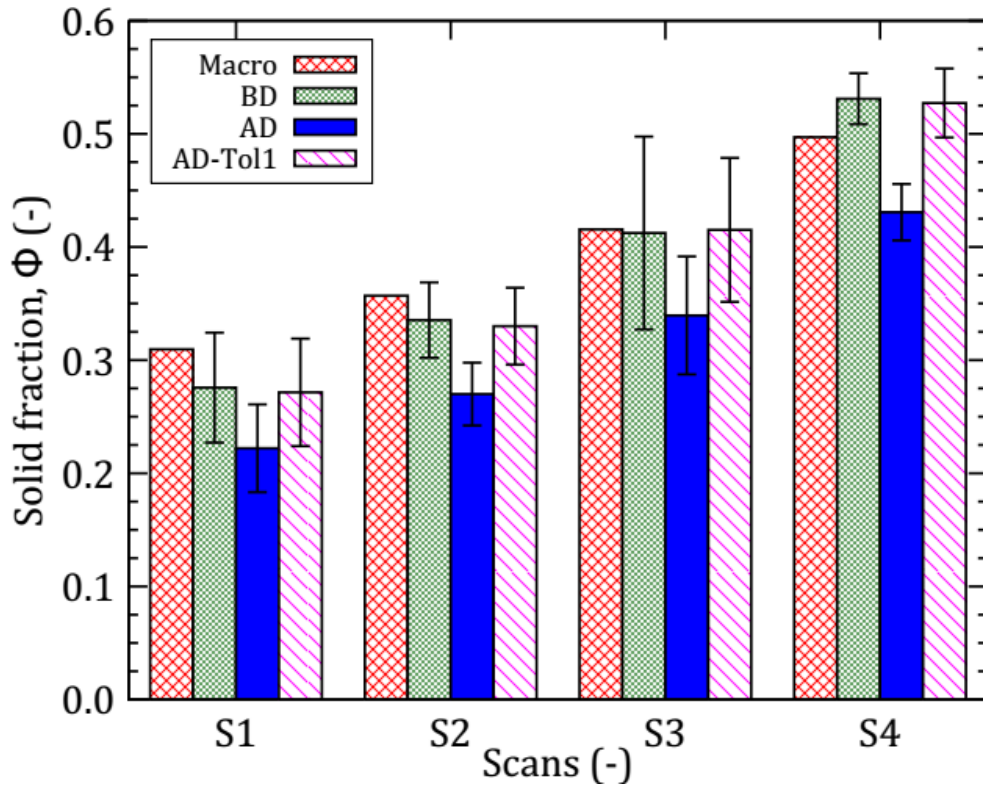
382

383 The contact between spheres is obvious in Figure 13(*b* and *d*). Some typical contacts are  
384 denoted by the positions 1 to 5. The corresponding spheres are kept in contact at these  
385 positions in the specimen after binarization (see Figure 13(*b* and *d*)). However, these spheres  
386 are no longer in contact after the detection process, as shown in Figure 13*f*. After adding a  
387 tolerance of 1 voxel size to the diameter of all spheres, these contacts appear to be similar to  
388 the ones observed before the detection. Clear evidence is shown in Figure 13*h*. In Figure 13(*e*-  
389 *h*), the color of spheres encodes their number of contacts as follows: the red, green, blue,  
390 yellow, magenta represent the spheres having zero, one, two, three, and four contacts,  
391 respectively. Comparison of Figure 13*f* and Figure 13*h* clearly shows the effect of the change  
392 of tolerance on the detected contacts.

393

394 The macroscopic solid fraction and the average solid fraction of ten SVs are illustrated in  
395 Figure 14. The “macro” values (red columns) are computed from the void ratio of the entire  
396 specimen (Figure 2). The green columns (BD) show the average values, which were directly  
397 calculated from the binary images (before detection). The results show that the macroscopic  
398 solid fraction is different from that estimated at the center of the specimen. That can be partly  
399 explained by the heterogeneity of the microstructure of the sample (as shown in Figure 4 and  
400 Figure 6). In addition, after the detection process, the solid fraction is computed by dividing  
401 the volume of all particles by the volume of the cube (blue columns). The solid fraction  
402 obtained after the detection process with zero tolerance (AD) is smaller than that before the  
403 detection. However, with one voxel size tolerance (AD-Tol1), the results (magenta columns)  
404 are very close to the ones before detection (green columns). This suggests that the use of a

405 tolerance of one voxel size for all analyses of the 3D images after the detection process is  
 406 quite suitable. For this reason, in the following analyses, only the detection results with  
 407 tolerance of one voxel size are considered.  
 408



409  
 410 Figure 14: Solid fraction at different compaction levels. AD denotes after detection, BD  
 411 denotes before detection.

412

#### 413 **4 Pore-scale characterization**

414

415 In this section, the pore-scale deformation of specimen under growing of compaction level is  
 416 characterized via the pore-distance distribution. To obtain this distribution, a large quantity of  
 417 points (about fifty times the number of particles) is randomly placed into the pore space of the  
 418 specimen. For each point, the shortest distance from this point to a grain is then identified. In  
 419 other words, this distance is the radius of a “virtual” sphere which has this point as its center  
 420 and has one contact point with a “real” grain. The cumulative probability of the identified

421 distances is then computed. A statistic of the identified distances is finally collected in the  
422 form of a cumulate probability. The pore distance distribution of the specimen at four  
423 compaction levels is illustrated in Figure 15 in which the distance is normalized by the  
424 average diameter  $\langle d \rangle$  as the following equation:

$$425 \quad \langle d \rangle = \frac{2d_{\max}d_{\min}}{d_{\max}+d_{\min}}. \quad (1)$$

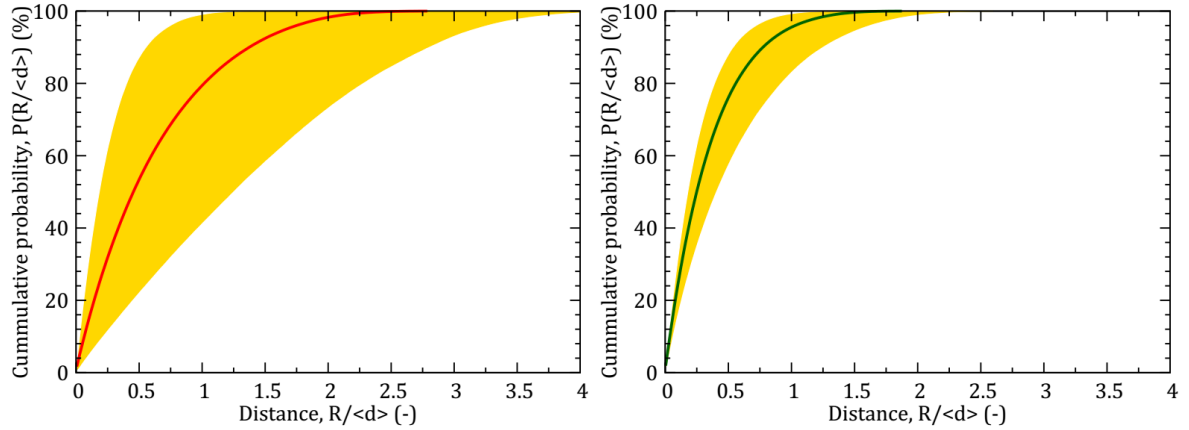
426 Here,  $d_{\max}$  and  $d_{\min}$  are the maximal and minimal diameters of grains, respectively.

427

428 In Figure 15, at each compaction level, the gold region denotes the range of pore distance  
429 distribution (for brevity, hereafter in the section referred to as the gold region) of ten  
430 different positions (ten cubes SV) in the specimen, and the continuous line denotes the  
431 average value of these ten distributions. In general, the pore distances strongly decrease,  $R_{\max}$   
432  $\approx 4\langle d \rangle$  in S1 reduces to  $R_{\max} \approx \langle d \rangle$  in S4, under growing of compaction level. In the loosest  
433 state S1 (Figure 15a), the gold region is rather large. That means the pore structure of the ten  
434 SVs is different from each other. For instance, the pore having size  $R \leq \langle d \rangle$  occupies  
435 approximately 40 % at the lowest boundary while its percentage is about 99 % at the highest  
436 boundary and is about 80 % for the average value; the very large pores,  $R \geq 3\langle d \rangle$ , still occupy  
437 a remarkable quantity (7 %) at the lowest boundary. An example of several large pores can be  
438 clearly seen in Figure 13a. In the denser states S2 and S3 (Figure 15(b and c)), the pore  
439 distance distributions significantly decrease,  $R_{\max}$  reduces to approximately  $2\langle d \rangle$ . In the  
440 densest state S4 (Figure 15d), the gold region is rather small. This means that the pore  
441 distance distribution of the ten SVs is rather similar in this state. The pore distances decrease  
442 twofold from the state S3,  $R_{\max} \approx \langle d \rangle$ . In addition, 99 % of the pores have a radius smaller  
443 than  $0.5\langle d \rangle$  and 50 % smaller than  $0.1\langle d \rangle$  in average. It should be noted that the pore  
444 distribution shown in Figure 15 corresponds to that of the ten SVs, the large voids observed in  
445 larger scale (Figure 4) are not considered. To assess the pore distribution of the whole  
446 specimen (shown in Figure 4), the binary x-ray 3D images can be used.



447

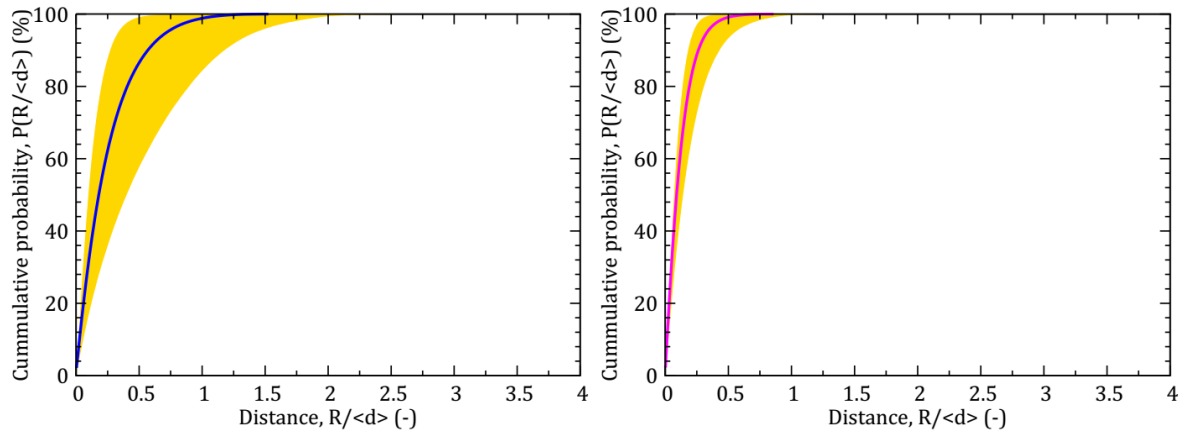


448

449

(a) S1

(b) S2



450

451

(c) S3

(d) S4

452 Figure 15: Pore distance distribution for four compaction levels. In each level, gold region  
453 denotes the values of ten cubes; continuous line denotes the average value.

454

## 455 5 Grain-scale analysis

456

457 The 3D reconstructed images are used in this section to perform grain-scale analysis in the  
458 SVs. As mentioned above, the agreement in solid fraction (Figure 14) suggests that the use of  
459 a tolerance of one voxel size for all analyses of the 3D images after the detection process is  
460 quite suitable. In this section, only the 3D reconstructed images with tolerance of one voxel  
461 size are considered.

462

463 **5.1 Total coordination number**

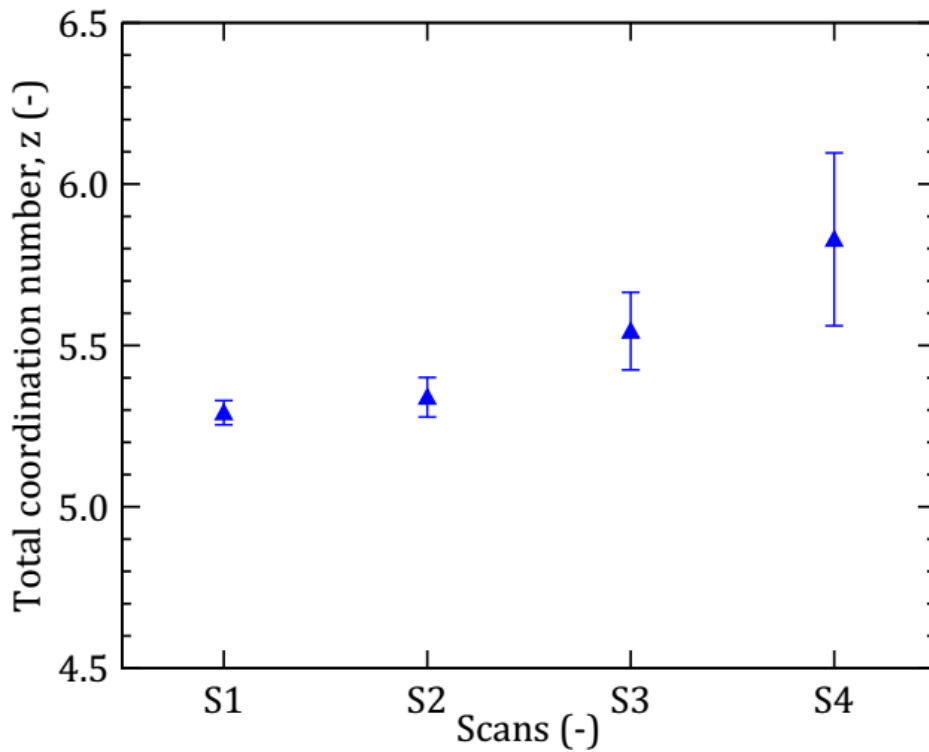
464

465 In granular materials, the coordination number  $z$  is defined as the average number of force-  
466 carrying contacts per grains. If  $N_C$  is the number of force-carrying contacts between  $N$  grains,  
467 then the coordination number is  $z = 2N_C/N$ . In the present analysis,  $N_C$  is redefined as the  
468 number of pairs of contacts between  $N$  grains. Pairs of contacts mean two particles that have a  
469 distance between the two centers smaller than or equal to the sum of their radii. Therefore, in  
470 the present part,  $z$  is called the total coordination number.

471

472 Figure 16 plots the average total coordination number  $z$  for the four compaction levels, S1 to  
473 S4. The error bar denotes the standard deviation of ten values of ten SVs at each scan. In the  
474 loosest state S1,  $z$  equals to 5.29; it increases at higher density and reaches to 5.83 at the  
475 densest state S4.  $z$  remarkably varies at higher density (see the error bars in Figure 16). This  
476 means that  $z$  is significantly different in the ten SVs. In other words, the average value of  $z$   
477 considerably depends on the structure of the specimen. As already shown in Figure 6, under  
478 growing of compaction level, the void ratio in the central elevation decreases nearly twofold  
479 (in the states S3 and S4) while it is negligibly decreased at the bottom elevation. This reflects  
480 a strong rearrangement of the structure. In other words, the central zone of specimen is denser  
481 than the bottom and the top ones. This explains why the  $z$  varies more largely in the states S3  
482 and S4.

483



484

485 Figure 16: Average total coordination number for different compaction levels.

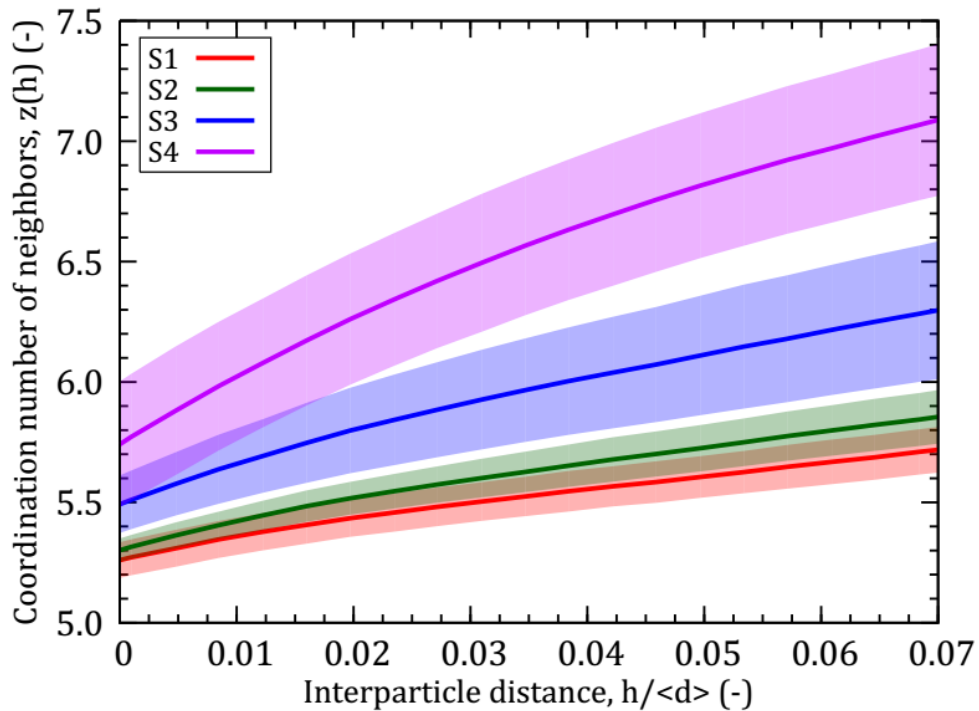
486

## 487 5.2 Coordination number of close neighbors

488

489 The coordination number of close neighbors,  $z(h)$ , is the average number of neighbors of one  
 490 particle separated by an interstice narrower than  $h$ . The interparticle distance  $h$  can be  
 491 normalized by the average diameter  $\langle d \rangle$ . Figure 17 shows, for the four compaction levels,  $z(h)$   
 492 versus the interparticle distance between grains; this distance varies between 0 and 1 voxel  
 493 size (that is  $0 \leq h/\langle d \rangle \leq 0.07$ ). The color region represents the variation of ten SVs, the  
 494 continuous line represents the average value. At  $h = 0$ ,  $z(h = 0)$  is equal to  $z$  (see Figure 16).  
 495 At the loosest state S1,  $z(h)$  increases from 5.29 to 5.75 (about 9 %) while it increases from  
 496 5.83 to 7.16 (approximately 23 %) at the densest state S4. Actually, this increased rate reflects  
 497 the structure of the specimen. This can be explained via the decrease in the pore distance  
 498 distribution, as depicted in Figure 15. Under growing of compaction level, when the specimen  
 499 is compressed, the large pores are strongly decreased (*e.g.*  $R_{\max}/\langle d \rangle = 4$  downs to  $R_{\max}/\langle d \rangle = 1$ ,  
 500 from the S1 to the S4). This means that the specimen becomes denser. That explains why the

501 number of close neighbors at the denser states increases more quickly than the one at the  
 502 looser states. In addition, similar to  $z$ , the variation of  $z(h)$  at the denser states is larger than  
 503 the one at looser states.  
 504



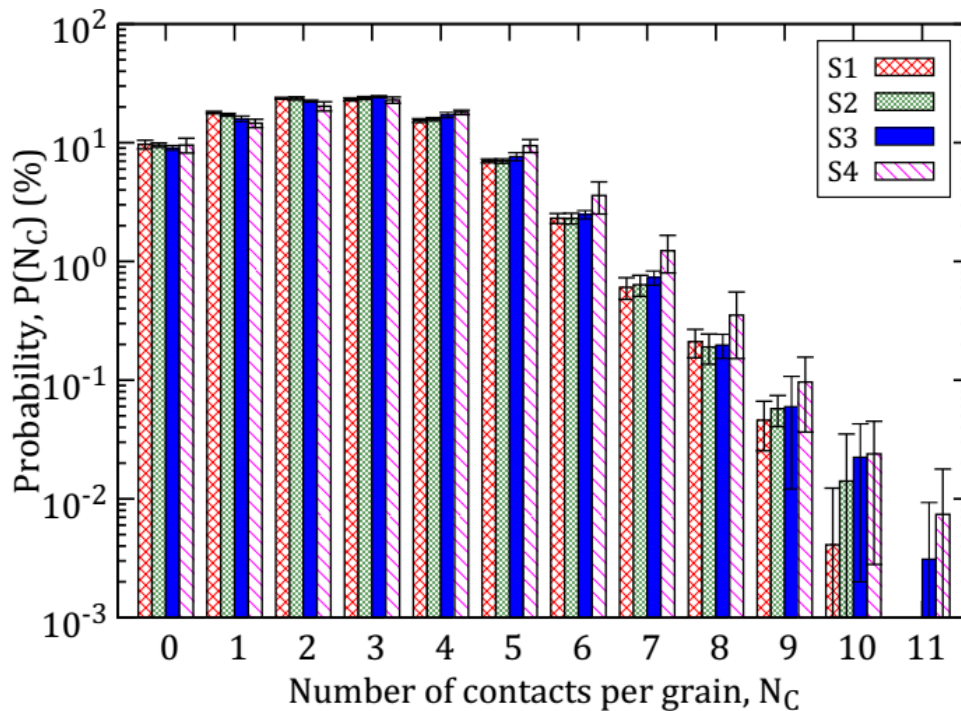
505  
 506 Figure 17: Average coordination numbers of close neighbors for different compaction levels.

507  
 508 **5.3 Number of contacts per grain**

509  
 510 Figure 18 shows the average number of contacts per grain for the four compaction levels. In  
 511 general, the grains have zero contact to eleven contacts, and the fraction of grains having two  
 512 or three contacts are predominant, *i.e.* 20 - 24 % for each of these categories. For the denser  
 513 state, the occurrence of high values of  $N_C$  ( $N_C \geq 3$ ) is higher than in the other states while at  
 514 low value of  $N_C$  ( $N_C < 3$ ), this occurrence is lower than in the looser states. This confirms the  
 515 influence of the granular structure on the contacts of grains. Under growing of compaction  
 516 level, the specimen's structure becomes denser and more contacts are created. Theoretically,  
 517 the maximal number of contacts per grain is 12. In the present case,  $N_C = 11$  is the maximal

518 value and it occupies a minimal probability, nearly 0.003 % (S3) and 0.007 % (S4). This  
 519 probability only occurs in the denser states (S3 and S4) and it is equivalently a few grains  
 520 having 11 contacts.  $N_C = 0$  still occupies a significant percentage, approximately 9.0 % to 9.5  
 521 %. In reality, the grain having zero contact does not exist in the granular structure. However,  
 522 in the present case, a remarkable quantity of the grains having zero contact appears after the  
 523 detection process.

524



525

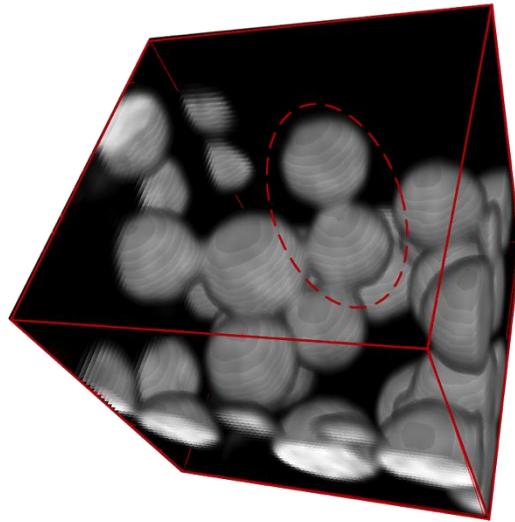
526 Figure 18: Average number of contacts per grain for different compaction levels.

527

528 Interestingly, spheres with one contact can be found in this structure, one example is  
 529 illustrated in Figure 19. This is a small cube extracted from the Figure 13a. For these cases,  
 530 after the detection process, these spheres have either one or zero contact. The presence of  
 531 water menisci and the possible rolling resistance [52] at contact between grains could explain  
 532 this. Indeed, with an average diameter of 100 microns, the particle weight loses relevance  
 533 with respect to capillary forces [53], which could alone stabilize these grains. Besides, as the

534 surface of the grain is not really perfect (see Figure 1), the rolling resistance can be  
535 significant.

536



537

538 Figure 19: An example of a sphere having one contact.

539

#### 540 **5.4 Radial distribution function**

541

542 The radial distribution function,  $g(r)$ , is the probability of finding a particle center in a  
543 spherical shell of a radius  $r$ , given that there is another particle at the center of the spherical  
544 shell [54].  $g(r)$  is related to the average number of particles by

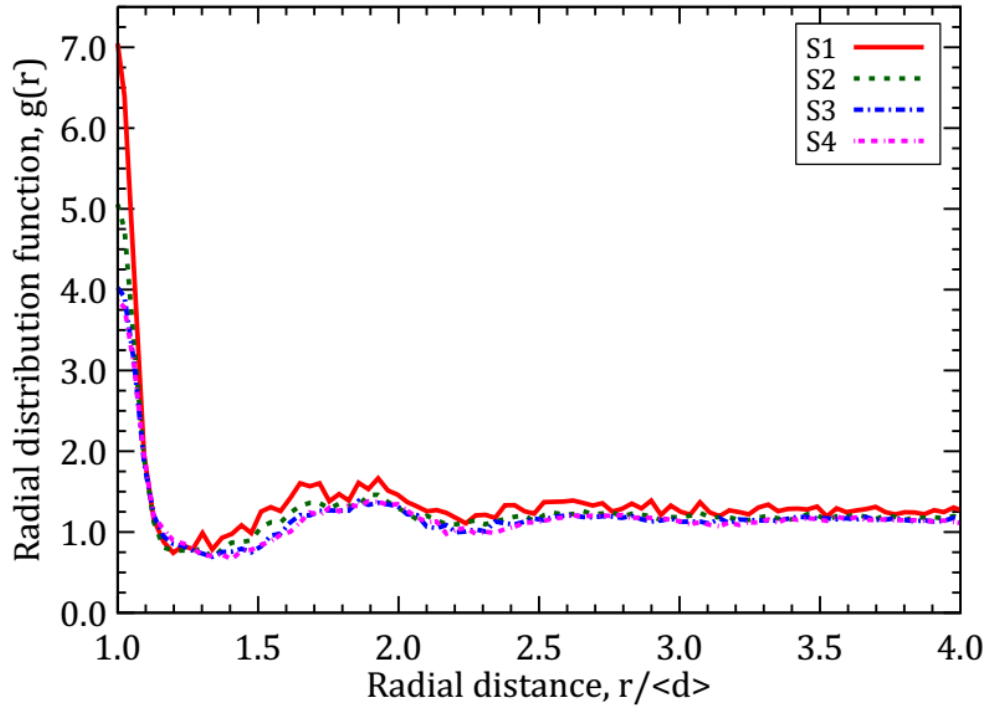
545 
$$p(r_2) - p(r_1) = \int_{r_1}^{r_2} g(r) 4\pi r^2 dr , \quad \text{(Erreur ! Signet non défini.)}$$

546 where  $p(r)$  is the average number density of particles in a spherical shell of radius  $r$ . In this  
547 formula, radial distances are normalized by the average diameter  $\langle d \rangle$  (given by Eq. (3)).

548

549 Figure 20 shows  $g(r)$  for the four compaction levels. The radial distance is normalized by the  
550 average diameter ( $r/\langle d \rangle$ ). The curves for the various states are nearly similar in their  
551 oscillations and peaks.  $g(r)$  starts from a maximum value at  $r = \langle d \rangle$  with different amplitudes;  
552 the loosest system presents the highest amplitude.  $g(r)$  then decreases to minimum probability

553 at  $r = 1.2\langle d \rangle$ ,  $r = 1.3\langle d \rangle$ ,  $r = 1.35\langle d \rangle$ , and  $r = 1.4\langle d \rangle$  for S1, S2, S3, and S4, respectively.  
 554 It increases subsequently to a local peak at  $r \approx 1.9\langle d \rangle$ . From this peak,  $g(r)$  continues to  
 555 fluctuate slightly until it approaches a rather constant value at  $r = 3.0\langle d \rangle$ .  
 556



557  
 558 Figure 20: Radial distribution functions for particle centers of four compaction levels.

559  
 560  $g(r)$  is a useful tool to describe the structure of a system, and it has been used widespread in  
 561 the study of granular materials, especially in the numerical simulations [55, 56]. At very  
 562 short  $r$  ( $r/\langle d \rangle < 1$ )  $g(r)$  must equal zero, because two particles cannot occupy the same space.  
 563 It is clear that  $g(r)$  should go to 1 for large  $r$ .  $g(r)$  shown in Figure 20 goes indeed  
 564 asymptotically to 1 for large  $r$ . As mentioned above, these analyses are performed on the ten  
 565 SVs (virtual cubical specimens with edge length = 1.95 mm). At this scale, the results would  
 566 suggest a representative volume element (RVE) size of about  $3d$  (300 micron). However,  
 567 characterization of the sample's homogeneity, based on the asymptotic behavior method for  
 568 air and solid fractions [57], would give a better knowledge on the heterogeneity of the whole  
 569 specimen (10 mm in height and 20 mm in diameter).

570

571 **5.5 Contact network**

572

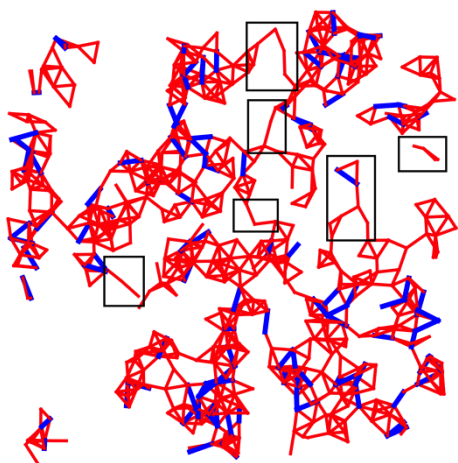
573 Figure 21 illustrates the contact networks (on the left-hand side) and the reconstructed 3D  
574 image (on the right-hand side) of a typical SV (for each compaction level) represented in a  
575 narrow slice having a thickness of three times the grain diameter ( $3\langle d \rangle \approx 40$  voxel sizes). The  
576 contacts between particles are represented by segments of different colors joining particle  
577 centers in two types (on the left-hand side of Figure 21): (i) the red lines link the center of two  
578 spheres in contact; (ii) the blue lines link the center of two spheres having an interparticle  
579 distance higher than zero but smaller than 1 voxel size (6.5 microns).

580

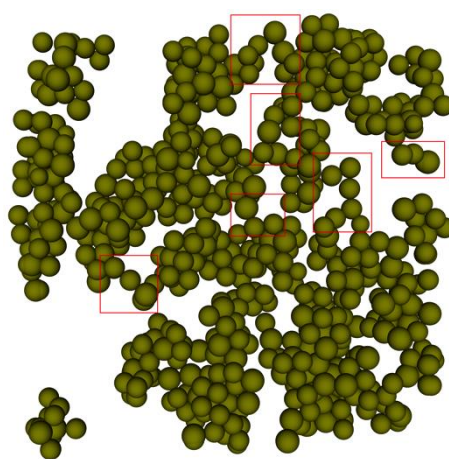
581 Under the lowest compaction level S1, structure exhibits strands (or chains) of single particles  
582 – see the rectangle regions of Figure 21(a and b). These chains still appear in the higher  
583 compaction level S2 (Figure 21(c and d)). This could be explained by the appearance of  
584 rolling resistance at contacts between grains as mentioned above. This resistance tends to  
585 react the collapse of structure under external applied forces. However, under higher  
586 compaction levels, these contacts are broken and the specimen is restructured; The chains of  
587 single particles no longer exist (see Figure 21(e-h)). This phenomenon is also observed in a  
588 numerical study of model cohesive grains using discrete element method [58].

589

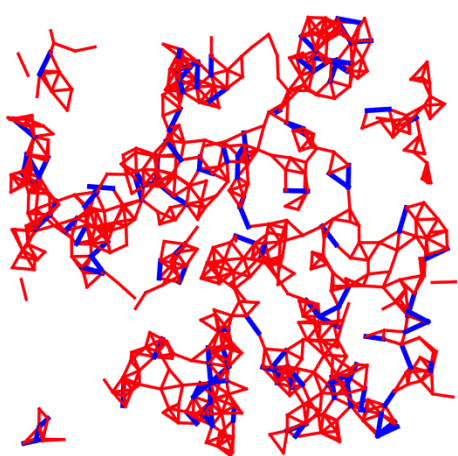




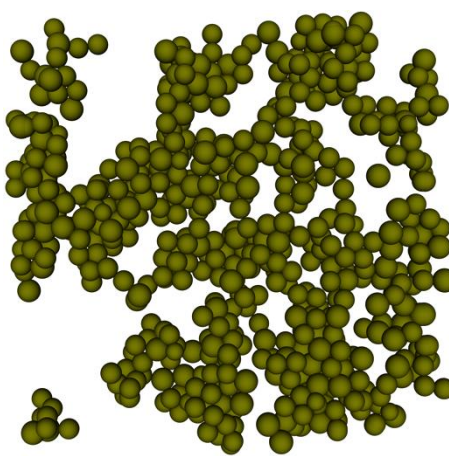
(a) S1



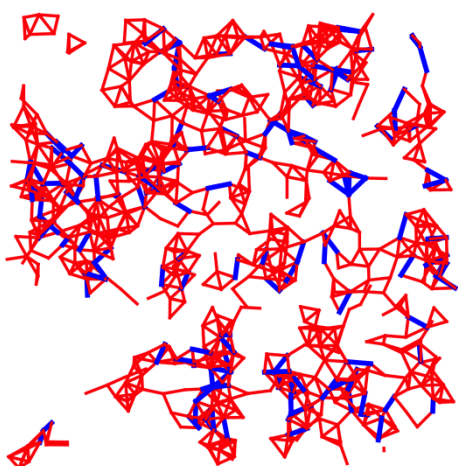
(b) S1



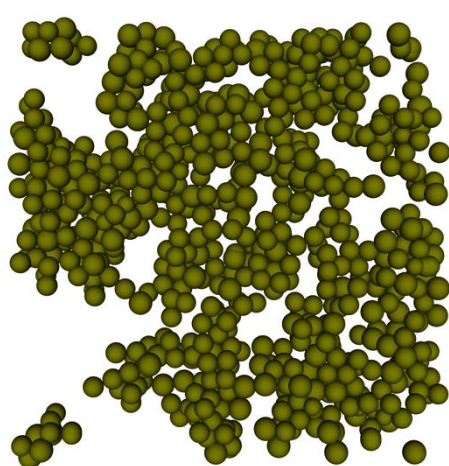
(c) S2



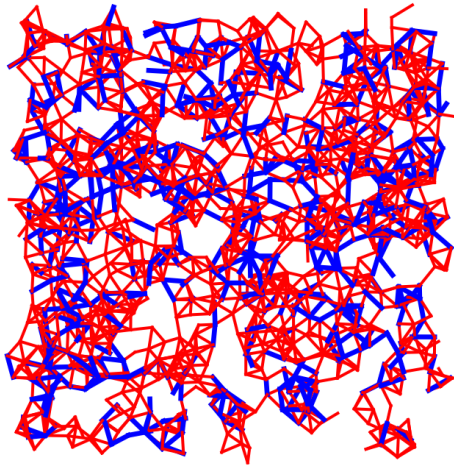
(d) S2



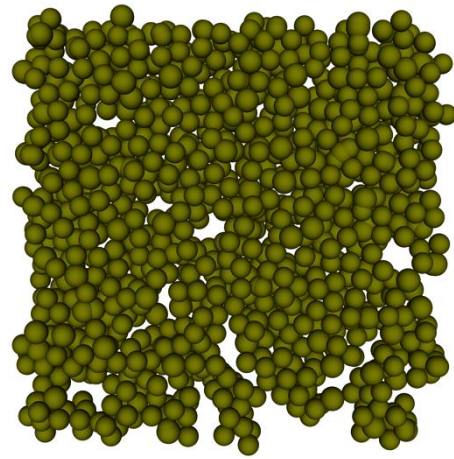
(e) S3



(f) S3



(g) S4



(h) S4

590

591 Figure 21: Contact network and reconstructed 3D image of a typical slice under growing of  
592 compaction level.

593

## 594 **6 Discussion**

595

596 A very loose yet stable-state of wet granular material was produced in laboratory even using  
597 very small water content. Evidently, the structure of the specimen is heterogeneous but it  
598 seems very difficult to make the specimen more homogeneous using such a low density.

599

600 The compression curves obtained show a 3-stages shape (see Figure 2). Following the usual  
601 concept of soil mechanics [5, 59], there are only two stages 1 and 2 on the oedometric  
602 compression curve. Interestingly, the stage 3 is also described in the S-shape curves obtained  
603 on agricultural soils [59]. In this stage, the void ratio is slightly decreased and maintained  
604 nearly constant at high stress (see Figure 2). That confirms the remarkable effect of the elastic  
605 deformation of grains at the contact points.

606

607 In the work of Than *et al.* [12], numerical simulations (by the discrete element method) of a  
608 model granular assembly, made of spherical balls, were used to investigate the influence of a  
609 small amount of an interstitial wetting liquid, forming capillary bridges between two adjacent  
610 particles on the plastic response in isotropic compression. The study was restricted to the  
611 pendular state of low saturation (degree of saturation smaller than 1%), in which the wetting  
612 liquid is confined in bonds or menisci joining contacting grains. By using glass beads of 115  
613  $\mu\text{m}$  (similar to those in the present work), a loose granular structure (void ratio equals 2.30)  
614 was created. Isotropic compression behavior of this structure showed equally a 3-stages  
615 shape. The agreement between the results obtained by the two methods (experiments in the  
616 present works and numerical simulation in the work of Than *et al.* [12]) confirm the key role  
617 of water menisci that stabilize the structure of specimen at very loose state. This statement is  
618 also in agreement with the results of Scheel *et al.* [41] showing a sharp rise of tensile strength  
619 of assemblies of wet glass spheres at very small water content.

620

621 In the present work the position and radius of the spheres were detected by the algorithm  
622 initially developed by Xie *et al.* [47] for 3D images, modified following Peng *et al.* [48]  
623 (proposed for 2D images) along with the Adaptive Hough Transform algorithm of Illingworth  
624 & Kittler [49]. Within this method, integer number of pixels was used to set the radius of  
625 particles, which would induce error on the characterization of the interparticle contacts. The  
626 results would be significantly improved by using the kalisphera-based technique [33] for  
627 sphere-matching in real 3D images of spheres. Besides, detecting interparticle contacts from  
628 x-ray microtomography 3D images by using a greyscale threshold that selects the correct solid  
629 volume would induce also error [60]. For the sake of simplicity, in the present work, the  
630 contacts were detected from the re-constructed 3D images and the above limitations should be  
631 considered when assessing the results.

632

633 The main contribution of the present work would be the links between macro and microscopic  
634 behaviors of specimen under growing of compaction level (Figure 2, Figure 6, Figure 14-  
635 Figure 17). In the stage 1 of the compaction curve (Figure 2), the void ratio remains nearly  
636 constant and slightly decreases at the end of this regime (equivalently the position of the scan  
637 S1). Under growing of stress in stage 2, the specimen structure quickly collapses. This is  
638 evidently reflected by a twofold increase in solid fraction from the loosest state (S1) to the  
639 densest state (S4) (see Figure 14) or by a twofold decrease of void ratio in the central layers of  
640 the specimen (see Figure 6). Besides, the strong decrease in the pore distance distributions at  
641 four compaction levels (see Figure 15) appropriately demonstrates this collapse. In the state  
642 S3, the structure is significantly disturbed. This is obviously confirmed via the largest error  
643 bar of solid fractions at the S3 (see Figure 14), the large gold region (see Figure 15c) and the  
644 void ratio distribution (blue curve in Figure 6). At higher densities, the number of contacts is  
645 larger than the one at lower densities (see Figure 16). In the stage 3 of the compaction curve,  
646 starting from S4, the structure collapses negligibly because of the elastic deformation of  
647 grains at contact regions. This is demonstrated by the highest density of S4 and the slight  
648 increase of  $z$  (see Figure 16 and Figure 17).

649

650 Moreover, the pore-scale deformation of specimen under growing of compaction level is  
651 clearly observed in Figure 15 and Figure 21. Initially, the structure can be stabilized in very  
652 loose state with a significant quantity of very large pores (see Figure 15a and Figure 21(a and  
653 b)). Several marked rectangle regions in Figure 21(a and b) show chains of single particles.  
654 Such a structure cannot exist and stabilize without rolling resistance at contacts [58]. Under  
655 growing of compaction level the structure quickly collapses and the pores size remarkably  
656 decreases. In the densest state S4, the pores having size  $R \geq 0.5\langle d \rangle$  (equivalently the grain  
657 size) occupy a tiny amount (see Figure 15d), and they can be seen in Figure 21(g and h).

658

659 Concerning the grain-scale investigations, several authors reported the distribution of number  
660 of contacts (*i.e.* the coordination number) [27, 35, 61–63] by using different methods to detect  
661 the contacts between the spheres. However, calculating the number of contacts is difficult due  
662 to the uncertainty when calculating the accurate position of the center of the particle,  
663 especially with the small size of grains and low resolution of the tomography images. That is  
664 why the tolerance of 1 voxel size is chosen for all calculations in the present study. This  
665 tolerance is very close to the interval tolerance of  $(1-2)^{1/3}$  voxel sizes used by Aste *et al.* [34,  
666 62] while their size of particles is bigger than that in the present work and the resolution is not  
667 too much different. In several packing studies [35, 38, 62], the spheres are in contact with 11  
668 particles at most and with 2 at least. Meanwhile, in the present study, the maximum number  
669 of contacts per grains is also 11 with a tiny percentage (0.03 % and 0.07 %) but this maximum  
670 value only occurs in the denser states. Besides, there is still a significant percentage of spheres  
671 that have zero (around 9.5 %) contact which should not exist theoretically. As mentioned  
672 previously, the rounded values of the detected spheres remarkably affect the accuracy of these  
673 analyses.

674

## 675 **7 Conclusion**

676 A very loose structure of granular materials (glass beads) is experimentally created thank to  
677 the presence of a small quantity of water forming capillary bridges. One-dimensional  
678 compression tests combined with X-ray computed tomography observations allow addressing  
679 the following conclusions:

680

681 Firstly, the macroscopic behavior of wet beads at loose state (initial void ratio of about 2.30)  
682 obtained from oedometric compression tests has usually three regimes: (*i*) at stresses lower  
683 than the apparent pre-compression stress, the initial structure sustains the applied load with a  
684 very small rearrangement; (*ii*) when the stresses exceed the pre-compression stress, the loose

685 structures collapse and restructure; and (iii) at higher stresses, the elastic deformation of  
686 grains at contact points dominates the specimen deformation. The XRCT observations (global  
687 scans) show that the initial loose structure is heterogenous, consisting of large voids, and its  
688 behavior during the second regime is mainly related to the structure rearrangement around  
689 these large voids.

690

691 Second, the position and radius of the spheres were detected by combining various algorithms  
692 proposed in the literature. Within this method, integer number of pixels was used to set the  
693 radius of particles. From the 3D reconstructed images, several fundamental microstructural  
694 properties are obtained such as coordination number, number of close neighbors, distribution  
695 of number of contacts, radial distribution function, contacts network. Changes of these  
696 properties at various stages of the compression curve allow better understanding the main  
697 mechanisms at grain-scale level that influence the behavior at the pore scale and the  
698 macroscopic scale. Although this experimental approach still needs to be improved, in  
699 comparison with some existing numerical and experimental works, it provides an appropriate  
700 characterization of the grain-scale behavior of wet granular soils at very loose states during  
701 compression.

702

703 Finally, our results emphasize the key role of water menisci that stabilize the structure of  
704 specimen at very loose state as well as the appearance of rolling resistance at contact between  
705 grains. Indeed, the intrinsic compression behavior is due to the collapse of pore's structure  
706 and the rearrangement of grains in the specimen under growing of external force.

707

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709

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714

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