

Agglomeration of powders with a new-coupled vibration-compaction device

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Abstract. Inorganic powder recycling should be a crucial process for the “smart factories” in the future. A complex three-phase system (bauxite mixed with ordinary Portland cement and water) with a new-coupled vibration-compaction device is studied. The compressive stress of compacts seems to be improved by using this device at low compaction pressure leaving the other characteristics unchanged. The tomographic study of macroscopic porosities shows differences in the pores repartitions inside vibrated and untreated compacts. Classic porosity repartition is shown in the classic compacted bauxite compacts whereas in the vibrated-compacted bauxite exhibits inhomogeneities. Despite this, we find these results quite promising for further investigations.

1 Introduction

« Smart Factory » should have production systems ever increasingly efficient with useless by-products. This “eco-efficient plant” should be energy efficient, so recycling fine inorganic particles can be an essential part of this endeavor.

Our aim is to study a new coupled vibration-compaction device in order to produce inorganic compacts made from used or waste powder and reintroducing these objects into current production. Moreover, developing a successful hybrid machine: by combining concrete vibration stirring with pharmaceutical use of high compaction pressures. Densification by compaction is widely used with pharmaceutical (1) ceramic (2) and metallic powders (3). The overall process is quite realistically described and some interesting models are developed (4-6). Vibration is also used for densifying or packing granular media (7,8) to achieve compacities up to 0,7. Recent work on vibrated granular media by X –ray tomography (9) shows interesting measurements of internal local characteristics of packing parameters. Just a few studies are made on recycling by compaction and vibration-compaction (10,11) of inorganic particles.

2 Material and methods

Initially, the characteristics of the powder used to simulate the recycling of inorganic powders will be described. Then all the methods to produce and characterize compacts are detailed.

2.1 Materials

The inorganic powder used is manufactured bauxite referred to simply as bauxite in this paper. We added normal hydraulic binder from Calcia, ordinary Portland cement (OPC).

The helium pycnometry of the two powders obtained with the Accupyc 1330 from Micromeritics gave the density in table 1.

Table 1. Powder densities.

powder	Density : g.cm ⁻³
bauxite	3,74
OPC (cement)	3,19

The bauxite powder density is not so far from pure alumina corundum (3,97 g.cm⁻³). There is only little other oxide inside this powder (Al₂TiO₅). The composition of the OPC is the standard cement used worldwide.

The granulometry of both powders is plotted in figure 2. The bauxite powder is multimodal with a bigger mode at 200 μm and a lower mode at nearly 20μm. The cement is also multimodal with a main mode at 20μm.

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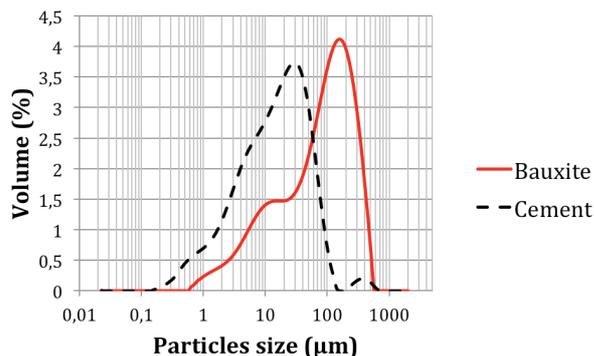


Fig. 1. PSD of the two powders

2.2 Methods

The dry mixture is composed of 85% of bauxite and 15% of cement. Added to this dry mixture is 10% in weight of water. Everything is mixed inside a turbula for 20 minutes and then the wet mixture is inserted into the die.

The Medelpharm' Styl'One Evolution tablet press was employed. This R&D "tableting tool" can develop a 50 kN force. The die dimensions are 23x31x29.5mm. The maximal pressure applied on the powders is 67 MPa. The vibration stage is set to have two frequencies of 40 hz and 60 hz and acceleration from 2 g to 16 g. The duration of this stage is 3 seconds.

After compaction, the cement is hydrated by storing for 48 hours at 5°C with relative humidity at 100% and then maturing for 28 days at 20°C with a lower humidity of 60%.

After this phase, the compacts characterizations: compressive stress, porosity and density are determined. * Compressive stress is provided by a normalized test called NF EN 196-1 on a uniaxial press LR 50 K from Lloyd Instruments. The punch velocity is 10 mm.min⁻¹, the value recorded is subsequently converted into stress using the horizontal area of the compact.

* Porosity is obtained by mercury intrusion porosimetry with the Micromeritics autopore IV 9500.

* Densities are calculated by direct measurement of the weight and volume of the compacts.

3 Results and discussion

The first result is the in-situ vibration obtained by special device made by Medelpharm company. In the next figure the vibration is shown in the variation of within the lower punch strength is zero during this time period.

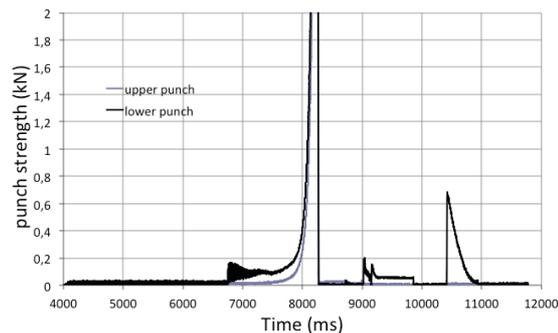


Fig. 2. Vibration just before compaction

3.1. Compacts properties

We only show results for compaction of non vibrated bauxite / OPC mixture, and compaction with 60 Hz vibration and accelerations of 11 and 16 g. The other experiments with lower frequency and acceleration exhibit no real differences from the non-vibrated compaction.

The first characteristic property of compacts is the compressive stress plotted in the Figure 3 versus compaction pressure (Pc).

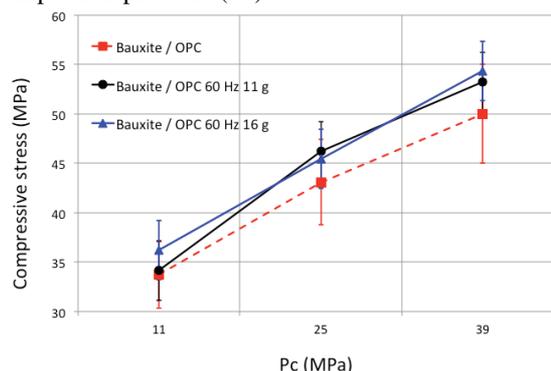


Fig. 3. Compressive stress versus compaction pressure of compacts.

This first result exhibits a small increase in the compressive stress with the vibration stage. For the three compaction pressures a 10% increase is obtained. This is a small but real impact of vibration on mechanical characteristic of compacts.

The densities are shown in the table below.

Table 2. Densities of compacts at different pressures and with and without vibration

Compaction pressure (MPa)	11	25	39
bauxite / OPC	2,29 g .cm ⁻³	2,41 g .cm ⁻³	2,44 g .cm ⁻³
bauxite / OPC 60 Hz 11g	2,28 g .cm ⁻³	2,38 g .cm ⁻³	2,43 g .cm ⁻³
bauxite / OPC 60 Hz 16g	2,26 g .cm ⁻³	2,37 g .cm ⁻³	2,45 g .cm ⁻³

We only can tell that the more acceleration the lower density we have (but not for 39 MPa). After hydration, we have a mixture of portlandite and C-S-H, the density is about $2,3 \text{ g.cm}^{-3}$. For a fully hydrated system we should have $3,4 \text{ g.cm}^{-3}$. So in compacts we have from 33 to 28 % of porosity, this is quite large. The lower density can be explained by both an increase of porosity or a better hydration of the OPC.

Then the repartition of pores diameter of compacts realized with 11 MPa of compaction pressure is shown in the figure 4 below.

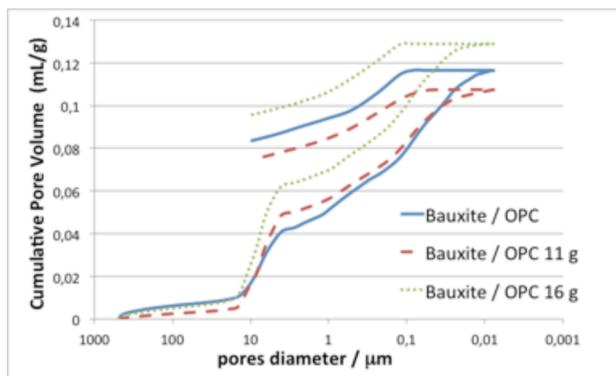


Fig. 4. Mercury intrusion porosimetry of compacts fragments

The porosity is quite the same for the three compacts. We only have a slight difference in the pores volume of the larger pores around $10 \mu\text{m}$.

To explain the differences between those compacts we choose to study in detail the macroporosity inside the compact by using X-ray tomography.

3.2 X-ray tomography

The microtomography experiments were performed in a General Electric Phoenix Nanotom providing an X-ray beam with high-energy voltage on the range between 0 and 180 kV. In the following experiments, 90 kV were used, enabling X-ray to pass through the compacts with a good contrast for the final images. A thin layer of copper was used as a filter of the X-ray beam to reduce the beam hardening effects and neither streaking, shading and ring artefacts were observed in the reconstructed 3D images. 2800 radiographies were taken at different angles all around the 360° and the distance between the sample and the detector or X-ray source was adjusted to get a voxel size of $17 \mu\text{m}$. The resolution in the images was chosen regarding the size of the whole sample and it was enough to get quantitatively information about the grains and porosity from a macroscopically point of view.

3D images were reconstructed (absorption coefficient) using the algorithm included in the Phoenix Nanotom software package. Then the whole 3D image volumes ($800 \times 1600 \times 2000$) were filtered with the median filters were used to enhance the contrast between grains and pores by using the AVIZO software from FEI.

A thresholding procedure was then applied to separate the solid phase from the porous one. We have this porosity first for a bauxite / OPC compact obtained with 11 MPa of compaction pressure. We only have a half compact in this figure 5 to have a better viewing (the top of the shortest thickness side).

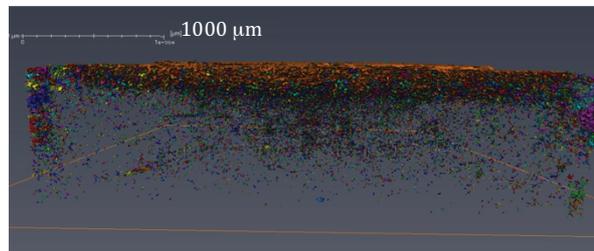


Fig. 5. X-ray tomography picture of porosity of the compact

We have a classic repartition of porosity in the upper part of the compact and in the corners. This tendency is also shown in the models in bibliography (4). We now want to calculate the homogeneity of the porosity. We choose only the bigger pores by labelling the porosity and separate pores by erosion. We only choose the 1000 bigger pores representing 50% of the overall porosity. In the figure 6 we plot the barycentre of all pores in the X axis. The same result is obtained in Y and Z axis.

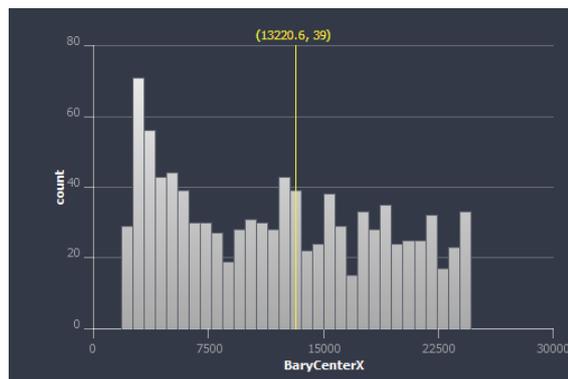


Fig. 6. Global porosity homogeneity inside reference compact

We have in this bar graph the same tendency observed in the picture in figure 5. We have high porosity near the edges of the compacts but the other pores are quite homogeneously positioned inside the compact.

The same procedure of image analysis is done for compact obtained with 60 Hz frequency with the acceleration of 11g and the compaction pressure of 11MPa.

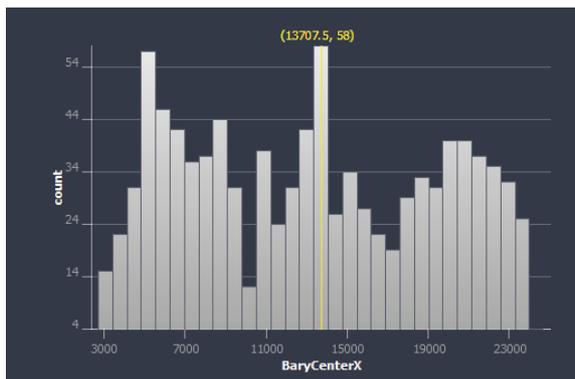


Fig. 7. Global porosity homogeneity inside vibrated compact (11 MPa and 11 g)

For the vibrated compacts it seems to have inhomogeneities. We will confirm this fact by viewing these pores inside the compact (the pores are labelled in colour and the solid phase in grey transparency)

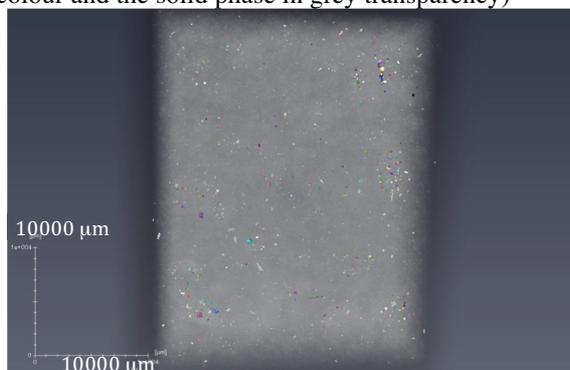


Fig. 8 Global porosity (coloured dots) inside vibrated compact at 11 MPa and 11 g

We do not have small coloured spots that represent pores inside the compact equally positioned but only in some areas. If we only show the pores inside the upper right corner of the compact we have the following pores (figure 9).



Fig. 9 Localised porosity at the right corner of vibrated compacts at 11 MPa and 11g

We have quite the same size and shape for these pores. In the compaction process, we have 10 % of water used to hydrate the cement. Those pores seem to be located where the water droplets were. The high acceleration during vibration may generate pellets with

free water inside that does not react with OPC. During maturation stage, water evaporates and leaves round shape porosity inside the compact as we can see in figure 8.

So despite inhomogeneities and water that do not react with cement we increase the mechanical strength of the compact with the vibration stage, we found this result quite promising.

4 Conclusions

This work on the compaction of complex mixture of inorganic powder with hydraulic binder and water by an in-situ new coupled vibration compaction device show encouraging results. We have a small increase in the mechanical characteristics of compacts with quite the same porosity and density. Macroscopic X-ray tomography study shows inhomogeneities inside the vibrated compacts.

Former studies on other mixture with natural bauxites and different binder may show better results without inhomogeneities and better mechanical properties.

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