Brazil Nut Effect and CONCRETE: Entering *Terra Incognita*

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This paper presents some evidence of the impact of the Brazil nut effect (BNE) on concrete's particulate structure on meso-level (aggregate) as well as on micro-level (cement paste). BNE is associated with long-range size segregation in particle mixtures due to vibration in slurry state of concrete, both near boundaries and in bulk. Presented evidences on meso- as well as on micro-level are coming from experiences in practice and in research, as well as from discrete element modelling (DEM) investigations.

Key words: Brazil nut effect, concrete, aggregate, cement paste, segregation, discrete element method

1 Introduction

This paper will highlight a relatively new and fundamental phenomenon that should or could influence a particulate material like concrete on different levels of the microstructure; a phenomenon that the last decades received quite some interest in journals like Science, Nature and Physical Review Letters, *i.e.,* the Brazil nut effect (BNE). Simply stated, a mixture of nuts in a container that is subjected to vibration at the bottom will segregate as to their respective sizes. In the most common situation, the largest nuts will migrate to the top of the mixture (Fig. 1). Cement grains of a wide range of sizes are in the fresh state dispersed in water between the aggregate grains. During the compaction-by-vibration stage, the surface of an aggregate grain will transmit the vibration to the cement grain mixture in its near neighbourhood. The same holds for the wide range of aggregate particles near the mould or even near reinforcement bars. When BNE influences particle structure, it will also have impact on material properties. Of course, BNE could be exploited only when this phenomenon is completely understood; we are however far away from that situation; we are just entering *terra incognita*. Research in concrete technology is therefore required to investigate conditions in concrete technology under which BNE may occur on different levels of the microstructure, and where it may manifest itself in new types of concrete, in particular in the (super) high performance range. Limited observations in concrete technology that seem to reflect BNE will be discussed in this paper. They are not presented as final proof; they may just be appreciated as a good reason for more systematically starting to explore this topic.

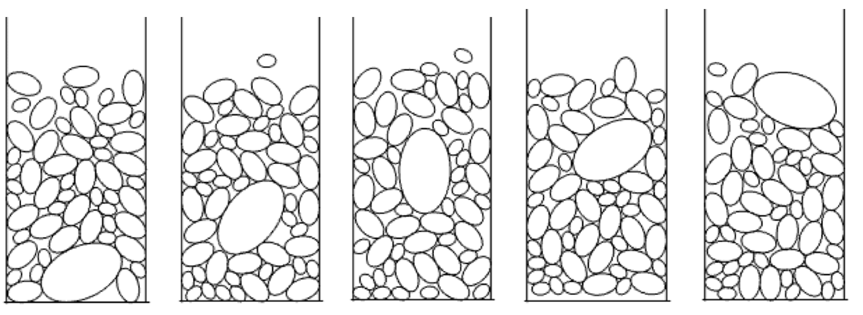

Figure 1. Traditional Brazil nut concept: mixture of non-spherical particles of various sizes is activated by vibration at the bottom of the container. Sequence is displayed of 2D simulations by DEM system HADES during the size segregation process (stills of video).

2 Segregation in concrete

Segregation is a well-known phenomenon in concrete technology: multi-size aggregates when transported over conveyor belts do segregate; bleeding during compaction of fresh concrete is the result of segregation. But no reference is generally made in such situations to BNE. Also wall effects are the manifestation of (size) segregation. This occurs in the aggregate mixture near moulds, potentially leading to surface cracking, or near major reinforcement bars. On a lower structural level, a similar phenomenon gives rise to gradient structures in the binder particles around aggregate grains; gradient structures that persist in the hydrated state (Stroeven, 1999; Hu, 2004) and lead to the formation of the interfacial transition zones (ITZs) (Stroeven and Stroeven, 2001a). As a matter of fact, segregation in aggregate due to differences in size, shape and density is recognized for quite some time in concrete technology. Three-quarters of a century ago, Weymouth (1933) already published a theory on segregation in concrete. The effects of compaction by vibration on aggregate segregation have been extensively studied since those days. But as Khan and Smalley (1973) state "much studied but little understood".

Segregation is made possible by vibration to which concrete generally is subjected in the production stage. Segregation in concrete is generally attributed to effects of gravity in not properly designed mixtures: aggregate tends to segregate in a cementitious matrix, but size segregation is not necessarily involved. Wikipedia defines segregation as: "The separation of constituents in a heterogeneous mixture creating a non-uniform distribution of concrete mixture". A wall effect is the inevitable impact of a nearby wall on the "natural" dispersion of the particles at issue. Theoretically, the wall effect zone will have a thickness of maximum grain size in multi-size aggregate or cement mixtures (Zheng, 2000) and it is solely based on size segregation due to geometric packing laws. In practice, the extent of the wall effect is mostly more restricted.

BNE involves various activation mechanisms (still under discussion in the international literature) leading to long-range size segregation opposing gravity (as in Fig. 1), or not, or even in a direction perpendicular to the gravity field. On meso-level, the wide size range of sand and gravel grains may potentially undergo enhanced size segregation at any rigid surface that can transmit the vibration energy to which the concrete is subjected during compaction. Generally, the aggregate can be compacted in bulk into a dense random state with volume percentages of 70 to 75, or even higher. However, near moulds and even near reinforcement bars relatively long range gradient structures may arise, the extent of which depends on the vibration regime including its duration, on workability of the cementitious matrix and on size, shape, surface texture and on different densities of the particles involved (coarse lightweight aggregate!).

On micro-level, the fresh cement paste is pocketed between aggregate grains, of which average surface-to-surface spacing has been shown of the same order of magnitude as the extent of the interfacial transition zone (ITZ) for density (Chen *et al*., 2006). Volume percentage in the cement paste can be as high as 60 at low water to cement ratios relevant for (super) high performance material. So, packing is far from dilute. In such case, sizes of the cement particles range over two orders of magnitude on the micrometre scale. The present authors are convinced that under the impact of vibrating surfaces of the aggregate particles, the cement particles are liable under favourable conditions to manifest BNE-driven size segregation. What "favourable conditions" are will require elaborate investigations - for the proper exploration of *terra incognita*, so to say.

The advances of BNE in concrete are probably exploited unaware of its very existence. Equally likely is that we also suffered from disadvantages imposed by BNE. Anyhow, impact of BNE on particle structure on meso- or on micro-level, as well as on material properties could be significant, sometimes even dramatic. If so, modifications in material structure (such as by cement blending into hybrid or ternary systems) or in production conditions (omitting compaction by vibration in the case of self-compacting concrete) may lead to undesired side effects. Scientific as well as economic arguments therefore plea for studying the impact of BNE on concrete structure, and the conditions under which it would occur. Enhanced insight would make it possible *designing* modifications. Since systematic research on structural level is very time-consuming, laborious and expensive, an increasing appeal is made on DEM (Discrete Element Method)-produced virtual concrete. This approach will also be employed in this article, whereby use is made of concurrent algorithm-based simulation systems, SPACE (Software Package for the Assessment of Compositional Evolution) (Stroeven, 1999) and HADES (HAbanera's Discrete Element Simulator) (He, 2010; Le and Stroeven, 2012). Compaction by vibration and the resulting particle interferences are simulated in a natural way in such dynamic DEMs. The DEM systems SPACE and HADES produce superior results on particle spacing or dispersion (relevant for the topic discussed herein) as compared to the relatively popular methods in concrete technology, i.e. random sequential systems (Stroeven *et al.*, 2009). Fig. 2 demonstrates the dramatic deviations between RSA and DEM simulations when particle dispersion is important. RSA is based on random generator simulation for which analytical curves are available and used (so, eliminating experimental scatter).

SPACE (developed during the 1990s) is based on spherical grains (Stroeven, 1999). Moreover, inter-particle contacts during the dynamic stage are *impulse-based* and they occur in an infinitesimal small timeframe. Hence, forces between particles cannot exist. As a consequence, force-based experiments, such as densification under pressure, could not be performed. With the newer discrete element package, HADES, particles can be of any shape and contacts are *force-based* rather than impulse-based. The surfaces of objects are no longer described by a mathematical function (such as in case of a sphere), but by a set of interconnected surface elements. Densifications of particle structure by shaking, flow, or impact constitute a few examples of the simulation possibilities (Stroeven *et al*., 2006).

However, after hydration simulation of the binder, the pore network system of concrete can also be investigated (Le and Stroeven, 2012; Stroeven *et al.*, 2012).

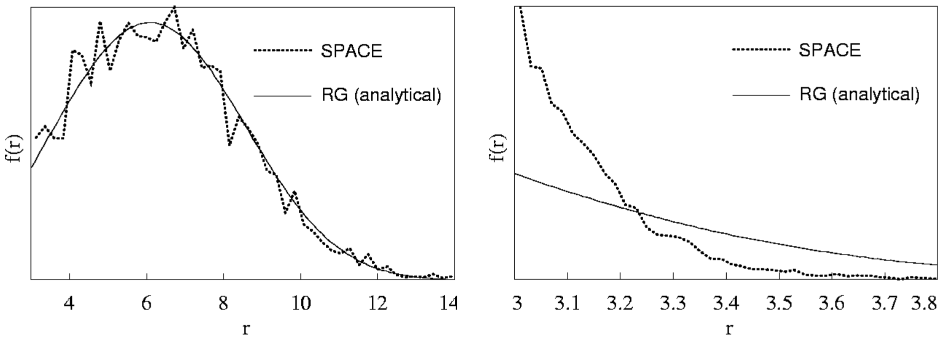

Figure 2. Deviations between predictions on nearest neighbor density distributions, f(r), at 1% (left) and 30% (right) by volume of 3 mm particles obtained by a random generator (RSA system), and by a concurrent algorithm based (SPACE) computer simulation system. For the RSA simulation the theoretical Poisson estimate is used. Already at a volume percentage of 30, the deviations in aggregate or cement grain distributions would be dramatic between RSA and DEM.

3 Structural evidence of BNE

The development of wall effects requires a limited degree of size segregation. The smaller particles in a multi-size particle mixture should be allowed filling up the vacancies left by nearby larger particles that cannot approach the rigid surface closer than half their particle sizes. Of course, the structural gradient merges gradually into bulk value; hence, thickness of the boundary zone can neither easily nor unambiguously be assessed. BNE augments this physical wall effect by more excessive size segregation *driven by various other mechanisms than gravity*. For some observations in concrete practice and in research, where BNE probably influenced segregation, reference can be given to:

- Density gradient in largest fraction of aggregate grains in concrete that was found extending over the full specimen section in research (Stroeven, 1973; Stroeven *et al.*, 2007);
- Practice and researches have revealed that brittleness of high performance concrete (HPC) increases dramatically upon reducing *w/c*-ratio in combination with application of a superplasticizer. This is due to disproportionately enhanced interfacial physical bonding strength as a result of size segregation (Stroeven, 1999; Stroeven and Stroeven, 1999; 2001b);

- A similar phenomenon is observed in HPC when cement is blended with ultra-fine silica fume (Tasdemir, 1996);
- Efficiency of cement blending was found depending on the relative fineness of the secondary component even when *inert* of nature (Detwiler and Mehta, 1989; Goldman and Bentur, 1993). Experiments showed that particle size segregation-enhanced physical (van der Waals) bonding in the ITZ (its extent exceeding wall effect) compensated for the reduced chemical bonding capacity;
- Efficiency of cement blending by a pozzolanic component was equally found relying on so called gap-grading with respect to the cement. Experiments as well as DEM simulations revealed gap-grading in binder components to optimize size segregation (Bui, 2001; Bui *et al.*, 2005).

3.1 Example of size segregation in aggregate due to BNE

Concrete mixtures were designed with the former Dutch standard N 480. Basically, a fine river gravel aggregate (sand) of which all grains passed through the 11.2 mm sieve was mixed with different amounts of 16 mm mono-sized spherical ceramic (steatite) aggregate. The relative density of the steatite was 2.71, which corresponds to the values in the literature mentioned for the type of aggregate used. The resulting sieve curves fell roughly inside the area indicated in the German building code of those days for proper mixtures. The three mixtures were based on 10%, 30% and 50% by weight of steatite in the total aggregate, respectively. Ratios of sand to cement (4.4) and water to cement (0.5) were similar for all mixtures. So, aggregate to cement ratio, and water and cement contents were different. Particularly, the coarsest grained mixture had a very low slump and had to be subjected to vibration for a considerable time for complete compaction on a traditional laboratory vibration table. 250 mm cubes were prepared per mixture. Boundary layers with a thickness of about 1.5 times the maximum grain size were removed from all sides to yield 200 mm wall effect-free cubes for the investigations. One of the cubes per mixture was serially sectioned perpendicular to the gravity direction during production. Tiles with a thickness of about 11 mm provided for 34-38 images per cube.

The distribution of volume fraction of coarse aggregate in the mixture of Fig. 3, at the left, represents a very slight amount of "traditional" segregation. The mixture in Fig. 3, at the right, however , revealed a gradient over the full 200 mm cube height (Stroeven, 1973; Stroeven *et al.*, 2007). This is due to long-range size segregation and will probably originate from BNE. Coarse aggregate has migrated to external sides of the specimen (note that boundary zones with wall effects were removed). So, there are the normal BNE at the top of the specimen and the reversed version at the bottom, where the large grains have segregated in the direction of the gravity field. Note that the tests were executed around 1970. However, the discussions in the literature during the past decade on BNE made us reviewing our results again to find such possible evidences for BNE, indeed.

Although this paper is not going to enter into the extensive discussions during the last decade, or so, on *mechanisms* underlying BNE, it should nevertheless be mentioned that there exists a great controversy concerning the upward or downward segregation in granular materials (Breu, 2003; Huerta and Ruiz-Suarez, 2004; Kudrolli, 2004; Rosato *et al*., 2002; Shinbrot, 2004). To complicate matters even more, also horizontal BNE exists with segregation in either one of two possible directions (Schewe *et al*., 2003; Trujillo and Herrmann, 2003; Schnautz *et al*., 2003). Fig. 4 is taken from Schnautz *et al*. (2003), available on Internet, showing effects of diameter ratio (intruder particle versus particles on a vibrating horizontal surface, with index *s*) and volumetric density ratio. Hence, experimentally observed phenomena depend on various parameters of the particle mixture and on external conditions.

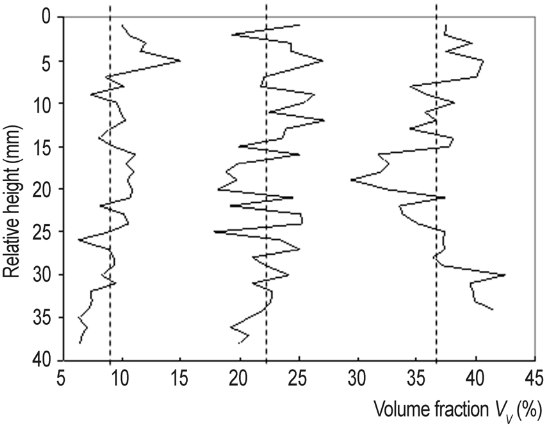

Figure 3. Gradient structures in volume fraction (V_V) of coarse aggregate (dashed lines indicate averages in different concrete mixtures). Top of specimen is at the bottom of the scale.

The concrete cube at issue in the present experiments was long vibrated on the laboratory table (because of the low workability level), whereby bottom and side mould surfaces transmitted the vibration to the mixture, complicating the interpretation of the results because of interferences. Anyhow, the large particles segregated outward.

Fig. 4 demonstrates that for a density ratio of about 1 (both, siliceous aggregate and steatite have a volumetric density of about 2.7g/cm^3) and a diameter ratio of about 2 (which is the size ratio of successive sieve openings in concrete technology!) we are on the transition of moving in either one of the two directions. So, our data on the multi-size concrete aggregate mixture are not contradicting the much simpler situation in Fig. 4. Aggregate to cement ratio was lower in the other two mixes that did not reveal BNE (shown in Fig. 3).

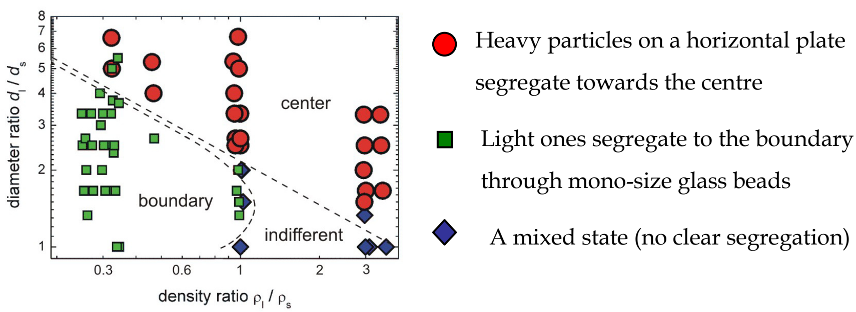

Figure 4. Phase space for particle properties. Each symbol represents one experiment. Shaker amplitude of an horizontal disk is 1.91 cm and frequency 1.67 Hz. Mechanisms were also depending on the area percentage of filling the disk with the glass beads (segregation was not observed at lower values).

*3.2 **Examples of size segregation in cement paste due to BNE***

The structure of the ITZ in concrete has been studied by producing virtual cement in containers with rigid boundaries (Stroeven and Stroeven, 1999; 2001a; He *et al.*, 2007). The rigid surface represents the aggregate grain surface. Gradients can be studied by quantitative analysis of sections parallel to this interface. This can be volume fraction, specific surface area, mean free spacing or grading, which all have their independent gradient structure and different thickness of the associated ITZ. Size segregation in the HPC range (low *w/c*-ratio, high fineness Portland cement) has been demonstrated causing disproportional increase in physical (van der Waals-type) strength in the ITZ (based on mean free spacing measurements)(Stroeven and Stroeven, 2001a; Stroeven and Hu, 2007). This favorable effect will be absent, supposedly, in case of self-compacting concrete. This would be in conformity with random sequential addition (RSA) simulations and theoretical predictions by Zheng

(2000) (ITZ thickness is in all cases equal to maximum grain size). As an example, Fig. 5 (left) reveals bonding capacity increasing (steeply) away from the interface to thereupon gradually decline, finally merging into bulk value at a distance significantly exceeding D_{max} (=20 μm) of the virtual cement grains. The separate grain size fractions have segregated with respect to each other due to wall effect (extending not more than, say, 10 μm) and possibly to BNE (Stroeven, 1999; Stroeven *et al.*, 2007). The composition parameter "volume fraction" does not prove the existence of BNE. However, the configuration-sensitive parameter "grading" (reflected by local particle size distribution, or local "sieve curve") extends as a result of BNE over a handful of maximum grain size diameters before arriving at a stable average (Fig. 5 - right), also probably evidencing BNE on micro-level.

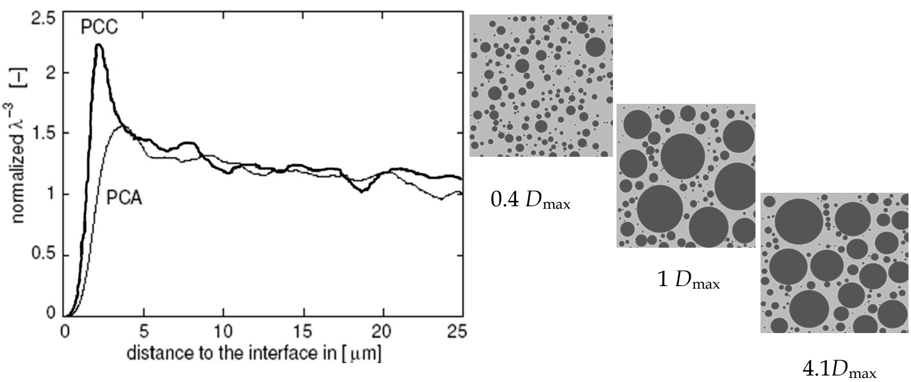

Figure 5. (right) Section patterns through fresh model cement paste parallel to aggregate grain's surface on indicated distances. The model cement paste was simulated by the Discrete Element Method SPACE (Stroeven, 1999). Grading gradient extends due to BNE-driven size segregation far deeper into the cement paste than that of the wall effect (≤ D_{max} =20 μm), witnessing a more extensive ITZ (left). Bonding capacity is assumed proportional to λ^{-3}, whereby λ stands for the mean free particle spacing; w/c=0.2; PCC is fine-grained cement; PCA is coarse-grained cement. Section patterns at the right (at a distance of 8, 20 and 84 μm, respectively, from the aggregate grain surface)

Another example will be discussed of long-range size segregation (BNE) in a DEM (Hades)-produced model gap-graded blended cement paste. In a container with two rigid and four periodic boundaries about 84% of 3-30 μm cement grains were blended by 16% of 1.5 to 2 μm grains of a mineral admixture (supposedly rice husk ash (RHA)). Thereupon, the specimens were hydrated by an improved version of the so called vector approach, IPKM (Navi and Pignat, 1999), to the ultimate degree of hydration of 0.715. The pore network structure was explored for pore continuity by double-random multiple tree structuring (DraMuTS), which is an improved technique of a method used in robotics (LaValle and Kuffner, 2000; Stroeven *et al.*, 2012; Le and Stroeven, 2012). The produced hydrating blended cement paste is shown in Fig. 6.

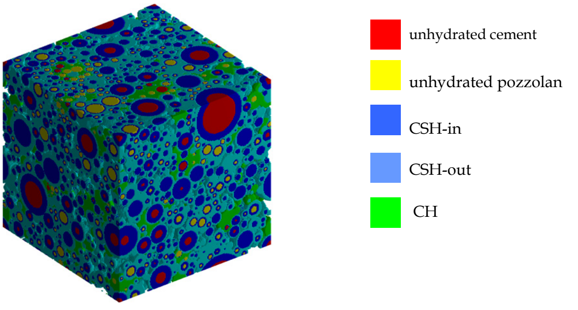

Figure 6. Visualization of the hydrating paste. Each unhydrated multi-phase core is represented by a sphere whose volume equals the total volume of the phases.
(This figure is in colour at www.heronjournal.nl)

Finally, the volume-based pore size distribution was obtained by measuring pore size in uniformly at random dispersed points *inside* the pores. This was accomplished by star volume measurements, a technique used in life sciences (Gundersen and Jensen, 1985). Fig. 7 reveals significant pore refinement in the ITZ due to disproportionately large numbers of RHA particles that have migrated over long distances to the ITZ (Stroeven *et al.*, 2012; Le

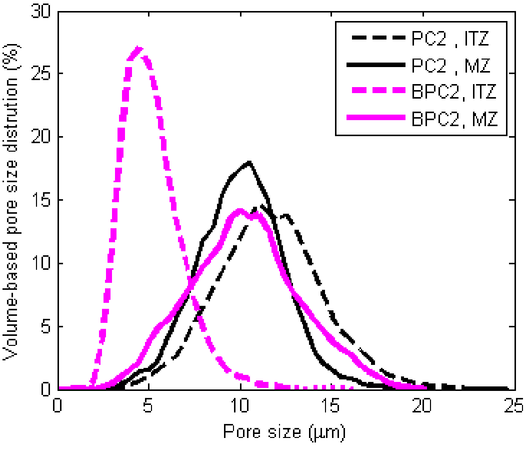

Figure 7. Pore size distribution functions for plain (PC2) and blended Portland cement (BPC2) pertaining to the ITZ (dashed line) and to the middle zone (MZ) between the ITZs (continuous line).

and Stroeven, 2012). Fig. 7 is the result of the afore-mentioned DEM packing and hydration simulation processes for (blended) cement. Application of DraMuTS renders possible studying topology and geometry of the pore network structure. Pore size in the ITZ is somewhat larger than in the middle zone in plain PC. Gap-graded blending has resulted in long-range segregation of the finer particles to the ITZ and the larger grains to the middle zone. This results in a significant pore size refinement in the ITZ.

3.3 Workability influence on BNE-driven size segregation

Influences of common particle characteristics like density (lightweight concrete!) and shape, in addition to effects due to production conditions (frequency, amplitude and duration of vibration) have been covered for dry mixtures by Khan and Smalley (1973). Here the effect of workability will be illustrated that distinguishes concrete from dry aggregate mixtures. In fact it confirmed the established effects investigated in dry mixtures; however, energy dissipation in the slurry required prolonged vibration. A virtual container with two rigid surfaces and four semi-periodic ones was filled in the center by the larger cement

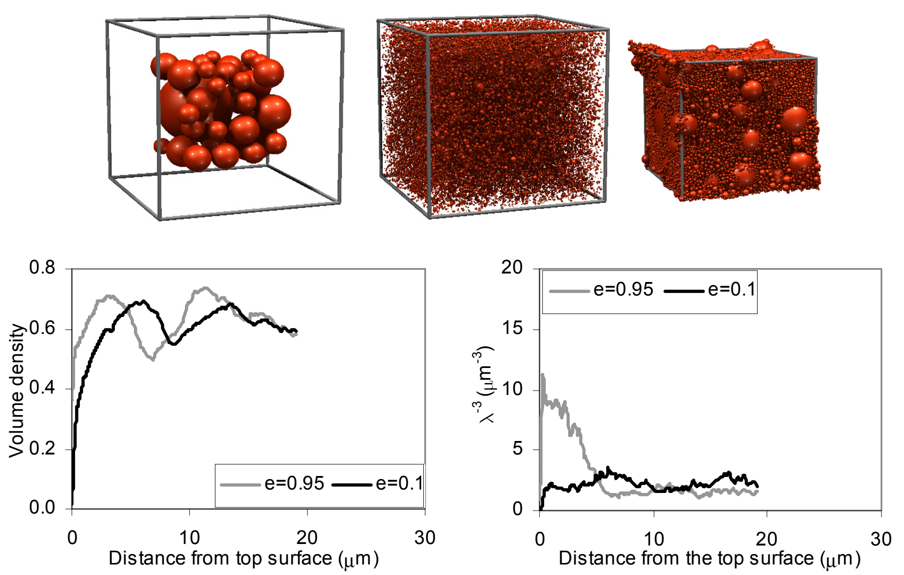

*Figure 8. Effect of workability conditions on volume density (left) and internal bonding capacity (right) of fresh binder with discontinuous grading. The parameter **e** represents in SPACE the energy dissipation during compaction; high value of **e** corresponds to better workability. Internal (van der Waals) physical bonding is supposedly proportional to λ^{-3}, whereby λ stands for the mean free spacing (Stroeven, 1999). Note the dramatic drop in λ^{-3} due to incomplete exploitation of BNE; particles need long-range segregation (BNE) for full exploitation!*

particles, while the outer part of the container was filled by the finer particles (represented by the two cubes in Fig. 8 at the top left). Next, the particles were agitated in the DEM system SPACE, whereupon they moved and collided in the container that was gradually reduced in size until the desired volume fraction was obtained, corresponding to the designed water to cement ratio (Fig. 8, top right). Details of these dynamic algorithms are described in the open literature to which the reader is referred (Stroeven, 1999; Stroeven and Stroeven, 2003; Stroeven and Guo, 2006). Results are plotted in Fig. 8 at the bottom.

4 Conclusions

BNE can be expected in particulate materials like concrete; conditions required for occurrence should still be experimentally assessed, of course. Such particulate materials will always reveal size segregation of limited extent near walls on different levels of the particulate structure, and sometimes may suffer from (normal) segregation (Safawi *et al*., 2004). For vibration-compacted concrete, some of the BNE-induced changes in the structure of concrete may be favourable. The long-range size segregation in gap-graded blends may increase density and particularly improve interfacial bonding capacity inside ITZs. Wall effects might do the same but only in an inner ring of the ITZ. The resulting denser fine-grained particle packing will also promote a refinement in the pore network structure in the ITZ, which in normal concrete constitutes the more porous zone (Hu *et al*., 2006; Stroeven *et al*., 2012). So, the HPC development - where high performance stands for strength and durability – may profit from the existence of BNE. The same can be concluded with respect to cement blending by a mineral admixture that is finer grained than the PC. This can additionally promote sustainability, particularly when the mineral admixture is obtained from a vegetable waste like rice husk ash. This would in all cases be due to relatively long-range size segregation that is inherent to BNE. Research is needed to confirm and generalize the present conclusions that are based on only limited evidences. Seeing the differences of opinion inherent to BNE research on mostly simpler set ups than would be encountered in concrete (see references in the literature list, to which can be added more publications in *Powder Technology*, such as Ripple *et al*., (1973)) inevitably leads to the conclusion that such research focussing on BNE in concrete will be complicated and probably also leading to various opinions: our *terra incognita* will not easily open up.

Yet, systematic research is necessary for the exploration of *terra incognita*. Starting is almost at scratch: search the Internet for "BNE and Concrete" and you will find not much more than Stroeven and He (2011). So, systematic research should add the necessary details and depth to the modest and sketchy voyage of discovery conducted herein. This should ultimately render possible *exploiting* BNE in the design of technological developments in the field of concrete technology.

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