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Internal erosion of chemically reinforced granular materials: A granulometric approach

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Internal erosion (IE) of chemically reinforced granular materials results in dislodgement of particles from their rigid skeleton due to seepage stresses. Whether or not these particles can be dislodged and flushed out by seepage depends on (i) the amount of binding agent used to reinforce the granular matrix, (ii) the structure of pore network and (iii) severity of seepage stresses. Results of IE tests are presented for compacted-sand reinforced with increasing amounts of silica gel. The silica gel is a binding agent that permits low-permeability materials reinforcement. IE tests provided information about eroded particles, macro/micro-structural changes and indicators of ongoing IE mechanisms. For instance, data highlighted the existence of up to three IE stages. This includes flushing of movable particles, binder removal and subsequent releases then self-filtering of fine particles within the rigid skeleton. From the careful monitorings of effluent particles (in terms of concentrations and sizes), it was possible to gauge the dynamics of binder removal. Besides, it was also possible to follow changes in dimensions of the smallest constriction that drives travel distances of dislodged particles.

Keywords:
Internal erosion
Reinforced granular material
Tests
Indicators
Constriction

1. Introduction

Erosion is ubiquitous in the natural and urban environments. Surface soils of the earth have been continuously changing as erosion sculpts the landscape. It reduces mountains to a fraction of their original size and carves out valleys and canyons between cliffs. Man-made infrastructures that support society, like dams or levees as well as agricultural lands also experience the processes of erosion and transport of detached material. For instance, in 2005 during Hurricane Katrina, the levee failures in New Orleans, USA, submerged 80% of the city causing death of >1100 inhabitants (Jonkman et al., 2009). The Federal studies that were initiated to provide answers about structures vulnerability stated that most levees sections that performed well were made of less erodible materials (Andersen et al., 2007; IPET, 2007).

Among all the erosion forms, internal erosion (IE) is especially dangerous since it is one of the most common causes of failure of embankment dams (Sherard et al., 1972a, 1972b; Arulanandan and Perry, 1983; Foster et al., 2000a, 2000b; Foster and Fell, 2001; Wan and Fell, 2002, 2004; Ronnqvist, 2005; Fell and Fry, 2007; Reiffsteck, 2007; Fox and Wilson, 2010). Four types of mechanisms have been identified in this context (Bonelli and Brivois, 2008): (i) development of defects in the primary fabric of large

particles that support imposed stresses (like grain removal inducing stacks faults or dislocations), (ii) suffusion, which is the process whereby the fines fraction is entrained internally through the primary fabric of large particles, (iii) elongated cavities or channels that are eroded backward toward the reservoir, and (iv) contact erosion, which occurs at the interface between two solid domains of distinct granulometric distributions provided the coarse layer is not appropriate filter for finer layer. Actually, there may be no apparent geomorphic evidence, or only subtle evidence (such as minor cracks, slides, and depressions), that one or several IE processes are taking place. Moreover, a dam may breach within a few hours after evidence of the IE becomes obvious: as a spring discharging at the downstream toe of an embankment. Therefore, it is relevant to monitor changes in physical characteristics of the solid structure at an early stage of erosion. Variations of pore conditions (e.g. like the water content and permeability) and dynamics of particles fluidization (i.e. dislodgement then transport) are expected to trace the processes that control IE.

So far, research on the IE mainly focused on low cohesion or cohesionless granular materials like sand, gravels and diverse non-cohesive soils (US Army Corps of Engineers, 1953; Loebotsjkov, 1969; Arulanandan et al., 1980; Kovacs, 1981; Kenney and Lau, 1985; Burenkova, 1993; Reddi et al., 2000; Bendahmane et al., 2008). The data from the literature showed that the potential for instability of low cohesion/cohesionless materials depends on the ability of their small particles to be flushed throughout the

constrictions of their pore network. However, unlike cohesionless granular materials, the reinforced granular materials are expected to endure seepage stresses even though their primary fabrics of particles prove to be unstable and/or their small particles in pores partially flushed out (Tam, 1996). Hence, granular material reinforcement has brought about a plethora of engineering applications: e.g. soil stabilization, slope protection, tunnels and underground excavations (Pitt et al., 2007). In this study, we performed IE tests on reinforced compacted-sand columns (CSCs) to probe the mechanisms inherent to IE in these particular granular materials. This paper, which is restricted to chemically reinforced CSCs, has three objectives. First, probing IE stages within CSCs reinforced with increasing amounts of stabilization agent. Second, the monitoring of particles released into the effluent with respect to their granulometry. Third, investigating the microstructural changes induced by seepage.

2. Material and methods

2.1. Internal erosion experiments

Three IE tests were performed at a constant inflow rate (0.1 L min^{-1} , fluid velocities around 10^{-3} m s^{-1}) from the bottom to the top of a transparent tube of height 0.1 m and 0.1 m in diameter filled with reinforced compacted Fontainebleau sand (Fig. 1). IE tests are different according to the reinforcement of the CSCs (i.e. amounts of binding agent). During every IE test, the weight of the CSC, the injection pressure (P_{inj}) and the granulometric distribution of eroded particles (from 0.01 to 100 μm) were measured at different time intervals. The experimental program also included a series of scanning electron microscope (SEM) micrographs.

2.2. Sample preparation

The Fontainebleau sand is a reference granular material that consists of sub-rounded grains with intermediate sphericity and a grain size range of 50–400 μm . Its particle and bulk densities are $\sim 2600 \text{ kg m}^{-3}$ and $1500\text{--}1600 \text{ kg m}^{-3}$, respectively, with an internal friction angle of 40–45° and a cohesion of 70 Pa (van Mechelen, 2004). A colloidal dispersion of high molecular polysilicic acids was used to reinforce the sand matrix. Indeed, preliminary IE tests done on unreinforced Fontainebleau sand columns highlighted the prompt destabilization of their granular structure (see below). Due to its colloidal nature and its high water content ($\sim 70\%$), the dispersion allows the reinforcement of low permeability materials ($< 10^{-10} \text{ m}^2$). The colloids size lies between 5 and

75 nm and the transformation of the colloidal silica dispersion into insoluble silica gel (i.e. the binder; $\sim 1300 \text{ kg m}^{-3}$ particle density), upon addition of a saline solution, is irreversible. The gelation time was set to $\sim 1 \text{ h}$. Once impregnated with the dispersion, and prior to gelation, sand compaction was achieved at the centimetric scale by using a dynamic impact method (load of 2.5 N; height of 0.1 m). Finally, prepared samples were kept at 20 °C and 50% relative humidity for 1 week to gain strength and harden fully until IE tests. In the experiments, the amounts of colloidal silica dispersion were set for the silica gel to occupy 33%, 50% and 66% of the porosity of the compacted Fontainebleau sand ($n = 0.38$). We thus referred to the prepared CSCs as N_{33} , N_{50} and N_{66} , respectively. The obtained (n ; k) (as determined from the added amount of binder and water permeability measurements) for N_{33} , N_{50} and N_{66} equalled (0.25; 10^{-13} m^2), (0.19; $5 \times 10^{-14} \text{ m}^2$) and (0.13; $5 \times 10^{-15} \text{ m}^2$), respectively. The bulk density of the sand matrix was kept constant (close to 1600 kg m^{-3}).

2.3. Probing IE intensity

To investigate the IE intensities, we monitored the evolutions of P_{inj} , mass changes of CSCs during erosion tests and granulometric distributions of particles from the effluents (Fig. 1). P_{inj} measuring system included a pressure transducer (Model 14 from AEP), which was used together with an automated data acquisition and processing software (WinATS 6.0 from Sysma®). The interval of measurable relative pressure values was between 50 kPa and 2000 kPa, and the accuracy of the measurements equalled 0.10%. The pressure at the top of the CSCs was set equal to the standard atmosphere. The volumetric water content (i.e. θ) was determined by monitoring the CSCs weights at a 0.1 g scale. θ accounts for two mechanisms: (i) the spreading of injected water within capillaries (i.e. this results in CSCs weights increase in the absence of notable particle releases) and (ii) the filling of newly formed pores (i.e. the evolution of CSCs weights is the net result of injected water and eroded material amounts). Hence, to adequately appraise the θ increases (resp. amounts) of 1–100 μm eroded particles were repeatedly assessed using the granulometric distributions of effluent particles. Finally, coarse eroded particles were collected using a certified 100 μm sieve placed above the lower effluent reservoir. Their mass was measured after drying at 90 °C for 24 h: the $> 100 \mu\text{m}$ particles primarily consisted of highly insoluble (silica gel coated) sand grains that exhibit limited wetting. This permitted to lower the drying temperature while keeping the data comparable. As a matter of fact, further drying at 110 °C for 24 h did not result in significantly lower dry weights ($< 0.5\%$).

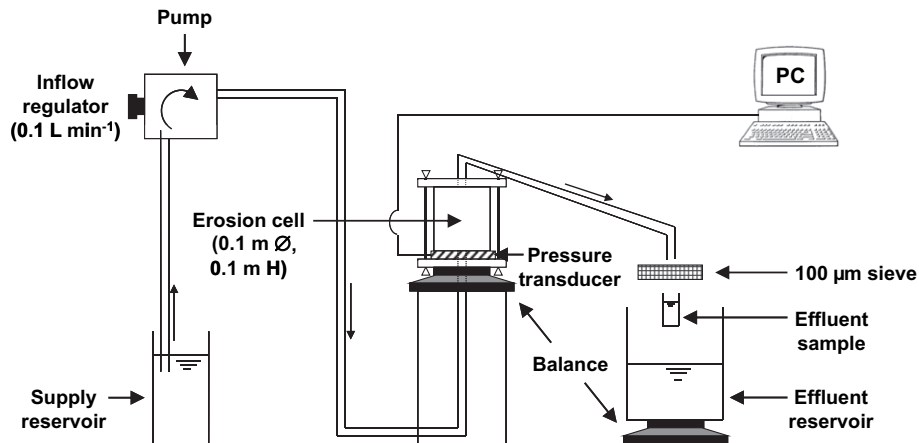


Fig. 1. Experimental set-up for internal erosion tests.

2.4. Characterisation of eroded CSCs and effluent particles

In order to probe the effects of IE on the microstructure of CSCs, the sand skeleton and the binder of unexposed (i.e. preserved from IE) and eroded samples were examined by using a SEM device. Initially, unexposed and eroded samples were homogeneously prepared by the same operator using analogous preparation and hardening methods. During IE tests, the unexposed samples were hermetically stored at 20 °C. Following IE tests, SEM micrographs were taken after carefully splitting the CSCs by hand. The granulometry of eroded particles was determined in the first few hours that followed the sampling of the effluents by means of two particle sizing systems (PSS) from CAD instrumentation. Size distributions (volume based) of small eroded particles from 1 to 100 µm diameter range were determined by single-particle optical sensing using an AccuSizer™ 780 optical PSS. These correspond to the finest fraction of Fontainebleau sand (<5% in mass) and coarse silica gel particles from the binder. At the nanometric scale, size distributions (volume based) of eroded nanoparticles ranging from 0.01 to 1 µm were assessed by dynamic light scattering using a submicrometric Nicomp™ 380 PSS. These correspond to the silica gel colloids from the binder. The accuracy of (sub)micrometric particles sizing systems was less than 1%. Data were corrected using the granulometry of particles initially present in the injected water as a blank.

3. Results and discussion

In advance of the IE tests on reinforced CSCs, we investigated how Fontainebleau sand columns without the chemical reinforcement resist against seepage force. The granular structure of the unreinforced Fontainebleau sand columns was promptly destabilized by the eroding fluid passing through them in less than a few seconds. Beyond 1 min of water injection, the cohesion between sand grains disappeared and notable amounts of sand were discharged. The observed mechanism was not IE but overall transport of fluidized sand grains such as sand boiling.

3.1. The three stages of internal erosion

Fig. 2A–C shows the evolutions of masses of N_{33} , N_{50} and N_{66} CSCs during IE tests, respectively. $t = 0$ s denotes the outflowing of the effluent. Mass evolution curves developed up to three zones: an early mass intake zone, which increases with the amount of binder {at $t = (0-85) \times 10^3$ s for N_{50} and $(0-30) \times 10^3$ s for N_{66} }, a later zone of plateau {at $t = (0-1) \times 10^3$, $(85-130) \times 10^3$ and

$(30-50) \times 10^3$ s for N_{33} , N_{50} and N_{66} , respectively} and a final mass loss zone. This observation is consistent with the initiation (loss of the impervious function), continuation (creation of pathways for eroding fluid) and progression (destabilization of the granular structure) phases described by Foster and Fell (2001) for embankment dam filters. The mass intake, which is observable only for N_{50} and N_{66} {average \pm SD; $(5 \pm 2) \times 10^{-7}$ and $(29 \pm 18) \times 10^{-7}$ kg s⁻¹, respectively}, proves the lasting infiltration of the injected water into the pore network. The plateau zone is developed particularly in N_{50} (duration of 45×10^3 s) and to a lesser extent in N_{66} and N_{33} (20×10^3 and 1000 s, respectively). It supports the existence of an IE episode during which both mass intake via water infiltration and flushing of eroded particles by seepage were low or balanced each other out. In the last zone, the masses of the reinforced CSCs decreased slower with increasing amounts of binder: $(8.3 \pm 1.1) \times 10^{-7}$, $(0.8 \pm 0.1) \times 10^{-7}$ and $(0.4 \pm 0.2) \times 10^{-7}$ kg s⁻¹ for N_{33} , N_{50} and N_{66} , respectively.

Fig. 3A and B display the evolutions of eroded volumes of 1–100 µm particles (as determined from the granulometry of effluent particles) and θ , respectively. θ of N_{33} , N_{50} and N_{66} CSCs exhibited analogous trends (Fig. 3B): (i) an initial period of rapid linear increase $\{(12.3 \pm 1.5, 5.6 \pm 0.4$ and $45 \pm 7) \times 10^{-7}$ s⁻¹, respectively}, which except for N_{33} $\{(0-10.8) \times 10^3$ s} corresponds to the mass intake zone and (ii) a later period of slower increase $\{(6.0 \pm 0.7, 0.8 \pm 0.1$ and $0.4 \pm 0.3) \times 10^{-7}$ s⁻¹, respectively} in the plateau and mass loss zones {at $(10-61) \times 10^3$ s for N_{33} , $(85-250) \times 10^3$ s for N_{50} and $(30-250) \times 10^3$ s for N_{66} }. The rapid initial increase of θ accounts for the filling of accessible pores with injected water and, as the volumes of 1–100 µm eroded particles indicate $\{(32.2 \pm 0.3, 0.8 \pm 0.1$ and $0.4 \pm 0.1) \times 10^{-10}$ m³ s⁻¹, respectively} (Fig. 3A), the flushing out of most movable particles (like loose and/or small particles that can easily exit the material). By comparison, in the plateau and mass loss zones, the slower increases of θ likely account for slower filling of the less accessible pores and lower effluent loading with 1–100 µm particles $\{(1.4 \pm 0.1, 0.3 \pm 0.1$ and $0.2 \pm 0.1) \times 10^{-10}$ m³ s⁻¹, respectively} and rigid skeleton constituents.

During all the IE tests, head losses between the bottom and top of the CSCs markedly decreased (ΔP of 10, 107 and 506 kPa, respectively) (Fig. 3C). This resulted in significant k increases ($k:k_0$ ratio values reached up to 40, 28 and 9, respectively) (Fig. 3D). Assuming that infiltration of injected water and removal of silica gel lead to local drain of the finest sand grains and further destabilization of CSCs structures, we used exponential regression models to fit P_{inj} and k evolutions: $P_{inj}(\text{Pa}) = 3 \times 10^7 e^{-3\theta}$ (r^2 , squared Pearson's correlation coefficient = 0.87) and $k(\text{m}^2) = 6 \times 10^{-16} e^{30\theta}$ ($r^2 = 0.81$) (p , value used to reject null hypothesis = 0.05). Since the intrinsic

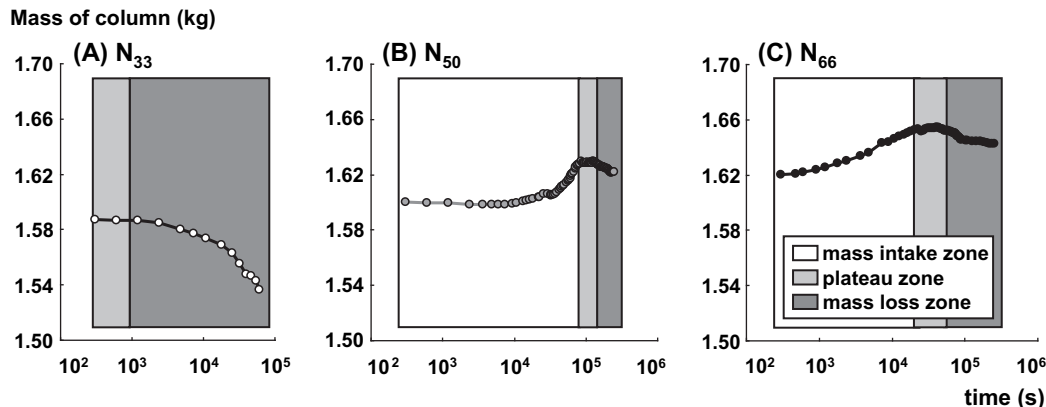


Fig. 2. Monitorings of the masses of (A) N_{33} , (B) N_{50} and (C) N_{66} CSCs (columns of 0.1 m height and in diameter that consist of compacted Fontainebleau sand reinforced with increasing amounts of silica gel) during IE tests. Water inflow is set constant and equalled 0.1 L min⁻¹ (time = 0 s denotes the outflowing of the effluent).

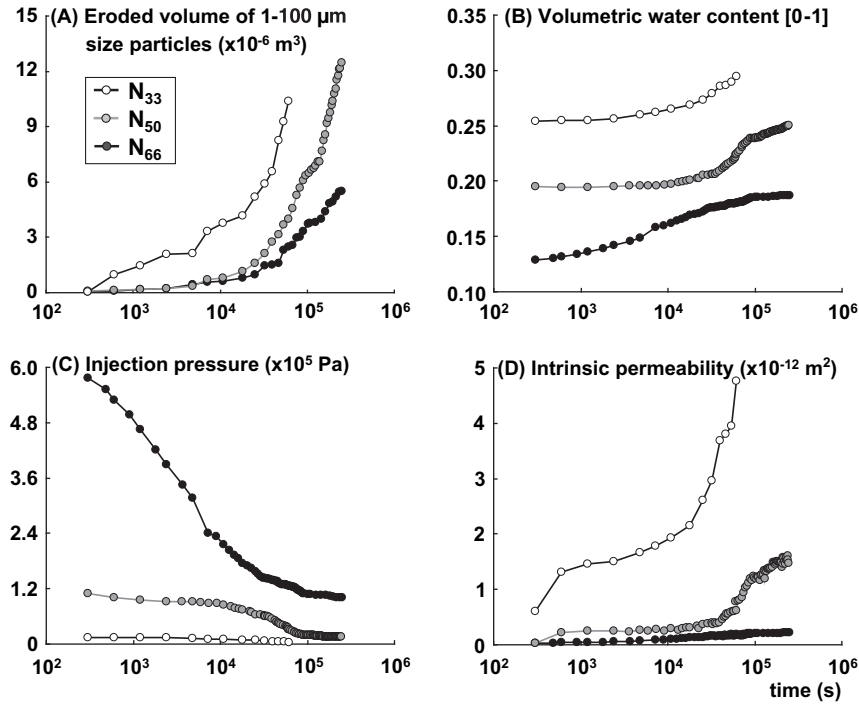


Fig. 3. Evolutions of: (A) eroded volumes of 1–100 μm size particles, (B) volumetric water contents, (C) injection pressures and (D) intrinsic permeabilities of N_{33} , N_{50} and N_{66} eroded CSCs, respectively.

permeability depends on the geometry of constrictions and inter-connections between pores, the obtained relationships suggest that the filling of pores and subsequent drain of fluidized particles could have initiated developments (either in terms of sizes or quantities) of channels and/or capillaries for water flowing throughout the material.

3.2. The finest Fontainebleau sand grains

Fig. 4A–C shows the contributions of micrometric particles to instantaneous discharged mass of solids for IE tests with N_{33} , N_{50} and N_{66} CSCs, respectively. They gather eroded silica gel particles (1–50 μm , average diameter $11 \pm 5 \mu\text{m}$) and finest Fontainebleau sand grains (50–100 μm , average diameter $65 \pm 7 \mu\text{m}$) that are easily flushed out by seepage. Put succinctly, obtained results prove the continuous erosion of the binder (darkest areas below 50 μm), which implies an increasing risk of CSCs failure due to the removal of finest Fontainebleau sand grains and the subsequent instability of coarse sand grains that support imposed stresses.

In the mass intake zone, effluent concentrations of 50–100 μm Fontainebleau sand grains underwent fast decreases $\{(150 \pm 20, 66 \pm 5 \text{ and } 42 \pm 2) \times 10^{-6} \text{ kg m}^{-3} \text{ s}^{-1} \text{ for } N_{33}, N_{50} \text{ and } N_{66}, \text{ respectively}\}$ and reached minimal values (all around 0.02 kg m^{-3}) as soon as $t = 1000 \text{ s}$. The corresponding mass losses were $(0.42 \pm 0.07) \times 10^{-3}$, $(0.19 \pm 0.02) \times 10^{-3}$ and $(0.15 \pm 0.01) \times 10^{-3} \text{ kg}$: i.e. $0.66 \pm 0.14\%$, $0.30 \pm 0.06\%$ and $0.24 \pm 0.02\%$ of the amount of fines used for every single CSC preparation. The monitoring of effluent loading with 50–100 μm Fontainebleau sand grains provides useful information about IE dynamics and the transport of fluidized particles into the CSCs. For instance, as previous results indicated, most of fines are initially immobilized in closed pore spaces or cavities (>99% of CSCs' proportions). Hence, the swift releases of 50–100 μm Fontainebleau sand grains that occur during the plateau and mass loss zones can be considered as indicators of the progressive deterioration of CSCs structure (like the expanding

of constrictions and/or interconnecting pores). Releases of fines are particularly visible in Fig. 4A (N_{33} at $t > 35 \times 10^3 \text{ s}$) and B (N_{50} at $t = 170 \times 10^3 \text{ s}$), while N_{66} , with its high binder content, displayed lowest contributions of 50–100 μm Fontainebleau sand grains (Fig. 4C). It may be concluded that, in regard to IE in chemically reinforced CSCs, the important criterion is not so much the movable sand grains contents but, rather, the suddenness of their mobilisation.

3.3. The silica gel particles

From an engineering point of view, delayed occurrence of 50–100 μm Fontainebleau sand grains into the effluent is a consequence of IE mechanisms that affect the binder (e.g. fracture and removal of the intergranular silica gel). Actually, silica gel particles constituted, either in terms of volume ($70 \pm 8\%$, $79 \pm 8\%$ and $73 \pm 15\%$, respectively) or mass ($54 \pm 10\%$, $67 \pm 11\%$ and $65 \pm 19\%$, respectively), most of the 1–100 μm eroded particles. It is thus pertinent to monitor their removal dynamics by injected water (fractions 1–50 μm in Fig. 4A–C).

In the first 1200 s of the tests, unlike the 50–100 μm Fontainebleau sand grains, the effluent concentrations of silica gel particles underwent their sharpest decreases for the CSCs with the highest binder contents: $(130 \pm 20, 220 \pm 10 \text{ and } 260 \pm 30) \times 10^{-6} \text{ kg m}^{-3} \text{ s}^{-1}$ for N_{33}, N_{50} and N_{66} , respectively. This induces losses of $3.3 \pm 0.2\%$, $2.7 \pm 0.1\%$ and $2.4 \pm 0.3\%$ of the amount of binder used for every individual CSC preparation. According to Kenney et al. (1985), the size of the predominant constrictions in a void network of a filter is approximately equal to one-quarter the size of the particles making the filter; here $\sim 220/4 \mu\text{m}$. This means that the eroded silica gel particles (average diameter of $11 \pm 5 \mu\text{m}$) may even pass through the constrictions formed by the finest Fontainebleau sand grains (average diameter of $65 \pm 7 \mu\text{m}$). Since most of the movable silica gel particles ignore the filtration mechanisms, the strong concentration decreases observed in most reinforced CSCs primarily reflect the lower

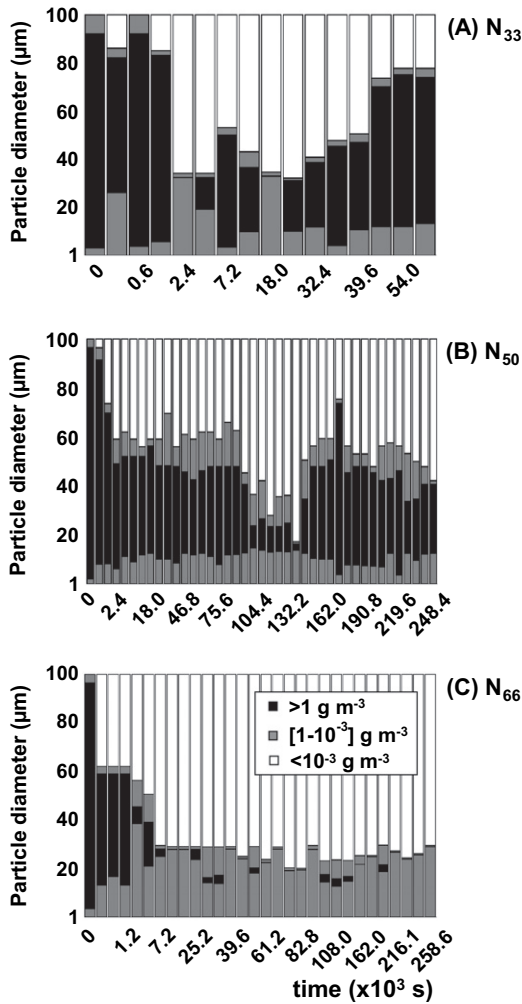


Fig. 4. Contributions of micrometric particles (finest Fontainebleau sand grains + eroded silica gel particles) to the instantaneous discharged mass of solids for IE tests with (A) N_{33} , (B) N_{50} and (C) N_{66} CSCs, respectively.

erodibility of their binder (mass effect). Another observation is that minima of concentration accompanied the developments of plateau or mass loss zones: at $(2-25) \times 10^3$ s in N_{33} ,

$(95-130) \times 10^3$ s in N_{50} and around 30×10^3 s in N_{66} . This means that silica gel fragments are then hardly dislodged from the binder $\{(0.5 \pm 0.1, 0.2 \pm 0.1 \text{ and } 0.3 \pm 0.1) \times 10^{-7} \text{ kg s}^{-1} \text{ for } N_{33}, N_{50} \text{ and } N_{66}, \text{ respectively}\}$ and, as a result, the rigid skeleton would effectively resist the seepage stresses. With increasing IE time, however, the effluent silica gel particles concentrations tended to increase either gradually $\{(0.5 \pm 0.1) \times 10^{-6} \text{ kg m}^{-3} \text{ s}^{-1} \text{ for } N_{33}\}$ or as peaks (up to 0.91 and 0.32 kg m^{-3} for N_{50} and N_{66} , respectively), which matched reasonably well with the swift releases of fine Fontainebleau sand grains $\{p = 0.05, r^2 = 0.66; [\text{sand}]_{50-100\mu\text{m}} = 0.34 [\text{binder}]_{1-50\mu\text{m}}\}$. It implies that beyond the period of low effluent loading with silica gel particles, instability of CSCs can be gauged using the occurrences of fine sand grains as indicators of IE of remaining binder.

3.4. The dynamics of particle transport within CSCs

Fig. 5A–C display the diameters of effluents' (i) smallest nanometric particles (SNP; right scale) and (ii) largest micrometric particles (LMP; left scale) that are strongly flushed out by seepage ($>10^{-3} \text{ kg m}^{-3}$). First, silica gel is practically insoluble ($<100 \text{ nmol L}^{-1}$ at pH 7–8; Iler, 1979) and thus SNP depict the finest silica colloids from the binder that can be mechanically dislodged by seepage. We thus assumed that SNP sizes should be proportionate with seepage stresses: i.e. coarser silica colloids would be dislodged at higher shear forces acting on the binder. Second, if sizes of eroded micrometric particles are smaller than CSCs' constrictions, they can be moved by water flow through the pore network until they are discharged into the effluent. We therefore assumed that LMP diameters provide some measure of the dimensions of the smallest constriction that permit their passage.

From LMP data, the minimal constrictions are largest in the very beginning of IE tests ($80-100 \mu\text{m}$, left scale). This is about twice the value obtained using Kenney's formulation ($\sim 55 \mu\text{m}$, see Section 3.3), likely due to the preferential leaching of most superficial, thus unfiltered, sand grains. In fact, except for N_{33} , which does not exhibit an initial mass intake zone, sizes of the minimal constrictions rapidly tend to stabilize around $50-60 \mu\text{m}$ (49 ± 3 and $59 \pm 1 \mu\text{m}$ for N_{50} and N_{66} , respectively). Later $\{at t = (0.3-1.2, 30-90 \text{ and } 2.4-18) \times 10^3 \text{ s for } N_{33}, N_{50} \text{ and } N_{66}, \text{ respectively}\}$, marked size increases of the effluents' SNP either precede or accompany additional narrowing of constrictions (resp. down to $32, 17$ and $16 \mu\text{m}$). This suggests that, previously to the plateau zone, shear forces that affect the binder would produce sufficiently large

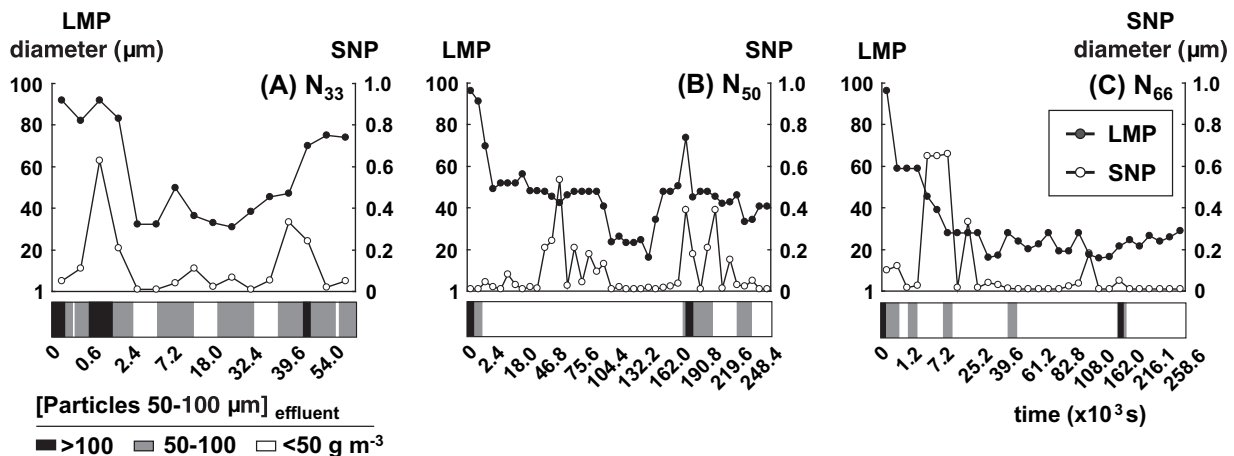


Fig. 5. Diameters of effluents' largest micrometric particles that are readily flushed out by seepage (LMP; left scales, filled circles) and smallest nanometric particles (SNP; right scales, empty circles) for internal erosion tests with (A) N_{33} , (B) N_{50} and (C) N_{66} CSCs, respectively. Greyish scales below the diameters account for effluents' concentrations of $50-100 \mu\text{m}$ size Fontainebleau sand grains (in g m^{-3}).

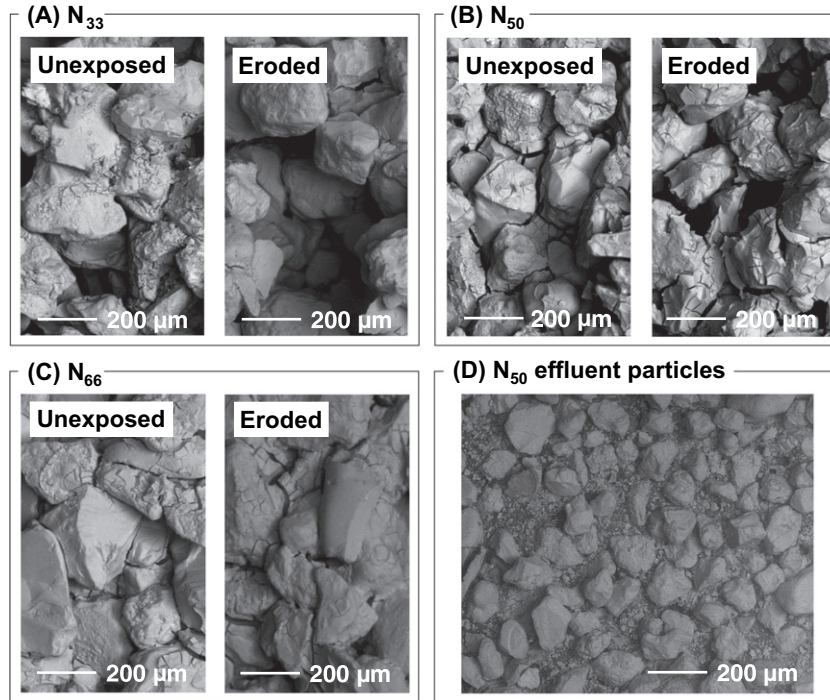


Fig. 6. Micrographs, obtained by means of a scanning electron microscope (SEM), showing the bulk microstructure of unexposed (i.e. preserved from IE, left) and eroded (right) (A) N_{33} , (B) N_{50} and (C) N_{66} CSCs, respectively and (D) displays the particles discharged from N_{50} CSC during a swift release of fine Fontainebleau sand grains.

particles in the pore network (via the dislodgement of coarse silica gel fragments and/or sand grains) to partially obstruct the existing constrictions. This is further confirmed by concomitant decreases in slope values of θ and thereafter k curves (see Section 3.1 and Fig. 3B and D). Based on the previous remarks and measured IE rates, the plateau zone not only depicts a domain of lower erodibility of binder but also of hindered transport of dislodged fine (50–100 μm) Fontainebleau sand grains. Finally, in the mass loss zone, peaks of SNP match to broadenings (resp. higher effluent concentrations of 50–100 μm size grains) {at $t = (7.2\text{--}10.8$ and $40\text{--}47) \times 10^3$ s for N_{33} , $(169\text{--}176$ and $212) \times 10^3$ s for N_{50} and 144×10^3 s for N_{66} } or narrowings (resp. lower effluent concentrations of 50–100 μm size grains) {at $t = 25 \times 10^3$ s for N_{33} , $(198$ and $234) \times 10^3$ s for N_{50} and 102×10^3 s for N_{66} } of constrictions (greyish scales in Fig. 5A–C). This demonstrates that recorded mass losses are the sum of dynamic releases and filterings of eroded fine sand grains.

3.5. The microstructural changes

Fig. 6A–C displays the bulk microstructure of unexposed and eroded N_{33} , N_{50} and N_{66} CSCs, respectively. The micrographs clearly show that, in spite of the microstructural changes inherent to SEM analyses (like the shrinkage of the binder), silica gel within eroded samples is more fractured and in part removed. As a matter of fact, micrographs of effluent particles exhibit multiple silica gel particles ranging from <1 to 50 μm (Fig. 6D). The extent of silica gel/sand grains removal (as estimated using the surface increase of the darkest areas) averages $12 \pm 3\%$, $6 \pm 1\%$ and $4 \pm 2\%$ for N_{33} , N_{50} and N_{66} , respectively. These exceed by up to an order of magnitude the values obtained using the total weight of $>100 \mu\text{m}$ eroded grains ($5.2 \pm 0.1\%$, $0.6 \pm 0.2\%$ and $0.5 \pm 0.1\%$, respectively): i.e. 50–90% of eroded particles are smaller than 100 μm and their contribution increases with the amount of binder. For instance, size ranges of apparent voids formed during IE are 25–500, 15–300 and 5–250 μm for N_{33} , N_{50} and N_{66} , respectively. Lowest

values are consistent with dimensions of the smallest constriction based on the granulometry of effluent particles (see Section 3.4) whereas the highest values match the maximal size of effluent particles (e.g. up to 300 μm for N_{50} at $t = 170 \times 10^3$ s during a swift release of fine Fontainebleau sand grains, Fig. 6D).

3.6. Data synthesis

At a constant inflow rate, seepage tests on chemically reinforced CSCs highlight the existence of up to three IE stages: (i) an early CSCs mass intake stage, which corresponds to the filling of accessible pores with injected water and flushing of most movable particles, (ii) a subsequent stage of low binder erodibility coupled with the clogging of constrictions, and (iii) a later CSCs mass loss stage of dynamic releases and self-filtering of sand grains from/within the rigid skeleton.

From a macroscopic point of view, the concomitant increases of θ and k indicate the development of channels and/or capillaries for the injected water. The dynamics of channels developments depend on the size of constrictions and IE stages: (i) it rapidly increases during the mass intake stage, (ii) decelerates because of material acting as a filter and in part retains dislodged particles during the low binder erodibility stage, and (iii) fluctuates according to releases and filtration of sand grains during the mass loss stage.

Microstructural changes firstly consist of the hydraulic fracturing of the binder. Binder deterioration significantly increases while lowering the amount of binder used for CSCs preparation (mass effect). Continuous drain of silica gel fragments and the dislodgement of fine Fontainebleau sand grains forms voids within the primary fabric that incurs shear stresses. Sizes of the largest voids are consistent with that of coarsest particles discharged during the swift sand grains releases. In turn, the destabilization of CSCs structures provides favourable conditions for further binder removal.

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