RESEARCH PAPER

Particle loss and volume change on dissolution: experimental results and analysis of particle size and amount effects

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Abstract Soil particles may be lost through dissolution, degradation or erosion. Regardless of the process of loss, there follows a change in soil structure both in terms of phase composition and grading. In this paper, the influence of size and amount of particle loss on phase composition at two stresses is investigated. The tests are performed on sand-salt mixtures, loaded in a modified permeation oedometer and subsequently dissolved. Changes in overall volume and void ratio are presented. Two significant observations about the volumetric consequences of particle loss can be made. First, overall volume changes are directly related to the amount of dissolved particles and to a lesser extent, the size of particle lost. Second, particle loss leads to an increase in void ratio; the magnitude of the increase is related to the amount of dissolved particles but appears not to be sensitive to either the size of particle lost or the predissolution void ratio. Based on the observed response and a dissolution-induced void change parameter, the influence of different mechanisms of volume change is discussed. Tests were performed at two different vertical stresses with no discernible influence on void ratio change.

Keywords Dissolution · Laboratory testing · Micromechanics · Particle loss

1 Introduction

The mechanics of particle loss are a phenomenon relevant to dissolving, degrading and eroding soils. Particle loss will

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lead to changes in volume and strength; the exact nature of these changes and the accompanying change in particle size distribution being dependent on the amount and size of lost particles. A number of investigations have been reported: Fam et al. [6] investigated shear behaviour and shear wave velocity changes due to dissolution in 2, 5 and 10 % salt–sand mixtures (D_{50} of 0.35 and 0.82 mm, respectively) under triaxial conditions. They observed negligible change in overall volume during dissolution and reported void ratio increases of between 5 and 32 % depending on the amount of salt dissolved. At the same time, reductions in shearing resistance of 26 % for 10 % added salt were noted.

Shin and Santamarina [18] looked at dissolution in 5, 10 and 15 % salt–ballotini mixes (D_{50} of 0.3 and 0.7 mm, respectively) under oedometric conditions. They found measured settlement was much lower than the strain corresponding to salt volume loss under constant void ratio¹ conditions and hence an increase in void ratio during dissolution.

More recently, Truong et al. [21] examined volume changes in 2, 5, 7 and 10 % salt-sand mixtures (D_{50} of 0.25 and 0.36 mm, respectively) under a range of vertical (oedometric) stresses. They showed settlements and void ratio increases directly related to the amount of salt particles lost. Under a vertical stress of 160 kPa, settlement increased from 0.5 % to about 1 % for 2 % to 10 % salt particle contents, respectively. Void ratio increases were similarly affected, increasing from 0.03 in 2 % particle mixes to about 0.16 with 10 % added salt. In the vertical

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 $^{^{1}}$ The constant void ratio condition depicts an amount of settlement induced by a solid volume loss and accompanying void volume change that leave the void ratio unchanged. Settlements that are less than the constant void ratio condition lead to void ratio increases and vice versa.

stress tests, settlement was 0.5 % at 40 kPa rising to about 1.2 % at 320 kPa. Void ratio changes were relatively unaffected by this vertical stress range revealing the 0.16 increase cited above at all vertical stress levels.

Studies such as those by Fam et al. [6], Shin and Santamarina [18] and Truong et al. [21] show that dissolution in coarse-grained soils leads to a change in soil fabric, i.e., structure. They focused primarily on the amount of particle loss in mixes with a relatively narrow range of sand to salt particle size diameter ratios $(D_{50 \text{ sand}}/D_{50 \text{ salt}})$ between 1.44 and 2.34). Consequently, there is little experimental data, and hence, understanding of the influence of particle size on fabric changes due to particle loss. This paper presents the findings of an experimental investigation into the volumetric consequences of both amount and size of particles lost. Salt particle sizes ranging from 0.063 to 1 mm, added to sand with a D_{50} of 0.85 mm, in amounts from 2 to 15 %, have been tested. In such an investigation, the initial packing of the mixes assumes greater importance.

The packing characteristics of particle size distributions extending over one or more orders of magnitude have been investigated extensively by ceramics scientists [11, 22]. Over such a range, the opportunity for small particles to fill void spaces exists. Lade et al. [8] elucidated the extent to which the presence of smaller particles increases density and reduces void ratio for a number of sandy soils and spherical particles.

The experimental procedures, results and methods of analysis presented here are of wider interest, being applicable to other processes of particle loss and grading change, e.g., microbial degradation of solid waste in landfill, internal erosion in dams and particle crushing during shear.

2 Test materials

The inert fraction is a coarse rounded to sub-rounded quartz sand from Leighton Buzzard in the UK ($D_{50} = 0.85$ mm; $C_U = 1.4$; $G_S = 2.65$). The salt is sodium chloride ($G_S = 2.165$), chosen for its high solubility in water, which after crushing to the required dimension, has a more angular form.

Particle size distributions are defined by a sieve stack having a sequence of aperture dimensions that double² in size, i.e., 0.063, 0.125, 0.25, 0.5, 1.0 and 2.0 mm. Hence, in this paper, 0.063 mm particle fraction refers to particles between 0.063 and 0.125 mm. Figure 1 shows the relative

sizes of the sand and salt particles for two of the gradings used as follows: 1 mm salt particles in Fig. 1a and 0.063 mm particles in Fig. 1b.

To assess the likelihood of salt particle breakage in the test programme, 1.0 mm salt particles (without sand) were incrementally loaded in an oedometer to 6.5 MPa with particle size distributions determined at selected increments. The first evidence of breakage occurred at a vertical stress of 880 kPa, whereupon the mass of salt particles passing the 1.00 mm sieve was about 1.5 %, rising to 3 % at 1760 kPa.

3 Equipment and procedures

3.1 Equipment and operation

The dissolution tests were performed in an oedometer that was modified to allow for (gravity-driven) permeation of water, see Fig. 2. The oedometer is 100 mm in diameter. It has an extended confining ring to accommodate a sample height of up to 30 mm and to retain a reservoir of water above the loading cap and sample. The Perspex loading cap has a number of small (1 mm) holes to improve the flow of water from the overlying reservoir to the sample. Permeation avoids the accumulation of ion-saturated solution in the pores and encourages a more even distribution of particle dissolution within the sample. Complete dissolution is usually obtained in less than an hour, and its termination is checked using the procedures described in Sect. 3.4. Pore fluid exits through two ports in the oedometer base into an external 4 L reservoir, which is continuously stirred. A peristaltic pump transfers solution from the external reservoir back to the oedometer. Both reservoirs are open to atmosphere, so pump flow rate is used to control the level in the oedometer reservoir.

3.2 Test programme

The programme comprised 40 dissolution tests in 2 groups of 20. In each group of 20 tests, 5 different single-salt particle size fractions (0.063, 0.125, 0.25, 0.50 and 1.00 mm) were added to Leighton Buzzard sand in 4 different proportions (2, 5, 10 and 15 %). These percentages are calculated as a fraction of the total *mass*. In one group, dissolution occurred under a vertical stress of 62 kPa; in the other group, vertical stress was 250 kPa.

3.3 Sample preparation

The dry sand and salt particles were mixed together then carefully 'spooned' into the oedometer. Light tamping of the sample mixes and careful handling of the oedometer

 $[\]frac{1}{2}$ This sieve stack allows the particle size distributions and the impact of mass loss to be defined in terms of grading entropy coordinates. Further information on the grading entropy concept can be found in Lőrincz et al. [9].

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Fig. 1 Photographs of salt-sand (Leighton Buzzard) mixes showing relative sizes and shapes: a 1.0 mm salt particles; b 0.063 mm salt particles



Fig. 2 Schematic diagram of modified oedometer allowing for circulation of pore fluid through sample and large (4 L) external reservoir (not shown to scale)

were important to avoid segregation of the dry mix components. This was a potential problem where the sand and salt particles were of significantly different size but visual inspection of the samples was possible through the Perspex cap and sufficient to verify sample homogeneity.

3.4 Test procedures

After loading to the planned vertical stress, distilled water from the external reservoir was circulated through the sample. Conductivity measurements were taken using a hand-held meter in the external reservoir to monitor dissolution progress. Figure 3a shows typical conductivity readings during the dissolution process. There was a simultaneous reduction in sample height, as shown in Fig. 3b, which in the case of NaCl was substantially complete in about 60 min. The correspondence between conductivity changes and vertical displacement was observed in all tests. Samples were subsequently loaded to 500 kPa and then unloaded, although post-dissolution loading is not addressed in this paper. Finally, samples were dried and weighed to confirm mass loss. An additional check for complete dissolution was made by evaporating the saturated saltwater solution and weighting the resulting salt mass.

4 Initial states

Mixtures comprising salt particles of various size fractions and amounts mean there is a wide range of initial sample void ratios in this test programme. Figure 4a, b show the void ratios of all 40 test mixes after initial loading, by salt particle size fraction and percentage of total mass. Also shown are the minimum and maximum void ratios for this Leighton Buzzard sand reported by Kingston et al. [7].

Initial void ratios show that the addition of small particles (0.063 and 0.125 mm) produces void ratios below the pure-sand minimum. The reduction in void ratio is clearly related to the amount and size of the particles added with the 0.063 mm particles achieving lower void ratios than the 0.125 mm particles. The addition of larger particles results



Fig. 3 a Electrical conductivity of solution during dissolution as measured in external reservoir and b associated displacement

in a range of initial void ratios having a minimum relative density of approximately 66 %.

The general picture is consistent with previous studies. Lade et al. [8] measured the void ratios of a number of binary mixtures of Cambria and Nevada sands. The relative size of the components of any mixture was denoted using a diameter ratio, i.e., the ratio D_{50} of the larger particle to that of the smaller particle. They found that for these soils, when diameter ratios are less than 3.0, there is no reduction in void ratio with increasing amounts of the smaller particles. The added particles are broadly the same size as the 'receiving' particles and are accommodated by interruption of the particle assembly. At higher diameter ratios, e.g., 7 or greater, added particles are fine enough to occupy the pore network without disturbing the 'receiving' particle skeleton; void ratio then decreases with added fine content.

Taking D_{50} of the Leighton Buzzard to be 0.85 mm and D_{50} of the 0.063 mm particle fraction to be 0.094 mm (midpoint of the fraction interval, 0.063–0.125 mm) gives a diameter ratio of 9.0. By contrast, the larger particle fractions have diameter ratios of 2.3 or less—approximately the same as the mixes tested by Fam [6], Shin and Santamarina [18] and Truong et al. [21]. The void ratio of



Fig. 4 Initial void ratio by salt particle size and percentage of total mass at pre-dissolution stress; **a** 62 kPa and **b** 250 kPa



Fig. 5 Initial void ratio (at pre-dissolution load) obtained for percentage of total mass added of 0.063 mm particle at predissolution loads of 62.5 and 250 kPa together with void ratio data reported by Lade et al. [8] for Cambria–Nevada 50/80 mix

Cambria–Nevada 50/80 mix reported by Lade et al. [8], which has a diameter ratio of 7.1, is shown in Fig. 5 and compares well with the pre-dissolution void ratio data for the 0.063 mm samples obtained here.

5 Settlement

Previous studies have reported relatively little settlement due to particle loss by dissolution. Fam et al. [6] conducted tests on salt–sand particle mixtures with diameter ratios of 2.30 noted virtually no settlement whilst Shin and Santamarina [18] measured settlement between 1.2 and 8.2 % for their range of salt contents (5–15 %) with diameter ratios of 2.33. Truong et al. [21] testing mixes with diameter ratios of 1.44 showed settlements of 2 % and less for mixes containing up to 10 % of salt particles.

Vertical settlements induced by dissolution for each of the sand–salt mixes tested here are shown in Fig. 6. Also shown are (1) the settlement at constant void ratio line, i.e., the settlement that would occur if solid volume loss and corresponding void volume change maintain a constant void ratio, and (2) the Shin and Santamarina data presented as mass fractions.

It is evident from the settlement data that small particles, which can occupy the inter-sand void spaces, can be dissolved with little corresponding settlement. For example, less than 2 % vertical strain was observed during dissolution in samples containing 10 % of 0.063 mm salt particles. Settlement increases with salt content and tends to be greater where the dissolved particles are relatively large.

The Shin and Santamarina [18] data, obtained from glass bead-salt mixtures with a diameter ratio of 2.33, all settle more than the Leighton Buzzard-salt mixtures. This may be due to the lower frictional resistance and shape of the glass beads [15] facilitating particle rearrangement. It is well recognised from experimental and numerical DEM studies that both inter-particle friction and particle shape have a significant effect on the behaviour of granular materials [e.g. 4, 5, 19]. The settlements are, however, significantly less than settlement under a constant void ratio condition. Hence, particle dissolution, for the amounts and sizes of the soluble particles tested here, leads to an increase in void ratio.



Fig. 6 Settlement of samples due to dissolution. Data not separated by pre-dissolution stress

6 Void ratio changes due to dissolution

In a dissolving soil, changes in void ratio are a function of both mass loss and void volume changes, calculated from total and solid volume changes. Figure 7 shows the preand post-dissolution void ratios (lower and upper lines, respectively) for each of the four salt particle amounts dissolved under 62.5 kPa. Also shown are the maximum and minimum void ratios for the pure Leighton Buzzard sand [7].

Figure 8 shows the pre- and post-dissolution void ratios obtained from the samples dissolved under 250 kPa vertical stress; a number of observations can be made from these and the data in Fig. 7:

- 1. There is a clear increase in void ratio with amount of salt particle dissolved.
- 2. The vertical translation from pre- to post-dissolution states of the almost congruent void ratio lines within any diagram indicates that the increase in void ratio is:
 - a. not influenced by particle size and
 - b. not influenced by the value of pre-dissolution void ratio.
- 3. Comparison of void ratio increases in Figs. 7 and 8 for the same percentage additions does not indicate any sensitivity to the stress states under which dissolution occurred.

In other words, the dominant influence on the magnitude of the void ratio change during dissolution is the amount of soluble particle loss.

7 Mechanisms of volume change and force chains

The data presented reveal the volumetric consequences associated with soil particle loss; more specifically, the influence of the amount, particle size, initial void ratio of the sand–salt mix and the load at which dissolution took place. The magnitude of settlement is related to both the amount and size of the particles lost, whereas the increase in void ratio is related to amount only.

The muted settlement response and the insensitivity of void ratio to particle size can be intuitively explained by the likely disposition of different salt particle sizes in the mixes and the impact of their loss on the sand skeleton.

7.1 Smallest particles

Small particles can nestle within the voids surrounding the larger sand particles. Dissolution of the smallest particles is then accompanied by a near equivalent increase in void space and minimal settlement.



Fig. 7 Dissolution induced increases in void ratios with particle size for each percentage mass of salt particle added under vertical stress of 62.5 kPa. Minimum and maximum void ratios for the pure Leighton Buzzard sand, after Kingston et al. [7], are shown as broken *lines*, respectively



Fig. 8 Dissolution induced increases in void ratios with particle size for each percentage mass of salt particle added under vertical stress of 250 kPa. Minimum and maximum void ratios for the pure Leighton Buzzard sand, after Kingston et al. [7], are shown as broken *lines*, respectively

7.2 Largest particles

Particles of this size, i.e., those approximately equivalent to the sand particles cannot nestle within the sand skeleton voids. If present in large amounts, salt particles are likely to be part of the load-carrying skeleton, which are then disrupted by dissolution. Particle rearrangement will lead to settlement and an increase in void ratio.

It is useful at this point to consider insights into particle assemblies gained from discrete element method (DEM) simulations. Figure 9 illustrates the inter-particle forces inside a typical section of a 3D assembly of spheres at the peak stress state of a DEM simulation under triaxial compression. Black lines indicate normal contact forces between particles carrying forces larger than average contact force magnitude with the thickness of such lines proportional to the magnitude of the contact force. It is clearly shown that stress transmission in granular materials is not homogeneous. Previous studies have demonstrated that load-carrying capacity is confined to a limited number of particles belonging to highly stressed force chains oriented in the direction of the major principal stress [20], which are in turn supported by multiple particles carrying a minimal proportion of the stress [16]. These 'supporting' particles contribute to prevent the buckling and collapse of the strong force chains [13], which are inherently supported by friction [3].

Larger particles, which are not part of a force column network, may therefore be dissolved without disruption to the load-carrying structure, i.e., without settlement. The volumetric changes associated with these particles will be a near equivalent increase in void ratio. Indeed, Fig. 6 shows settlement of 0.5 % or less in the 2 % salt sample mixes for all particle sizes. Such behaviour is consistent with the idea



Fig. 9 Distribution of normal contact forces of triaxial specimen under anisotropic stress conditions, from Barreto [2]

of small particles nestling between the sand skeleton and large particles nestling between the force columns. When the number of large particles is greater (10 and 15 %), the likelihood of them participating in force columns is greater and disruption of the sand skeleton more likely. Settlements then become more significant as indicated by the results in Fig. 6, although inspection of the Truong et al. [21] data points to little settlement, even with 10 % added salt.

Two-dimensional DEM simulations of particle dissolution performed by Shin and Santamarina [18] showed that dissolution concentrates an initial predominantly vertical regime of force columns into fewer columns (strong force chains), arching around a honeycomb fabric. These observations were made for 20 % salt content and a diameter ratio of 2.33.

8 Volume changes due to dissolution

The effect of small particles (or fines content) on the behaviour of soil under various loading conditions has received considerable attention. For example, Polito and Martin [14] suggest using the so-called skeletal void ratio defined by Shen et al. [17] to assess the effect of fines content on liquefaction potential for sands. The intergranular void ratio [12] is another alternative used by various researchers for the same purpose. However, these volumetric state variables are not well-suited to the interpretation of dissolution in the salt particle mixes being considered here. The problem is that these state variables are predicated on the existence of a 'fines' content; only the 0.063 mm salt particle mix can be considered as having a fines content, so the variables cannot be applied to the full range of salt particle mixes tested. Furthermore, the proportion of fines is not a fixed characteristic; particle dissolution means that a skeletal or intergranular void ratio would be constantly changing. Hence, an alternative approach is required.

In this section, a more quantitative interpretation of the volumetric consequences of dissolution is made using Λ , a dissolution-induced volume change parameter,

$$\Lambda = dV_V/dV_S \tag{1}$$

where V_V is the volume of voids and V_S is the solid volume. Here, the change in solid volume is associated with loss due to dissolution of salt particles. An advantage of Λ is that by normalising the change in void volume with respect to the amount of mass dissolved, particle size effects are more clearly drawn. An expression relating total volume (V_T) changes due to dissolution can be derived, e.g.,

$$dV_T = (1 + \Lambda) \, dV_S \tag{2}$$

and a change in void ratio due to dissolution by,

$$de = (e - \Lambda) \, dV_V / V_S \tag{3}$$

Key values of Λ are summarised in Table 1. For example, when $\Lambda = -1$, the loss of solids results in a direct and equal increase in void volume, i.e., there is no change in overall volume and the increase in void ratio is at a maximum. An increase in void ratio occurs at all values of $\Lambda < e$, with the magnitude decreasing until $\Lambda = e$, whereon there is no change in void ratio. At higher Λ values, the loss of solid matter leads to a more compact particle arrangement. In this case, significant settlement would occur since there is a reduction in both solid and void phase volumes. A fuller derivation and explanation of Λ can be found in McDougall and Pyrah [10].

Figure 10 shows values by particle size for each percentage amount added. In the 2 % salt particle addition tests, A values range between -0.9 and -0.7. Void volume increases per unit loss of solid volume are at their greatest but appear relatively insensitive to particle size. This insensitivity is consistent with the mechanics of dissolution of different particle sizes elucidated in the previous section of this paper. That is, small particles are able to nestle within the voids adjacent to the strong force chains and dissolve with little disruption to the load-carrying particle network whereas large particles are too large to 'nestle.' However, the distribution and orientation of force chains discussed by Thornton and Anthony [20] and others, as well as the small number of large particles, mean that the probability of large soluble particles being part of a strong force chain is low. So, at small percentage salt additions, dissolution leads to a small increase in void ratio i.e. independent of particle size and with little overall volume change.

In the 15 % particle addition tests, Λ values are relatively low (≈ -0.7) for the small salt particle mixes, rising to values of up to -0.35 where the salt particles are larger. There appears to be enough space within the sand skeleton voids to accommodate 15 % of small particles (diameter ratio = 9). Indeed, McGeary [11] and Lade et al. [8]

Table 1 Key values of Λ and associated volumetric responses

Reference values of Λ	Void ratio	Volumetric response
$\Lambda = -1$	Maximum increase	No change in overall volume
$\Lambda = 0$	Increasing	No change in void volume
$\Lambda = e$	Constant	_
$\Lambda > e$	Decreasing	Maximum change in overall volume



Fig. 10 Volumetric response to particle loss as depicted by Λ in terms of particle size and amount lost

indicate that the minimum achievable void ratio due to the addition of fines with comparable diameter ratios occurs at about 25 % by volume of fines addition. In the case of large salt particles at 15 %, the likelihood of participation in, and disruption of, the strong force chains is greater, so particle rearrangement and overall volume change occur. Data from both 10 and 15 % particle addition tests reveal settlements that are related to salt particle size and changes in void ratio that are insensitive to salt particle size. A constant void ratio increase, regardless of salt particle size, is consistent with the range of values of e and Λ that would be entered into Eq. 3. In the case of the small salt particle mix, where Λ is approximately -0.7, e is low due to nestling particles. For the larger salt particle mixes, Λ rises to about -0.45, which is higher than the small particle case because of the void volume loss occurring as strong force chains containing salt particles buckle or collapse. Initial void ratio would also be higher due to fewer nestling particles. Since e and Λ change in the same direction and by similar amounts, the $(e-\Lambda)$ term is relatively unchanged rendering the change in *e* relatively insensitive to particle size.

9 Summary and conclusions

The data presented here reveal how both the amount and size of particles control overall volume and void ratio changes in soils from which particles are lost. Settlement was observed to be related primarily to the amount of particle loss and secondly to particle size. Void ratio increases were observed in all tests with the magnitude being related to amount of particles lost but insensitive to particle size.

Two mechanisms of volume change have been presented to explain the observed effects of dissolution of particles. One concerns particles that are relatively small; the other, particles that are at least as large as the inert sand particle network. The first is founded on the existence of small particles nestling within the voids surrounding strong force chains with high load-carrying capacity. Removal of these particles has little impact on the remnant structure producing a near equivalent increase in void space. There is a significant increase in void ratio with minimal settlement. The second mechanism, for large particles, is controlled by the existence of force chains and the amount of particles in the mix. With small amounts of salt, few (large) particles occupy the force chains, and dissolution will occur with little effect on the load-carrying structure. Again, void ratio increases are significant, and settlement is minimal. However, where larger amounts are lost, particle size effects emerge. Particle rearrangement occurs due to buckling and collapse of strong force chains; void ratio increases are smallest, settlement is greatest and Λ attains its highest value, although it is still negative.

Finally, there is no obvious influence of vertical stress in the range 62–250 kPa on void ratio increase in these quartz sand–salt mixtures.

As well as dissolution, these findings have relevance to erosion and degradation phenomena. They are a continuum-based interpretation of the volumetric consequences of particle loss calling on micromechanics to support the postulated mechanisms. Recent work in micromechanics, on observation and analytical methods such as 3D tomography combined with particle tracking [1], would make possible direct investigation into the mechanisms proposed.

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