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Tensile strengths of flocculated compacted unsaturated soils

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Abstract

Flocculating agents can be introduced to soils through a number of natural or anthropogenic processes. This paper investigates the effect of flocculant addition (aqueous CaCl₂) on the tensile strengths of two soils of differing flocculation susceptibility. Tensile strengths were found using the Brazilian (direct splitting) test for a range of suction values. A decrease in tensile strength was found for a soil with a high clay content, which was consistent with previous literature findings. However, the strength of the lower clay-content soil unexpectedly increased. Results were interpreted using the Extended Mohr Coulomb (EMC) Yield Criterion comprising two planar yield surfaces, fitted to data above and below the residual suction value. Changes in EMC parameters were used to infer changes in material behaviour on CaCl₂ addition. Results have important implications for the design of geotechnical structures, for example engineered cover systems, exposed to flocculating conditions.

Keywords: Flocculation, tensile strength, Brazilian test, suction

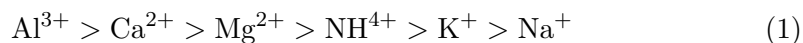
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1 **1. Introduction**

2 Many processes introduce flocculating agents to soils. For example, they are
3 widely used in the mining industry to improve tailings dewatering, consolidation
4 times and transportability (Williams and Jones, 2005). Closure and rehabili-
5 tation of tailings storage sites typically involves the placement of unsaturated
6 soil cover layers in direct contact with this treated material. How flocculants
7 effect unsaturated soil properties must therefore be understood; for example, un-
8 expected cracking can lead to increased water infiltration or decomposition gas
9 emission. Root penetration resistance may also be affected, with beneficial or
10 problematic implications depending on the cover's function.

11 **2. Soil flocculation**

12 Flocculating agents modify the behaviour of soil clay particles by modifying
13 cation adsorption and surface charge. The *lyotropic series* gives the order of
14 the strength of bonding to the cation exchange surface (i.e. cation exchange
15 preference):



16 Flocculants lie to the left of (1), dispersants to the right (Grant et al., 1992).
17 Previous investigations have indicated that tensile strengths of clayey soils are
18 improved through the addition of dispersants (Dexter and Chan, 1991; Rycroft
19 et al., 2002; Sou/Dakouré et al., 2013; Deng et al., 2014), but reduced through
20 flocculation (Barzegar et al., 1994a,b, 1996; Dontsova and Norton, 2002). Criti-
21 cally, however, these works comprised several shortfalls:

- 22 • soil microstructure was not considered during sample preparation: material
23 was either in a field condition or reconstituted, with no record of dry density;

- 24 • testing was conducted on aggregate-scale specimens ($\leq 12\text{mm}$), which do
25 not represent bulk soil properties;
- 26 • soil suction was generally not considered: results may have been affected
27 by poor suction equilibration or hysteresis.

28 This paper addresses some of these issues. Here, the splitting tensile (“Brazilian”)
29 test is used to investigate the effect of flocculant addition on the tensile strength
30 of specimens of two engineered soils equilibrated to a wide range of suction con-
31 ditions. Results are interpreted according to Extended Mohr-Coulomb theory, in
32 order to identify changes in unsaturated material properties responsible for the
33 observed behaviour.

34 **3. Experimental procedure**

35 *3.1. Materials*

36 ‘Engineered’ soils were used for this investigation in preference to natural
37 soils to improve material grading and particle mineralogy control. Two soils were
38 formed by combining *a priori* known quantities of silty-clay (“Birtley clay”, LL
39 58.8%, PL 25.7%, 50% kaolinitic clay by mass (Smith and Augarde, 2014)), sand
40 and gravel, sieved to remove particles larger than 10mm. Soil constituents (by
41 mass) are given in Table 1 and particle grading curves are shown in Figure 1.
42 As flocculants interact with the soil clay particles, engineered soils were designed
43 to investigate the effect of changing clay contents on strength change on CaCl_2
44 addition. A low gravel content was used to improve specimen compaction and
45 testing (Beckett, 2011). Soils are labelled according to their silty-clay:sand:gravel
46 content ratios by mass, where $[x]$ in Table 1 gives the maximum percentage devia-
47 tion between designed and actual mix proportions (Hall and Djerbib, 2004; Smith

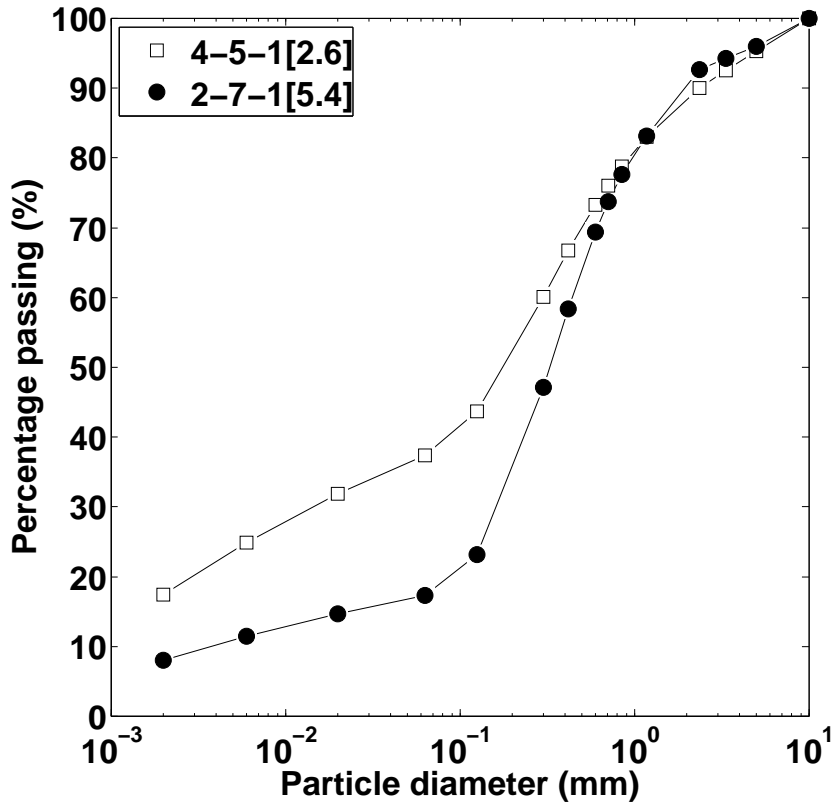


Figure 1: Particle grading curves for Soils 4-5-1 and 2-7-1 (untreated)

48 and Augarde, 2013). *Untreated* material optimum water contents (OWCs) and
 49 maximum dry densities ($\rho_{d_{max}}$), determined using the Light Proctor compaction
 50 test (BS 1377:1990), are also given in Table 1.

51 (Insert Figure 1 somewhere near here)

52 (Insert Table 1 somewhere near here)

Table 1: Soil constituents, OWC and $\rho_{d_{max}}$

Soil	Silty-clay (%)	Sand (%)	Gravel (%)	OWC (%)	$\rho_{d_{max}}$ (kg/m ³)
4-5-1[2.6]	40	50	10	11.0	1940
2-7-1[5.4]	20	70	10	12.0	1960

53 3.2. Tensile test specimen manufacture

54 Tensile strength was selected to compare treated and untreated material be-
55 haviour due to the concern of cracking in engineered cover systems. Specimens
56 were tested using the Brazilian test; it is envisaged that its low cost and conve-
57 nience would make it ideal for cover material bulk testing. Although originally
58 developed for hard materials, for example rocks, the Brazilian test is also capa-
59 ble of testing low-plasticity soils and specimens comprising multiple compaction
60 planes (Frydman, 1964; Dexter and Kroesbergen, 1985). Direct testing methods
61 (e.g. Lu et al. (2007)) were not deemed suitable due to the high anticipated soil
62 strengths.

63 3.2.1. Specimen size

64 Specimen size greatly affects the tensile strength inferred from the Brazilian
65 test, due to the relative size of the loaded surface (Rocco et al., 1999). Specimens
66 of different diameters were manufactured to investigate size effect on measured
67 tensile strength. Untreated $\varnothing 50\text{mm}$ and $\varnothing 100\text{mm}$ (2:1 diameter-to-length ratio)
68 Soil 4-5-1 and 2-7-1 specimens were compacted to $\rho_{d_{max}}$ given in Table 1, at the
69 material OWC. Six specimens were tested per soil and specimen size. Specimens
70 were air-dried to water contents of $4.0 \pm 0.1\%$ and wrapped in clear plastic for
71 48 hours for suction equilibration. 4.0% was selected as it produced sufficient
72 strength for transportation in a relatively short drying time. Specimens were
73 then tested to failure at a constant displacement rate of $0.2\text{mm}/\text{min}$, suggested

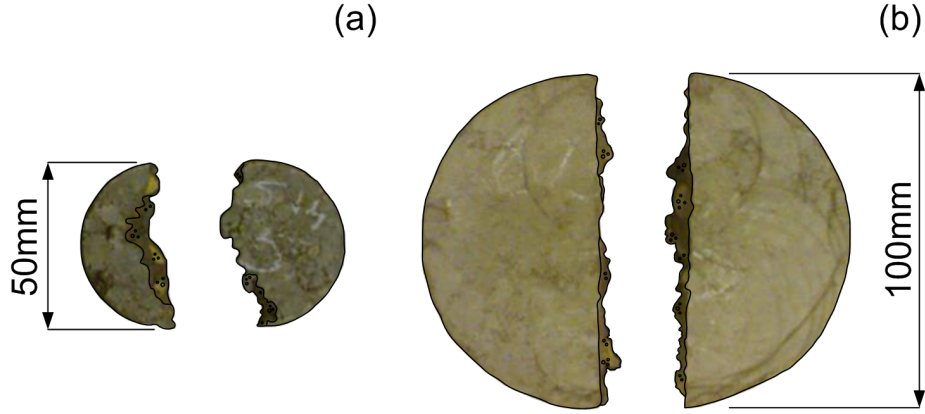


Figure 2: Failure of Brazilian test samples indicating non-vertical and vertical failure cracks: a) 50 mm diameter 25 mm high; b) 100 mm diameter 50 mm high. Outlines and shading added for clarity.

74 by Stirling et al. (2015) to be sufficiently slow for repeatable tensile strength
 75 testing. Tensile strength (σ_t) was determined via

$$\sigma_t = \frac{P}{\pi RL}, \quad (2)$$

76 where P is the peak applied compressive load and R and L are the specimen
 77 radius and length respectively. For convenience, tensile strength has been taken
 78 as *positive*.

79 Significant variability was found for σ_t for $\varnothing 50\text{mm}$ specimens, whilst good
 80 consistency was found for all $\varnothing 100\text{mm}$ specimens (Table 2). Typical failure cracks
 81 for these specimens are shown in Figure 2. Higher $\varnothing 50\text{mm}$ variability was due to
 82 the greater influence of larger soil particles on crack formation and propagation
 83 (Eqn 2 assumes a planar failure crack). $\varnothing 100\text{mm}$ specimens were therefore used
 84 for further testing.

85 (Insert Figure 2 somewhere near here)

Table 2: Average tensile strength and standard deviation for different specimen diameters. s : standard deviation; CV: coefficient of variation.

Soil	Diameter (mm)	σ_t (kPa)	s (kPa, CV (%))
4-5-1	50	59.1	5.6, 9.5
4-5-1	100	86.4	4.7, 5.4
2-7-1	50	50.2	8.5, 16.9
2-7-1	100	63.8	2.7, 4.2

86 (Insert Table 2 somewhere near here)

87 3.2.2. Flocculated specimens

88 Calcium chloride (CaCl_2) was used as a flocculating agent due to its ready
 89 availability and non-toxic nature. It is noted, however, that it is unlikely that
 90 pure CaCl_2 would be purposefully added to soil as a source of calcium, given its
 91 expense and the availability of cheaper sources, for example gypsum.

92 $\varnothing 100\text{mm}$ specimens of soils 4-5-1 and 2-7-1 were manufactured using either
 93 deionised water or a solution of 40dS/m CaCl_2 (3.1% CaCl_2 content by water
 94 mass). 40dS/m was selected to be representative of saline groundwater concen-
 95 trations (Clayton et al., 1995). Wetted soil was left in a sealed bag to equilibrate
 96 for 48 hours after wetting prior to manufacture. All specimens were compacted to
 97 the *untreated* soil $\rho_{d,max}$ (at the corresponding OWC) given in Table 1, using the
 98 previously-described procedure. Once compacted, specimens were transferred to
 99 wire racks and air-dried to the desired water content for testing, whereupon they
 100 were wrapped in clear plastic for 48 hours for suction equilibration (all specimens
 101 were therefore tested under drying conditions). Specimens were then removed
 102 from their wrapping and tested, using the Brazilian test, to failure at a constant
 103 displacement rate of 0.2mm/min. σ_t was then determined via Eqn 2.

104 *3.3. Retention property testing*

105 Treated and untreated soil retention properties were determined through a
 106 combination of filter paper and vapour equilibrium (for suctions >10MPa) test-
 107 ing. These methods were selected in preference to direct suction measurement
 108 (e.g. tensiometers) as they were able to cover the large range of suctions an-
 109 ticipated. Filter paper testing was conducted as per ASTM D5298-10 . The
 110 relationship

$$\ln \psi_t = -4.6234 - 3.6454 \ln(w_{fp}) \quad (3)$$

111 was used to calculate total suction (ψ_t) values from suspended filter paper gravi-
 112 metric water content, w_{fp} . Eqn 3 was determined via a best-fit solution to filter
 113 paper data presented in Hamblin (1981). Soil water retention curves (SWRCs)
 114 for Soils 4-5-1 and 2-7-1 are shown in Figures 3 and 4 respectively.

115 Total suction values were approximated using

$$S_r = \left(1 + \frac{\log \left(1 + \frac{\psi_t}{10^9} \right)}{\log(2)} \right) \times \frac{1}{\left(\ln \left(e + \left(\frac{\psi_t}{a} \right)^n \right) \right)^m} \quad (4)$$

116 where S_r is the degree of saturation and e is the Euler number (≈ 2.7183). Fitting
 117 parameter a , m and n , residual suction ($\psi_{t,res}$) and saturation ($S_{r,res}$) values
 118 are given in Table 3 (Fredlund and Xing, 1994). Eqn 4 was used to determine
 119 specimen suction conditions at testing (i.e. after drying) from measured S_r values.

120 Figures 3 and 4 show an increase in ψ_t for given S_r values; this is due to the
 121 addition of CaCl_2 and an increase in osmotic suction (Dao et al., 2008). The ef-
 122 fect of the addition of the flocculant agent is evident through increased $S_{r,res}$ and
 123 $\psi_{t,res}$ values for both soils, which indicate a change in soil structure. Complemen-
 124 tary information regarding soil structural changes, for example as obtained by

Table 3: Fitting parameters used with Eqn 4

	Soil	a	m	n	$\psi_{t,res}$ (MPa)	$S_{r,res}$
4-5-1	0 dS/m	1.60	0.90	3.50	4.0	0.26
	40 dS/m	2.10	0.60	2.50	10.0	0.33
2-7-1	0 dS/m	1.00	0.85	4.75	2.2	0.22
	40 dS/m	1.25	0.85	2.50	6.0	0.28

125 Scanning Electron Microscopy (SEM), X-Ray CT (XRCT) or Mercury Intrusion
 126 Porosimmetry (MIP) is, unfortunately, not available.

127 (Insert Figure 3 somewhere near here)

128 (Insert Figure 4 somewhere near here)

129 (Insert Table 3 somewhere near here)

130 4. Results and discussion

131 4.1. Tensile strengths

132 Variations in σ_t with changing S_r are compared in Figures 5 and 6 for treated
 133 and untreated Soils 4-5-1 and 2-7-1 respectively. Linear relationships have been
 134 added to indicate the rough data trends. All specimens displayed nominally-
 135 linear failure cracks, as shown in Figure 2(b), indicating that the use of Eqn 2 is
 136 valid.

137 Figure 5 shows a general reduction in σ_t on CaCl_2 treatment, which is typical
 138 of results for clayey soils found in previous investigations. However, the opposite
 139 is shown results in Figure 6: an unexpected *increase* in σ_t is observed. Notably, σ_t
 140 results and trend gradients are similar for both treated soils (Figure 7); additional
 141 data is, however, required to determine whether this similarity is significant.

142 (Insert Figure 5 somewhere near here)

143 (Insert Figure 6 somewhere near here)

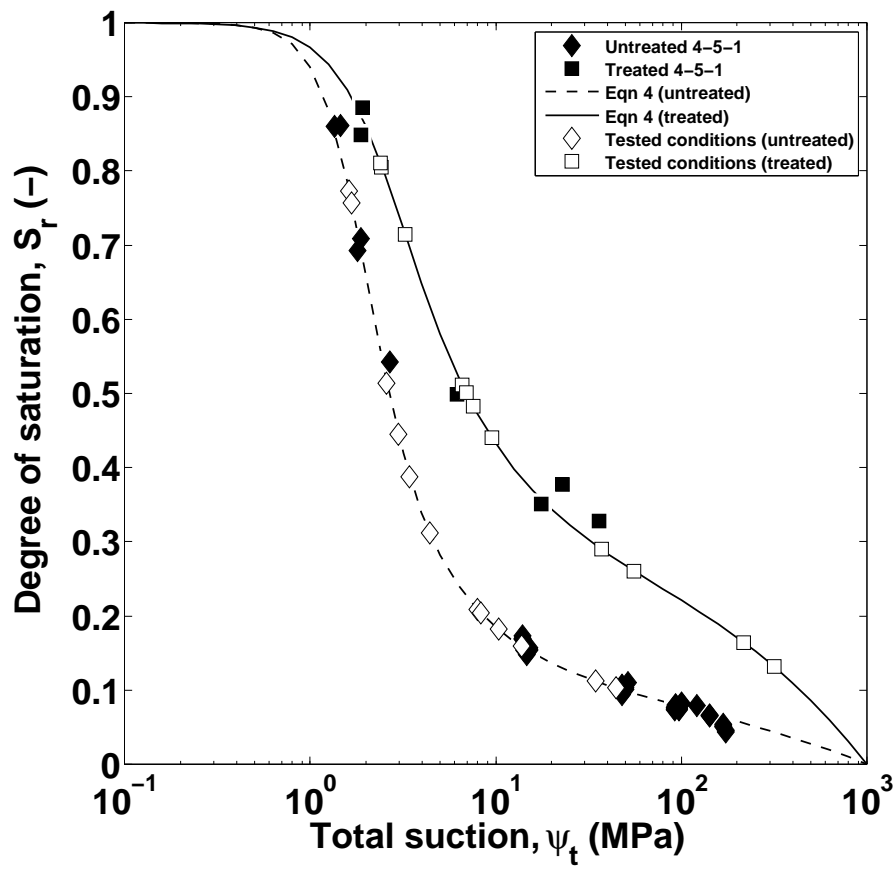


Figure 3: Treated and untreated SWRCs for soil 4-5-1. White markers show drying suction conditions for treated and untreated Brazilian test specimens.

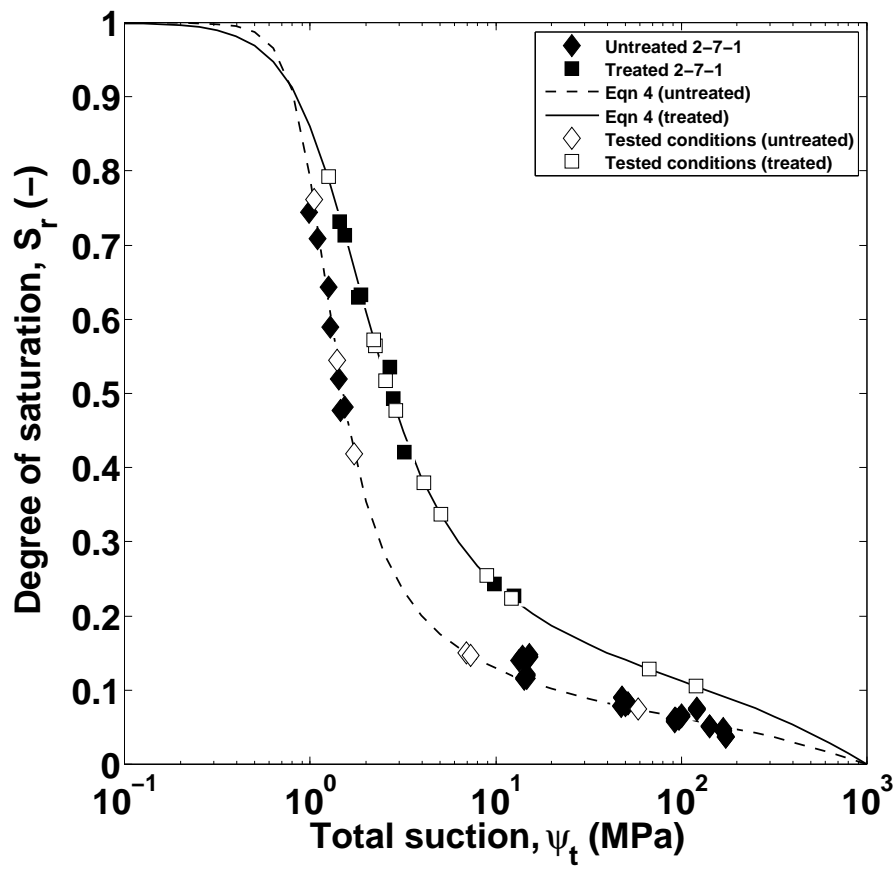


Figure 4: Treated and untreated SWRCs for soil 2-7-1. White markers show drying suction conditions for treated and untreated Brazilian test specimens.

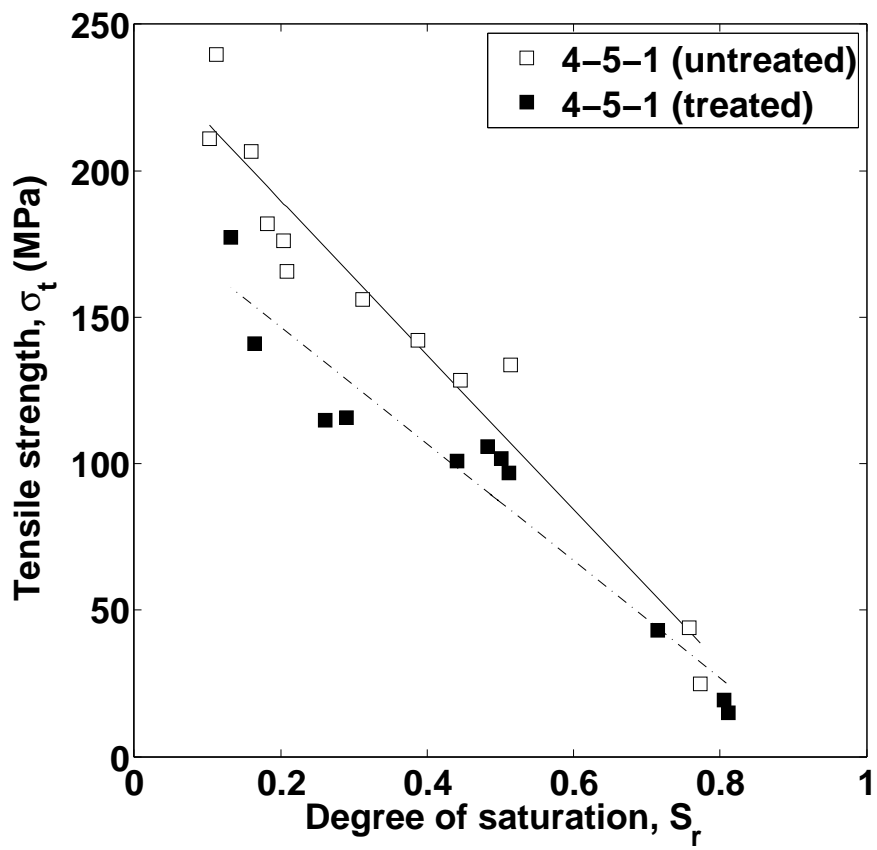


Figure 5: Tensile strength against degree of saturation for treated and untreated Soil 4-5-1 specimens

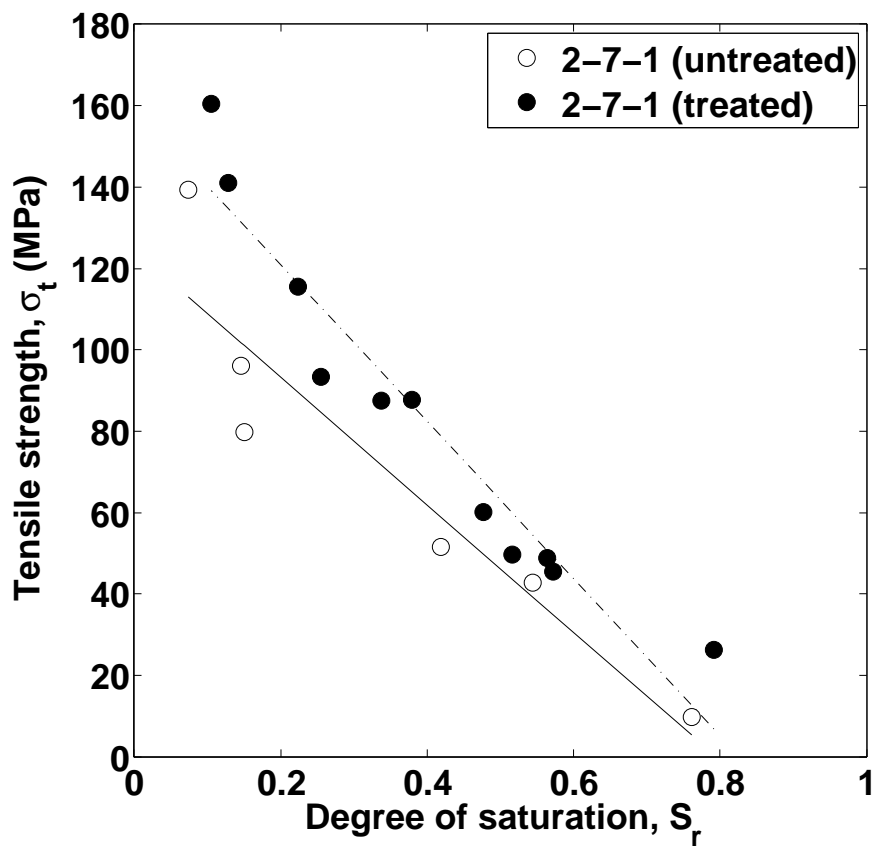


Figure 6: Tensile strength against degree of saturation for treated and untreated Soil 2-7-1 specimens

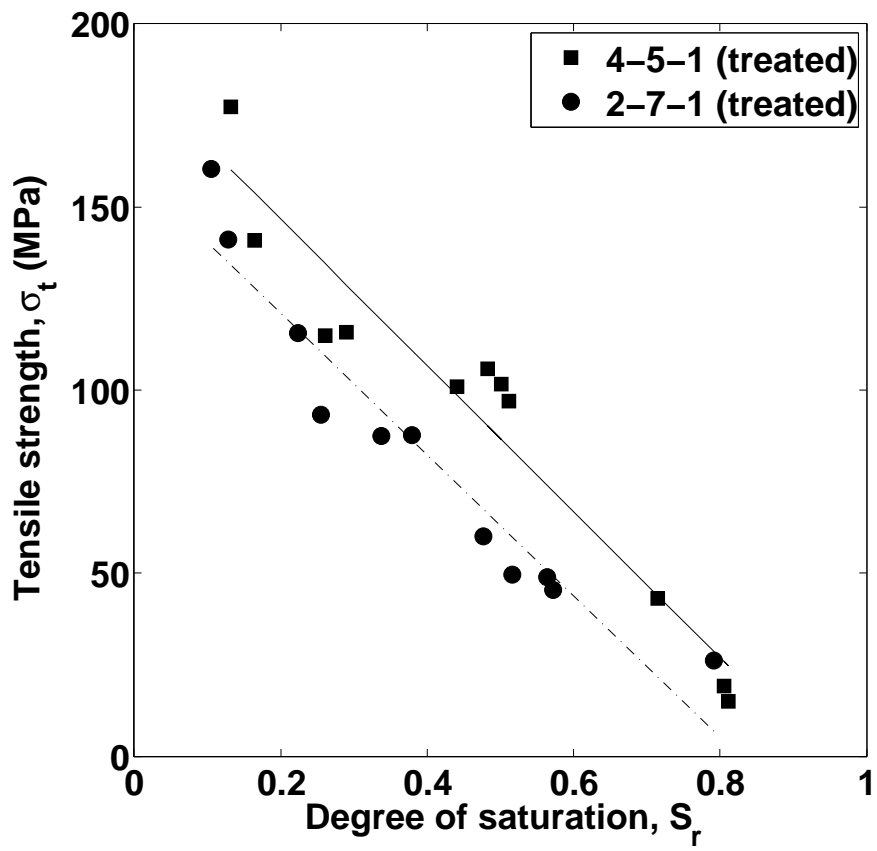


Figure 7: Comparison of treated Soil 4-5-1 and 2-7-1 results, with descriptive linear trends.

144 (Insert Figure 7 somewhere near here)

145 4.2. Extended Mohr-Coulomb analysis

146 The Extended Mohr-Coulomb Yield Criterion (EMC) was used to interpret
147 observed changes in σ_t with associated changes in suction. The EMC approach
148 adds a suction dimension to the familiar Mohr-Coulomb criterion. Shear strength,
149 τ_f , is given by:

$$\tau_f = c + (\sigma_f - u_a) \tan(\phi) + (\psi) \tan(\phi^b) \quad (5)$$

150 where c is apparent cohesion, σ_f is the normal stress corresponding to τ_f , u_a
151 is pore air pressure (usually assumed to equal zero) and ψ is suction. ϕ and
152 ϕ^b are friction angles describing the change in τ_f with σ (as for the saturated
153 case) and τ_f with ψ respectively (Fredlund et al., 1978). It is generally accepted
154 that ϕ^b is a function of S_r and diminishes to small values as S_r approaches zero.
155 Construction of the EMC failure envelope for a Brazilian test conducted at some
156 non-zero suction value ψ is shown in Figure 8 (Jaeger et al. (2007), reported in
157 Consoli et al. (2014)). The relationships $\sigma_3 = \sigma_t$ and $\sigma_1 = -3\sigma_t$ are derived in Li
158 and Wong (2013) and are valid provided $\frac{a}{y_1}$ is less than 0.27, where a is the width
159 of the deformed contact region and y_1 is the change in axial diameter (Frydman,
160 1964). These relationships are used here as little deformation (≤ 1 mm reduction
161 in diameter, negligible flattening of contact area) occurred during testing.

162 (Insert Figure 8 somewhere near here)

163 Figure 9 shows calculated Mohr's circles for untreated Soil 4-5-1 specimens.
164 A distinct change in the rate of circle growth with increasing ψ_t is seen either side
165 of $\psi_{t,res}$ (demarcated in Figure 9). The residual suction can be considered to be
166 the transition between continuous (funicular) and discontinuous (pendular) water
167 phases (Schubert (1975), reported in Song et al. (2012)); it is therefore reasonable

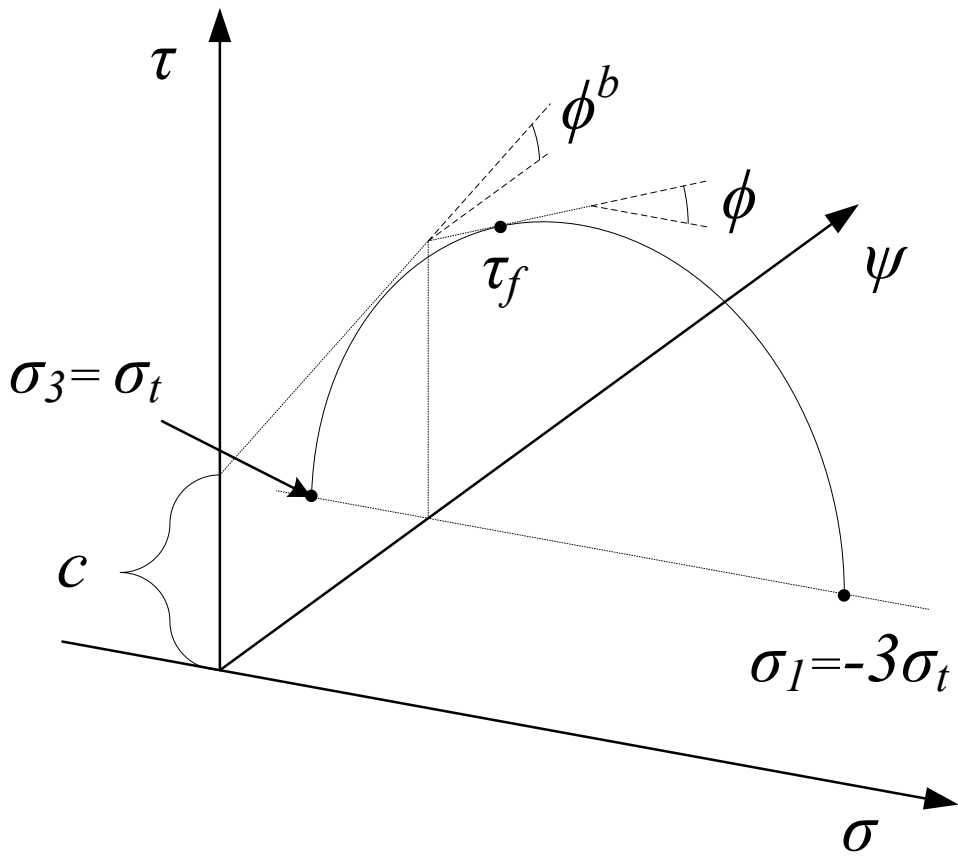


Figure 8: Example Brazilian test EMC failure surface construction

168 that a mechanistic change in the contribution of suction to strength occurs at this
169 point. Fitting planes to data $< \psi_{t,res}$ produced negative c values, which is not
170 consistent with established EMC theory. A single plane was therefore fitted to
171 data $\geq \psi_{t,res}$. Fredlund et al. (1996) showed that changes in ϕ^b are related to
172 those in $\frac{dS_r}{d\psi}$; as S_r changes little with ψ_t in the residual suction range, a single
173 plane is suitable to describe data in this region. The significance of negative c
174 values below $\psi_{t,res}$ is a topic for further study. Shear strengths predicted by the
175 fitted failure plane, $\tau_{f,p}$, can be calculated via

$$\tau_{f,p} = c_{res} + (\sigma_f - u_a) \tan(\phi_{res}) + (\psi_t) \tan(\phi_{res}^b) \quad (6)$$

176 where subscript res denotes parameters for suctions above $\psi_{t,res}$. σ_f is found
177 via $\sigma_f = \sigma_t (1 - 2 \sin \phi)$, using the appropriate value of ϕ and where σ_t is the
178 measured value of tensile strength, found using Eqn 2. Predicted tensile strength
179 ($\sigma_{t,p}$) can then be found via

$$\sigma_{t,p} = \text{centre} - \text{radius} = \sigma_f + \tau_{f,p} \left(\sin(\phi_{res}) - \frac{1}{\cos(\phi_{res})} \right) \quad (7)$$

180 using the appropriate value of τ_f . Fitted EMC parameter and r^2 values are given
181 in Table 4. Geometrical relationships used in the derivation of Eqn 7 are shown
182 in Figure 10.

183 Measured against predicted values of σ_t for all tested soil conditions are shown
184 in Figure 11. That a good approximation to σ_t was found in Figure 11 is to be
185 expected, given the fitting nature of failure plane selection. However, the distinc-
186 tion between changes in σ_t with ψ_t above and below $\psi_{t,res}$ is readily apparent,
187 supporting the interpretation of a mechanistic change in suction's contribution

Table 4: Fitted EMC failure surface parameter values

Soil	Treatment	c_{res} (kPa)	ϕ_{res} ($^{\circ}$)	ϕ_{res}^b ($^{\circ}$)	r^2
4-5-1	0 dS/m	272.1	24.6	0.15	0.92
	40 dS/m	173.3	23.1	0.02	0.94
2-7-1	0 dS/m	134.6	23.6	0.10	0.98
	40 dS/m	166.7	23.6	0.05	0.97

188 to strength between these two regions.

189 (Insert Figure 9 somewhere near here)

190 (Insert Figure 10 somewhere near here)

191 (Insert Figure 11 somewhere near here)

192 (Insert Table 4 somewhere near here)

193 Changes in EMC parameter values in Table 4 can explain observed changes in
 194 σ_t with S_r on CaCl_2 addition in Figures 5 and 6. The effect of flocculant addition
 195 on EMC parameters was mostly consistent between soils:

- 196 • ϕ_{res}^b decreased (more severely for Soil 4-5-1);
- 197 • little change ϕ_{res} occurred.

198 However, c_{res} decreased for Soil 4-5-1 but increased for Soil 2-7-1. A decrease
 199 in c_{res} demonstrates a reduced contribution to strength for suctions $> \psi_{t,res}$.
 200 Contrariwise, this contribution increased for Soil 2-7-1. These effects combined
 201 to produce the unexpected increase in σ_t for Soil 2-7-1 on CaCl_2 addition, but
 202 the traditional decrease in σ_t for Soil 4-5-1, suggestibly due to its higher floccula-
 203 tion susceptibility. This observation has important implications for geotechnical
 204 structures: if soils are only marginally susceptible to flocculation, an increase
 205 in tensile strength may occur instead of an anticipated decrease which may, for
 206 example, prevent root penetration.

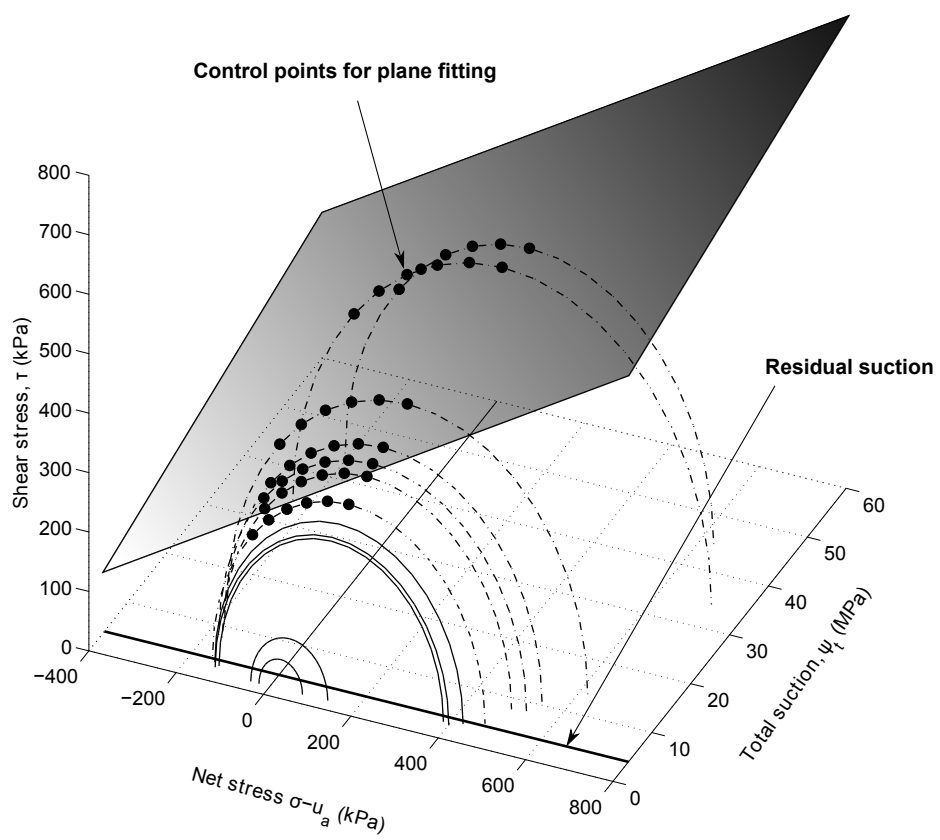


Figure 9: EMC failure surface for untreated Soil 4-5-1 above $\psi_{t,res}$. Solid lines (—): Mohr's circles below $\psi_{t,res}$; dashed lines (---): Mohr's circles above $\psi_{t,res}$; solid markers (●): points on the circles used for plane-fitting.

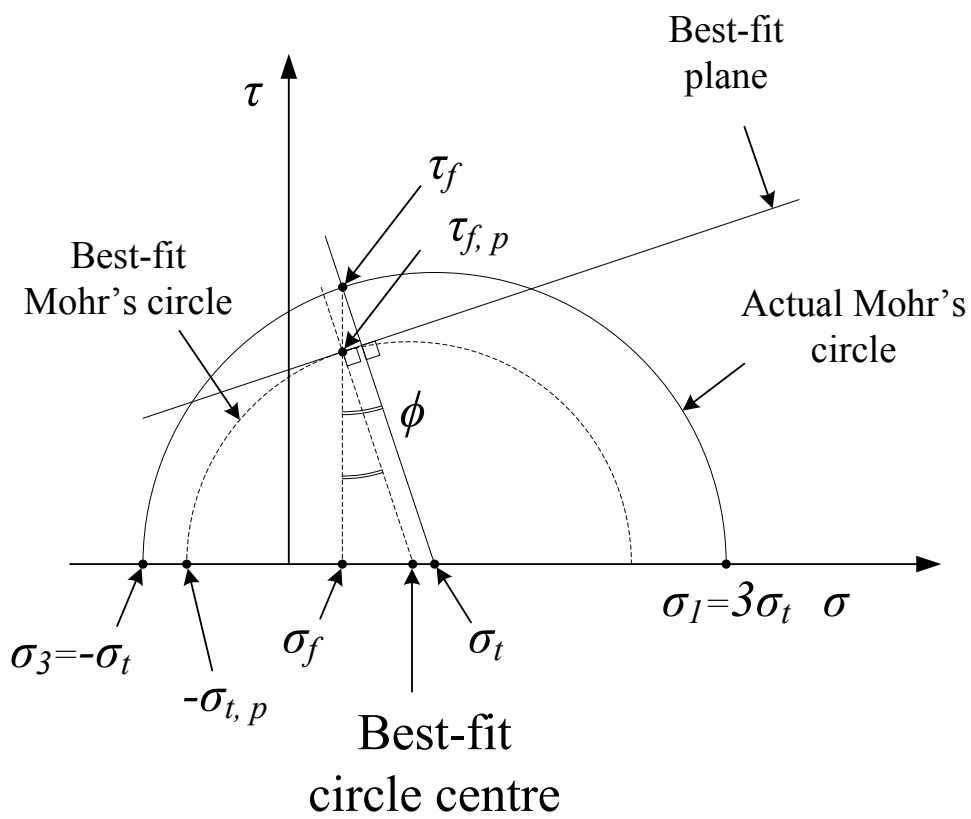


Figure 10: Geometrical relationships between predicted and measured strength values

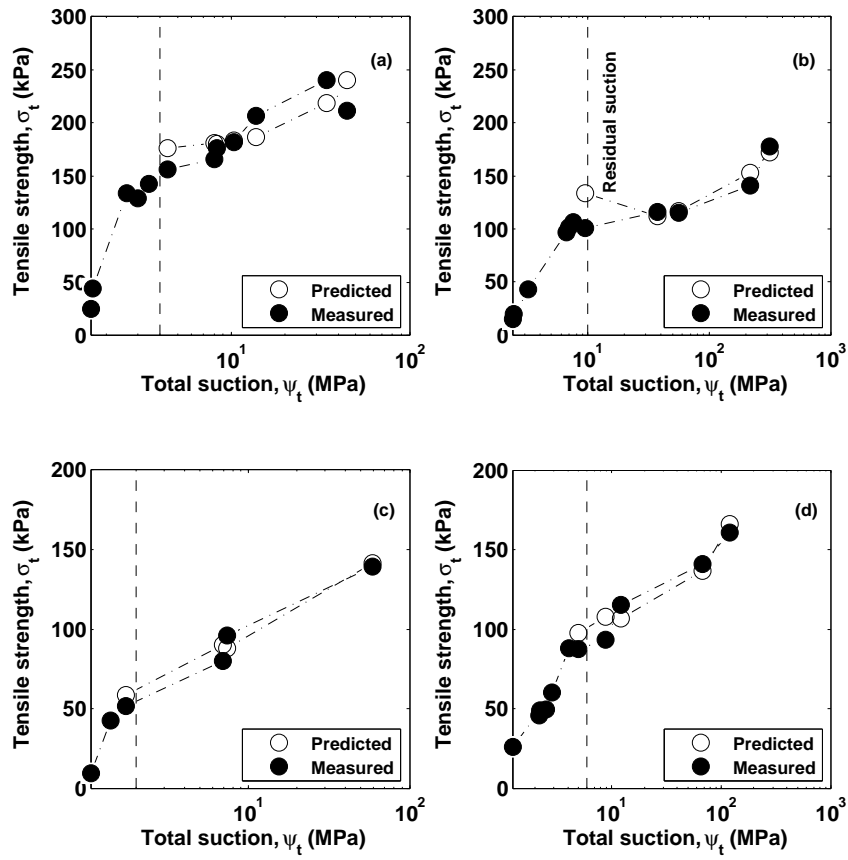


Figure 11: Measured and predicted ($\geq \psi_{t,res}$) tensile strengths for tested soil conditions: a) 4-5-1, untreated; b) 4-5-1, treated; c) 2-7-1, untreated; d) 2-7-1, treated.

207 **5. Conclusions**

208 This paper investigated the effect of CaCl_2 addition on the tensile strength
209 of two compacted engineered soils equilibrated to a range of suction conditions.
210 Contrary to literature results, the traditional decrease in tensile strength on flocculant
211 addition was not found for a soil with a low clay content. Soil retention
212 properties were used in combination with an EMC analysis to examine this behaviour.
213 It was determined that CaCl_2 addition decreased the contribution of
214 suction to strength (ϕ_{res}^b) in both soils. However, material cohesion (c_{res}) for
215 suctions $> \psi_{t,res}$ increased for Soil 2-7-1 but decreased for Soil 4-5-1, resulting in
216 the observed changes in σ_t as the soils dried. Changes in soil flocculation susceptibility
217 therefore have a significant effect on resulting soil properties. Similarities
218 between treated material tensile strength and detailed analyses of soil structural
219 changes using SEM, XRCT and/or MIP are subjects for further investigation.

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296 **Figure captions**

- 297 1. Particle grading curves for Soils 4-5-1 and 2-7-1 (untreated)
- 298 2. Failure of Brazilian test samples indicating non-vertical and vertical failure
299 cracks: a) 50 mm diameter 25 mm high; b) 100 mm diameter 50 mm high.
300 Outlines and shading added for clarity.
- 301 3. Treated and untreated SWRCs for soil 4-5-1. White markers show drying
302 suction conditions for treated and untreated Brazilian test specimens.
- 303 4. Treated and untreated SWRCs for soil 2-7-1. White markers show drying
304 suction conditions for treated and untreated Brazilian test specimens.
- 305 5. Tensile strength against degree of saturation for treated and untreated Soil
306 4-5-1 specimens
- 307 6. Tensile strength against degree of saturation for treated and untreated Soil
308 2-7-1 specimens
- 309 7. Comparison of treated Soil 4-5-1 and 2-7-1 results, with descriptive linear
310 trends.
- 311 8. Example Brazilian test EMC failure surface construction
- 312 9. EMC failure surface for untreated Soil 4-5-1 above $\psi_{t,res}$. Solid lines (—):
313 Mohr's circles below $\psi_{t,res}$; dashed lines (---): Mohr's circles above $\psi_{t,res}$;
314 solid markers (●): points on the circles used for plane-fitting.
- 315 10. Geometrical relationships between predicted and measured strength values
- 316 11. Measured and predicted ($\geq \psi_{t,res}$) tensile strengths for tested soil condi-
317 tions: a) 4-5-1, untreated; b) 4-5-1, treated; c) 2-7-1, untreated; d) 2-7-1,
318 treated.

319 **Table captions**

- 320 1. Soil constituents, OWC and $\rho_{d_{max}}$
- 321 2. Fitting parameters used with Eqn 4
- 322 3. Fitted EMC failure surface parameter values