

1	Assessment of lime treatment of expansive clays with different mineralogy at low and high
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19	Re-submission
20	26 <sup>th</sup> June 2019

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# Assessment of lime treatment of expansive clays with different mineralogy at low and high temperatures

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24 Abstract: This paper examines the impacts of clay mineralogy on the effectiveness of lime stabilisation 25 at different temperatures. A comprehensive experimental programme was conducted to track down 26 the evolution of lime-clay reactions and their durations through monitoring the evolution of strength 27 gain at predetermined times using the Unconfined Compressive Strength (UCS) test. The study 28 examined clays with different mineralogy compositions comprising Na<sup>+</sup> Bentonite and Ball (Kaolinite) 29 clay. Four different clays were tested including 100% bentonite, 100% Ball clay and two clay mixtures 30 with ratios of 1:1 and 1:3 by mass of bentonite to Ball clay. All clays were treated using a range of lime 31 content up to 25% and cured for a period of time up to 672 h at two different temperatures of 20 and 32 40°C. The results showed that the continuity of the fast phase (stage 1) of strength gain was dependent 33 on the availability of lime in particular at the higher temperature. Whereas, for the same lime content, 34 the duration of the fast phase and the kinetic of strength gain were significantly related to the clay 35 mineralogy and curing temperature. Except for the initial strength gain at 0 h curing time, the lime-36 treated Ball clay specimens at 20°C appeared to show no strength gain throughout the curing period 37 that extended up to 672 h. However, when curing occurred at 40°C, the no strength gain stage only lasted for 72 h after which a gradual increase in the strength was observed over the remaining curing 38 39 period of time. The addition of Bentonite to Ball clay succeeded in kicking off the strength gain after a 40 short period of curing time at both curing temperatures.

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42 Keywords: Lime stabilised clays, clay mineralogy, Unconfined Compressive Strength, Curing
 43 temperature, Pozzolanic reaction.

#### 45 **1. Introduction**

Natural soils in work-sites are sometimes detrimental to the construction of engineering projects. 46 Problematic soils such as soft and expansive soils are a real source of concern to the long term stability 47 48 of structures if care is not taken. Expansive soils could generate huge distress due to their volume 49 change in response to a slight change in their water content. On the other hand, soft soils are 50 characterized by their low shear strength and poor workability. In earthwork, replacing these soils is 51 sometimes economically and sustainably unjustifiable in particular if they can be stabilised to improve 52 their behaviour. Several techniques have evolved to enable construction on problematic soils such as reinforcement using fibre and planar layers (see for example; Mohamed 2010; Mirzababaei et al., 2017 53 54 and 2018), piled reinforced embankments (see for example; Aqoub et al., 2018) and chemical agent 55 (see for example; Alrubaye et al., 2018; Coudert et al., 2019; Kang et al., 2019; Yaghoubi et al., 2019)

56 Chemical treatment using e.g. lime and/or cement is an alternative method to seize the volume 57 change of swelling clays. The use of lime as a binding agent is becoming a popular method due to its 58 abundant availability and cost-effectiveness. When mixed with swelling clays, lime enhances the 59 mechanical properties, workability and reduce sensitivity to absorption and release of water. The lime 60 in both states; Hydrated lime Ca(OH)<sub>2</sub> and Quick lime CaO, have been used to stabilised swelling clays. 61 Cation exchange, flocculation and agglomeration, and pozzolanic reaction in addition to carbonization 62 are well-reported mechanisms that are in charge of causing changes in the clay characteristics after the addition of the lime in the presence of water. These mechanisms have been subjected to 63 64 numerous investigations (see for example; Diamond and Kinter, 1965; Rogers and Roff, 1997; 65 Boardman et al., 2001; Puppala et al., 2005; Di Sante et al., 2014; Zhao et al., 2015; Vitale et al., 2016; 66 Vitale et al., 2017; Chemeda et al., 2018; Gao et al., 2018; Di Sante et al., 2019). Precisely, the added 67 lime dissolves partially into calcium ions and hydroxyl ions in the pore water. The calcium ions as 68 divalent cations resort to subrogate the lesser charge cations surrounding the surface of clay particles 69 in a mechanism so-called cation exchange. The surfaces of clay particles inherently carry negative

70 charges which are balanced by native cations forming a diffuse double layer surrounding each clay 71 particle. The cation exchange leads to a reduction in the thickness of the diffuse double layer, hence 72 the charge on the surface of clay particles is balanced by a smaller number of cations (Strawn et al., 73 2015). Immediately, neighbouring clay particles become closer and interact with each other leading 74 to the reconfiguration of their positions into flocs and clusters in a so-called flocculation and 75 agglomeration mechanism. In contrast, the release of hydroxyl ions creates an alkaline environment 76 in the pore water. Such an aggressive alkaline environment attacks the surface of clay particle, causing 77 a launch of alumina and silica ions in the pore water. These ions react with the available calcium and 78 hydroxyl ions to form the cementitious compounds in a process called "pozzolanic reaction". However, 79 a point of controversy remains as to whether these mechanisms take place consecutively or 80 simultaneously (Boardman et al., 2001).

81 The effect of the aforementioned mechanisms and reactions is tangible through observing the 82 changes that occur in the soil characteristics such as swelling behaviour, plasticity indices, hydraulic 83 conductivity, compaction and strength. The strength of lime-stabilised clays is one of the key 84 parameters required in the engineering design of earthworks. Necessities for assessing the evolution 85 of strength, long-term stability and desirable lime content require the need for not only a practical but also a relatively quick test. Unconfined Compressive Strength (UCS) was reported to satisfactorily suit 86 87 the requirement to determine the lime content that is desirable to cause an optimum change in the 88 strength properties. A few studies used the UCS tests as a mean to monitor the evolution of lime 89 reaction. Locat et al. (1990) monitored the development of strength gain in four types of sensitive 90 clays that were treated by different lime contents reaching up to 10%. The results showed that the strength gain passed through three distinct phases. The strength gain showed small improvement 91 92 during the first phase, followed by a significant growth during the intermediate phase before 93 slowing down or even coming to a halt through the final phase. Locat et al. (1990) attributed the 94 behaviour of strength over the final phase to the completion of the pozzolanic reaction. Hashemi

95 et al. (2018) observed similar three phases for the strength development over a curing period of up 96 to 28 days on sand-bentonite mixtures that were treated by various percentages of quick lime ranging 97 from 3 to 8%. However, both studies did not indicate the role of curing temperature on the evolution 98 of strength gain. Using chemical approaches, Al-Mukhtar et al. (2010a) and Al-Mukhtar et al. (2010b) 99 demonstrated that the rate of pozzolanic reaction at a curing temperature of 50°C was six-fold higher 100 than that observed to occur at a curing temperature of 20°C. This was in agreement with the finding 101 of an experimental investigation that was conducted by De Windt et al. (2014) to evaluate the impact 102 of curing times up to 98 days and two ambient temperatures of 20 and 50°C on lime treated bentonite. 103 The results showed that the ambient temperature of 50°C multiplied the kinetic of pozzolanic reaction 104 by five times compared with that observed at a curing temperature of 20°C.

105 . The effectiveness of lime stabilization was found to be dependent on many key parameters such 106 as mineralogy composition of clay, lime content, moisture content, mixing time and technique, mellowing time, mellowing temperature, compaction method, dry unit weight, curing 107 108 temperature and curing time (Bell, 1996; Bozbey and Garaisayev, 2010; Kitazume and Terashi, 109 2013; Ali and Mohamed, 2017; Ali and Mohamed, 2018; Al-Alwan, 2019; Jahandari et al., 2019). These key parameters require systematic testing and evaluation. In the current study, four clays with 110 111 different mineralogy compositions, covering a wide range of liquid limit were used. Lime with a range 112 of 5 to 25 % by dry weight was added to clays with different mineralogical compositions to assess its 113 effect on the evolution of strength and the continuity of chemical reactions at 20 and 40°C throughout a curing period that lasted up to 672 h. Earlier results of (Ali and Mohamed, 2018) on lime stabilised 114 115 bentonite with up to 13% lime showed the occurrence of two distinct stages in the strength gain that 116 are dependent upon lime content and temperature. However, authors did not assess different clays 117 and the continuity of the distinct stages, in particular, the short-term fast strength gain, at higher lime contents and extended period of curing time. Therefore, this paper aims to provide a comprehensive 118 119 assessment of the kinetic of strength gain in the short- and long-term stages so as to enable deep

120 understanding of the key factors that govern the kinetics of strength gain over time in different soils.

121 Furthermore, the results are coupled with and supported by earlier studies on the microstructure and

122 chemical reactions to strengthen the discussion on the changes in the strength characteristics.

123 2. Methodology

124 2.1. Materials

125 Two different types of clay, namely Bentonite and Ball clays, were used in the current study. The main 126 clay mineral in the Bentonite is the montmorillonite mineral, whereas the kaolinite mineral is the 127 major clay mineral in the Ball clay. All clays were supplied by Potclays Ltd, UK in a powder form. The chemical analysis of both Bentonite and Ball clays are shown in Table 1. Both clays were mixed in 128 129 different proportions to obtain two additional clay mixtures with a ratio of 1:3 and 1:1 Bentonite to 130 Ball clay by dry mass, as illustrated in Table 2. The Bentonite-Ball clay mixtures were selected to assess the accumulation of calcium on the surface of kaolinite particles which might cause delay of 131 132 disassociation of alumina and silica ions by preventing the alkaline environment from attacking the 133 surface of kaolinite particles. In this case, Bentonite, which is an active clay, is introduced in the mix 134 as a rival consumer for the calcium to reduce the potential accumulation of calcium. The potential 135 reduction in the accumulation of calcium cations allows the alkaline environment to attack the surface 136 of clay particles to begin the dissolution of alumina and silica and thus, to initiate the formation of 137 cementitious compounds and strength gain in a shorter time. The geotechnical properties of the four used clays in this investigation are illustrated in Table 2. The experimental data for the characterization 138 139 of clay materials showed that the Liquid Limit (LL) of M1 which is pure Bentonite clay is 320% whereas 140 that of Ball clay is 58%. The other two mixtures that were created by different ratios retained LL of 141 115 and 189% for M3 and M4 respectively. These data demonstrated that the four materials 142 represented a vast range of liquid limit and plasticity index spanning from 320% down to 58% and 143 277% to 26% respectively. Data for the maximum dry unit weight and optimum moisture content were 144 obtained using the developed compaction mould by Ali and Mohamed (2017). The data showed that

as the liquid limit of the clay material was lowered, an increased maximum dry unit weight and
decreased optimum water content were recorded as illustrated in Table 2. Non-hydraulic high calcium
hydrated lime that satisfied the requirements of BS EN 459-1 was used. About 97 to 99.5% of lime
powder is finer than 90 microns. The availability of lime in the form of calcium hydroxide ranges from
95 to 97%.

150 Table 1: Chemical analysis of primary clays

Component, %	Bentonite clay	Ball clay
SiO <sub>2</sub>	63.02	52.0
$AI_2O_3$	21.08	31.5
Fe <sub>2</sub> O <sub>3</sub>	3.25	1.0
K <sub>2</sub> O	-	2.3
Na <sub>2</sub> O	2.57	0.3
MgO	2.67	0.4
CaO	0.65	0.2
FeO	0.35	-
TiO <sub>2</sub>	-	1.1
L.O.I. @ 1000C*	5.64	11.3
Carbon	-	1.6
Trace	0.72	-

151

# 152 Table 2: Geotechnical properties for utilised clay materials

		Material			
		M1	M2	M3	M4
Composition*	Bentonite clay (%)	100	0	25	50
	Ball clay (%)	0	100	75	50
Liquid limit (%)	-	320	58	115	189
Plastic limit (%)		43	32	36	40
Plasticity Index (%)		277	26	79	149

Maximum dry unit weight (kN/m <sup>3</sup> )	12.16	14.14	13.48	12.95
Optimum moisture content (%)	40	29	32.5	37.5

153 \*The ratio of Bentonite to Ball clay is by mass.

154 2.2 Test procedure and programme

155 In order to minimise potential scattering of UCS results, full attention was given to the experimental 156 procedure in order to avoid possible sources of the scattering including; i. adopting a cautious and 157 thorough mixing technique to eliminate the formation of lime lumps, to ensure a high degree of pulverisation and to reduce the disparity in mixing time, ii. selection of a feasible and efficient 158 compaction method to ensure the uniformity of dry density throughout the specimens and 159 160 repeatability of specimens and iii. conduction of a curing protocol that provided a stable and constant 161 curing temperature and humidity to avoid partial drying out of specimens. It should be noted that an 162 identical procedure was followed for the preparation of all specimens. Each mixture was prepared by 163 adding a predetermined amount of clay with the intended amount of lime and mixed mechanically. 164 Then, a given amount of water was added, and the mixing was continued manually to distribute the 165 water as uniformly as possible at this stage. Subsequently, the mixture was passed through the 2 mm 166 sieve. Immediately, the retained clay-lime mix was kneaded by hand and passed through the same 167 sieve. The mixture was then re-mixed mechanically to ensure homogeneity. In order to avoid the 168 adverse impacts of the mellowing period, all mixtures were compacted directly after finishing the 169 mixing process. All mixtures were compacted statically in five layers to produce specimens with a 170 diameter of 38 mm and a height of 76 mm. Specimens were prepared to achieve a designated 171 maximum dry unit weight at the optimum moisture content as illustrated in Table 2 using the 172 developed compaction mould and the compaction procedure by Ali and Mohamed (2017). The 173 adopted compaction method was found to be effective and efficient in preparing specimen with less 174 than 4% scattering in UCS results (Ali and Mohamed, 2017). This shows a remarkable degree of 175 improvement in reducing the UCS results than the 10% recommended acceptable scattering by 176 (Consoli et al., 2011). Upon completion of compaction, specimens were extracted from the mould and

177 measurements of specimen's mass, and dimensions were taken. Each Specimen was then wrapped 178 properly using a cling film and placed in a double sealed bag. The specimens were then stored in an environmental cupboard at the desired temperature of 20°C or 40°C and 90% relative humidity for 179 180 curing except those that were tested immediately after the compaction process. Table 3 presents the 181 full details of the experimental programme. In total, 336 specimens were prepared to assess the effect 182 of different parameters. Also, 140 specimens were prepared as replicates for quality assurance of UCS 183 values and to confirm the effectiveness of preparation method in alleviating the scattering in the 184 results especially with curing time of 168 h and 672 h. Results of UCS on replicate specimens showed 185 that the scattering in the results did not exceed the 4% even with prolonged curing time. All UCS tests 186 were performed using an automatic loading machine. Data for the axial strain and axial stress were 187 registered automatically every second. The loading velocity at which specimens were tested was 188 1mm/min.

Sarias	Material		Parameters	ters		
Jenes		Variables	Fixed	Note		
1	M1	C = 0, 3, 6, 12, 24, 48, 72,	$\gamma_{d}$ = 12.16 kN/m <sup>3</sup>	Additional specimens for L		
		168 and 672 h	MC= 40%	= 11% after 192 & 216 h		
		T = 20°C and 40°C	MP = 0 h	and L = 13% after 96, 216,		
		L = 5, 7, 9, 11 and 13%		& 240 h at 40°C		
2	M1	C = 0, 3, 6, 12, 24, 48, 72,	$\gamma_d$ = 12.16 kN/m <sup>3</sup>	Excessive lime content		
		96, 144, 168, & 672 h	MC= 40%	Additional specimens for L		
		T = 20°C and 40°C	MP = 0 h	= 17% after 216 h, L =21%		
		L =17, 23 and 25%		after 192 & 240 h and L =		
				25% after 240 & 288 h at		
				40°C		
3	M2	C = 0, 24, 48, 72, 168 and	γ <sub>d</sub> =14.14 kN/m <sup>3</sup>	Additional specimens for		
		672 h	MC= 29%	all lime contents after 3 h		
		T = 20°C and 40°C	MP = 0 h	at 40°C		
		L = 5, 7, 9, 11 and 13%				

189 Table 3: Testing programme

4	M3	C = 0, 24, 48, 72, 168 and	γ <sub>d</sub> =13.48 kN/m <sup>3</sup>	Additional specimens for
		672 h	MC = 32.5%	all lime contents after 3 h
		T = 20°C and 40°C	MP = 0 h	at 40°C
		L = 5, 7, 9, 11 and 13%		
5	M4	C = 0, 24, 48, 72, 168 and	$\gamma_d$ =12.95 kN/m <sup>3</sup>	Additional specimens for
		672 h	MC = 37.5%	all lime content after 3 h
		T = 20°C and 40°C	MP = 0 h	at 40°C
		L = 5, 7, 9, 11 and 13%		

190 where; C = curing time, MP = mellowing period, L = lime content, T = temperature, MC = moisture

191 content and  $\gamma_d$  = Dry unit weight

192

#### 193 3. Results and discussion

#### 194 3.1 M1 Clay (Bentonite clay)

The strength values for all lime treated bentonite specimens that were tested immediately after compaction were higher than double the strength value of the untreated specimen, which was 0.5 MPa. The sudden increase in the strength of lime stabilised bentonite is consistent with earlier observations by Vitale et al. (2017). This increase could be caused by a reduction in the specific surface area which can be attributed to the flocculation and aggregation mechanisms that were prompted by cation exchange phenomena and enhanced by the immediate formation of initial cementitious compounds that takes place instantly after the addition of lime in the presence of water.

The evolutions of strength gain over the curing period at different temperatures of 20°C and 40°C are illustrated in Figure 1 and 2, respectively. The figures revealed that the evolution of strength passes through two phases, depending on the rate of strength gain and can be named first and second phases. During the first phase, the rate of strength gain was extremely high compared with that recorded in the second phase in particular at the higher temperature. The first phase can then be defined as the interval of time after which the rate of strength gain commences to slacken drastically. 208 Due to the role that the higher temperature of 40°C plays in accelerating the strength gain and thus 209 the lime consumption, it is easier to distinguish the onset of the second phase at the higher 210 temperature, unlike at the temperature of 20°C.







213

211

214 Figure 2: Evolution of strength gain with time for lime treated M1 specimens cured at 40°C

Although the strength gain appeared to develop remarkably over the first phase, the rate of strength
gain slightly decreased as time elapsed. Therefore, a polynomial equation was found to best describe

the strength gain over the first phase. The best fit lines that are plotted in Figures 1 and 2 represent
the polynomial relationships that govern the strength evolution at 20 and 40°C, respectively. At 40°C,
the strength gain during the first phase can be given by the Equation 1 until 6, 24, 48, 72, 96, 144, 192,
and 240 h on specimens with lime content of 5, 7, 8, 9, 11, 13, 17, 21 and 25% respectively.

$$UCS_{First \, phase} = -0.0674C^2 + 42.77C + 1360.6 \qquad R^2 = 0.99 \qquad 1$$

The data suggested that the continuity of the first phase was strongly dependent on the lime content, and its duration increased with the increase in the lime content. Here it should be mentioned that higher lime contents from 17 to 25% were considered to assess the continuity of the first phase.

224 However, the data showed that the strength of lime stabilised bentonite during the second phase can 225 be represented by logarithmic relationship reaching strength values of 2, 2.78, 4, 6.6, 8.37, 9.6 and 226 11.3 MPa after 672 h of curing time for lime contents of 5, 7, 9, 11, 13, 17, 21 and 25% respectively. 227 Unlike the relatively shorter first phase at 40°C, the results showed that the first phase at 20°C 228 continued to 672 h with the addition of the substantial amount of lime, e.g. 11, 13, 17, 21, and 25% 229 reaching nearly a strength value of about 4 MPa. In contrast, the first phase was shorter with the 230 addition of 5, 7 and 9% of lime at 20°C achieving 2, 2.5 and 3.1 MPa respectively but it was reached 231 after extremely long periods of curing time in comparison with those recorded at 40°C on specimens 232 treated with the same lime content. During the first phase at 20°C, the strength is governed by 233 Equation 2.

$$UCS_{First phase} = -0.0028C^2 + 5.89C + 1242.6 \quad R^2 = 0.98$$

However, careful inspection of data presented in Figure 2 on specimens cured at 40°C suggested that most of the difference in the strength was gained during the first phase and was a function of the lime content. The rate of strength gain during the second stage was significantly lower but increased with the further addition of lime. In an attempt to describe the evolution of strength over phase 2 at 40°C, Equation 3 was developed based on the attained data. The strength given by Equation 3 evolves logarithmically as a function of lime content (L) and curing time (C) during phase 2 at 40°C.

$$UCS_{second \ phase} = (4L^2 + 41.9L - 235)ln C + (-35.1L^2 + 464.1L - 87.1)$$

240 To aid the discussion on examining the lime consumption, Figure 3 was plotted to present the attained 241 strength results on specimens after 672 h of curing time against the lime content at 40°C and 20°C. 242 The data attained on specimens cured at 40°C showed that there was a linear relationship between 243 strength and lime content up to a lime content of 13% and that the difference in the strength value 244 between two consecutive lime contents was about 1 MPa. This means that the amount of lime was 245 fully consumed within the 672 h of curing time under 40°C. Extrapolating the best fit line at higher 246 lime content would assist with the estimation of the final strength at the time of full consumption of 247 lime. The best fit line for the full range of lime content used in this investigation was plotted in Figure 248 3. The resulting linear equation (Equation 4) from this relationship was used to predict the presumed 249 final strength for other lime contents of 17, 21 and 25%.

$$UCS_{Presumed final strength} = 546.2L - 791$$
 4

The values showed that at 40°C the available lime was wholly consumed during the 672 h of curing time on specimens with lime content up to 17% whereas specimens with lime content of 21% and 25% might not have fully consumed the lime. Furthermore, data attained on specimens that were cured at 20°C indicated that just lime contents of 5 and 7% were nearly consumed whereas other lime contents would require a prolonged period of curing time more than 672 h to consume the available lime. The data suggested that the addition of lime of more than 13% would remain unconsumed in the stabilised clay when cured at 20°C.



Figure 3: comparison between the presumed final strength and measured strength after curing for
672 h at 20°C and 40°C on specimens of treated M1 clay.

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#### 3.2 M2 Clay (Ball clay)

262 Results on the specimens that were tested directly after compaction process showed attainment of 263 UCS strength of 0.82, 0.89, 0.92, 0.95, and 1.03 MPa for 5, 7, 9, 11 and 13% of lime contents 264 respectively compared with just 0.33 MPa for untreated specimens. This illustrated that the addition 265 of lime could also enhance the strength of kaolinite material to up to 3 times in comparison with the 266 strength of untreated clay. The sudden surge in strength could be attributed to the fast initial calcium 267 adsorption and sodium desorption in cation exchange process within the first five minutes on kaolinite 268 soil which was reported by (Singh et al., 1996; Chemeda et al., 2018). Chemeda et al. (2018) also 269 observed that as the concentration of Ca(OH)<sub>2</sub> increased, the adsorbed calcium by Kaolinite became 270 higher according to measurements taken for the calcium concentration after 3 h from submerging 271 equal amounts of kaolinite in various concentration of Ca(OH)<sub>2</sub>.





Figure 4: Evolution of strength gain with time for lime treated M2 specimens at 20°C





275

Figure 5: Evolution of strength gain with time for lime treated M2 specimens at 40°C

The evolution of strength gain for lime treated kaolinite specimens under a temperature of 20°C and 40°C were plotted against the curing time in Figures 4 and 5 respectively. The attained strength values on specimens that were cured at 20°C indicated a very marginal increase in the strength within the first 72 h subsequently the strength remained constant irrespective of the lime content and curing time as seen in Figure 4. This would be due to a delay in the consumption of lime and the absence of formation of cementitious compounds after treating kaolinite clay, which was observed by Vitale et 282 al. (2017). Though, Bauer and Berger (1998) reported that in alkaline solution, the dissolution rate of 283 kaolinite was higher than its counterpart with the bentonite. (Konan et al., 2009; Chemeda et al., 2015; 284 Chemeda et al., 2018) attributed the observed behaviour of kaolinite to the accumulation of various 285 adsorbed calcium species on the surfaces of kaolinite mineral forming a coating layer which isolates 286 the surface of the kaolinite clay particles from the alkaline environment, curbs the dissolution of 287 alumina and silica compounds and thus inhibits the pozzolanic reaction. In contrast, when curing at 288 40°C, the strength remained nearly stable during the first 72 hours followed by a gradual but 289 remarkable gain in the strength up to reaching values of 2.2, 2.54, 2.6, 2.66 and 2.68 MPa for 5, 7, 9, 290 11 and 13% of lime content respectively after 672 h as illustrated in Figure 5. This gave a clear 291 indication of the temperature role (40°C) in accelerating the strength gain. Furthermore, the lowest 292 strength value for 5% lime content indicated that the available lime content was nearly consumed during the 672 h (28 days) of curing at 40°C. The role of higher temperature, e.g. 50°C in re-initiating 293 294 the strength gain in lime-treated kaolinite after a period of stability (7 days) was also reported by 295 Maubec et al. (2017). Maubec et al. (2017) coupled this behaviour with the re-initiation of the calcium 296 absorption and the beginning of forming hydrates compounds, e.g. Calcium Aluminate Hydrates and 297 Carboaluminate Hydrates. However, the mechanism by which the accumulating calcium layer is 298 eliminated, after a long time at 20°C and shorter time at 40°C, so that the alkaline environment could 299 attack the surface of kaolinite, has not been clarified yet. A possible elucidation could be referred to 300 the calcium disposal mechanisms which depend on the specific surface area of kaolinite over time at 301 20°C that showed a prolonged increase as observed by Vitale et al. (2017). Whereas a relatively faster 302 increase in the specific surface area at 40°C is likely to occur, which enables the accommodation of 303 the calcium accumulation. Further investigations would be needed to assess the evolution of specific 304 surface area over time at different temperature.

305

307 3.3 M3 Clay (a mix of 1 portion of Bentonite to 3 portions of Ball clay)

The UCS data attained on M3 specimens treated with 5, 7, 9, 11 and 13% of lime and cured for a period of time up to 672 h at 20°C and 40°C are shown in Figures 6 and 7, respectively. Immediately after compaction, the UCS was nearly equal for all specimens irrespective of the lime content and was about double of the UCS of the untreated specimen (0.5 MPa). Unlike lime treated Ball specimens, the treated M3 specimens showed marginal strength gain of about 0.4 MPa after 672 h at 20°C, reaching UCS value of about 1.4 MPa for all specimens. Equation 5 governs the evolution of strength during the initial phase at 20°C.

$$UCS_{First \ phase} = -0.0003C^2 + 0.78C + 1031.5 \quad R^2 = 0.96 \qquad 5$$

315 In contrast, the cured specimens at 40°C achieved UCS values of 1.4 MPa in only 48 h, which 316 highlighted the significant role for the curing temperature in accelerating the chemical reaction. After 317 a period of curing of 168 h at 40°C, the measured UCS values for all specimens with different lime 318 contents were nearly the same at about 2.1 MPa except a specimen that was treated with 5% lime 319 content which showed a slowdown in the strength gain entering in the second phase after 72 h of 320 curing. The UCS values observed on specimens with 5% lime content experienced no appreciable 321 change after 168 h of curing achieving a value of almost 1.9 MPa. The no significant change in the 322 strength suggests that the addition of 5% lime is not enough to support further reactions between 323 lime and clay after 168 h of curing at 40°C. The UCS values at 40°C increased notably with the increase 324 in lime content reaching 2.7, 3.6, 4.0 and 4.2 MPa on M3 specimens treated with lime content of 7, 9, 325 11 and 13% respectively after 672 h of curing. Equation 6 governs the evolution of strength during the 326 initial phase at 40°C.

$$UCS_{First \, phase} = -0.0039C^2 + 7.13C + 1072 \quad R^2 = 0.99 \qquad 6$$

Results of measured UCS values at 672 h of curing time were plotted against lime content in Figure 8. The data suggested that UCS values attained at 672 h at 40°C is directly related to the lime contents of 5, 7, and 9%, which means that lime was fully consumed during the curing period. Consequently, a

- 330 linear relationship (Equation 7) between UCS and lime content is obtained and used to predict the
- final UCS values for specimens treated with higher lime contents of 11 and 13%.

$$UCS_{Presumed final strength} = 405.7L - 115.9$$
 7

Comparing estimated strength values with measured UCS values after a period of curing of 672 h at 40°C illustrated that specimens treated with 11 and 13% of lime would not have reached their maximum strength yet which means that lime might not be fully consumed. Nevertheless, curing at 20°C slowed the consumption of lime and resulted in markedly lower values of strength. In addition, the relationship indicated that a 1% increase in the lime content would result in an increase of 0.4 MPa in the final strength value when cured at 40°C.



339 Figure 6: Evolution of strength gain with time for lime treated M3 specimens at 20°C





Figure 7: Evolution of strength gain with time for treated M3 specimens at 40°C



344 Figure 8: comparison between the presumed final strength and the measured strength after curing

for 672 h at 20°C and 40°C for treated M3 specimens

349 3.4 M4 Clay (A mix of 1 portion of Bentonite to 1 portion of Ball clay)

350 The UCS evolution of compacted M4 specimens that were treated with 5, 7, 9, 11 and 13% of lime and 351 cured for a period of time up to 672 h at 20°C and 40°C are depicted in Figures 9 and 10 respectively. 352 The measured UCS values for all treated specimens that tested directly after compaction were about 353 two times that attained on the untreated specimen (0.43 MPa). Results of UCS on specimens treated 354 with 7, 9, 11 and 13% of lime and cured at 20°C showed a gradual increase in strength over the whole 355 duration of curing. This means that the first strength phase continued until 672 h for specimens with 356 lime content of 7% and higher. The relationship between strength and curing time seems to be 357 governed by the polynomial Equation 8, achieving the same UCS value of about 2.2 MPa at 672 h. 358 However, M4 specimens treated with 5% lime did not follow the same path for the evolution of 359 strength. The strength did not increase after 168 h of curing time, indicating the commencement of 360 the second phase.

$$UCS_{First phase} = -0.0015C^2 + 2.8C + 1059.9$$
  $R^2 = 0.99$  8

On the other hand, specimens cured at 40°C showed a typical relationship through which the strength gain was initially fast followed by a slower second phase. It is clear that the continuity of the fast phase was dependent on the availability of lime. The strength gain over the first phase at 40°C is governed by Equation 9. The achieved strength at the end of curing period at 40°C was directly related to the lime content attaining 2, 2.9, 4.2, 4.9 and 5.8 MPa on specimens treated with lime content of 5, 7, 9, 11 and 13% respectively.

$$UCS_{First \, phase} = -0.043C^2 + 22.52C + 1089 \quad R^2 = 0.99 \qquad 9$$



369

Figure 9: Evolution of strength gain with time for M4 specimens at 20°C



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371

Figure 10: Evolution of strength gain against curing time for M4 specimens at 40°C

The attained UCS at 20 and 40°C after 672 h of curing were plotted in Figure 11 against the corresponding lime content. The data suggest that lime would be consumed entirely on specimens treated with lime content of 5, 7 and 9% at 40°C since Equation 10 can fit the data accurately. Extending the linear equation (Equation 10) to higher lime contents indicated that the higher lime content need further curing time to be completely consumed even at ambient temperature of 40°C. The best fit line indicated that after 5% of lime content, an increase of 1% in the lime content would result in an increase in the final strength value by about 0.55 MPa. The data suggest that lime was not consumed when specimens were cured at 20°C. The data in Figures 3, 8 and 11 highlighted the responsibility of lime content on determining the final strength value and the role of mineralogy composition and temperature on determining the time needed to reach the final value. It was also noticed that each increase of 1% lime above 5% lime content offers an increase in the final strength ranging from 0.4 to 0.55 MPa.

$$UCS_{Presumed final strength} = 552.15L - 813.15$$
 10

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385

672 h at 20°C and 40°C for treated M4 clay

388

389 3.5 Mineralogical effects

In this investigation, four different types of clay namely; M1 of pure bentonite, M2 of pure kaolinite,
M3 which is a mix of Bentonite and Kaolinite with a ratio of 1:3 by mass and M4 which is a mix of
Bentonite and Kaolinite with a ratio of 1:1 by mass were used to represent soils with a vast range of

liquid limit from 330% down to 58%. Based on the UCS results that were presented earlier for the four
different types of clay, thorough assessment and comparison were conducted to highlight the impact
of clay mineralogy on the reaction process and kinetics of strength gain when mixed with hydrated
lime.

397 The data showed that testing lime treated specimens with a range of lime contents immediately after 398 compaction would result in a relatively narrow range of UCS values. By and large, the UCS values on 399 treated specimens were 2~3 times that achieved on untreated specimens irrespective of the amount 400 of added lime. The UCS values increased slightly with the increase in the bentonite content in the 401 specimens. The immediate changes in the structure and bonding between treated particles could be 402 attributed primarily to cation exchange, flocculation and aggregation mechanisms and enhanced by 403 the immediate formation of initial cementitious compounds. Since the surface area of bentonite clay 404 is much higher than that of kaolinite clay, it is more likely that the lime would react with the bentonite 405 particles at a higher rate resulting in a significant reduction in the surface area of bentonite and in a 406 relatively higher strength immediately after compaction. The results demonstrated that the amount 407 of added lime at zero h curing has no impact on the evolvement of strength which could be attributed 408 to the small amount of lime that is required to satisfy the needs for cation exchange and flocculation 409 mechanisms.

410 Careful inspection of UCS data for all clays indicated that the kinetic of strength gain throughout curing 411 is dependent on curing temperature, lime content and curing time. Two stages were very noticeable 412 in the evolution of strength of the lime-treated clays in particular at the high temperature of 40°C. 413 Quadratic equations were proposed for stage 1 of strength gain (fast-growing) and presented in 414 Figures 2 and 3 for the bentonite clay. It was noted that during the first days of curing both equations 415 behave mostly linear due to the small negative value of the numerical coefficient in the second order 416 parameter compared with the higher positive numerical coefficient in the first order parameter. So, it 417 can be inferred that the numerical coefficients in the first order parameters reflect the kinetic of

strength gain under both temperatures. Consequently, the results suggested that during the first
hours (stage 1), the kinetic of strength gain at curing temperature of 40°C was about 8 times that
experienced when curing at 20°C.

421 To appreciate the effect of bentonite inclusion with the Ball clay, the kinetic of strength gain of lime-422 treated treated M3 and M4 were also assessed using the numerical coefficients in the quadratic 423 equations (5, 6, 8 and 9) (. Discarding the minor numerical coefficients in the second order parameters, 424 it became clear that the strength gain was a function of the clay mineralogy and increased with 425 elevating the curing temperature as seen in Figure 12. The kinetic of strength gain of M3 and M4 426 experienced at curing temperature of 40°C was found to be also about 8 times that recorded when 427 curing at 20°C. At a given temperature, the rate of strength gain with M3 and M4 during stage 1 was 428 about 15% and 50% of that recorded for M1 which highlighted a significant role of the bentonite in 429 the reaction with lime and evolution of strength.





Two possible elucidations could be brought forward to clarify the changes leading to the increase in
strength gain with the increase in the bentonite content; i. increasing the amount of bentonite content

435 pushes towards consideration that bentonite is predominantly responsible for the degree of 436 improvement in the strength gain since the increase in strength is directly related to the proportional 437 of bentonite in the material and ii. bentonite might act as a rival consumer for calcium ions in the lime 438 leading to reduced accumulation of calcium ions on the surface of kaolinite particles. Consequently, 439 the alkaline environment was allowed access to the surface of kaolinite layer. As a result of attacking 440 the alkaline environment to the surface of kaolinite and montmorillonite minerals, silica and alumina 441 would be released leading to the formation of cementitious compounds in the form of Calcium Silicate 442 Hydrates (CSH), Calcium Aluminate Hydrates (CAH) and Calcium Aluminate Silicate Hydrates (CASH). 443 It is well known that kaolinite minerals comprise of the octahedral sheet (AL site) and tetrahedral 444 sheet (Si site) whereas, in the case of montmorillonite, there are two tetrahedral sheets sandwiched 445 an octahedral sheet (Brigatti et al., 2006). Taking into account the differences in the structure of 446 minerals, the launch of alumina and silica would be synchronised in the case of kaolinite, and the 447 release of silica would be followed by the release of alumina in the case of montmorillonite minerals. 448 Bauer and Berger (1998) concluded that unlike the preference of releasing the silica in the case of 449 montmorillonite mineral, the preference of the dissolution of alumina was prevalent in the case of 450 kaolinite minerals. Using X-ray diffraction analysis, the presence of CAH with lime-treated kaolinite 451 was observed by Maubec et al. (2017) and Vitale et al. (2017) after 28 days at 20°C, whereas CSH was 452 observed by (Maubec et al., 2017) after 98 days of curing at 50°C. With respect to the montmorillonite 453 mineral, the presence of CSH was observed since the very short time of the treatment whereas the 454 presence of CAH and CASH were observed after a prolonged period of time as reported by (Pomakhina 455 et al., 2012; Vitale et al., 2016; Maubec et al., 2017; Vitale et al., 2017). Bauer and Berger (1998) also 456 reported that the rate of dissolution of kaolinite was higher than that in montmorillonite minerals in 457 a strong base solution (potassium hydroxide).

458 On the other hand, the results of two studies conducted by (Al-Mukhtar et al., 2010a; Al-Mukhtar et 459 al., 2010b) on expansive soils naturally contain 38% of kaolinite, and 58% of smectite minerals 460 indicated that the formation of CAH was observed using X-ray diffraction after 1 and 7 days at 50 and

461 20°C respectively. In contrast, the formation of CSH was observed at 50°C after 7 days. Hence, the 462 availability of alumina and/or silica at the time when the reaction takes place controls the outputs of 463 the pozzolanic reactions and the development of CAH, CSH and/or CSAH depending on the abundant 464 reactants, e.g. alumina or silica (Beetham et al., 2015). The formation of CAH; 1. refers to the 465 responsibility of kaolinite in the formation of the cementitious compounds and thus on the strength 466 gain, 2. confirms the role that played by the smectite mineral as competitive consumer which prevents 467 the accumulation of calcium ions on the surface of kaolinite and the faster dissolution of kaolinite in the alkaline environment. Based on that in the current study, it can be stated that the increase in 468 469 bentonite content in M3 and M4 offered faster elimination of the calcium accumulation, the earlier 470 appearance of cementitious compounds and initiation of greater kinetic of strength gain.

471

## 472 3.6 Collapse pattern and desiccation cracks

473 Careful inspection of the failure pattern of all lime-stabilised clay specimens suggested that the failure 474 mechanism was markedly dependent upon the type of clay material and its strength which was a 475 function of the amount of lime and curing conditions. Figure 13 shows pictures of specimens at failure 476 after being cured for 672 h (28 days). In all specimens, the failure pattern was in the form of a cone-477 split that was well formed at one end only. The physical observations suggested that the cone-split equally occurred at either the top or the bottom of the specimens. Curing lime stabilised clays for a 478 479 long period resulted in a brittle behaviour which can be noticed by failure at a relatively small strain 480 of less than 2% as shown in Figure 14. The cone-split is very similar to that classified by ASTM C39 481 (2018), type 2 for the typical collapse in the cylindrical brittle concrete specimen.



a. M1 b. M2 c. M3 d. M4 Lime treated Lime treated ball clay Lime treated 1:3 Lime treated 1:1 bentonite LP = 13% and bentonite - ball clay bentonite – ball clay LP = 9% and T = 40°CLP = 17% and T = 40°C  $T = 20^{\circ}C$ LP = 13% and T= 40°C



Figure 13: typical cone-split failure pattern on lime treated clays after 28 days



484 Figure 14: stress-strain relationships on lime treated clays: LP = 13%, T = 40°C and C = 672 h

Figure 15 shows an example of the failure of the M1 at different times of curing. It was observed that a classical shear collapse was imminent on specimens that were cured for a short period of time up to 12 h whereas a combined cone-shear collapse appeared to occur on specimens that were cured for a period of time between 12 and 72 h. Furthermore, the failure pattern on specimens cured for longer periods showed a cone-split failure, as illustrated in Figure 15. The collapse pattern is related to the strength of the specimen at the time of testing, which is related to the type of clay and treatment and curing conditions. Results for the stress-strain relationships on treated M1 specimens at different curing times are presented in Figure 16. The results confirmed that the behaviour of lime treated clay specimens changed from ductile to brittle with the curing time. The ductile behaviour of limestabilised bentonite was accompanied by a classical shear failure, whereas the cone-split is dominant on high strength specimens that showed brittle behaviour.







a. Shear collapse At zero curing time LP=21%

b. Cone-shear collapseAt 24 h of curing at 40°C, LP=25%

c. Cone-split collapse after 72 h of curing at 40°C, LP=21%

Figure 15: the type of collapse patterns over the curing time on M1 specimens



498 Figure 16: stress-strain relationships on lime treated M1 specimens as a function of curing time 499 Another distinctive feature was observed during the drying process during which all tested specimens 500 were dried in the oven at 105°C, to ascertain the water content of cured specimens. This was a final 501 quality assurance step that was important to ensure the effectiveness of controlling and maintaining 502 a target water content throughout the curing period. During the drying process, it was observed that 503 the appearance of desiccation cracks on the surface of lime treated bentonite (M1) is different from 504 that observed on lime treated ball clay (M2). The desiccation cracks on the lime-treated ball clay, M3 505 clay, and M4 clay specimens appeared at the onset of the drying process within 1 h (see figure 17a) 506 and then gradually closed by the end of 24 h of drying as shown in Figure 17b. Only some hair cracks 507 can still be visible on the specimens. Whereas the substantial amount of cracks were generated within 508 1 h of drying on lime treated bentonite M1 specimens (see, Figure 17 a) and some cracks were 509 widened with time and remained after completion of drying as shown in Figure 17b. Nevertheless, 510 there was no significant volume change on the lime-treated clays.



a. Treated M1 after1 h of drying



- c. Treated M4 clay after 1 h of drying
- d. Treated M4 clay after 24 h of drying

512

Figure 14: behaviour of desiccation cracks during the drying process of 7% lime treated clays: a and b
for M1 clay and c and d for M4 clay

## 515 **4.** Conclusions

516 In this investigation, four different types of clay with a wide range of liquid limit were mixed with 517 different amounts of lime up to 25% by mass to examine and evaluate the mineralogical effects on 518 the chemical process and the evolution of strength. Five series of experiments were undertaken to 519 test specimens with different lime contents at two different curing temperature for a period of curing 520 time up to 28 days. The key outcome of the current investigation is that the beginning and vitality of 521 changes in strength characteristic are related to the formation of cementitious compounds and its 522 kinetics which depend primarily upon the mineralogy composition of clay and ambient temperature. 523 Furthermore, at a given ambient temperature, the continuity of such changes in the characteristics of 524 a given lime-treated clay depends on the availability of lime. In addition, several conclusions could be 525 drawn out of the experimental study;

An immediate effect of lime on the strength of lime treated clays was evident in all specimens
 that were tested directly after compaction in comparison with those recorded on untreated

specimens at the same dry unit weight. The results suggested that an increase of 2 ~ 3 times
could be achieved with the addition of lime. However, the degree of improvement in the
strength was not related to the amount of lime.

2. Lime treatment of bentonite and kaolinite clays showed recognisable differences during the 531 532 period of curing and immediately after the initial strength gain at zero h of curing period. 533 Bentonite clay reacted swiftly with lime leading to a significant and sustained degree of improvement in the strength as time passed at 20°C and 40°C but with different rates. 534 535 Nevertheless, specimens of treated Ball clay (Kaolinite) showed that the strength gain entered 536 in an idle phase in which no growth in strength was observed over the 672 h of curing in 537 particular at 20°C. Whereas the idle phase was shortened to only 72 h when the curing temperature was raised to 40°C. 538

539 3. Since the Ball clay comprised mainly of kaolinite minerals, the phenomenon of the 540 accumulation of calcium cations species on the kaolinite surface caused obscuring the surfaces 541 of mineral from the alkaline environment. This accumulation of calcium cations led to a delay 542 in the release of alumina and silica and thus delaying the formation of cementitious 543 compounds. However, the mechanism by which the accumulation of calcium was reduced or eliminated at 40°C so that the alkaline environment was allowed to attack the surface of 544 545 mineral and thus to launch the alumina and silica in order to form the cementitious 546 compounds that are responsible for the strength gain, deserves further investigations.

547
4. The addition of Bentonite to Ball clay with a ratio of 1:3 and 1:1 to form M3 and M4 materials
548
548 was found successful in eliminating the idle phase in the strength gain over the curing period.
549 Bentonite would act as a competing consumer for the incoming calcium ions to the system
550 reducing and/or eliminating the accumulation of the calcium ions on the surface of kaolinite
551 minerals. This led to a gradual improvement in the strength but at a slower rate.

552 5. The results showed that the strength gain throughout curing went through two stages 553 process. The first stage was recognised by a fast strength gain, followed by a second stage in

which a slower strength gain occurred. The two stages were very prominent at the high curing temperature of 40°C. The time for stage 1 of strength gain was dependent upon the curing temperature, lime content and mineralogy of clay. It increased with higher lime content and increased bentonite portion.

- 558
  6. Despite the use of quadratic equations to best fit stage 1 of strength gain, the numerical
  559
  coefficients for the second order term were found to be negligible. Simplifying the equations
  560
  into straight lines assisted with comparing the rate of strength gain.
- During stage 1 of strength gain, the rate of strength gain at the high curing temperature of 40°C was found to be about 8 times that observed at the low curing temperature of 20°C. At the same temperatures, the ratio between the rates of strength gain was very dependent upon the clay mineralogy. The kinetic of strength gain of lime treated bentonite clay was about 2 and 7 times the kinetic of strength gain of lime treated clays with 50 and 25% bentonite content, respectively.
- 567 8. The failure pattern was found to change throughout the curing period owing to the strength 568 of treated specimens. Classical shear failure was imminent on all specimens that were cured 569 for a short period up to 24 h. A combined shear and cone-split occurred on specimens that 570 were cured for up to 72 h and then cone-split failure pattern was observed on all specimens 571 that were cured for long periods. This was in harmony with the change in behaviour from 572 ductile to brittle with further curing.
- 573 5. Acknowledgements

This research did not receive any specific grant from funding agencies in the public, commercial, ornot-for-profit sectors.

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