

Innovative cementing agents, used as alternatives to conventional soil stabilisers

Ciments innovants pour le traitement des sols comme alternatives aux agents chimiques conventionnels.

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ABSTRACT: The paper studies innovative cementing materials and processes such as alkali-activated (AA) types of cement or enzymatically induced mineral precipitation for soil stabilisation, as alternatives to traditional binders such as Portland cement or lime. Parametric studies have been performed in the laboratory on two problematic fine-grained soils, treated by mixing with different activators at different molarities and dosages. Materials retrieved from waste have also been considered for both processes (i.e. as activators or as enzyme source). Tests included unconfined compressive strength (UCS) and p-wave velocity measurements using Portable Ultrasonic Non-destructive Digital Indicating Tester (PUNDIT). In general alkali-activated cements outperformed enzymatically induced mineral precipitation cements in terms of strength improvement. However the latter process has potential and is worth investigating further in order to gain a better understanding of the complex mechanisms involved. This can lead to the development of improved procedures that will give better results in terms of soil stabilisation.

RÉSUMÉ: Nous étudions des matériaux et processus innovants pour le traitement des sols, c.-à-d. des matériaux à activation alcaline (AAM) et la précipitation de calcite induite par l'intermédiaire d'une enzyme d'origine végétale, comme alternatives au ciment Portland classique ou à la chaux. Nous avons mené en laboratoire une étude paramétrique sur deux sols fins problématiques et divers mélanges, étudiant l'effet de la molarité des solutions et du dosage en agent de traitement. Nous avons aussi utilisé des matériaux provenant de déchets. Nous présentons des résultats de compression uniaxiale ainsi que des résultats d'essais ultrasoniques de la vitesse d'impulsion (UPV). La performance des mélanges à activation alcaline était supérieure à celle des mélanges tentant d'induire la précipitation de calcite mais cette technique mérite l'attention. Nous proposons donc de continuer la recherche dans ce sens pour développer une meilleure compréhension de ce processus, qui nous permettra d'optimiser les mélanges et par conséquent d'améliorer leur performance comme agents de traitement de sols.

Keywords: ground improvement; alkali-activated cements; enzymatically induced calcite precipitation

1 INTRODUCTION

With the increase in the world population, engineers need to build over soils which, as found on site, may have unsuitable properties for construction. Soil stabilisation with a variety of techniques, of which chemical stabilisation, has been introduced as a more viable alternative to the replacement of unsuitable soil by imported aggregate. Although this is a step towards more sustainable solutions, common chemical stabilisers such as Ordinary Portland cement (OPC) or lime are not environmentally-impact free, as their production at temperatures over 1200-1400 °C is energy intensive, and produces large amounts of greenhouse gases (e.g., 0.95 t carbon dioxide per ton of OPC) and other air-polluting chemicals (e.g., sulphur dioxide, carbon monoxide, and nitric oxide). It also consumes non-renewable natural resources (e.g. 1.5 t limestone and clay per ton of OPC, Du et al, 2016). Alternative cementing materials are therefore actively sought, towards higher sustainability.

Of these, alkali-activated (AA) cements are increasingly gaining the attention of researchers worldwide. The use of such materials in concrete has been widely researched; even so, improved systems for concrete AA cement are continuously sought and studied. For soils, mixing the usual chemical stabilisers such as lime with industrial by-product aluminosilicate materials e.g. ground granulated blast furnace slag (GGBS) or Pulverised fuel ash (PFA), can be considered as a form of activation of these materials. On the other hand, the use of binders based on alkali-activated cement systems as those used for concrete has been less researched for soils. Some examples of relevant research include: (a) the study of the effectiveness of alkali-activated low-calcium and high-calcium fly ash as silica and alumina amorphous sources for soil stabilisation (Cristelo et al., 2011, 2012, 2013); (b) the activation of metakaolin to produce geopolymer for shallow depth stabilisation (Zhang et al., 2013) or (c) the use of

a GGBS-NaOH binder to improve a soft alluvium for high-speed rail (Sargent, 2015).

Another emerging soil stabilisation technique is the calcite in-situ precipitation method. Following research by Kucharski (see e.g. Kucharski et al, 1996) a number of researchers studied the process of calcite precipitation mediated mainly by the action of ureolytic bacteria (microbially-induced calcite precipitation, MICP) (e.g. Whiffin, 2004; Whiffin et al, 2007; van Paassen et al, 2010 amongst many others). Via the catalytic action of urease enzyme, these bacteria dissociate urea into ammonium and carbonate ions; in the presence of calcium ions in the system, calcium carbonate/calcite crystals are then precipitated. These act as cementing agents that bond the particles of the soil together, thus increasing its strength and stiffness.

In a more limited number of studies, cementation through calcite precipitation was attempted using free enzymes, in particular urease (Hamdan, 2014; Knorr, 2014; Dilrukshi and Kawasaki, 2016). The main reason for this was to overcome disadvantages of the microbially induced calcite precipitation, in terms of difficulties in controlling and sustain the growth of appropriate bacteria under in situ conditions and also size restrictions limiting the use of bacteria to coarse grained soils or at most, silts. On the other hand, researchers have identified a number of potential disadvantages of using free enzyme compared to microbial urease, namely: (a) the lack of nucleation points on the soil surface for CaCO₃ precipitation if a free enzyme is used (as opposed to the biomediated process, where microbes typically attach themselves onto the soil particles thus providing nucleation points for mineral precipitation); (b) the more rapid precipitation rates of carbonate minerals when free urease enzyme is used, which can lead to smaller and less-structured (more amorphous) crystals that are not effective for cementation (Hamdan, 2014); (c) the high costs of commercially supplied urease enzyme; (d) the stability of the

enzymes and their soil-specificity. Therefore further research is needed to establish the viability of the free enzyme urease calcite precipitation process as an effective and sustainable ground improvement technique for a number of different soil types (so far plant-derived urease enzyme was mostly used for sands, although it was recognised as potentially suitable for fine-grained soils).

The aim of this paper is to assess the effectiveness of these relatively novel methods of ground improvement, i.e. alkali-activation (AA) and enzymatically induced calcite precipitation (EICP), for two problematic fine grained soils, namely a silt soil and an expansive clay soil. In some of the AA cement systems waste paper sludge ash (PSA) has been used in the alkaline activator solution, as a low cost activator coming from waste, with the related solid management advantages.

2 MATERIALS AND METHODS

The soils used in this study were (a) an industrially supplied silt soil and (b) a mixture of 70% kaolin clay from the South West of England and 30% sodium activated bentonite. Tests for the total and water soluble sulphate content based on the gravimetric method of BS 1377-3:1990 (BSI, 1990a) showed no evidence of sulphates in the soils; this allows for the use of calcium-based stabilisers, such as PSA, without the risk of deleterious reactions.

The sodium silicate Na_2SiO_3 (waterglass-WG) solution of a modulus $M=\text{SiO}_2/\text{NaO}_2=2$ and the potassium hydroxide KOH pellets were supplied by Fisher Scientific. GGBS was provided by Hanson Regen. For slag to be suitable for alkali activation it needs to have a high vitreous content of 90% or more and a large specific surface of 400-600 m^2/kg . According to information provided by the supplier the slag had a vitreous content of 98% and a specific surface of 450-550 m^2/kg (i.e. the optimal fineness range for AA cement according

to Wang et al, 1994); thus both requirements were satisfied. Moreover, the slag should also be preferably pH-basic and have an adequate modulus of activity or quality coefficient (Mavroulidou and Martynková, 2018); the higher the modulus of activity or quality coefficient is, the higher the amount of alkaline compounds present in the slag, giving better hydraulic properties. We checked the basicity coefficient K_b and the quality coefficient K_q defined respectively as:

$$K_b = (\text{CaO}+\text{MgO})/(\text{SiO}_2+\text{Al}_2\text{O}_3) \quad (1)$$

$$K_q = (\text{CaO}+\text{MgO}+\text{Al}_2\text{O}_3)/(\text{SiO}_2+\text{MnO}) \quad (2)$$

The slag used was found to be basic ($K_b>1$) and of a $K_q=1.65$, thus of moderate activity ($K_q=1.6-1.9$) (Hanykýř & Kutzendörfer, 2002) which is adequate. Therefore the slag satisfied all suitability criteria.

PSA was provided by a newspaper recycling company in the South-East of England. It is the ash produced from the incineration of non-hazardous paper sludge (a semi-solid slurry collected in the effluent treatment units), which is the main waste stream of the paper recycling industry. The sludge is incinerated primarily to reduce the volume of sludge waste for landfilling (80-90% reduction) and partly to recover energy through co-combustion with biomass (although mechanically dewatered paper sludge has a low calorific value of 2.5-6.0 MJ/kg, Spathi, 2015). PSA is subsequently landfilled in large part.

In this study the PSA was used as received, i.e., it was not milled (hence less reactive); in this form it has an average particle size (d_{50}) of ca. 90 μm (Spathi, 2015) i.e. larger than clay size particles (of $<2 \mu\text{m}$); it is thus coarser than the average particle size d_{50} of the tested soils i.e. 4.5 μm and 20 μm for kaolin and silt respectively (based on hydrometer testing, BSI 1990b); bentonite is even finer, consisting of 92% montmorillonite clay with only 5% of the particles $>150 \mu\text{m}$ (based on supplier's data).

Table 1 shows the chemical composition of the solid materials used in the AA cement mixes, according to information from the supplier or literature (e.g. Mavroulidou, 2018).

Table 1. Chemical composition of the solids used in cementing mixes

	Chemical composition (%)			
	PSA	GGBS	KOH Pellets (impurities)	
SiO ₂	19.2	34.68	Na ₂ CO ₃	< 2
Al ₂ O ₃	8.7	14.16	Ca	< 0.005
CaO	60.7	38.74	Cu	< 0.002
MgO	2.8	7.74	Fe	< 0.002
Fe ₂ O ₃	0.5	0.05	Pb	< 0.002
Na ₂ O	0.15	0.46	Mg	< 0.002
K ₂ O	0.2	0.55	Na	< 1
SO ₃	0.48	0.21	Zn	< 0.005
P ₂ O ₅	0.17		S	< 0.01
TiO ₂	0.2		P	< 0.02
SrO	0.09		Si	< 0.02
MnO	0.02			
BaO	0.04			
Li ₂ O	0.01			

From Table 1 it can be seen that the chemical compositions of GGBS and PSA have some similarities. PSA is mainly a calcium aluminosilicate, as the principal compounds are lime (CaO), silica (SiO₂) and alumina (Al₂O₃). The PSA used in this study is richer in CaO and SiO₂ compared to the PSA used elsewhere (e.g. Gluth et al. 2014 or Frías et al. 2008). PSA is not a pozzolan as the total content of the three major oxides (SiO₂ and Al₂O₃ and Fe₂O₃) is typically less than 50%. Conversely it is cementitious and due to its high free CaO content it has a pH=12.3-12.4 (Mavroulidou, 2018). Attempts to activate PSA itself in the context of structural concrete did not show evidence of such activation (Martynková and Mavroulidou, 2015). Therefore the PSA was considered here mostly as an alkaline activator of the GGBS in the system, where free CaO hydrates to Ca(OH)₂ in the presence of water. Thus, the alkaline activators used were: (a) Potassium hydroxide solutions (KOH) of 4M

and 6M respectively; (b) KOH solutions of 4M and 6M mixed with waterglass (WG) solution; (c) Ca(OH)₂ from the hydration of CaO contained in the PSA; (d) WG and Ca(OH)₂ from the PSA (in the latter systems PSA was used in a slurry form). For AA systems, different mixes were prepared, in which activator solutions were thoroughly mixed with the soil and the GGBS mixture (both mixed in dry powder form). The aim of the mix design was to perform a parametric study on the effect of the following parameters: (a) percentage and ratio of activator solution mass/GGBS mass; (b) molarity; (c) effect of WG. To decouple the possible effect of GGBS, specimens treated with GGBS only were also prepared.

For the EICP specimens, two different sources of urease enzyme were used; namely a commercially supplied Urease Type III (low grade) from Jack bean (Aldrich Sigma) and urease that we extracted at London South Bank University (LSBU) laboratories from water melon seeds. The solution formulations were based on findings by Hamdan (2015) and Knorr (2014). Different ratios of CaCl₂/Urea were used and solutions with different molarities. To decouple possible effects of these two substances from those of urease-containing mixes (for EICP), specimens with these two solutions without any urease enzyme were also made. Urease was added at two different mass proportions and was stabilised with the use of non-fat dried milk powder (Knorr, 2014).

Compacted cylindrical specimens at fixed target dry densities ρ_d and water contents w (adjusted for solution water) were used for all mixes of each soil i.e. $\rho_d=1.56 \text{ g/cm}^3$ and $w=25\%$ for the silt and $\rho_d=1.43 \text{ g/cm}^3$ and $w=40\%$ for the clay soil. The specimens were then left to cure at ambient temperature as required. As a minimum, duplicate to triplicate specimens were prepared. Two different curing methods were used, namely water-curing method with specimens subjected to capillary soak and air-curing (i.e. constant moisture curing with specimens wrapped in cling film and

stored in an insulated cabinet for the specified curing period). For the silt, air curing was applied for both 7 and 28 days; for the clay, 7 days of air curing was followed by water-curing (this curing method with exposure to water was done to complement an ongoing project assessing the effect of stabilisers on the swelling characteristics of the expansive soil). The soil stiffness evolution was assessed from ultrasonic pulse velocity (UPV) measurements (p-wave velocity) using a Portable Ultrasonic Non-destructive Digital Indicating Tester (PUNDIT). At the end of the required curing periods (7 and 28 days respectively), the dimensions and the mass of the specimens after curing were measured; Unconfined Compressive Strength (UCS) testing was then performed at a constant rate of strain of 1mm/min. Note that the untreated clay specimens disintegrated upon soaking; for this reason, there are no UCS or UPV readings in Table 3 for the untreated soil.

3 RESULTS AND DISCUSSION

3.1 AA mixes

Indicative results of our work presented in Tables 2-3 clearly show that all alkaline activators increased the strength of the soils (also the strength of the clay, as treated specimens could be handled and tested after soaking, unlike the untreated soil). GGBS was found to have a minor effect on the strength after 7 days; 28 days later however, the strength had approximately doubled compared to the untreated silt. However, compared to the strength of the alkali-activated mixes with $\text{Ca}(\text{OH})_2$ (from PSA) and KOH acting as the base activators, the strength increase with GGBS only, was very small. Whereas KOH led to higher strengths than PSA (with or without WG in the respective mixes), the strength gains with only 3% to 6% PSA in the mixes were very considerable, namely, (a) for mixes without WG 17 and 30 times higher for 7 and 28 days of

curing respectively; (b) for mixes with WG 34 and 79 times higher for 7 and 28 days of curing respectively. KOH mixes without WG showed little evolution between 7 and 28 days (so that 3%PSA+GGBS 28 day strength is very close to that of 3 % 6M KOH+GGBS). It should be noted that mixes with WG as the sole activator, had only about 2-6 times the strength of the untreated silt. This shows that PSA contributes indeed to the strength gain/acts as an alkaline activator of GGBS. An interesting observation is that increasing KOH molarity from 4M to 6M consistently led to a decrease in strength in silt mixes; further study is required to establish the reason for this reduction. Finally, it can be seen that combinations of KOH+WG spectacularly increased the 28 day strength of the silt soil. Although comparisons of strength gain with respect to the untreated clay cannot be made, the overall trends concerning the success of AA-clay mixes are generally similar to the AA-silt mixes. However PSA is shown to be clearly more successful with clays compared to KOH, potentially due to modification reactions with the clay.

Table 2. Unconfined compressive strength (q_u) of AA treated silt: 7 vs 28-day curing

Soil/soil mix	7-day	28-day
	q_u (kPa)	q_u (kPa)
Untreated silt	65	
10% GGBS	70	119
3% PSA + 10% GGBS	1121	1921
3% PSA+ 3% WG +10% GGBS	2234	5157
3% WG +10%GGBS	145	121
6% WG +10%GGBS	170	426
6% 4M KOH+10% GGBS	3202	3943
3% 4M KOH+ 3% WG+ 10% GGBS	4405	11226
3% 6M KOH+10%GGBS	1937	2106
6% 6M KOH+10%GGBS	1776	2010
3% 6M KOH+3% WG +10% GGBS	2439	8873

Indicative stress-strain curves of the AA activated mixes (Figure 1) show these to have a very brittle behaviour, in particular for the PSA treated soil. Indicative ultrasonic pulse velocity UPV (p-wave velocity) results from PUNDIT measurements are shown in Table 3. The p-wave velocity can be expressed as the square root of the stiffness/density ratio; therefore for a constant density the higher the ultrasonic pulse (p-wave) velocity, the higher the stiffness.

Table 3. 28-day curing results for AA clay (UCS and ultrasonic pulse velocity UPV)

Soil/soil mix	q_u (kPa)	UPV (m/sec)
Untreated clay	N/A	N/A
8% PSA	1271	909
3% PSA + 10% GGBS	798	613
6% PSA + 10% GGBS	1014	1205
3%PSA+3%WG+10% GGBS	391	588
6% 4M KOH + 10% GGBS	697	1031
3% 6M KOH + 10% GGBS	145	926
6% 6M KOH + 10% GGBS	277	858
6% 6M KOH+3%WG+10% GGBS	637	866
9% 4M KOH + 15% GGBS	2953	1156
9% 6M KOH + 15% GGBS	3707	1333

3.2 EICP mixes

Preliminary results of ongoing research on EICP are shown in Table 4 for the silt soil. The clay soil results did not show improvement with EICP application (in fact a reduction in strength was noted) and are not shown for brevity. The results for the silt are variable; in some instances some improvement in the unconfined compressive strength of the silt soil is noted. Consistently with Knorr (2014) the best CaCl_2 /urea ratio was 2/3, which got close to doubling the strength of the soil (as expected in soil stabilisation). However even without urease some strength gain is noted (one mix for instance, has a higher strength than the corresponding urease-containing mix); this

difference in strength may however not be significantly compared to the specimens treated also with urease. Better results were obtained with urease extracted from the watermelon seeds compared to the equivalent specimen treated with commercial urease, potentially linked to urease activity effects on crystal growth. In ongoing work we will measure urease activities and CaCO_3 contents and will perform micro-structural analysis of EICP treated samples for a better understanding of the strength increase mechanisms.

Table 4. Unconfined compressive strength of EICP treated silt: 7 vs 28-day curing

Soil/soil mix	7-day q_u (kPa)	28-day q_u (kPa)
Untreated silt	65	
0.5M CaCl_2 :0.5M Urea ratio 1/1	102	34
0.5M CaCl_2 :0.5M Urea ratio 1/1 +0.25g commercial urease +milk powder	72	108
0.25M CaCl_2 :0.375M Urea ratio 2/3	75	111
0.25M CaCl_2 :0.375M Urea ratio 2/3+0.5g commercial urease + milk powder	79	87
0.5M CaCl_2 :1.5M Urea ratio 2/3	108	123
0.5M CaCl_2 :1.5M Urea ratio 2/3+0.5g commercial urease+ milk powder	89	88
0.5M CaCl_2 :1.5M Urea ratio 2/3+0.5g water melon urease+ milk powder	133	139

4 CONCLUSIONS

We performed a systematic parametric study on two fine grained problematic soils treated by mixing with different activators at different molarities and dosages. Waste materials have also been considered for both processes (i.e. as activators or as enzyme source). The studies were based on unconfined compression and p-wave velocity tests. All AA mixes showed great

improvements in the strength. EICP was less successful and variable in terms of strength improvement. However the latter process has potential and is worth investigating further in order to gain a better understanding of the

complex mechanisms involved. This can lead to the development of improved procedures that will give better results in terms of soil stabilisation.

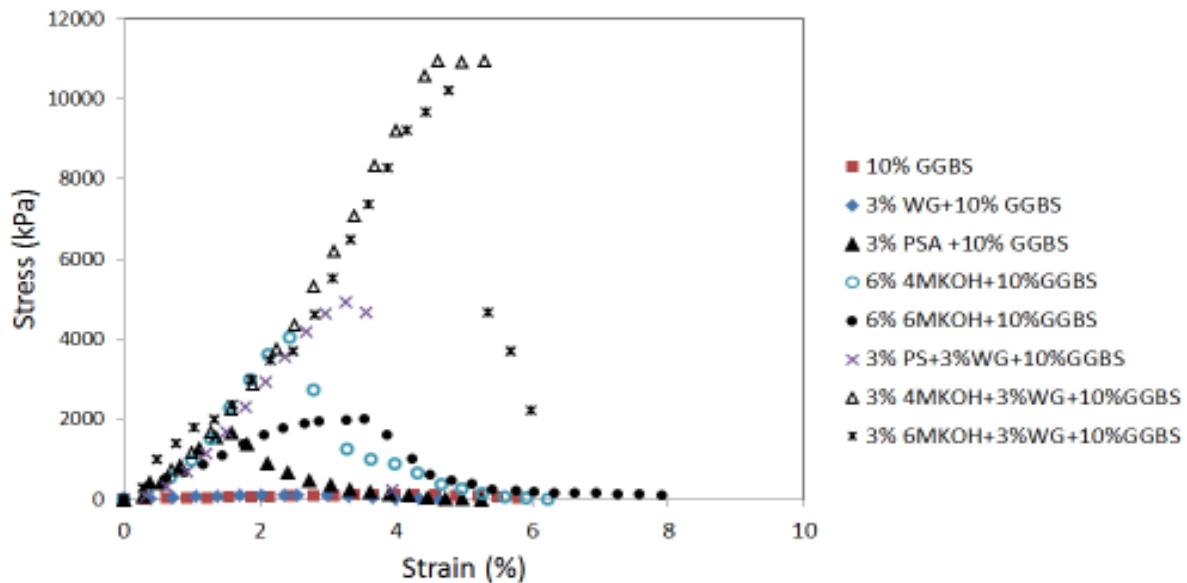


Figure 1 Indicative stress-strain curves based on UCS testing (silt soil)

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