Deep soft soil improvement by alkaline activation

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This research studied the use of alkaline activation of fly ash, as a silica and alumina amorphous source, to improve soft soils. A laboratory programme – including tests to study strength and deformability development, alternative curing methods, the effect of the different components of the grout, effect of raising initial temperature and comparison with a cement grout – was carried out. Laboratory tests revealed that the use of fly ash and alkaline activator resulted in a soil strength improvement up to 11.4, 16.7 and 43.4 MPa, at 28, 90 and 365 days curing, respectively. The most effective combinations obtained in the laboratory were chosen for the field application with jet grouting. The grout performed adequately to pass standard engineering specifications for soil mixing, achieving up to 26.4 MPa at 90 days curing. The main conclusion is the potential of alkaline activation for soil improvement, and therefore this research has created a basis for further studies.

1. Introduction
The alkali activation of materials containing silica + alumina has been researched and offers a potential alternative to cement in construction. Materials formed using reactions between silica and alumina and alkali cations such as sodium or potassium are very similar, at a molecular level, with natural rocks. Alkaline-activated materials have been shown to have improved mechanical characteristics at higher levels than cement. The amorphous chemical structure of fly ash makes its high levels of silica and alumina particularly applicable to alkaline activation, since its components are more susceptible to combination with sodium or potassium cations in this form. Therefore, alkali activation of fly-ash offers a potential financial and environmental cost saving when used as a cement replacement. Jet grouting is an advanced technology, using cement-slurry to improve the strength and stiffness of soft soils in situ and therefore offers a means of applying alkali activation as a ground improvement method. The aim of this study was to determine the potential of an alkaline-activated waste material to improve soft soils, particularly in depth, and the influence of several variables on strength gain. The research aims to understand if the activator/waste slurry is suitable to be used as a binder for the engineering improvement of a range of soils, and particularly as a substitute for Portland cement. It also included the evaluation of the effectiveness of the binder when used with jet grouting.

2. Alkaline activation of fly ash
The name ‘geopolymerisation’ refers to the dissolution of alumina (Al$_2$O$_3$) and silica (SiO$_2$) from some materials in an alkaline environment and their subsequent polymerisation to form a new material. It was first used by Davidovits in 1976 (Pacheco-Torgal et al., 2007) when he generically designated ‘geopolymers’ as a term related to the products achieved by alkaline activation of kaolin. A geopolymer is basically a three-dimensional aluminosilicate mineral polymer formed by several amorphous to semicrystalline phases. These alkali-activated aluminosilicate binders are formed by reacting raw solids which are rich in silica and aluminium with a solution of alkali or alkali salts resulting in a mixture of gels and crystalline compounds that eventually harden into a new strong matrix (Feng et al., 2004). The polycondensation reaction occurs in a high alkaline environment that reorganises aluminium and silica in a more stable Si–O–Al type structure, resulting in materials with high mechanical strength and chemical stability (Wang et al., 2005).

The development of alkali-activated cements, which have a significant proportion of the alkali metals Na or K (between 3 and 20%) in their composition, can be achieved by two main methods (Granizo, 1998).

(a) Compounds rich in calcium, such as lime, Portland cement, blast furnace slag, etc., combined with alkaline earth metals, with composition:

$Na_2O$–CaO–Al$_2$O$_3$–SiO$_2$–H$_2$O

(b) Soluble compounds of alkali metals combined with aluminosilicate materials (without the presence of any calcium), such as fly ash, metakaolin, burned clay and others, with composition:

$Na_2O$–Al$_2$O$_3$–SiO$_2$–H$_2$O

Both systems can be activated with sodium or potassium...
hydroxide, and the reactions will lead to fundamental modifications on the structure of the component materials. The research presented herein relates to the second group, since no calcium is involved. Palomo et al. (1999) summarises the main differences between both models in the following points.

(a) Composition of the material to be activated, essentially Si and Ca in the first case and Si and Al in the second.

(b) Concentration of the activator, being low to mild for the first model and high for the second.

As soon as the aluminosilicate material is mixed with the alkaline solution the hardening process begins, leaving insufficient time and space for the gel to transform into a well crystallised structure, as is the case in zeolite formation (Hua and Deventer, 2000). These authors compared X-ray diffraction (XRD) patterns of different minerals before and after activation and concluded that after geopolymerisation all of the main characteristic peaks of Al–Si decreased slightly in intensity, but still remained, suggesting that there was not a total dissolution into the gel phase. Furthermore, the fact that there were no new peaks suggested that no new major crystalline phases formed.

The great majority of aluminosilicates are crystalline in nature, making them highly stable in all but extreme environments. To promote the conditions for alkaline activation and to enhance the amount of polymerisation that takes place, the aluminosilicate raw materials should first be submitted to thermal treatment (Hua and Deventer, 2003). This will induce the loss of constituent water and the re-coordination of the aluminium and oxygen ions, which ultimately transforms the previously crystalline material into an amorphous one. These structural changes promote an environment in which chemical combinations more easily take place. For that reason, raw materials with a natural or artificial thermal history, such as fly-ash, blast furnace slag, Portland cement residues, pozzolanic wastes or metakaolin, are more suitable for alkaline activation than non-calcined materials such as clay or feldspars. For this reason, and also because of the environmental benefits of using a waste by-product (Hu et al., 2009), fly ash was used in this investigation. Another possibility was metakaolin, but its cost is much greater than fly ash, which makes it much less advantageous for potential commercial applications of this technique. The laboratory investigation into the development of alkali-activated binders for soil stabilisation and a field trial using these binders are described herein.

3. Laboratory research design

The aim of the laboratory programme was to develop suitable alkali-activated binders for ground improvement using jet grouting. The specific objectives are listed here.

(a) Establish the most advantageous fly ash percentage for both strength and stiffness development rate.

(b) Identify the effect of activator temperature on strength and stiffness.

(c) Understand how curing conditions affect the reactions.

(d) Determine the optimal activator concentration, as it constitutes the most expensive material involved in the process.

(e) Compare the effectiveness of alkali-activated grout with a common cement grout.

The soil used in the laboratory experiments was collected from the site where the field trials were later performed, sieved down to fractions below 1-18 mm. The Class F (low calcium content) fly ash used was obtained from a thermo-electric plant. Its chemical composition is given in Table 1.

The chosen alkaline activator solution was a combination of sodium silicate solution and sodium hydroxide solution. The sodium silicate was in solution form, with a specific gravity of 1.5 and a SiO2 : Na2O ratio of approximately 2 : 1 by mass. The sodium hydroxide was supplied in flake form, with a specific gravity of 2.13 at 20°C and 95–99% purity. The cement used to prepare laboratory samples was a Portland cement Type I, class 42.5 R. The mixer used was a 600 W kitchen appliance.

The activator/(soil + fly ash) ratio was kept constant during preparation of the samples, which meant that the activator/fly ash ratio was changed by variation of the fly ash percentage in the mixture. This ratio was of major significance for maintaining sufficient liquid phase to enable effective mixing, while preventing excessive liquidity. A ratio between 0.40 and 0.50 proved to be ideal. In order to determine the effect of the alkali–metal concentration, three different sodium hydroxide (NaOH) concentrations were used: 10, 12.5 and 15 molal. Preparation of the samples with 15 molal hydroxide concentration was far less straightforward than with lower concentrations. This was because the SiO2/Na2O weight ratio for the 15 molal mixtures was approximately 1, making the metasilicate solution very unstable, such that at higher temperatures it remains in solution form, but when cooled down it usually crystallises. This crystallisation occurred several times during preparation of the cold activator samples, and in most cases by the end of the mixing phase the mixture had become very viscous. Warming of the activator was found to prevent this problem occurring. Based on general alkaline activation research available in the literature (Hardijito and Rangan, 2005) and previous experience (Teixeira Pinto, 2006), it was decided to use a ratio of sodium silicate solution to sodium hydroxide solution, by mass, of 2. This was fixed because the sodium silicate was considerably cheaper than the hydroxide, and no significant gains have ever been reported with smaller ratios.

When sodium hydroxide and sodium silicate solutions are mixed...
together, the chemical reactions developed are highly exothermic. Furthermore, according to Fernández-Jiménez et al. (2006), the Si/Al ratio of aluminosilicate gel that is obtained from alkaline activation of fly ash F is highly dependent on, among other factors, the synthesis temperature. This is important because several authors (Duxson et al., 2005; Fletcher et al., 2005; Provis et al., 2005) highlight the importance of the Si/Al ratio on the mechanical behaviour of any alkaline-activated material. However, the heat generated in the laboratory mixing (± 30°C) was significantly lower than that expected for the larger quantities mixed in the field (> 50°C). This is why the warm activator, used in half of all the samples tested in this work, was kept in an oven at approximately 50°C.

Three 76 mm long, 38 mm diameter samples were tested for unconfined compressive strength (UCS) per mixture. One complete set of samples made were wrapped in cling film and stored at ambient temperature and humidity conditions. The other set was buried in wet sand (approximately 20% moisture content) at a depth of about 20 cm. The cement grout samples were prepared with water/cement ratios of 0.5, 0.75 and 1.0, with cement percentages of 20, 30 and 40%, in order to be able to directly compare the effect of cement content on strength.

### 4. Laboratory results

#### 4.1 Characterisation of the original soil

Geotechnical characterisation and mineralogical tests were performed, in accordance with BS 1377 (1990) Parts 1, 2, 4 and 7 (BSI, 1990a, b, c, d) on the soil collected from the site and are summarised in Figure 1 and Table 1. The soil was a sandy clay of low plasticity. The undrained triaxial tests were performed on saturated remoulded samples at the optimum water content. The XRD pattern showed the presence of quartz, kaolinite, muscovite (mica) and albite. All of these minerals are very common in this type of soil.

#### 4.2 Strength and stiffness of the stabilised soil

Each of the compressive strength test data plotted corresponds to the mean value of the compressive strengths of three tests. Figure 2 shows the UCS results obtained with 40% fly ash, cold activator and normal and buried curing for the 10, 12.5 and 15 molal hydroxide concentration. The addition of the grout increased the strength of the original dry soil progressively with curing time. The strength gain after 1 year curing is still increasing at the same rate as it was in the first 28 days. In both types of curing, and for all concentrations, there was no relevance of a peak value, which probably means that further strength developments can be expected with increased curing periods. The values for 12.5 and 15 molal were generally quite similar, suggesting that it is not effective, in terms of cost/improvement ratio, to use 15 molal concentrations. Although in the short term the 15 molal concentration mixture strength was higher than the 12.5 molal, the later produced higher strength at 90 and 365 days, for both types of curing. That, together with the already-mentioned problems regarding the chemical stability of the 15 molal mixture, seems to indicate that the 12.5 molal mixtures are indeed the best option.

In terms of fly ash percentage, Figure 3 clearly indicates that the higher the fly ash content in the mixture the better the performance of the mixture in terms of strength development. This was verified in both curing conditions.

In addition to the 20, 30 and 40% fly ash mixtures a 50% mixture was also considered, with activator/fly ash ratios of 0.8 and 0.9 at 7 and 28 days curing. Figure 4 shows an increase in strength at 7 days curing for both activator/fly ash ratios and for both curing conditions. However, at 28 days curing (normal and buried curing) the 0.9 ratio resulted in a decrease in strength for the 50% fly ash mixture.

The decrease in activator/fly ash ratio was achieved without any increase in the amount of activator, which is a positive factor since the activator is comparably more expensive than fly ash. Further tests are necessary to confirm the idea that a reduction in activator/fly ash ratio achieved by just reducing the amount of activator could also lead to a strength increase. However, these

<table>
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<th>Component</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>Fe₂O₃</th>
<th>CaO</th>
<th>MgO</th>
<th>K₂O</th>
<th>TiO₂</th>
<th>Others</th>
<th>L.O.I.*</th>
</tr>
</thead>
<tbody>
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<td>Percentage</td>
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<td>23.62</td>
<td>8.9</td>
<td>5.56</td>
<td>2.12</td>
<td>1.13</td>
<td>1.25</td>
<td>4.68</td>
<td>2.59</td>
</tr>
</tbody>
</table>

* L.O.I., loss on ignition.

Table 1. Chemical composition of the fly ash used
tests cannot be performed under the same conditions established during the research reported herein. This is because the only water added to the mixture was the water already present in the activator components, and preliminary tests showed that lower quantities of activator would not provide enough liquid to obtain a homogeneous mixture. Therefore the tests would have to be made in the field, first mixing the activator and the fly ash and only then injecting and mixing it with the soil.

The UCS development rate was similar for the samples made with cold and warm activator, however there was a significant difference regarding absolute values, with cold activator mixtures reaching higher values especially at 90 days curing and beyond (Figure 5). When using a potassium-based activator to stabilise a class F fly ash, Van Jaarsveld et al. (2002) reported that initial curing (no more than a couple of hours) at higher temperatures, which can be considered a situation similar to the warming of the activator immediately prior to the mixing, did not increase compressive strength substantially above that achieved with curing at room temperature. Only when the increase in temperature lasts longer do the positive effects on compressive strength become apparent. The same authors also concluded that curing for longer periods of time (more than 24 h) at elevated temperature possibly weakened the structure and suggest that small
amounts of structural water are needed in order to reduce cracking and maintain structural integrity.

It is also possible to conclude that, in general, burying the samples did not significantly reduce their strength performance, but only up to 90 days curing (Figure 3). The 20 and 30% fly ash samples indeed revealed higher UCS, after 90 days, than the equivalent normal-cured samples. After that time though, normal curing had a significantly better influence on strength development, as these values were more than double the values registered for the buried curing at 365 days. This might be due to temperature differences, as the temperature of the wet sand where the samples were kept was colder (between 2 and 5°C) than the ambient temperature.

4.3 Comparison with a cement grout

The curing period of all the cement samples tested was 28 days since it coincides with one of the curing periods used during the study of the alkaline-activated mixtures, and also because it is a reference curing period in soil stabilisation (cement and lime) and in the cement and concrete industry in general. The results obtained after such a period can be a good indicator of the final strength characteristics of the mixtures. The cement content was defined as the dry mass ratio between cement and soil.

A direct comparison between cement and alkaline grouts is shown in Figure 6. A water/cement (w/c) ratio of 1.0 was used as this is the most common w/c ratio in general practice. Comparison for curing periods longer than 28 days was necessary because it is well known that binders based on traditional Portland cement achieve most of their final strength after 28 days curing, whereas for the alkaline activator samples that period was revealed to be short for significant strength development.

A concrete-related mathematical expression to estimate the strength gain for the cement mixtures at 90 and 365 days curing was used. This was mostly because an estimation that was specific for a typical soil–cement material could not be found, but also because in terms of chemical and mineralogical composition the two materials are not so different, which makes the concrete approach significantly similar. The compressive strength of concrete at an age $t$ depends on the type of cement, the temperature and the curing conditions. For a mean temperature of 20°C, BS 1992-1-1:2004 (BSI, 2004) recommends the following relationship for moist-cured concrete made with normal Portland cement (Classe CEM 42.5 N), where $f_{cm}$ is the mean compressive strength at 28 days curing:

$$f_{cm}(t) = \beta_{cc}(t) \cdot f_{cm}^{1}$$

with

$$\beta_{cc}(t) = \exp\left\{s \cdot \left[1 - \left(\frac{28}{t}\right)^{1/2}\right]\right\}$$

where $f_{cm}(t)$ is the mean compressive strength at an age $t$ (in days); and $s$ is a coefficient which depends on the type of cement (0.25 for class CEM 42.5 N).

This comparison is not meant to be considered precise or capable of generating final conclusions about the capabilities of the alkaline activator technique, but only as a mere reference for the
strength gain rate and final values of this new binder. Since the buried curing can be considered most similar to a real field situation, and because cement grout samples achieved better results in that curing condition, the significantly superior results delivered by the alkaline activator normal cure mixtures should be acknowledged with some moderation, and so the buried curing results are considered more relevant for this study.

For the 12.5 molal concentration the results for 30 and 40% fly ash at 90 days were already at the same level as those estimated for the cement mixtures (Figure 7). At 365 days they were 33% (30% fly ash) and 73% (40% fly ash) higher than the estimated strength of the soil-cement material. Furthermore, the best cement result was reached at 30% content, which is not the highest content tested. This probably means this maximum strength cannot be raised by just increasing the cement content, whereas it was already established that by increasing the fly ash content on the alkaline activator grout above 40%, further strength limits could be achieved. Even if at short term (28 days) the cement samples showed better behaviour in terms of strength development, and even considering the possibility that the strength gain estimation of the soil/cement mixtures is too conservative, there is a great probability that the maximum strength of the soil/alkaline activator material is superior.

The following conclusions may be drawn from the laboratory tests.

(a) There was a strength gain with an increase in fly ash.

(b) Better results were obtained when a cold activator was used, which means that the activator should only be used when the heat generated from the exothermic reactions has dissipated to ambient temperature.

(c) An increase in activator concentration up to 15 molal was not beneficial, as the 12.5 molal concentration gave at least similar results, with the advantage of being cheaper and more chemically stable.

(d) Buried curing resulted in lower strength overall when compared with curing at ambient temperature and humidity; nevertheless there was a similar pattern in terms of strength evolution, with significant final values.

(e) The comparison with cement grouts showed that although at short term the alkaline-activated samples developed lower strength, at 3 months curing the strength was already higher than that of the cement samples.

5. Field tests

5.1 Design and construction

The field tests were performed using two different grouts corresponding to the 30 and 40% fly ash mixtures tested in the laboratory. The grout compositions are thus presented in Table 3.

The jet single system (BS EN 12716:2001) was chosen to produce the columns, as it is the cheapest and easiest system to use in practice (BSI, 2001). A set of cement grout columns was also constructed in order to evaluate the effect of each of the jet parameters on strength.

After careful analysis of the cement columns, and comparison

![Figure 7. Comparison between 12.5 molal and cement (w/c = 1.0) estimated strength (normal curing)](image)

<table>
<thead>
<tr>
<th>Material</th>
<th>Percentage of total slurry</th>
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<tbody>
<tr>
<td></td>
<td>30% fly ash</td>
</tr>
<tr>
<td>Sodium hydroxide</td>
<td>6.7%</td>
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<tr>
<td>Water</td>
<td>13.3%</td>
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<td>Sodium silicate</td>
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<tr>
<td>Fly ash†</td>
<td>40.0%</td>
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</table>

* This percentage refers to the grout + soil mixture.
† This percentage refers to the grout without the soil.

Table 3. Grout composition for 30 and 40% fly ash*
with the respective jet parameters used, the parameters to build the alkaline columns were defined (Table 4).

5.2 Field tests results
In order to perform mechanical tests on the samples recovered from the column sections after 3 months curing, the excavation work began 11 weeks after the construction of the columns (Figure 8).

The strength of the first columns recovered was minimal, allowing breaking by hand, without much effort. By this time it was obvious that something went wrong regarding the formation of a column of soil with a strong alkaline matrix and a visual inspection of the columns revealed that little, if any, mixing between soil and grout occurred (Figure 9).

Several hypotheses were formulated on why this segregation happened, but after the laboratory work and the field tests with cement, it was obvious that it must be something to do with the jet parameters as some of the columns achieved good visual consistency levels (Figure 10), and the samples extracted from these achieved significant UCS values (Table 5).

It seems that the fact that these particular columns had a consistency and geometry that was closer to that expected had more to do with chance than with any specific subtleties of their grout composition or jet parameters. This idea is reinforced by the fact that between the columns that achieved satisfactory strength levels there were significant differences in the jet parameters used. Another factor contributing to this hypothesis is the similarity of jet parameters between these columns and some other columns that achieved very poor results.

It is important to emphasise that the UCS values are merely an indication of the possible results that might be achievable if the issues related to the jet parameters are resolved. Due to the insufficient number of samples recovered and consequently

<table>
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<tr>
<th>Ref.</th>
<th>Nozzle diameter (× 2); mm</th>
<th>Rotation speed; rpm</th>
<th>Lift speed; cm</th>
<th>Stage gap; cm</th>
<th>Time/stage; s</th>
<th>Ls: m/min</th>
<th>Flow rate; l/min</th>
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Table 4. Parameters used for the alkaline activator columns
insufficient strength results obtained, they cannot be considered as statistically reliable. Furthermore, because of the reduced length of the sections that could be recovered, the direction of the samples for UCS tests, related to the column axis, was chosen with the intention of maximising the number of samples. This explains why most samples do not have a height/diameter ratio of 2, which is the normal value for unconfined compression tests of stabilised soil samples (BSI, 1990).

The comparison with the UCS results obtained from the cement grout samples from one of the columns is also relevant. The average strength for the cement column, 6.6 MPa, which was derived from values of 4.65, 5.33 and 9.8 MPa, was inferior to the average values obtained from column C (9.1 MPa) and column G (15.3 MPa).

Figure 11 shows a comparison between the results from Table 5, the cement columns and the results from the laboratory tests which best fit the conditions used to build the columns during the field trials. These laboratory results were obtained for the following conditions.

(a) Curing period: 90 days.

(b) Sodium hydroxide concentration: 12.5 molal.

(c) Fly ash percentages: 30 and 40%.

(d) Curing conditions: ‘cold–normal curing’ and ‘cold–buried curing’.

For both fly ash percentages of 30% (column K) and 40% (columns C and G) it is clear that, outlier points excluded, the...
results obtained in the field were in accordance with those from the laboratory tests. The field/laboratory strength ratio was 1.05, 0.87 and 0.32 for columns K, G and C, respectively. Although there is not enough data for a statistical approach and analysis, these results give a good reference regarding the potential of the technique, if jet grouting-specific issues are addressed and solved.

6. Conclusions

Extensive laboratory testing led to the following conclusions.

(a) Fly ash class F activated with sodium produced a paste which when mixed with soil and hardened formed a new material with significantly improved mechanical strength and deformation. This improvement was time dependent, revealing a significant increase after 1 year curing, in a process completely different from those using more common binders, such as Portland cement, which gain most of their strength at 28 days curing.

(b) The alkaline activation of this waste by-product is a simple process and it does not involve any sophisticated or costly procedures, or even any specialised or dangerous techniques.

(c) Use of the activator increased temperature – due to the exothermic reactions between its components – reduced strength when compared with ambient temperature-activated samples.

(d) In comparison with cement results, alkaline grout mixtures showed higher strength. However, in the short term, cement had a faster strength gain rate.

The field trial concluded that regarding the preparation of the grout, there was not much difference between alkaline-activated grout and cement, meaning that there is no significant time delay involved in the fabrication of this new grout. Furthermore, in terms of strength results it is important to remember that contrary to cement, which reaches approximately 80 to 85% of its final strength after 28 days, alkaline-activated fly ash strength, after 3 months, was only at 40 to 60% of its 1 year strength. However, UCS tests on samples obtained from sections recovered from alkaline and cement columns showed that at 3 months curing the strength level of the alkaline grout columns was greater than cement columns. Therefore, further research is needed to determine whether the difference in the strength gain rate is significant and, if that is the case, to develop ways to increase the speed of the reactions of the alkaline grout.

The main conclusion, based on the results obtained during this study, is that alkaline activation is a viable technique to be applied to soil stabilisation, more specifically to jet grouting columns, and is competitive with more traditional grouts, such as cement.

REFERENCES


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