

The activation energy of stabilised/solidified contaminated soils

B. Chitambira^{a,*}, A. Al-Tabbaa^b, A.S.R. Perera^b, X.D. Yu^c

^a Haskoning UK Ltd., Peterborough, UK

^b Department of Engineering, University of Cambridge, Cambridge, UK

^c Beijing Geotechnical Institute, Beijing, China

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Abstract

Developing an understanding of the time-related performance of cement-treated materials is essential in understanding their durability and long-term effectiveness. A number of models have been developed to predict this time-related performance. One such model is the maturity concept which involves use of the 'global' activation energy which derives from the Arrhenius equation. The accurate assessment of the activation energy is essential in the realistic modelling of the accelerated ageing of cement-treated soils. Experimentally, this model is applied to a series of tests performed at different elevated temperatures. Experimental work, related to the results of a time-related performance on a contaminated site in the UK treated with in situ stabilisation/solidification was carried out. Three different cement-based grouts were used on two model site soils which were both contaminated with a number of heavy metals and a hydrocarbon. Uncontaminated soils were also tested. Elevated temperatures up to 60 °C and curing periods up to 90 days were used. The resulting global activation energies for the uncontaminated and contaminated soils were compared. Lower values were obtained for the contaminated soils reflecting the effect of the contaminants. The resulting equivalent ages for the uncontaminated and contaminated mixes tested were 5.1–7.4 and 0.8–4.1 years, respectively. This work shows how a specific set of contaminants affect the E_a values for particular cementitious systems and how the maturity concept can be applied to cement-treated contaminated soils.

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1. Introduction

Developing an understanding of the time-related performance of cement-treated materials is essential in understanding their durability and long-term effectiveness. Studies have shown that the strength of cement-treated soils, both contaminated and uncontaminated, generally increases with time [1–5]. The rate of increase generally depends on the type and properties of the soil, type and quantity of the binder and the type and concentration of contaminants if present. Such published data extends to up to 5 years after treatment. For much longer time-related performance accelerated tests or accelerated ageing techniques are usually employed. These include durability tests, leaching tests, mechanical loading, chemical acceleration, carbonation and elevated temperatures. The use of elevated temperature, with and without carbonation, is the technique considered in this paper.

Limited research has been undertaken to study the use of elevated temperatures in stabilised materials [6]. Studies reported by Clare and Pollard [7] on stabilised uncontaminated soils and by Fuessle and Taylor [8] on stabilised waste showed that for elevated temperatures of up to 60–75 °C the hydration products are the same as those at lower temperatures but that their development and formation is accelerated and hence the hardening process is accelerated. Although elevated temperature curing of cementitious materials results in initial rapid strength development, the ultimate strength is lower and the permeability is higher. These effects can be related to the increased rate of silicate polymerisation at elevated temperatures which results in denser and stiffer reaction products and a coarser capillary pore structure in comparison with materials cured at a lower temperature [9]. Low curing temperatures lead to a uniform distribution of hydration products and interstitial pores. This can be used to explain why the strength for samples cured at elevated temperatures tend to cross-over that of similar samples cured at lower temperatures [10,11].

When carbon dioxide (CO₂) is introduced, carbonation, which is a naturally slow process, is also accelerated. The accelerated process would consume calcium ions (Ca²⁺) available in

* Corresponding author. Tel.: +44 1733 334455; fax: +44 1733 333360.
E-mail addresses: b.chitambira@royalhaskoning.com (B. Chitambira), aa22@eng.cam.ac.uk (A. Al-Tabbaa), asrp2@eng.cam.ac.uk (A.S.R. Perera), herayu@163bj.com (X.D. Yu).

certain anhydrous cement phases and hydration products to form calcium carbonate (CaCO_3). This leads to lowering of the pore fluids pH, to volume changes due to conversion of portlandite to CaCO_3 , and to the decalcification of C–S–H [12] and probably gain in strength and weight.

2. Elevated temperatures and the maturity concept

There are a number of approaches which characterise the strength development of cementitious systems with time. This includes the degree of hydration concept and maturity concepts. The latter is more frequently used and is considered here. Freiesleben Hansen and Pedersen [13] proposed one of the most popular maturity equations which is based on the activation energy concept (Eq. (1)). The equation calculates the time required at a reference temperature (also known as equivalent age, t_e), for a cementitious material to achieve the same degree of hydration under the influence of the actual time–temperature history:

$$t_e = \sum_0^t e^{(E_a/R)((1/T)-(1/T_0))} \Delta t \quad (1)$$

where t_e is the equivalent age at the reference temperature (days), T an arbitrary temperature in Kelvin ($=273.15 + T$ (°C)), T_0 the reference temperature (K) (usually taken as 20 °C, i.e. 293.15 K), Δt the change in time (days), E_a the activation energy (J/mol) and R is the Universal gas constant ($=8.3144$ J/mol K).

The activation energy concept derives from the Arrhenius rate theory for chemical reactions [14] which states that the rate of a reaction can be expressed using the following equation:

$$k = A \exp \left\{ -\frac{E_a}{RT} \right\} \quad (2)$$

where k is the rate constant (t^{-1}), A the pre-exponential factor (t^{-1}), E_a and R are defined as before and T is the absolute temperature (K). Since the hydration of cement is thermo-activated, it is widely acceptable to use this relationship to enable the transformation of the time under accelerated conditions to the time in a real (in-service) application to determine concrete maturity [15,10,11,9].

The hydration of cement involves several chemical reactions due to its composition; in addition to the transport of water and other agents, hence the value for E_a is termed the *apparent activation energy*, which is the average of the sum total of all the reactions taking place. In using the Arrhenius equation, it is assumed that all the important chemical reactions and transport mechanisms during the hydration of cement are affected by approximately the same extent due to an increase of temperature.

From the definition of the rate constant, the time for any chemical event is inversely proportional to the rate of that chemical reaction, as expressed in the following equation:

$$\frac{t_1}{t_2} = \frac{c/k_1}{c/k_2} = \frac{k_2}{k_1} \quad (3)$$

where t_1 is the age of samples cured at a temperature T_1 , t_2 the age of samples cured at a temperature T_2 , k_1 the rate constant for

the strength increase at T_1 , k_2 the rate constant for the strength increase at T_2 and c is a constant. The ratio t_1/t_2 given in Eq. (3) is also known as the time *shift factor* (a_T), and it describes the level of acceleration achieved by increasing the exposure temperature from T_1 to T_2 , leading to the following equation:

$$a_T = \frac{t_1}{t_2} = \exp \left\{ \frac{E_a}{R} \left(\frac{1}{T_2} - \frac{1}{T_1} \right) \right\} \quad (4)$$

The *apparent activation energy* (E_a) defines the temperature sensitivity of the hydration process of a particular cementitious mix [16]. A number of different techniques have been developed and applied for the evaluation of E_a values [17–21].

Knowing the E_a value of the strength development, Eq. (1) can be used to evaluate the equivalent age. The extent of reactions can be evaluated by simply monitoring chemical properties, physical properties or both as a function of time [17–21]. Equations that express the measured chemical or physical parameter as a function of time are used to determine the temperature-dependent rate parameters from which E_a can be evaluated. As the unconfined compressive strength (UCS) is the most commonly measured parameter for cementitious materials, it was the parameter chosen for this study. The E_a value is usually assumed to be constant over the experimental range of temperatures [13,22,23], and this assumption is also the basis of a number of standards [24,25] and in this case E_a is termed the ‘global’ activation energy.

Based on Eq. (4), a simple graphical approach for determining E_a is presented based on shifting the UCS profiles along the horizontal axis to obtain shift factors. This is similar to the approach of constructing a *master curve* from compliance curves using time–temperature–superposition principles [26,27]. First the UCS versus age results are plotted on a logarithmic scale and then by shifting all the other curves in relation to the curve at the reference temperature, the shift factors are obtained. A plot of the shift factors against the reciprocal of temperature in degree Kelvin (or the difference in the reciprocal of the temperatures in question) produces a straight line graph whose slope is proportional to E_a . Mathematically this is equivalent to plotting the natural logarithm of the rate constant ratios $\ln(k_2/k_1)$ for the two given temperatures T_1 and T_2 against the inverse of the temperature difference in Kelvin ($(1/T_2) - (1/T_1)$). Once the E_a is evaluated, maturity concepts are used to obtain the equivalent age according to Eq. (1). Full details of this technique are given in Chitambira [28].

The Arrhenius equation should accurately describe the time–temperature relationship for the overall hydration process if similar hydration mechanisms were taking place at the different temperatures. This is indicated by curves or straight lines of similar shape. If this condition is not met, there are possibilities that different hydration or water transport mechanisms could have been taking place for the different temperatures concerned. Factors which affect the value of the apparent activation energy of cementitious mixes include cement type and chemical composition, cement fineness, cement content, water:cement ratio, supplementary cementing materials, admixtures, mixture proportions, degree of saturation and other physical properties

such as density, thermal conductivity and specific heat [21,29]. Using semi-adiabatic testing, Schindler [16] and Schindler and Folliard [30,29] evaluated and produced the following ranges and variations in the E_a values:

- (i) For Type I cement from three different sources, the E_a value ranged between 41,977 and 46,269 J/mol.
- (ii) For Types II–V cements the E_a values obtained were 41,788, 49,955, 39,978 and 37,329 J/mol respectively.
- (iii) For Type I cement with 15–45% Class C fly ash addition, the E_a value changed from 43,148 to 37,461 J/mol, hence the presence of the ash reduced the E_a value compared to that of Type I cement alone and as the ash content increased, the E_a value decreased further.
- (iv) For Type I cement with 15–45% Class F fly ash addition, the E_a value changed from 40,703 to 30,127 J/mol, hence the addition of Class F ash gives a lower range of E_a values compared to the addition of Class C ash. This could be attributed to the fact that Class C fly ash contains a higher percentage of CaO in comparison with Class F fly ash which is purely pozzolanic.

Work carried out by Ma et al. [31] based on isothermal calorimetry performed at temperatures ranging from 10 to 55 °C showed that Type I cement gave $E_a = 39,000$ J/mol, while Type I with 17% Class F fly ash replacement gave $E_a = 26,700$ J/mol. These results show similarities but also differences with the results obtained by Schindler and Folliard above which highlights a high level of sensitivity of the E_a values to a wide range of variables; the higher the E_a the more sensitive the hydration process is to temperature changes. Carino and Lew [10] reported apparent activation energy values for concrete between 30,000 and 60,000 J/mol for water/cement ratios of 0.45–0.6. This above range of different E_a results simply highlights the system-specific nature of the E_a value.

Studies on the thermal activation of lime-pozzolan cements have also shown that the E_a value is affected by the presence of accelerating admixtures. In a study of cementitious mixes, with a water:cement ratio of 0.5 at a range of temperatures between 23 and 65 °C, Shi and Day [19] found lime-pozzolan cement mixes to have $E_a = 66,000$ J/mol compared to 75,000 J/mol for lime-pozzolan with 4% sodium sulphate (Na_2SO_4) and 99,000 J/mol for lime-pozzolan with 4% calcium chloride (CaCl_2).

3. Materials and methods

The work reported here is related to an in situ stabilisation/solidification (S/S) trial conducted at a site in West Drayton near Heathrow Airport in the UK [32–34,5]. The results presented are for two cement-stabilised soils. One is a made ground, consisting of 20% clay, 20% silt, 15% sand and 45% fine gravel and the other is a sand and gravel, consisting of 6.5% clay, 6.5% silt, 37% sand and 50% fine gravel. These are laboratory-based models of the site soils. The two soils, contaminated and uncontaminated, were mixed with three different cement-based grouts as detailed in Table 1. Mix B grout contained cement and pulverised fuel ash (PFA), mix E grout contained lime in

Table 1
Details of the three cement-based soil-grout mixes used

Mix	Cement:PFA:lime: bentonite	Water:dry grout	Soil:grout	Soil:dry grout
B	3:8:0:0	0.42:1	5:1	7:1
E	2.5:8:0.4:0	0.42:1	3.5:1	5:1
G	8:0:0:0.8	1.6:1	3.7:1	10:1

Table 2
The contaminant compounds and concentrations used

Contaminant	Compound	Concentration of contaminant (mg/kg dry soil)
Lead (Pb)	$\text{Pb}(\text{NO}_3)_2$	2800
Copper (Cu)	$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$	1300
Zinc (Zn)	ZnCl_2	1600
Nickel (Ni)	$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	105
Cadmium (Cd)	$\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$	9
Mineral oil	Liquid paraffin	1700 in made ground, 8700 in sand and gravel

addition and mix G grout contained cement and bentonite only. The contaminated soils contained a cocktail of heavy metals and organics and were modelled using the contaminants detailed in Table 2. The contaminants and their concentrations were chosen to simulate similar conditions to those in the site soils. Mixing of the grout and model soil was performed by means of a food mixer to a homogenous consistence and the mixture was subsequently placed into moulds. After a few days, the samples were demoulded and waxed to minimise moisture loss. The mixes were subjected to three temperatures of 21 °C (control), 45 and 60 °C for three durations of 1, 2 and 3 months, following an initial period of up to 1 week in control conditions. At each of the test durations, the samples were tested in triplicate for UCS and the results reported have a margin of error of $\pm 10\%$. The test specimens were 50 mm in diameter and 100 mm high. Full details can be found in Chitambira [28]. In addition one mix was subjected to those elevated temperatures in a 20% carbon dioxide atmosphere to accelerate the carbonation process.

4. Modelling the UCS of blended-cement-soil mixes

In the absence of reported E_a values for stabilised soils, the results published by Porbaha [4] are used to illustrate how the E_a value is evaluated from unconfined compressive strength (UCS) development data. Fig. 1(a) shows the UCS–age curve, up to 30 days, for five different temperatures of between 10 and 50 °C for a marine clay stabilised with 20% cement. Fig. 1(b) shows the same data plotted on a natural log–log scale. By taking the results at 20 °C as the reference, all the other curves were shifted along the x -axis to obtain a smooth curve as shown in Fig. 1(c). The shift factors so obtained are then plotted against $((1/T_0) - (1/T))$ as shown in Fig. 1(d) so that the slope of the resultant curve is proportional to E_a/R . This gives an E_a value of 69,080 J/mol. Corresponding results for 10% and 30% cement addition produced E_a values of 60,570 and 74,450 J/mol, respectively. Hence the E_a value increases with increasing cement content. Therefore as the cement content increases, the temperature sensitivity

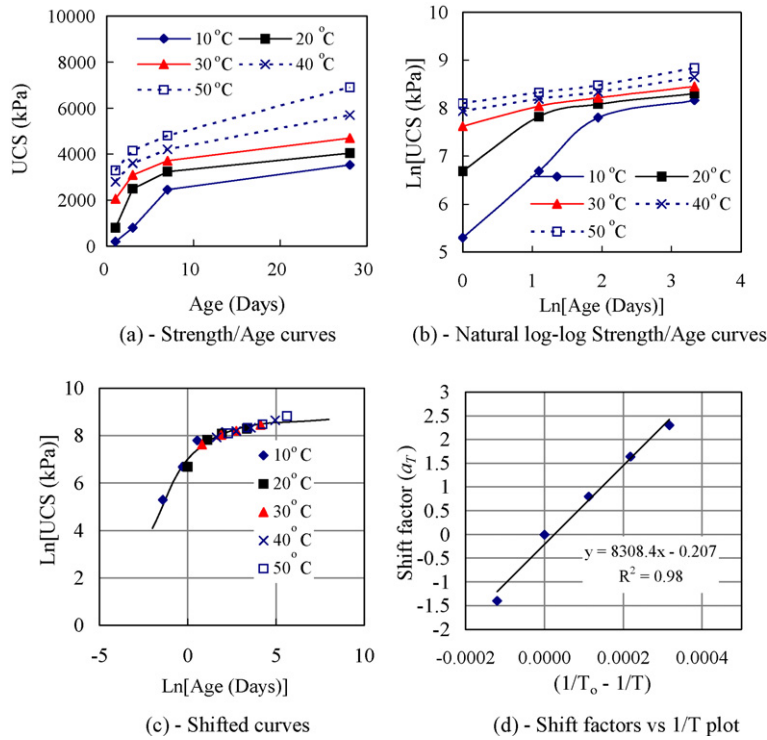


Fig. 1. Modelling graphs for soil stabilised with 20% cement (modified from Porbaha [4]).

of the hydration process (due to different hydration reactions) increases. These values are at the upper end of the range of values reported above for cement and concrete mixes indicating a higher temperature sensitivity.

Once the activation energy (E_a) is estimated, lines representing shift factors at any arbitrary exposure (or service) temperature can be drawn as shown in Fig. 2 for the results in Fig. 1 using Eq. (4). These are then used in conjunction with other physical and chemical tests to assess the durability of the S/S materials under varying service temperatures. For example, if the ground temperature is taken to be 10 °C, an accelerated test performed at 60 °C would therefore give a shift factor of 81.6 from Fig. 2. This implies that for instance a test carried out for 30 days will represent a mix in the field aged for 6.7 years.

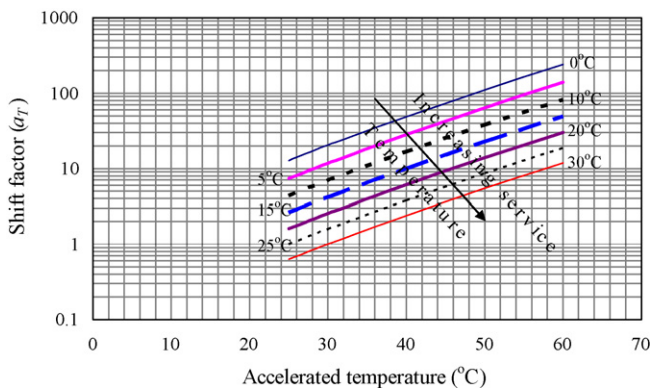


Fig. 2. Shift factors as a function of accelerated exposure temperature and service temperature for soil stabilised with 20% cement, based on results from Fig. 1.

The shifting technique described above is accurate if all the lines considered have approximately the same shape or are affine [35]. If the curves do not display nearly the same shape, the evaluated E_a value would only be indicative and cannot be an accurate representation of the hydration kinetics of the cementitious mix concerned. In other words more than one mechanism is required to fully characterise the hydration behaviour of such a mix at all the curing temperatures.

The variation of the UCS with time and temperature for the three mixes in the two uncontaminated soils is shown in Fig. 3(a)–(c) and (d)–(f), respectively. The corresponding profiles for the contaminated mixes are presented in Fig. 4. The expected increase in UCS with an increase in curing temperature and time can be observed in most of the graphs. Few mixes showed some signs of reduction in the UCS with time. This was not necessarily related to moisture loss as the density was relatively unchanged. The deterioration in the later age strength of elevated-temperature-cured mixes is widely documented [36,37]. At longer curing ages, while mixes cured at low temperatures continued to gain strength, their counterpart elevated-temperature-cured mixes fell, crossing them over as shown in all the graphs for mixes cured at 60 °C and is usually referred to as the *cross-over effect* [10]. Except for mix E sand and gravel, most of the UCS at higher temperatures was developed within the first 28 days, after which there was a small steady increase in the UCS. This agrees with other studies which have shown that temperature effects are more pronounced in the early ages but less so at later ages [8]. The differences in the behaviour between the different mixes are likely to be related to the cement content and to the presence of the other binder constituents.

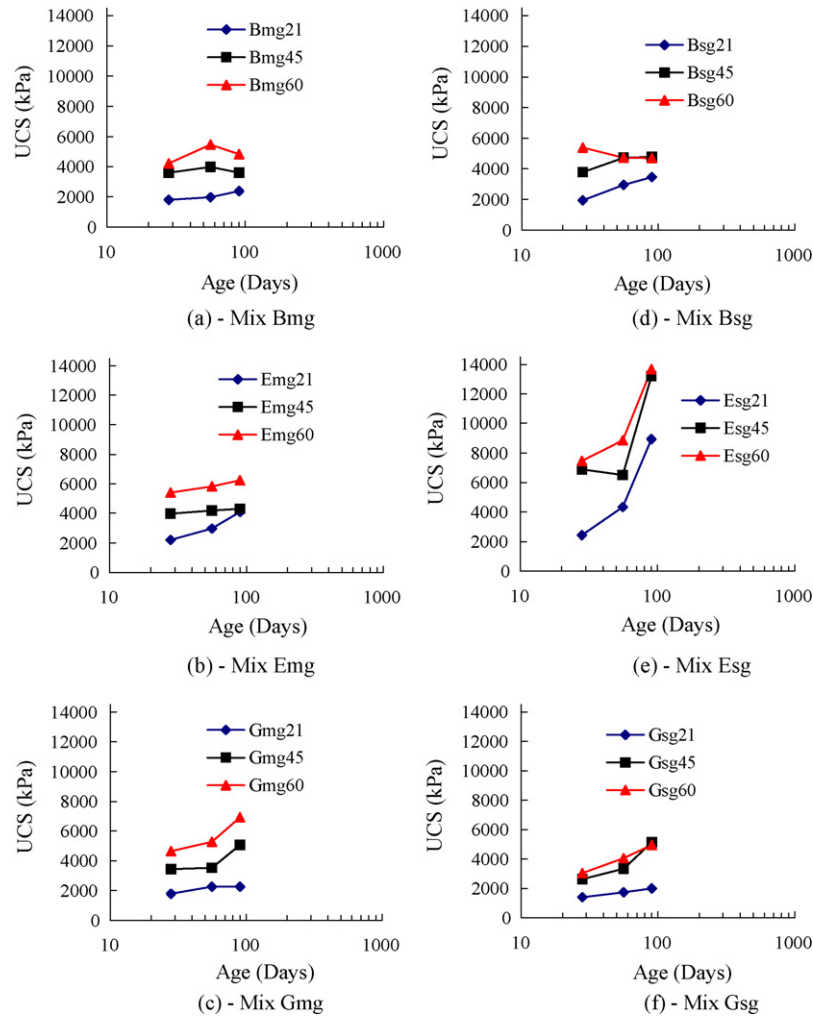


Fig. 3. The UCS profile of six uncontaminated mixes at the different temperatures and curing times where ‘mg’ refers to the made ground and ‘sg’ refers to the sand and gravel soils and the numbers 21, 45 and 60 refer to the curing temperatures (°C).

Since the UCS–age trends observed here are not all similar or affine and hence the conditions are not ideal for applying the Arrhenius equation to, the results reported below should be used with caution. Nevertheless they do provide a rough indication of the values and trends to expect from such mixes. The E_a values for the six mixes calculated from the data in Figs. 3 and 4, as illustrated in Fig. 1, are given in Table 3. Graphs of the shift factors plotted against $((1/T_0) - (1/T))$ produced straight lines of the form $y = ax + b$ with a calculated coefficient of correlation (R^2) of at least 0.98 and the constants a and b are shown in Table 4. For the uncontaminated mixes, the results are generally similar and are within a small range of between 63,220 and 70,990 J/mol. The results show that the difference in the E_a val-

ues for the made ground and sand and gravel is small suggesting that in this particular study the differences in the soil constituents did not influence the E_a value. They also show that the difference between the different binder mixes is also small. This is probably due to the relatively small grout content within the mixes (Table 1). However the trends in those small changes are consistent with changes in the binders presented above [16,29,30] in that mix E has a 33% higher PFA content and a slightly lower E_a value than mix B while mix G has twice the cement content, compared to mixes B and E, and has a slightly higher E_a value. These values are also within the range of values calculated earlier using the Porbaha [4] results suggesting that soil-cementitious binder mixes, which usually have lower binder contents and den-

Table 3
The apparent activation energies (E_a) (in J/mol) for the six stabilised soils, both contaminated and uncontaminated

Mix soil	Bmg	Bsg	Emg	Esg	Gmg	Gsg
Uncontaminated soil at elevated temperatures	67620	67625	65055	63220	70390	70990
Contaminated soil at elevated temperatures	43680	43430	43875	51320	58910	25020
Contaminated soil at elevated temperature + accelerated carbonation				63500		

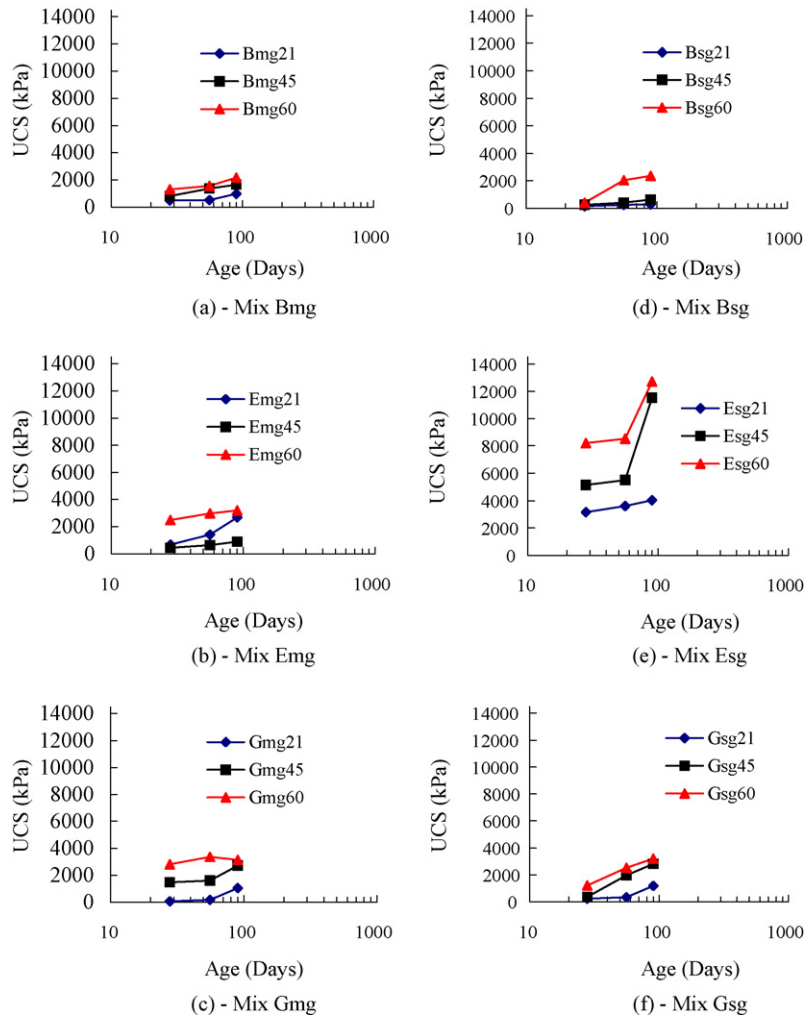


Fig. 4. The UCS profile of all six contaminated mixes at the different temperatures and curing times where ‘mg’ refers to the made ground and ‘sg’ refers to the sand and gravel soils and the numbers 21, 45 and 60 refer to the curing temperatures (°C).

sities and higher water contents, have a higher level of sensitivity of the hydration reactions to temperature changes than cement pastes and concrete.

The E_a values for the contaminated mixes are much lower than those of the corresponding uncontaminated mixes and with the exception of mix G sand and gravel also lie within the range of 43,430–58,910 J/mol. Since the only difference between those mixes and the uncontaminated mixes is the presence of the contaminants, this gives an indication of their effect on the global apparent activation energy. The E_a values in this case reduced to

up to an average of 70% of their values for the uncontaminated mixes. The values for the two soils for each mix are generally still similar except for mix G in which the E_a value for the sand and gravel is much lower than that for the made ground. The reason for this is not clear and requires further investigation. The reduced E_a value reflects the reduction in the sensitivity of the hydration process to temperature changes, hence reflecting a much slower or retarded hydration with time. Retardation of the hydration process is a common effect of many contaminant cocktails on cementitious binders [38] and its effect as expected

Table 4

The constants in the shift factor equation for the six stabilised soils, both contaminated and uncontaminated (R^2 for all plots was 1.0)

Mix	Uncontaminated soil at elevated temperatures		Contaminated soil at elevated temperatures		Contaminated soil at elevated temperature + accelerated carbonation	
	<i>a</i>	<i>b</i>	<i>a</i>	<i>b</i>	<i>a</i>	<i>b</i>
Bmg	8132.7	−0.0047	5253.2	0.0		
Bsg	8133.5	−0.0047	5223.1	−0.0018		
Emg	7824.4	+7E−07	5277.0	0.0		
Esg	7603.6	+6E−07	6172.7	−0.0036	7640.0	0.0
Gmg	8466.3	+7E−07	7085.5	−0.0041		
Gsg	8538.6	+7E−07	3009.5	−0.0017		

is opposite to the effect of accelerating admixtures discussed above [19].

In addition to the above mixes, contaminated mix E sand and gravel was subjected to a combination of elevated temperature and accelerated carbonation. The mix samples were initially subjected to the elevated temperatures alone for 1 week to allow hydration to be accelerated without interference from early carbonation. The samples were then subjected to the combined conditions in incubators at 90% relative humidity. The UCS–age results are shown in Fig. 5. The corresponding global apparent activation energy for this mix is also shown in Table 3. The E_a value of 63,500 J/mol is higher than that of the corresponding contaminated mix subjected to elevated temperatures alone of 51,320 J/mol. This shows that the effect of the accelerated carbonation was to accelerate the hydration process, similar to accelerating admixtures, and hence accelerate the ageing process with temperature. This means that the temperature sensitivity of the hydration process was increased in the presence of carbonation.

The above E_a values were then used to predict the equivalent age for the respective mixes. Those results lead to the conclusion that with contaminants, the accessed accelerated times are shorter compared to the uncontaminated mixes and longer accelerated times can be accessed with accelerated carbonation. The range of accelerated ages for the above uncontaminated soils is 5.1–7.4 years and that for the contaminated soils is 0.8–4.1 years. The single accelerated carbonation result gives an equivalent time of 5.2 years. This gives an indication of the accelerated equivalent ages obtained from curing those samples at elevated temperatures of up to 60 °C for up to 3 months. Full details of how the equivalent age was calculated for those mixes are given elsewhere [29,39]. These results are generally consistent with the real-time UCS–age performance at the West Drayton site [5]. A constant multiplier was found to relate the West Drayton data to the laboratory data [28]. This work shows that the derivation of the apparent activation energy from the UCS–temperature results presented here using the maturity concept can be used for the modelling of accelerated ageing of stabilised/solidified contaminated soils.

It is worth pointing out that although only three points are shown on each graph, which is the minimum number of points needed for such a study, the results managed to show the effect of the contaminants and carbonation on the apparent activation energy values.

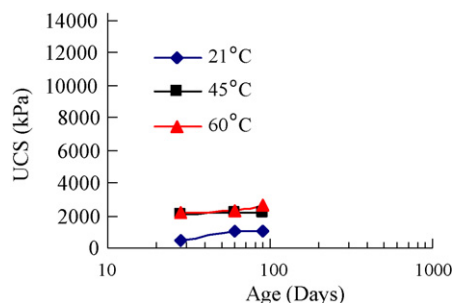


Fig. 5. The UCS for mixes subjected to combined elevated temperature and accelerated carbonation: mix E sand and gravel.

It should also be pointed out that the effect of contaminants on the E_a value is expected to be extremely complex due to the significant range of contaminant compounds and their concentrations that could be present in soils. For the study reported here the specific contaminant mixes were selected so that the results can be compared with real-time field behaviour. Clearly for a full understanding of the effect of specific contaminants on the E_a values, contaminants need to be added individually to soils and in different concentrations. However, contaminants are also known to be affected by the presence of other contaminants, which makes a generic study of this kind very complex. This also applies to the effect of the binder and native soil properties. Hence such a study is best suited to site specific conditions as performed in the study reported here where known contaminants and binder conditions are being investigated.

5. Conclusions

A derivation of the apparent activation energy, E_a , for any curing soil–cement mix is important in understanding its service life. This parameter is sensitive to different conditions such as water:cement ratio, binder content and type, temperature and content of mineral admixtures like PFA. Global apparent activation energies were determined for both uncontaminated and contaminated soil mixes treated with three cement-based grouts. The mixes were subjected to elevated temperatures, 45 and 60 °C, for up to 3 months and in one case also exposed to carbonation. The obtained E_a values for the uncontaminated mixes ranged between 63,220 and 70,990 J/mol while those for the contaminated mixes were much lower ranging between 25,020 and 58,910 J/mol. The E_a values for the uncontaminated mixes were found to be at the upper end of the range reported in the literature for cements and concrete. The much reduced values due to the presence of the contaminants reflect the reduced level of temperature sensitivity of the hydration process. Carbonation of one of the contaminated mixes was found to increase the E_a value hence accelerating the hydration process with temperature. This leads to the conclusion that with the specific contaminants tested, the accessed accelerated times are shorter compared to the uncontaminated mixes and longer accelerated times can be accessed with accelerated carbonation. Equivalent ages evaluated on the bases of the derived E_a values ranged from 5.1 and 7.4 years for the uncontaminated mixes, 0.8–4.1 years for the contaminated mixes and 5.2 for the carbonated contaminated mix, all for curing at temperatures up to 60 °C for 90 days. This work shows that the derivation of the apparent activation energy from the UCS–temperature results presented here using the maturity concept can be used for the modelling of accelerated ageing of stabilised/solidified contaminated soils. The results reported here are specific to the conditions investigated and the same procedure will need to be repeated for different site conditions.

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