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TITLE Carbonation of stabilised mixtures

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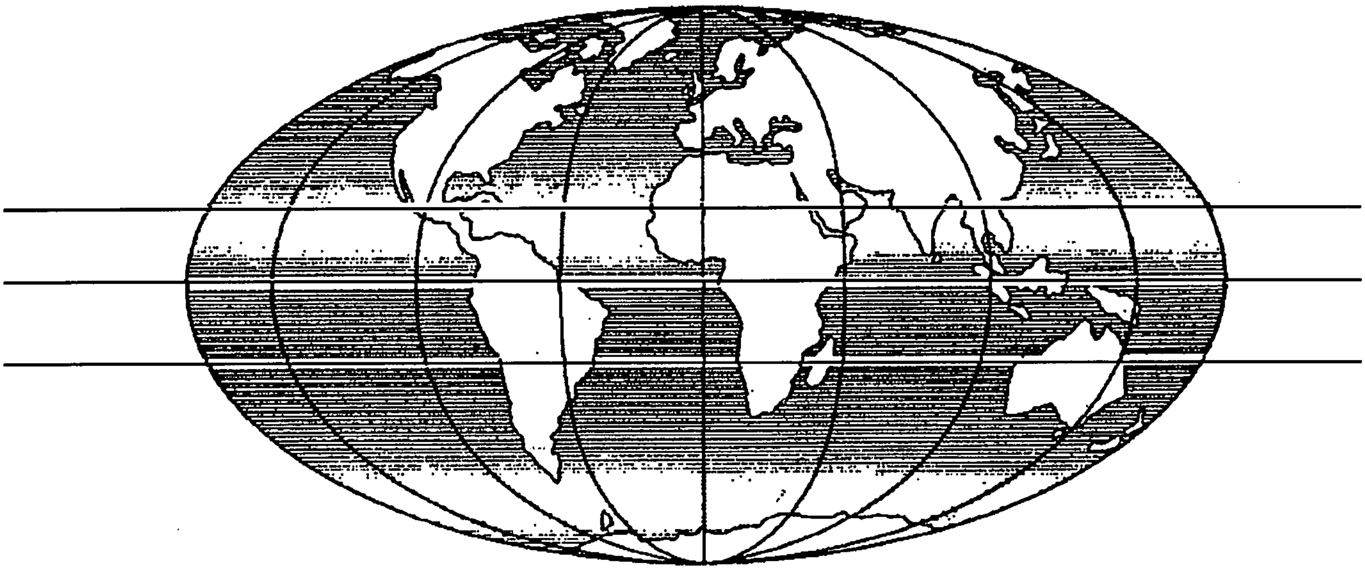

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CARBONATION OF STABILISED SOIL-CEMENT AND SOIL-LIME MIXTURES

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ABSTRACT

Soil stabilisation with cement or lime is used in many countries in the construction of road bases and sub-bases and for the treatment of subgrade soils. Recent studies have shown that the performance of stabilised layers can be affected by carbonation which inhibits the formation of cementitious products in soil-cement and soil-lime reactions.

This paper examines the evidence for carbonation in road trials constructed in a hot, arid area in Botswana and describes the laboratory study that followed using a clayey sand (a poor quality calcrete) from Botswana to investigate the effect of different curing conditions on soil-cement and soil-lime mixtures. Humidity, temperature and carbon dioxide in the environment were varied and the affect on strength and properties of the stabilised soils were assessed by the unconfined compressive strength and plasticity tests. Different curing periods were examined and the extent of carbonation was identified by the use of phenolphthalein indicator.

The results showed that carbonation to varying degrees and loss of strength occurred in all the strength-age relations except those that were cured under sealed conditions. Samples that were first allowed to harden also subsequently lost strength when exposed to carbon dioxide. It was also shown that changes in plasticity which occur in soil stabilisation and are an essential requirement in specifications for modified soils could also be reversed by carbonation.

There remains a need to obtain more field evidence of carbonation and its relation to pavement performance, curing conditions and climatic environment. In addition a better understanding of the mechanism of carbonation is required especially of those reactions which involve cemented products.

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1. INTRODUCTION

Soil stabilisation with cement or lime is used in many countries in the construction of road bases and sub-bases and for the treatment of subgrade soils. The process which enables a wider range of locally available materials to be used relies on chemical changes to give gains in strength or modification of soil properties to achieve satisfactory performance. Recent studies of stabilised road bases have shown that performance can be affected by carbonation which inhibits the formation of cementitious products in soil-lime and soil-cement reactions (1). Carbon dioxide in the atmosphere reacts with lime to form calcium carbonate. This reaction, known as carbonation has been identified mostly in hot dry climates where control of curing is difficult. As lime is a product of soil-cement reactions, cement stabilised soils are also vulnerable to carbonation.

Carbonation occurred in road trials conducted by the Overseas Unit of TRRL in a hot arid area in Botswana. Road base sections using a calcareous clayey sand, known locally as calcrete, failed to harden when stabilised with cement and lime. This paper, after examining the evidence for carbonation in the road trials describes a laboratory study at the University of Birmingham, using the calcrete from Botswana, to investigate the effect of different curing conditions on soil-cement and soil-lime mixtures. Humidity, temperature and carbon dioxide in the environment were varied and the affect on strength and properties of the stabilised soils was assessed by unconfined compressive strength and plasticity tests. In one series of tests some of the above treatments were applied to stabilised soils that had already hardened to determine whether carbonation could cause a loss of strength. The extent to which carbonation occurred in the tests was identified visually by spraying with phenolphthalein indicator which was a reflection of a change in pH of the stabilised soil.

2. SOIL STABILISATION AND CARBONATION

The two most commonly used soil stabilising agents are lime and cement. Lime is either the oxide or hydroxide of calcium although magnesium oxide or hydroxide may also be present. The oxide is usually known as quicklime or unslaked lime and the hydroxide is hydrated lime. Lime is usually made by burning limestone (calcium carbonate) which converts the carbonate to the oxide. If water is added to the oxide by the process of slaking the hydroxide is formed. It is in the form of the oxide or hydroxide that reactions with soil take place and it is for this reason that lime is normally specified by the amount of 'available lime' that is present. If lime is left exposed it will react with carbon dioxide in the atmosphere and gradually revert to calcium carbonate. It is this process that occurs during carbonation. When soil and lime are mixed together in the presence of water two main reactions occur. One is relatively rapid and involves ion exchange which results in the flocculation of clay particles in the soil and a consequent reduction in plasticity. The other is a pozzolanic reaction which if sufficient lime is added to the soil to reach a pH of 12.4 will usually continue for a considerable time. In this reaction lime and the clay mineral constituents combine and form hydrated calcium and aluminium silicate gels which on crystallisation are largely responsible for the hardening and development of strength. Lime is also produced in the hydration of cement when water is added. However, with cement which contains finely ground calcium silicates and aluminates other reactions also occur. In addition to the reduction in plasticity, intergrowths with soil particles are formed and these give more rapid increases in strength than the slower pozzolanic reactions. It is this bonding by the cement which explains why non-plastic soils will generally harden with cement but not with lime.

3. FIELD EVIDENCE OF CARBONATION

In the Botswana road trials calcrete, which is a naturally-occurring calcareous material of pedeogenic origin, was assessed as base material for lightly-trafficked bituminous surfaced roads. Four different types of calcrete were included in the trials which comprised a total of nine sections (2). The poorest quality calcrete in addition to being examined in the untreated condition was also treated separately with hydrated lime and Portland cement. Initial laboratory tests had shown that the addition of 3 per cent of stabiliser was sufficient to achieve the strength required for cement and lime stabilised road base materials. This was further confirmed at the time of construction when samples were taken from the road sections after mixing was complete but before field compaction was carried out. The samples were then taken to the laboratory and compacted by the 4.5Kg rammer method (3) into CBR moulds at their field moisture contents. The results of the CBR tests on samples cured under sealed conditions for 7 and 28 days are shown in Table 1. Generally considerable gains in strength occurred between 7 and 28 days indicating that hardening of the soil-cement and soil-lime mixtures had taken place normally under laboratory conditions. In the road trials a gain in strength was observed during the first six months following construction. However, subsequent measurements carried out at intervals during the next five years showed a fall in strength (see Figs 1 - 3). The initial strength gain was mirrored by changes in the moisture content in the base materials. During this time the base was left covered with an MC30 prime coat and a surface dressing seal was only applied after six months. The fact that drying occurred would indicate that the MC30 prime was not an effective moisture barrier and, consequently, not an effective curing membrane as has been shown in other studies (4). The drying that occurred during this time would have increased the risk of carbonation. It is also likely that the strength gain during this initial period may have been the result of drying. Subsequently, lower CBR values were measured at higher, in service moisture contents.

The plasticity characteristics of samples taken from the road trials are given in Table 2. These show that the addition of lime and cement did effect a considerable reduction in the plasticity index (PI) following field mixing. Tests on samples taken from the base after a number of years indicate little change in their plasticity with time.

Carbonation in the stabilised bases was detected when tests were conducted using phenolphthalein indicator. Phenolphthalein which has been used for many years to detect carbonation in concrete (5) is an indicator which changes from colourless to bright red when the pH (hydrogen ion concentration) rises above the value of 9. Lime which is present in both soil-lime and soil-cement mixtures has a pH greater than 12. Calcium carbonate, the product of carbonation, has a pH between 8.0 and 8.5. It follows then that carbonation has probably taken place if there is no colour change when the phenolphthalein solution is sprayed onto a freshly exposed surface of the soil-stabilised layer. The tests on both the soil-lime and soil-cement trial sections behaved in this way. Further confirmation of carbonation can usually be made by the addition of a small amount of dilute hydrochloric acid which in the presence of carbonate will effervesce because of the liberation of carbon dioxide. Clearly in the case of calcretes which already contain calcium carbonate this cannot be used as a confirmatory test. The application of the phenolphthalein test to lime and cement treated materials has been developed by Netterberg (6).

It was largely as a result of the performance and behaviour of the stabilised sections in the Botswana road trials, and other similar experiences in the region (7), that it was decided to undertake a laboratory study to investigate some of the causes and effects of carbonation.

4. DESIGN OF THE LABORATORY STUDY

4.1 Objectives

The main objectives of the study were to determine the conditions under which carbonation takes place in lime and cement stabilised or modified soils and to what extent this affected firstly the hardening and secondly the plasticity. It was also important to determine whether stabilised soils which had already gained strength through adequate curing could subsequently be affected by carbonation.

The investigation was carried out in two separate but generally similar series of tests, firstly on soil-lime mixtures and then on soil-cement mixtures. The respective proportions of lime and cement additive were 3 per cent and 4 per cent based on the dry weight of the soil.

4.2 Properties of the soil used

The soil used in the study was the same calcrete that was used for the cement and lime treated sections in the Botswana road trials. It was described as a plastic calcified sand and its main properties are shown in Table 3.

4.3 Strength tests

4.3.1 Unconfined compressive strength tests. Unconfined compressive strength tests on cylindrical specimens 100mm in height and 50mm in diameter were used to measure the strengths of the soil-lime and soil-cement mixtures (8). The densities and moisture contents of the samples prepared were those obtained from preliminary compaction tests on the soil-lime and soil-cement mixtures using the British Standard 4.5Kg rammer method (3). The values used are shown in Table 4.

4.3.2 Non-destructive strength tests. For the soil-cement programme of testing a series of non-destructive strength tests were carried out by measuring the time taken for ultrasonic pulses to pass through the length of the cylindrical specimens before they were tested in unconfined compression. The instrument used was known as the Portable Ultrasonic Non-Destructive Digital Indicating Testing (PUNDIT)(9). The pulse velocity (Km per second) was calculated from the ratio of the length of the specimen and the transit time as measured by the instrument.

4.4 Curing conditions

4.4.1 Main curing programme. A programme of different curing conditions to examine the effects of carbonation was carried out. This involved varying the humidity, temperature and level of carbon dioxide. Each of the curing conditions listed below was separately monitored for periods of 3 days, 7 days, 14 days and 28 days and applied to both the soil-lime and soil-cement mixtures.

(i) Normal curing - symbol NC: samples were either sealed in polythene bags or coated in paraffin wax to maintain them at constant moisture content and prevent contact with atmospheric carbon dioxide. The temperature was maintained at 20°C.

(ii) High humidity (HH): samples were unsealed and cured in an environmental chamber at 20°C and a relative humidity of 100 per cent.

(iii) Low humidity (LH): samples were unsealed and cured at 20°C in a desiccator containing silica gel.

(iv) High temperature (HT): samples were unsealed and cured at 40°C and a relative humdity of 60 per cent. The humidity was maintained constant by using a saturated solution of potassium chloride in the environmental chamber.

(v) High carbon dioxide (HC): samples were unsealed and cured at 20°C in a desiccator which was first evacuated and then restored to normal pressure by filling with industrial carbon dioxide.

Sufficient samples were prepared and cured under the above conditions for some to be tested immediately after the curing period and others to be subjected to a further four day period of immersion in water. Soaking was done to bring the samples to a similar moisture condition so as to determine whether any strength gains were the result of cementation and not from drying.

4.4.2 Double curing. This series of tests was designed to determine the effect of high temperature (HT) and a carbon dioxide environment (HC) on samples which had already hardened. Different regimes were used for the soil-lime and soil-cement mixtures as set out below.

(i) Soil lime: a) 7 days (NC) + 3 days (HC) + 4 days soaking
b) 3 days (NC) + 7 days (HT) + 4 days soaking

(ii) Soil cement: a) 56 days (NC) + 7 days (HC) + 4 days soaking
b) 56 days (NC) + 7 days (HT) + 4 days soaking.

4.4.3 Curing by water spraying. A series of tests was carried out on the soil-cement samples to simulate curing conditions often used in construction practice. Samples were not sealed but were sprayed with distilled water twice a day for seven days. Some samples were tested at this age and others were left exposed without spraying for a further 21 days before testing.

4.5 Carbonation penetration

Carbonation penetration was measured by spraying phenolphthalein indicator across the diameter of unsoaked samples after they had been tested in the unconfined compressive strength test. The extent of carbonation could be clearly identified by the boundary of the colour change and the penetration from the outside towards the centre of the sample was measured in millimetres (mm).

4.6 Plasticity tests

The changes in plasticity of soil-lime and soil-cement mixtures subjected to certain of the curing conditions were determined by either liquid and plastic limit tests or the linear shrinkage test (3). The curing regime before measuring the plasticity is shown below.

(i) Soil-lime: NC, HT, and HC for 7 days

(ii) Soil-cement: NC, HT and HC for 7 days and 28 days.

The plasticity tests were carried out on samples prepared after the unconfined compressive strength tests had been completed.

5. RESULTS AND DISCUSSION

5.1 Effect of different curing conditions

The results of the different curing treatments on the strengths of the soil-lime mixtures are given in Figs 4 and 5 and on the soil-cement mixtures in Figs 6 and 7. It was shown that:-

(i) Under the ideal curing conditions of constant temperature and moisture (NC) increases in strength occurred during the 28 days of the laboratory investigation for both the soil-lime and soil-cement samples. Unusually the soil-lime reactions were more rapid than those of the soil-cement and much higher early strengths were obtained even though only 3 per cent of lime was used compared with 4 per cent of cement. This effect has been observed before with some calcretes and may be a feature of these soils which requires further investigation. In this series of tests soaking had no adverse effect on strength and no carbonation was detected.

(ii) In a high carbon dioxide environment there was no increase in strength in either the soil-lime or soil-cement mixtures. The phenolphthalein test indicated that carbonation had penetrated throughout the samples even at the age of 3 days (see Figs 8 and 9). Table 5 compares the strengths of the carbonated samples (HC) with those normally cured (NC).

(iii)Samples which were left unsealed and cured in conditions of high and low humidity (HH and LH) and high temperature (HT) showed that the most significant loss of strength between unsoaked and soaked tests occurred with the high temperature samples. These samples as expected lost most moisture during curing and partial carbonation also occurred during this period.

5.2 Effect of double curing

The effect of first allowing the stabilised soil mixtures to harden under normal curing conditions (NC) and then exposing them to high temperature (HT) and high carbon dioxide (HC) conditions is shown in Tables 6 and 7 for soil-lime and soil-cement respectively. The soil-lime mixtures were only cured initially for 3 and 7 days compared with 56 days for the soil-cement mixtures. The high strengths obtained were subsequently reduced by the secondary treatments. The strength of the soil-lime mixtures reverted to values similar to those before any hardening had occurred (see Figs 4 and 5). The soil-cement mixtures did not lose strength to the same extent although they were only half of the strength of samples tested at the end of the initial curing. Complete carbonation occurred in samples subsequently exposed to the high carbon dioxide environment. Partial carbonation (up to 7mm) was observed in the samples exposed to higher temperature. The value of 7mm represents a penetration of about 1mm a day.

In this series of tests it has been shown that carbonation can clearly affect soil-lime and soil-cement mixtures which have already hardened. In the case of soil-cement however complete carbonation did not lead to a complete loss of strength. Carbonation only affects certain cementitious products and further research is needed to determine what happens with stabilised soils in practice.

5.3 Effect of curing treatment on plasticity

Comparisons of the effect on plasticity of high temperature (HT), high carbon dioxide (HC) and normal curing (NC) conditions for soil-lime and HC and NC for soil-cement are shown in Table 8. From the earlier results in Figs 4 - 9, it is clear that the cementitious reactions are most affected by carbonation brought about by exposure to high temperature and high carbon dioxide environments. Under these conditions some reduction in the original plasticity occurred but not to the same extent as that under normal curing.

Of further interest are the results of carbonation on soil-cement samples that had been cured for 56 days under normal conditions. These are shown in Table 7. Partial carbonation under high temperature conditions did not cause any change in plasticity but in the completely carbonated samples partial reversal of plasticity appears to have occurred. This could indicate that soil stabilisation for modifying soil properties could be affected by carbonation both in the initial stages just after adding stabiliser and in the longer term after high strengths have developed.

5.4 Effect of curing by water spraying on soil-cement samples

An additional series of tests were performed on soil-cement samples which were cured by spraying with water. Soaked tests on samples which had subsequently been exposed indicated that no permanent hardening had occurred under these conditions (see Table 9) and that the samples were vulnerable to carbonation penetration. These conditions were somewhat similar to the full-scale trial sections in Botswana where no cementation of the stabilised sections occurred.

5.5 Use of non-destructive methods of testing: measurement of pulse velocity.

Although measurement of ultrasonic pulse velocity has been used mainly in assessing the quality of concrete, results obtained using the Pundit in this project, showed that this technique can be applied to the assessment of the quality of stabilized soils.

As the quality of the material improves the velocity of ultrasonic pulses through the specimen increase. The deterioration of material is accompanied by a reduction of the velocity of ultrasonic pulses. The pulse velocity of carbonated specimens (HC) dropped with time from about 65% to 45% of that in the uncarbonated specimens (NC) (see Fig 10). The decrease in pulse velocity indicated the gradual deterioration through loss of cementation due to carbonation.

It was shown that pulse velocity correlates well with the strength of the stabilized specimens, at almost the same moisture content and bulk density. Fig 11 shows the strength - pulse velocity correlation for all the soaked soil-cement specimens tested. It is not known whether the relationship developed has general application since, as with concrete, the strength - pulse velocity correlation is likely to be affected by the type and grading of the soil, the cement content, the age of the material and curing conditions. Pulse velocity can therefore only be expressed as equivalent unconfined compressive strengths if a calibration is carried out on specimens made of a particular soil-cement mix and preferably soaked before testing.

6. CONCLUSIONS

The main conclusions from the study were:-

(i) Carbonation in which lime is converted to calcium carbonate in the presence of carbon dioxide affects the strength of both soil-cement and soil-lime mixtures. In the laboratory investigation a carbon dioxide environment led to carbonation which completely penetrated samples in less than three days and prevented cementitious products being formed. In a more natural environment in which the temperature and the humidity were varied carbonation also occurred. It was considered that the extent to which moisture was lost and hence air voids were increased, controlled the level of carbon dioxide that penetrated the soil. Only the sealed, normally-cured samples were unaffected.

(ii) Samples that were cured under well-sealed conditions to achieve high strengths subsequently carbonated under high temperature and high carbon dioxide environments. This implies that carbonation can have a long term affect on pavement layers that have already been successfully stabilised. Further studies are required to determine whether loss of strength in this way will lead to failure. Some field studies have shown that a road base can be fully carbonated without showing signs of physical distress.

(iii) Soil treatment especially with lime is used to modify soil properties by reducing or eliminating plasticity. The investigation showed that modification can be affected by carbonation and that the rate is affected by environmental conditions. Plasticity can be partially reversed when an already modified soil is exposed to carbonation.

(iv) In the laboratory tests spraying with water at intervals to simulate one curing technique which is often used in the tropics was not effective in preventing carbonation. Strengths gained during seven days of this treatment were drastically reduced when followed by four days immersion in water.

(v) Soil lime reactions were more rapid than those of soil-cement and higher strengths were achieved. Similar results have been obtained with other calcretes and further investigations need to be carried out to determine whether this affects construction practice.

(vi) A non-destructive test method of measuring the strength of soil-cement mixtures by pulse-velocity was a useful means of following strength-age behaviour of laboratory samples.

(vii) The laboratory investigation and field evidence from Botswana and neighbouring countries clearly indicates that carbonation can affect stabilised soils. Further information is needed on the occurrence of carbonation and how this relates to the performance of stabilised layers and climatic environments. A better understanding is also required of the mechanism of carbonation especially in the way that it affects cemented products.

7. ACKNOWLEDGEMENTS

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TABLE 1

Laboratory tests on stabilised samples taken after field mixing

Material description	Curing period (days)	Moisture content (%)	BS 4.5 kg rammer test	
			Dry density (Kg/m^3)	CBR (%)
Calcrete and 3% cement	7	9.3	1803	117
		9.8	1814	122
	28	8.1	1742	135
		8.2	1849	194
Calcrete and 3% hydrated lime	7	8.5	1751	65
		10.5	1736	126
	28	7.6	1686	124
		9.8	1741	197

TABLE 2

Plasticity characteristics of the stabilised materials used in the road trials

Material description	Before field mixing			After mixing and compaction			After 3 years in the road			After 4 years in the road		
	LL	PI	LS	LL	PI	LS	LL	PI	LS	LL	PI	LS
Calcrete and 3% cement	41	17	7.6	28	4	2.1	23	5	2.5	27	6	3.6
Calcrete and 3% lime	41	17	7.6	29	5	2.1	26	10	4.1	27	8	2.1

TABLE 3

Properties of the soil

Liquid limit - per cent	39
Plastic limit - per cent	19
Plasticity index	20
Linear shrinkage - per cent	7.5
Sieve analysis: less than 2 mm (per cent)	84
less than 0.425 mm (per cent)	77
less than 0.063 mm (per cent)	18
Specific gravity	2.66
Calcium carbonate content, per cent	16
pH	8.0
BSSC classification	S C.

TABLE 4

Dry density and moisture content values used for the unconfined compressive strength tests

Mixture	Dry density - Mg/m^3	Moisture content-per cent
Soil only	1.94	12.0
Soil + 3% lime	1.85	14.0
Soil + 4% cement	1.87	13.5

TABLE 5

Comparison of per cent loss of strength on carbonation in the soil-cement and soil-lime mixtures

Age (day)	4% cement stabilized calcrete soaked strength (MN/m^2)			3% lime stabilized calcrete soaked strength (MN/m^2)		
	NC*	HC*	HC*/NC*	NC	HC	HC/NC
3	1.76	0.74	42%	4.06	0.96	24%
7	1.85	0.65	35%	4.40	1.02	23%
14	2.67	0.81	30%	5.15	1.34	26%
28	3.10	0.71	23%	4.87	1.34	28%

* In this series another but similar calcrete was used for the cement-stabilised samples

TABLE 6

Results of the double-cured soil-lime samples

Curing conditions and age	Unsoaked strength MN/m^2	Soaked strength MN/m^2
NC 7 days	3.6	4.40
NC 7 days + HC 3 days	-	1.27
NC 3 days	2.33	4.07
NC 3 days + HT 7 days	-	1.01

TABLE 7

Results of the double-cured soil-cement samples

Curing conditions and age	Unsoaked compressive Strength (MN/m^2)	Soaked compressive Strength (MN/m^2)	Carbonation penetration (mm)	Linear shrinkage (%)
NC 56 days	4.48	3.47	0	0
NC 56 days + HT 7 days	4.15	1.79	7	0
NC 56 days + HC 7 days	2.12	1.72	25	2

TABLE 8

Effect of curing treatment on plasticity

Material description	Curing conditions and age	Liquid limit per cent	Plastic limit per cent	Linear shrinkage per cent	Plasticity index
Untreated calcrete	-	39	19	7.5	20
Soil + 3% lime	NC 7 days	33	-	2	4
Soil + 3% lime	HT 7 days	-	-	3.3	7
Soil + 3% lime	HC 7 days	33	18	-	15
Untreated calcrete*	-	29	20	3	9
Soil + 4% cement*	NC 7 days	29	-	0	NP
Soil + 4% cement*	HC 7 days	30	-	2	4

* For this series of test a different sample of calcrete was used for the soil-cement tests.

TABLE 9

Effect of curing by water spraying and exposure on soil-cement samples

Length of curing period (days)	Length of exposure period (days)	Test condition	Unconfined compressive strength (MN/m^2)	Carbonation penetration (mm)
7	0	Unsoaked	2.65	6
7	0	Soaked for 4 days	0.68	*
7	21	Unsoaked	3.13	15
7	21	Soaked for 4 days	0.58	*

* Carbonation penetration of soaked samples could not be measured because of the diffusion of the stabiliser.

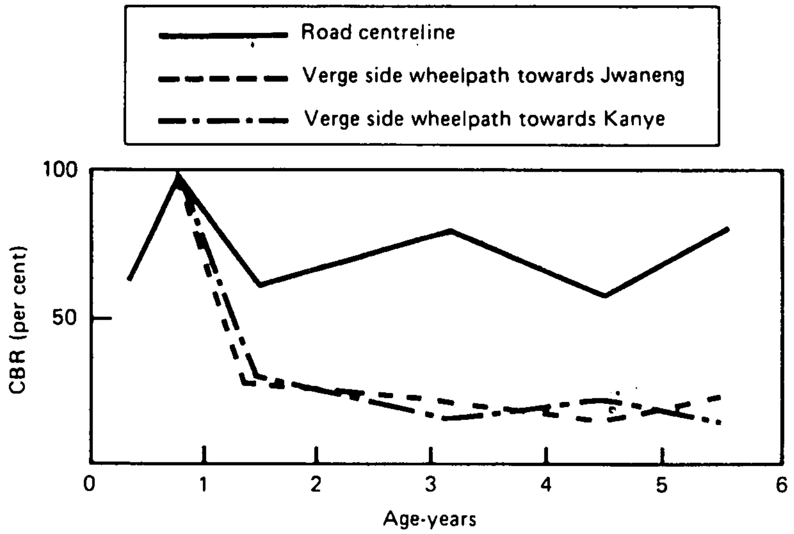

Fig.1 In-situ CBR's of untreated calcrete base

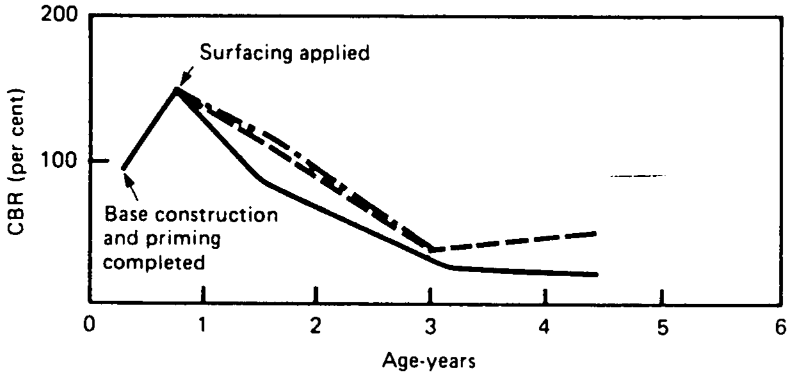

Fig.2 In-situ CBR's of cement-treated calcrete base

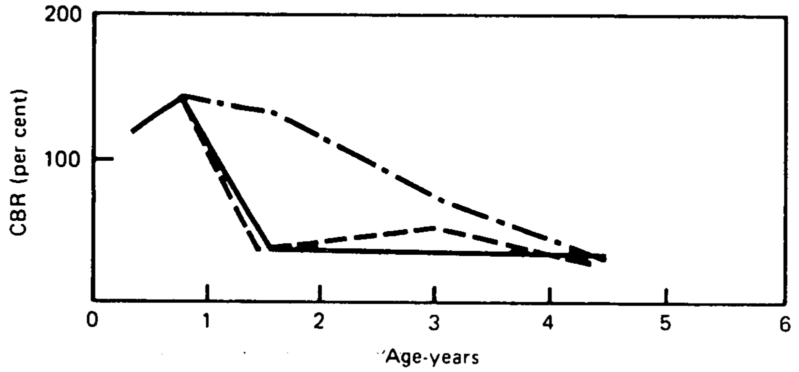

Fig.3 In-situ CBR's of lime-treated calcrete base

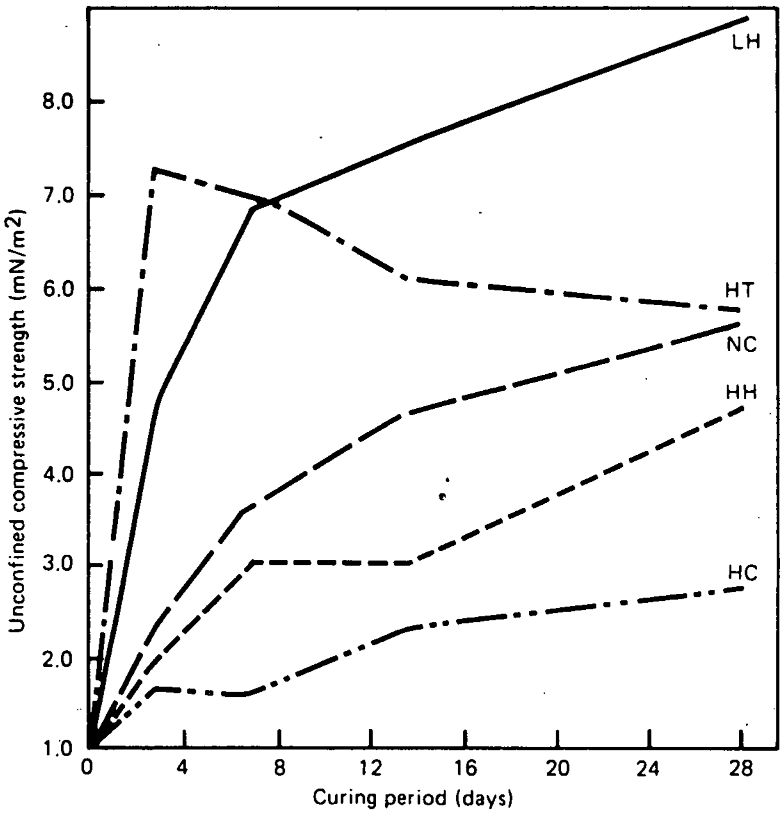

Fig.4 Soil-lime mixtures: strength-age relations for unsoaked samples

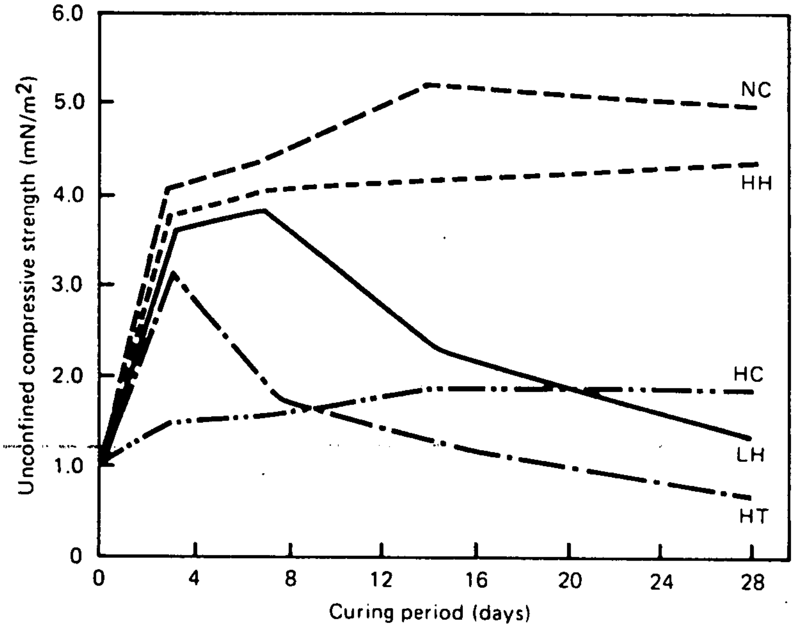

Fig.5 Soil-lime mixtures: strength-age relations for soaked samples

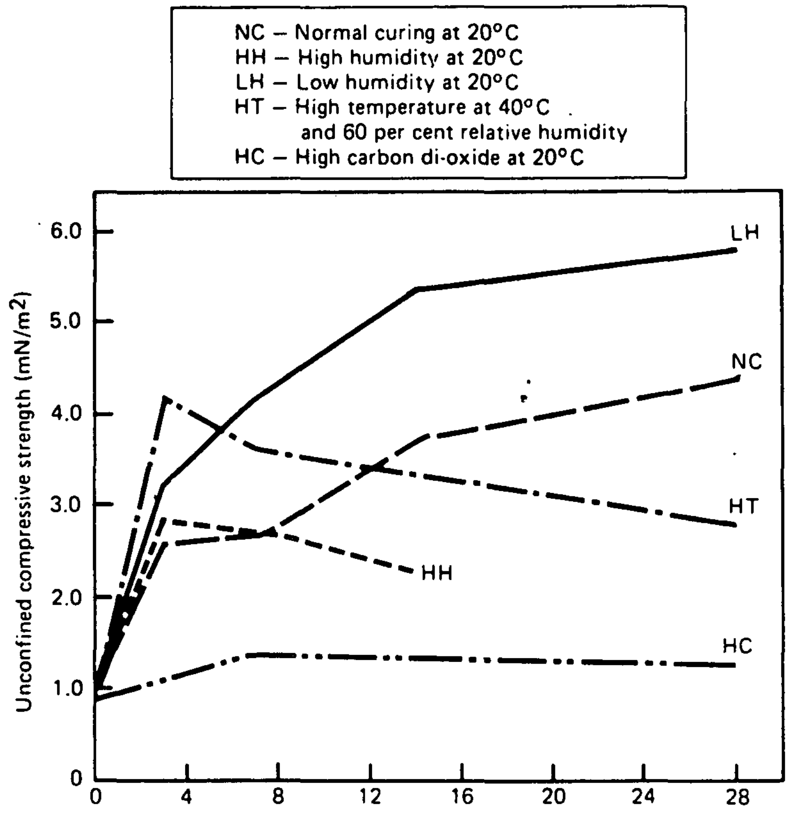

Fig.6 Soil-cement mixtures: strength-age relations for unsoaked samples

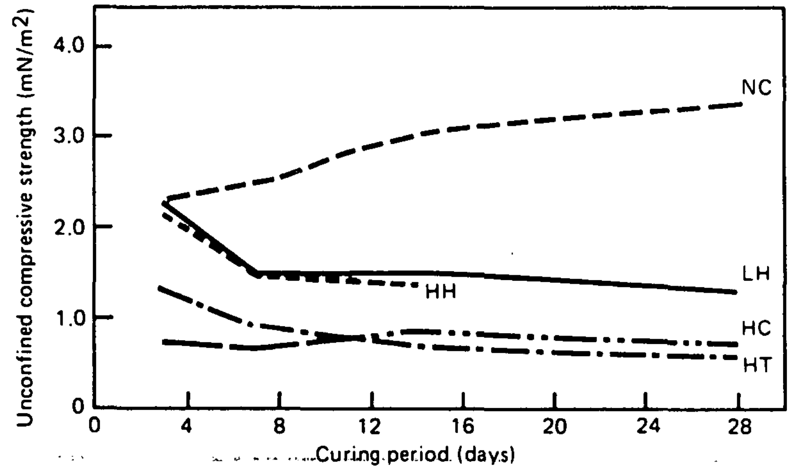

Fig.7 Soil-cement mixtures: strength-age relations for soaked samples

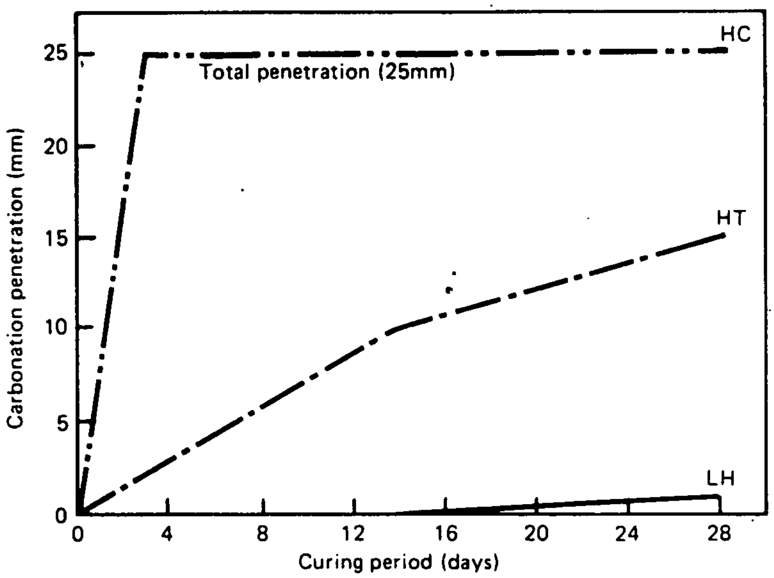

Fig.8 Soil-lime mixtures: carbonation penetration with time

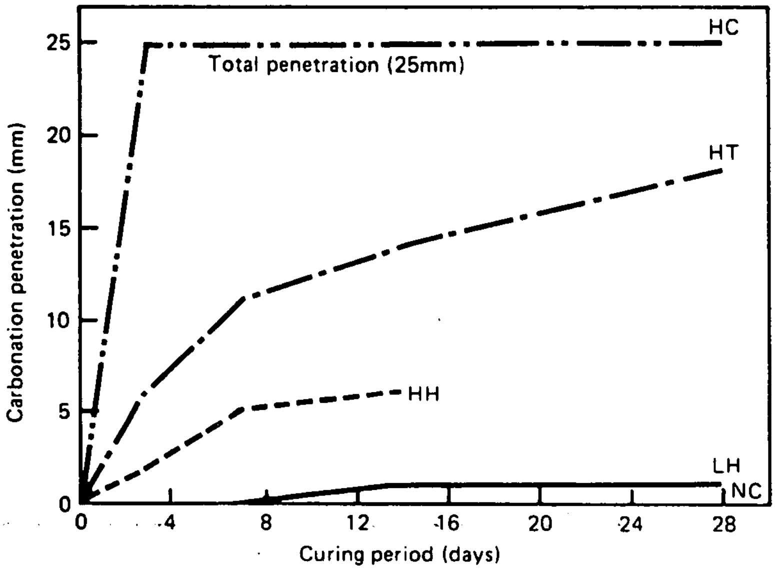

Fig.9 Soil-cement mixtures: carbonation penetration with time

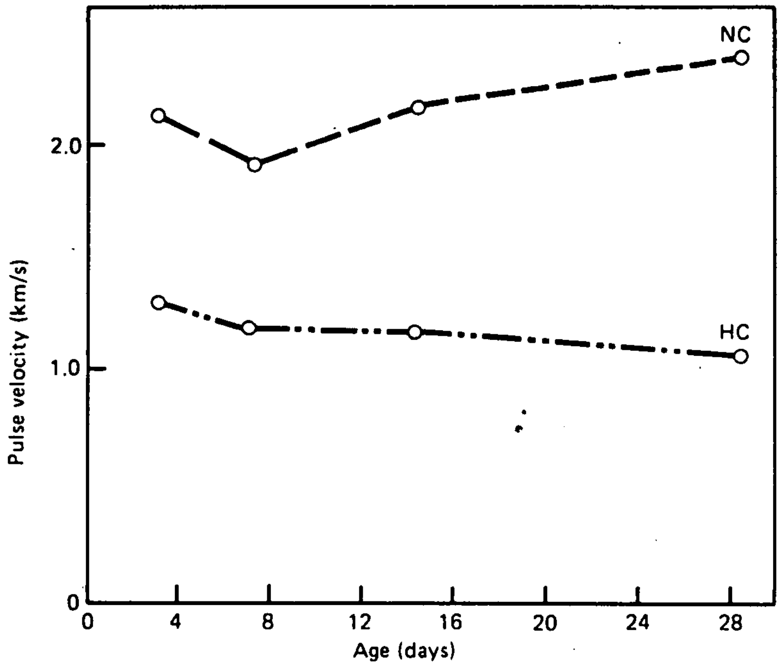

Fig.10 Pulve velocity-age relations for the soil-cement samples after curing and 4 days soaking in water

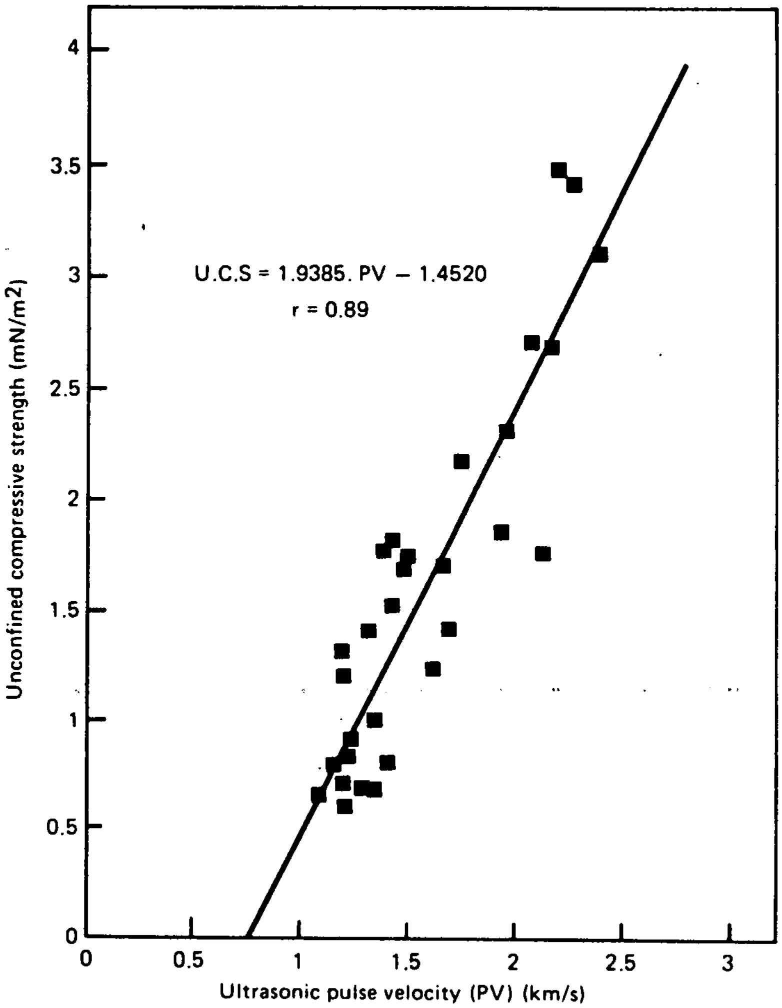

Fig.11 Strength-pulse velocity relationship for soaked stabilized specimens