

THE CARBONATION OF CHEMICALLY STABILISED ROAD CONSTRUCTION
MATERIALS: GUIDE TO ITS IDENTIFICATION AND TREATMENT

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Synopsis

The carbonation of pavement layers stabilised with lime, cement, slagment and combinations of these has become a significant problem in recent years, especially in roads constructed of marginal quality materials. This guide discusses the causes and effects of carbonation on the stabilised layers and summarises means of identifying potential carbonation situations. New techniques for assessing the durability of stabilised materials in the laboratory are discussed and recommendations on the prevention and rehabilitation of damage to pavements caused by carbonation are provided.

Sinopsis

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1 INTRODUCTION

Most pavements incorporating layers stabilised with lime, cement, slagment or combinations of these have performed satisfactorily in southern Africa over the years. However, a number of investigations into problems associated with road construction materials which had been chemically stabilised with these inorganic stabilisers had been carried out by the then National Institute for Transport and Road Research (NITRR) prior to 1982. Most of the problems were associated with the use of marginal materials (usually weathered natural gravels) and were manifested as flaking, blistering or scabbing of the prime, loss of strength in the surface layers, a return of the plasticity index or movement of the surfacing relative to the base course.

At the time of many of these investigations, the causes were mainly attributed to various aspects such as poor construction, poor materials, overcompaction, possible reaction of the stabiliser with the prime, poor lime quality, the influence of soluble salts and numerous others.

During 1982, the NITRR was requested to investigate extensive failures of a two year old road in Namibia (then South West Africa), constructed with lime stabilised calcareous sand base and subbase, sealed with a South West African "Cape seal". A similar problem to those described previously was observed with arcuate cracking of the seal, obvious signs of movement of the seal and extensive patching in the outer wheel tracks. Beneath the intact seal a loose layer of calcareous sand occurred above the relatively hard stabilised material. The stabilised material was mainly a calcified sand with varying proportions of hard calcrete gravel. In addition, weakening of the top and bottom of the subbase and in places the total subbase was observed. The findings from this investigation together with a number of follow-up investigations in other areas shortly afterwards indicated that carbonation of the stabilised layer was the primary cause of the problem.

This report discusses the mechanisms and chemistry of stabilisation reactions, the physical effects of stabilisation with inorganic chemicals, the effects of carbonation on stabilised layers and means of identifying and overcoming potential carbonation problems. It should not be taken as a design manual for stabilised layers but is a guide to assist with avoiding the problems associated with carbonation of stabilised layers during and after construction. This guide is based partly on a National Institute for Transport and Road Research internal report by Netterberg (1987) which had a somewhat wider scope including aggregate durability and the influence of salts and acids.

2 SCOPE OF THE PROBLEM

Contrary to what has often been stated in the literature, stabilisation is not always permanent, in spite of many examples of such permanence being cited. Some of the reactions involved are reversible and the reaction products are only stable under certain conditions. With time, even concrete can be destroyed by acids (including carbonic acid), salts and leaching (Addis, 1986).

Distress or failure due to loss of stabilisation has occurred in at least 100 cases in southern Africa over the last 30 years (Table 1). This represents an average rate of about three per year. The most common form of distress is surface disintegration of the primed base during construction and scabbing of the seal in service due to inadequate bond with the base. In many cases a loose layer of disintegrated base course material was noticed between the surfacing and the base. A less frequent occurrence has been a partial or even complete loss of cementation and a large decrease in strength, leading to rutting, cracking and shearing or pumping. In a few cases an increase in the plasticity index has been found. Lime, cement and lime-slag were used as stabilisers, in amounts ranging between 2 and 5 per cent by mass. Carbonation has been confirmed or is strongly indicated to have been a factor in about half of the cases. In the remainder of cases the available information does not permit an assessment of whether or not carbonation was involved.

Loss of stabilisation or the presence of carbonation does not necessarily lead to distress (Table 2). However, comparison of Table 2 with Table 1 indicates that distress upon loss of stabilisation or carbonation is more likely than not (100 cases out of 119 and 44 cases out of 64 respectively). Possible reasons for a lack of distress after loss of stabilisation or after carbonation include light or even no traffic (in some cases stabilisation was probably unnecessary), abnormally high stabiliser contents, durable aggregate and possibly even a more durable cement. A most important factor appears to be the quality of material before stabilisation: the poorer it is the more likely carbonation and destabilisation is likely to lead to distress.

Cases of surface weakening or disintegration, loose layers under the seal and/or general loss of stabilisation have also been reported from Australia, Britain and the United States. They have variously been attributed to frost, poor curing and poor mixing. Only the National Lime Association (in Alexander, 1976) and the FHWA guide (1979) have attributed some of them to carbonation. However, with the carbonation experience gained recently it is considered that carbonation may have been an unrecognised factor in many of these cases.

Table 1: Known cases of loss of stabilisation in southern Africa (1957-1986) which have led to distress⁽¹⁾

Area	Lime	Cement	Lime-slag	Loose prime &/or surfacing scabbing	General loss of strength	Return of PI	Carbonation ⁽²⁾
Cape	1	1	1	3	1		1
Natal	10	2		7	7	1	8
OFS	1	2	2	10	1	1	3
SWA	11			9	2	1	3
Tvl	3	9	6	19	9	2	13
Total	26	14	9	48	20	5	28
Botswana	3	2		4	3	1	6
Lesotho	1			2			1
Malawi	2	7		9	8		7
Swaziland	1		1	2			1
Zambia		1		1	2?		2?
Zimbabwe	1	1		3			1
Total	8	11	1	21	11	1	16
Grand Total	34	25	10	69	31	6	44

- (1) Compiled from DRTT records, discussions, and a survey of stabilisation durability in South Africa. A "case" may be an airport or length of road from about 800 m to over 20 km.
- (2) Confirmed by a decrease in pH to 8-9 and an increase in CaCO₃ content and similar symptoms to known cases of distress associated with carbonation.

Table 2: Known cases of loss of stabilisation, or carbonation, in southern Africa which have not led to distress⁽¹⁾

Area	Cement	Lime	Lime-slag	Loss of strength	Return of PI	Carbonation confirmed ⁽²⁾
Cape	3					3
Natal						1
OFS	3	1		2	1	4
SWA	1	3		2	1	4
Tvl	1		1	3		2
Total	8	4	1	7	2	14
Botswana		1				1
Lesotho		1		1		1
Malawi	2	1		3		4
Swaziland			1	1		1
Total	2	3	1	5		7
Grand total	10	7	2	12	2	21

- (1) Compiled from DRTT records, discussions, and a survey of stabilisation durability in South Africa. A "case" may be an airport or length of road from about 800 m to over 20 km.
- (2) Notable cases in which the base appeared to be totally carbonated according to the phenolphthalein test, but which were still hard and undistressed after 40 years include the Fisantekraal and Langebaanweg airfields. Construction test data for these have been given by Biesenbach (1973).

3 DEFINITIONS

Durable: capable of lasting, resisting wear (Oxford dictionary). This includes resistance to moisture absorption, strength reduction, freezing and thawing, and wetting and drying (Ballantine & Rossouw, 1972). In the case of concrete, durability is defined as the ability of concrete to retain its strength, impermeability, dimensional stability and appearance over a prolonged period of service under the conditions for which it was designed (Addis, 1986). It is suggested that the durability of stabilised materials for roads can be similarly defined with the omission of "appearance" (this ignores the problem of cracking).

Stabilise: increase strength and/or reduce plasticity. This includes modification and cementation. Some authorities, eg McDowell (1972), imply that stabilisation is permanent but that modification may be temporary.

Some materials (eg some laterites (Osula, 1989) and other soils (Thompson, 1966) often containing certain types of organic matter) cannot be easily stabilised with lime or cement: this problem is not dealt with in this report.

The strength of the stabilised material and the durability of stabilisation are not synonymous and the correlation between the unconfined compressive strength (UCS) and durability is not good (Figure 1). For example, according to United States experience (Portland Cement Association, 1971) (Figure 2), a material with a Proctor UCS of 2,25 MPa (the equivalent minimum for our C2 material) only has a 20 per cent chance of satisfying the PCA freeze/thaw and wet/dry durability requirements. A limited number of results of local tests plotted on this figure support this observation. As only 97 or 98 per cent Mod AASHO is usually specified in southern Africa (NITRR, 1986a) and, according to TRH 14 (NITRR, 1985b), the strength at 97 per cent Mod AASHO is two-thirds of that at 100 per cent, the South African C2 material at 2-4 MPa in place may only have between a 5 and 50 per cent chance of passing the PCA criteria. Our commonly-used C3 and C4 materials have little or no chance at all. Nor is the matter improved significantly if the grading is also considered (Leadabrand and Norling, 1953). However, a correlation between grading and density versus UCS exists and has been used in the PCA "short-cut" procedure for sandy materials (PCA, 1971). For materials with non-durable aggregates the position will be even worse. As has been pointed out by Ballantine and Rossouw (1972), if insufficient lime is added for complete modification (ie to satisfy the initial consumption of lime (ICL)) the imperfectly modified material may fail

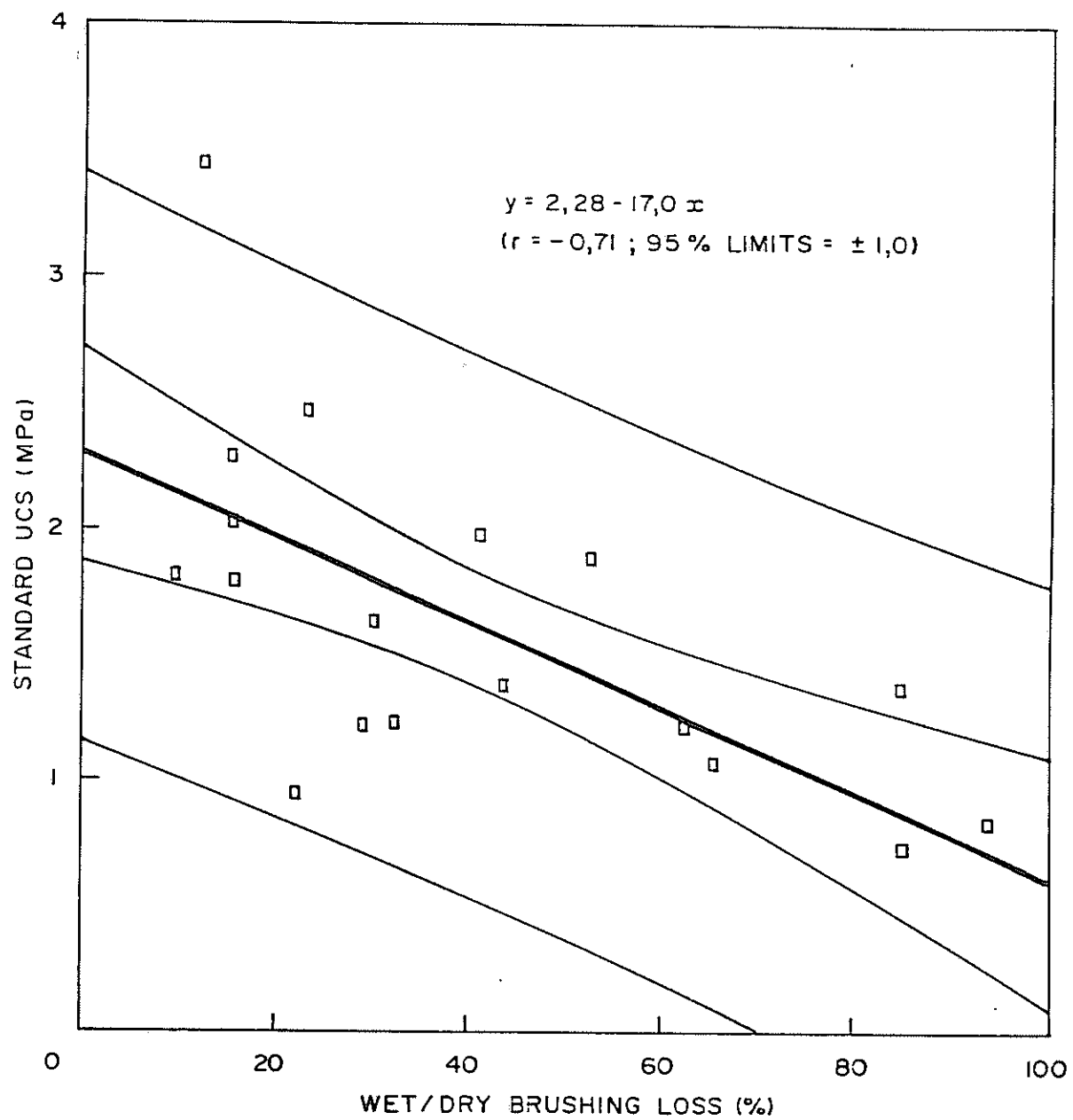


FIGURE I

REGRESSION ANALYSIS OF STANDARD UCS WITH WET/
DRY HAND-BRUSHING LOSS (97 % MOD. AASTO COMPACTION)

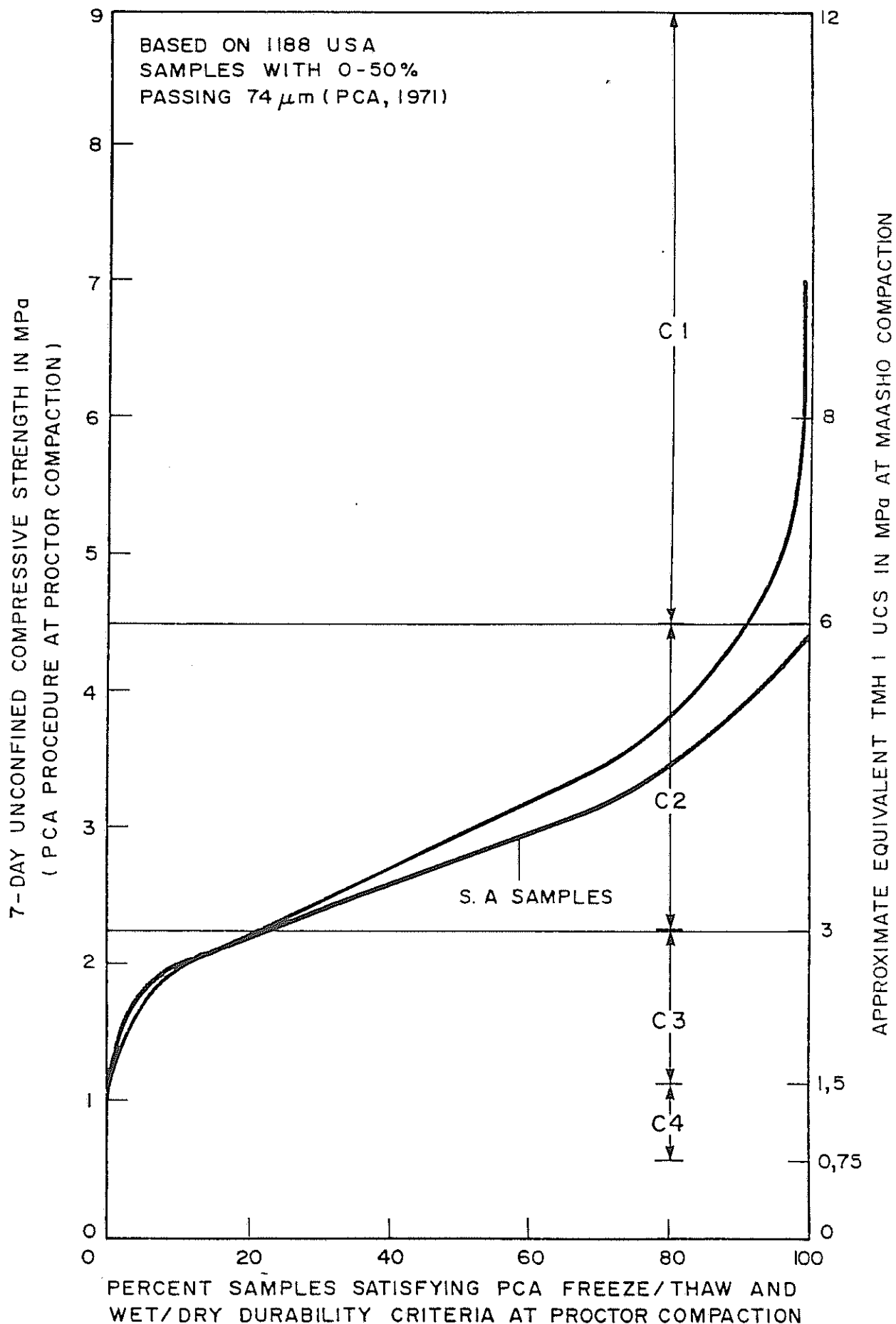


FIGURE 2

STRENGTH AND DURABILITY OF UNITED STATES CEMENT
STABILIZED SOILS IN RELATION TO TRH 14 (1985) REQUIREMENTS
AT 100 % MAASHO DENSITY.

in service despite other requirements such as plasticity index (PI) and California Bearing Ratio (CBR) having been met.

As compaction procedure, specimen density and specimen dimensions used by the PCA to prepare these figures differ from those in TMH 1 (NITRR, 1986b) the above assessment may not be very accurate. However, it serves to illustrate the point that the unconfined compressive strength and durability are not synonymous. It also implies that the strength of materials not meeting the durability criteria is not necessarily permanent.

Both the durability of the coarse and fine aggregate and that of the stabiliser and its reaction products must be considered. This report concentrates on the influences of carbonation and does not deal in any depth with the importance of aggregate durability. However, it is important to stress that the aggregate durability is extremely important and should always be considered. Any disintegration of coarse aggregate will expose new faces of untreated material which require additional stabiliser for neutralisation of the plasticity. The wisdom of carrying out an ICL test on a pulverised sample (NITRR, 1984) is thus confirmed. The durability limits in Sampson (1990) should be met for materials which are to be modified with no relaxation permissible.

The method of selecting the optimum stabiliser content has in the past concentrated on providing sufficient stabiliser to ensure that the design 7 day cured unconfined compressive strength is achieved. Although the standard method for determining this is not discussed a more realistic method based on durability and not strength is included in this guide.

4 WHAT IS CHEMICAL STABILISATION?

Chemical stabilisation is the process by which the addition of a chemical stabilising agent results in a chemical reaction which changes the surface molecular properties of the particles or cements the particles together.

Although the overall chemical stabilisation process is similar for lime and cement, the mechanism of reaction is generally considered to be different for the two types of stabilisation.

The composition of lime produced for road stabilisation is closely specified and controlled, but lime will not react without a suitable silicate, aluminate, amorphous or other pozzolanic component being available in the material being treated. This latter component is totally

uncontrolled in typical stabilisation projects. Lime stabilisation is considered to consist mainly of ion exchange, flocculation and agglomeration (Kézdi, 1979), although it would appear from local research that other mechanisms may be at least equally important. Laboratory testing of calcium-saturated soils has produced a significant increase in strength on the addition of lime. Ion exchange in these cases would theoretically be minimal. The effect of the highly alkaline lime solution on the clay platelets should not, however, be underestimated. It is well known that the edges of these plates may be attacked by the alkalinity and the ionic balance of the platelet disrupted (Gillott, 1987). This disruption would result in significant restructuring of the platelets and an overall change in the soil structure. In addition the alumina and silica components of the clay minerals will be more readily dissolved at the higher pH values and may react with the calcium ions in the lime to form hydrated calcium silicates, similar to the cement reactions. The ion exchange and flocculation/aggregation reactions occur relatively quickly whilst the pozzolanic reactions are fairly slow.

Even the reaction processes and products of cement, which is of a known and controlled composition, is not fully understood. Cement stabilisation resembles lime stabilisation in many ways, the major difference being that the pozzolanic component is an integral part of the cement and control of the reaction is thus better. It is generally hypothesised that hydration of the calcium silicates and aluminates, which are the primary components of the cement, form cementing agents.

Effectively, the stabilisation process consists of the reaction of the lime, silicate and aluminate components of the lime or cement and soil combination in an aqueous environment. These reactions are discussed fully in concrete and stabilisation manuals such as Lea (1970), Transportation Research Board (1976), Kézdi (1979), Bensted (1983) and Addis (1986).

For adequate stabilisation using lime, sufficient lime should be added to ensure an excess after the reactions are complete ie the Initial Consumption of Lime (ICL) (NITRR, 1984) of the soil should be satisfied and an excess provided. During stabilisation with cement, on the other hand, lime is produced as a by-product of the reaction. It is essential that this lime remains in the stabilised material in order to maintain the pH of the material at a level of at least 10 and preferably greater than 11. This is necessary to ensure the stability of the reaction products.

In the interest of simplicity, the term lime is often used in this report, although it should be read in the context that all the traditional chemical stabilisers (lime, cement, granulated blast-furnace slag, flyash, and combinations of these) do, to all intent and purposes, behave in a similar manner. All of these stabilisers rely on a pozzolanic reaction and/or ion exchange for their effectiveness. Asphalt stabilisation on the other hand is not dealt with in this report as it is not a truly chemical stabilisation method. No reversible chemical reaction occurs between the aggregate and the asphalt. The strength of the asphalt stabilised material is increased by a flexible particle to particle bond with asphalt forming the bond.

5 THE CARBONATION PROBLEM

5.1 Background

The carbonation of lime during stabilisation has been recognised for many years and in 1953 Borisova (1953) in fact suggested that the mechanical strength of soil/lime mixtures was derived from the carbonation of the calcium hydroxide. This theory was disproved in 1957 (Clare and Crutchley, 1957) when samples stored in air-tight containers produced strength increases. Eades et al (1962) in a full-scale experiment to investigate the reaction products in lime stabilised soil noted 2,5 per cent calcium carbonate in the material which was originally treated with 5 per cent lime 3 to 4 years earlier. The cementing products were identified as calcium-silicate-hydrates but they stated that "it would be interesting to know how quickly the carbonate formed".

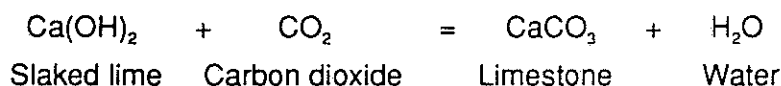
Diamond and Kinter (1965) discussed the work by Eades et al (1962) and concluded that carbonation was deleterious rather than helpful. During later work on soil stabilisation, researchers both overseas (Terrell et al, 1979) and locally (Ballantine and Rossouw, 1972) briefly note the possibility of carbonation occurring without providing much detail. Terrell et al (1979) note that carbonation is an undesirable reaction and construction should be carried out in such a fashion that lime carbonation is minimised. Sprinkling of water to maintain a moist condition often causes carbonation of the top 6 to 25 mm (Terrell et al, 1979) and the layer should be removed prior to construction of the next layer or surfacing. This layer may in fact be weakly cemented by calcium carbonate. Membrane curing was recommended by Terrell et al (1979). Ballantine and Rossouw (1972) on the other hand note that lime may become carbonated during storage and stabilised layers should be sealed as soon as possible after spreading.

A number of instances of apparently untreated material beneath the surfacing on lime stabilised roads have been reported from California (Alexander, 1976). These layers were attributed to the leaching of lime during water spraying for curing. A similar case was reported from Missouri (Thomas et al, 1965). Though the problems were attributed to leaching, in retrospect, carbonation was probably the cause. A representative of the National Lime Association in the United States (Alexander, 1976) recorded similar situations and attributed them to carbonation of the lime or flushing of the lime by water application.

Only cursory remarks are made about carbonation in such important works on soil stabilisation as those by Ingles and Metcalf (1972), Kézdi (1979) and Ballantine and Rossouw (1972; 1989). Generally, other than the work carried out locally (Netterberg and Paige-Green, 1984; Paige-Green, 1984; de Wet and Taute, 1985), very little existed in the literature on the effects and implications of carbonation in stabilised roads prior to about 1985.

5.2 Processes

Lime and to a lesser extent Portland cement are unstable under normal environmental conditions and carbonate readily under the right conditions. For example, lime (Ca(OH)_2) is only stable at a partial pressure of CO_2 (P_{CO_2}) of less than 3×10^{-14} atmospheres. Under normal conditions such as in atmospheric air (P_{CO_2} of 3×10^{-4} atm. or 0.03 % by volume), CaCO_3 is the stable phase (Table 3). The same should apply to cement, although some of the compounds present are stable at somewhat lower pHs, and therefore higher P_{CO_2} s of up to about 10^{-9} atmospheres. It is well accepted in concrete technology that complete carbonation of Portland cement is chemically possible even at the low concentration of carbon dioxide in the atmosphere. Very broadly, the reactions involve a reversal of those which took place during their manufacture. For example, lime simply reverts to the limestone from which it was made:



Cement similarly reverts to the original components from which it was made or similar combinations:

Table 3: pH of the system $\text{CaO-H}_2\text{O-CO}_2$ at 25 °C

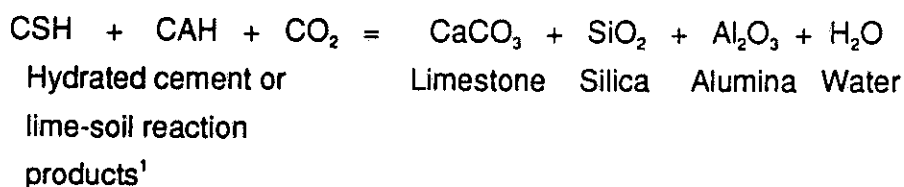
CO ₂ in air ⁽¹⁾		Dissolved		CaCO ₃		Remarks
P (atm.)	% v/v	gaseous		Excess ⁽²⁾ Absent ⁽⁴⁾		
		CO ₂ ⁽²⁾ mg/l ³	Dissolved CaCO ₃	pH	pH	
≤ 3 x 10 ⁻¹⁴	< 3 x 10 ⁻¹²	< 4.5 x 10 ⁻¹¹	-	-	12,4	Only Ca(OH) ₂ stable
3 x 10 ⁻⁷	3 x 10 ⁻⁵	4,5 x 10 ⁻⁴	13	10,0	7,3 min	CaCO ₃ solubility point
3 x 10 ⁻⁵	0,003	0,045	24	9,0	6,3 max	CO ₂ usually found in uncarbonated stabilised material
3 x 10 ⁻⁴	0,03	0,45	52	8,3	5,7	Atmospheric air
3 x 10 ⁻³	0,3	4,5	115	7,8	5,2	Well aerated soil
3 x 10 ⁻²	3	45	240	7,1	4,7	Poorly aerated soil
0,1	10	151	380	6,8	4,5	
0,14	14	212	430	6,7	4,3	Max. CO ₂ measured under pavement
1,0	100	1514	900	6,1	4,0	Pure CO ₂

(1) With which the system is in equilibrium.

(2) Solubility of CO₂ in pure water (ie CaCO₃ absent) or that required to stabilise the solution if saturated with CaCO₃.

(3) Pure water saturated with CaCO₃.

(4) pH of pure water saturated with CO₂.



The "limestone" can be either in the form of calcite or aragonite or both (Lea, 1970; Bensted, 1983). The change from lime (Ca(OH)₂) to calcite or aragonite is associated with a volume increase of about 10 and 3 per cent respectively and hydrated calcium silicates with an unquantified shrinkage (Lea, 1970). The alumina is usually in the form of a gel or Gibbsite mineral.

¹C = CaO, S = SiO₂, A = Al₂O₃, H = H₂O. However, the actual compounds present vary.

It is clear from Boynton (1980) that the problem of recarbonation of lime, especially quicklime, at all stages of its manufacture, storage, sampling, and testing is well appreciated by the industry and the American Society for Testing Materials (ASTM). What is new is that the same problem exists during construction and in service. Little et al (1987) note that carbonation of soil-lime mixtures is undesirable and construction should be carried out in such a manner that carbonation is minimised. Little elaboration of this is found in the text.

The hydrated calcium silicates (CSH) and aluminates (CAH) formed in lime-soil mixes are broadly similar to those in hydrated portland cement. A minimum pH of about 11,0 is necessary to initiate significant attack on clay minerals and is also the minimum pH at which C_1S_1H is stable. Only about 0,005 per cent $Ca(OH)_2$ dissolved in the pore water is required to give this pH. Work by the Transport and Road Research Laboratory (Sherwood, 1968) showed clearly that significant strengths (of more than about 1 MPa at 7 days) could not be attained with soils treated with 10 per cent cement unless the pH at 1 hour was at least 12,1. Strengths of up to 5 MPa were measured at a pH of 12,1 or more and up to 7 MPa at 12,4 or more. Most of the CSH in hydrated cement probably consists of $C_3S_2H_3$ (Addis, 1986) which contributes most of the strength of concrete. This mineral is only stable at a pH of 12,5 or more (Moskvin et al, 1983). A more or less saturated solution of $Ca(OH)_2$ (ie 0,16 per cent at 25 °C) is required for this.

Although nominally "insoluble" when placed in water CSH and CAH decompose to a small extent by hydrolysis and produce just enough $Ca(OH)_2$ in solution to prevent further decomposition. A certain concentration of $Ca(OH)_2$ dissolved in the pore water is therefore necessary to maintain the stability of the reaction products and this is reflected in a high pH, which is more convenient to measure. It follows therefore that if for any reason the concentration of dissolved $Ca(OH)_2$ (and hence the pH of the environment) is continually maintained below about 12,5 all the $C_3S_2H_3$ will eventually decompose to form silica gel (Goodbrake et al, 1979a). As the solubility of CSH and CAH at high pHs is very low, this is a slow process. In the case of concrete the usual causes of a reduction in pH are leaching, carbonation and sulphation (Addis, 1986). It is mainly the impermeable nature of good concrete that contributes to its durability (S H Diamond, pers. comm.) Carbonation of concrete is usually only a problem in the short term if floors are cured under conditions of higher than normal CO_2 and, in the long term, in reinforced concrete when the depth of carbonation reaches the steel (in the absence of chlorides it is depassivated at a pH below 10,6 (Addis, 1986)). Carbonation increases the density and strength of concrete made with OPC but decreases them in the case of other cements (Moskvin et al, 1983). Artificial carbonation of neat lime putties and lime-aggregate mixes under optimum conditions by the

Zalmanoff process (very high CO_2 content, pressure, temperature, moisture content, etc) has produced strengths of 35-70 MPa in a matter of hours (in comparison with 0,7-1,4 MPa by atmospheric carbonation in years (Boynton, 1980). However, it is clear that it is almost always detrimental in soil stabilisation. Although the reason for this is not clear, the poorer aggregate, higher clay content, lower stabiliser content and probably higher permeability of stabilised soils are obvious differences.

Although the carbonation of concrete, including bridges, has recently become of concern in Europe there does not appear to be any recorded case of a carbonation-induced problem with reinforced concrete in South Africa. However, the same was said of alkali-aggregate reaction only a few years ago. One local case is known in which carbonation may have contributed to the collapse of a no-fines concrete drain on the Estcourt-Frere section of the N3 (Sampson, 1985). This supports the conclusion of Bensted (1983) that carbonation of concrete only achieves significance if the permeability or porosity is high.

The development of strength by pure lime-sand mortars and the permanence of some such ancient structures has been cited as evidence against the negative effects of carbonation on stabilised permanent layers. However, such mortars develop strength by carbonation very slowly to a maximum of 0,7-1,4 MPa after many years and usually even then are incompletely carbonated (Boynton, 1980). Their final strength is only 2,5-3 per cent of that of cement mortars.

For comparison, a 1:3 non-hydraulic lime:sand mortar at 28 days would only possess a strength of 0,4-0,5 MPa as against 28 MPa for a 1:3 portland cement:sand mix (Boynton, 1980). Even allowing for the fact that on a mass basis the lime mortar would only contain about 15 per cent lime as against about 30 per cent cement in the case of the cement mortar it is clear that any strength afforded by carbonation is negligible in comparison with that by cement.

The success and former popularity of lime mortars and plasters is due to their availability (portland cement has only been readily available during the 20th century), workability and the low strength required of them. For example, a mortar strength of only 0,6 MPa is sufficient to support a four-storey solid brick building (Boynton, 1980).

Further research is necessary in order to confirm current evidence that the lime-soil and cement-soil reaction products can actually become carbonated. This would lead to a better understanding of the exact nature and mechanisms involved in the reactions.

However, recent laboratory investigations (Paige-Green, 1990) have shown that even after more than 7 years of controlled laboratory curing the strength of lime stabilised samples can be reduced by more than 60 per cent by 24 hours of carbonation (100 per cent carbon dioxide atmosphere) (Figure 3).

The influence of relative humidity on the rate of carbonation, although extremely important as a source of moisture is necessary for the reaction to occur, is not clearly defined. Addis (1986) includes a figure showing that carbonation shrinkage is a maximum at a relative humidity of about 50 per cent and decreases to zero at 100 per cent. Goodbrake et al (1979) have determined that the percentage of calcium silicate reacted with carbon dioxide declines steeply below a relative humidity of 50 per cent and is more or less constant (about 75 per cent) at a relative humidity in excess of 50 per cent). Roberts (1981) states that carbonation is greatest at ordinary temperatures in the relative humidity range of 50 to 75 per cent.

5.3 Rates of carbonation

The following rates of carbonation have been measured over a number of years in both laboratory and field situations (partly after Sampson et al, 1987):

Atmospheric: 0,5 - 2 mm/day on all exposed faces (notably downwards during curing and before sealing).

Upwards and sideways in soil air: 2-50 mm/year (notably on the bottom and sides of stabilised layers, but also from the top downwards of stabilised subbases under unstabilised bases).

Concrete in atmospheric air: 0,1 - 3 mm/year

Lime-sand mortars: incomplete even after hundreds of years (Boynton, 1980).

A brief laboratory experiment to investigate whether the wet/dry brushing test simulates carbonation has been carried out. After compaction and curing, 1, 3, 6, 9, and 12 wet/dry cycles, the surface and centre of specimens of three different materials were qualitatively inspected for carbonation using phenolphthalein indicator. The results are summarised in Table 4.

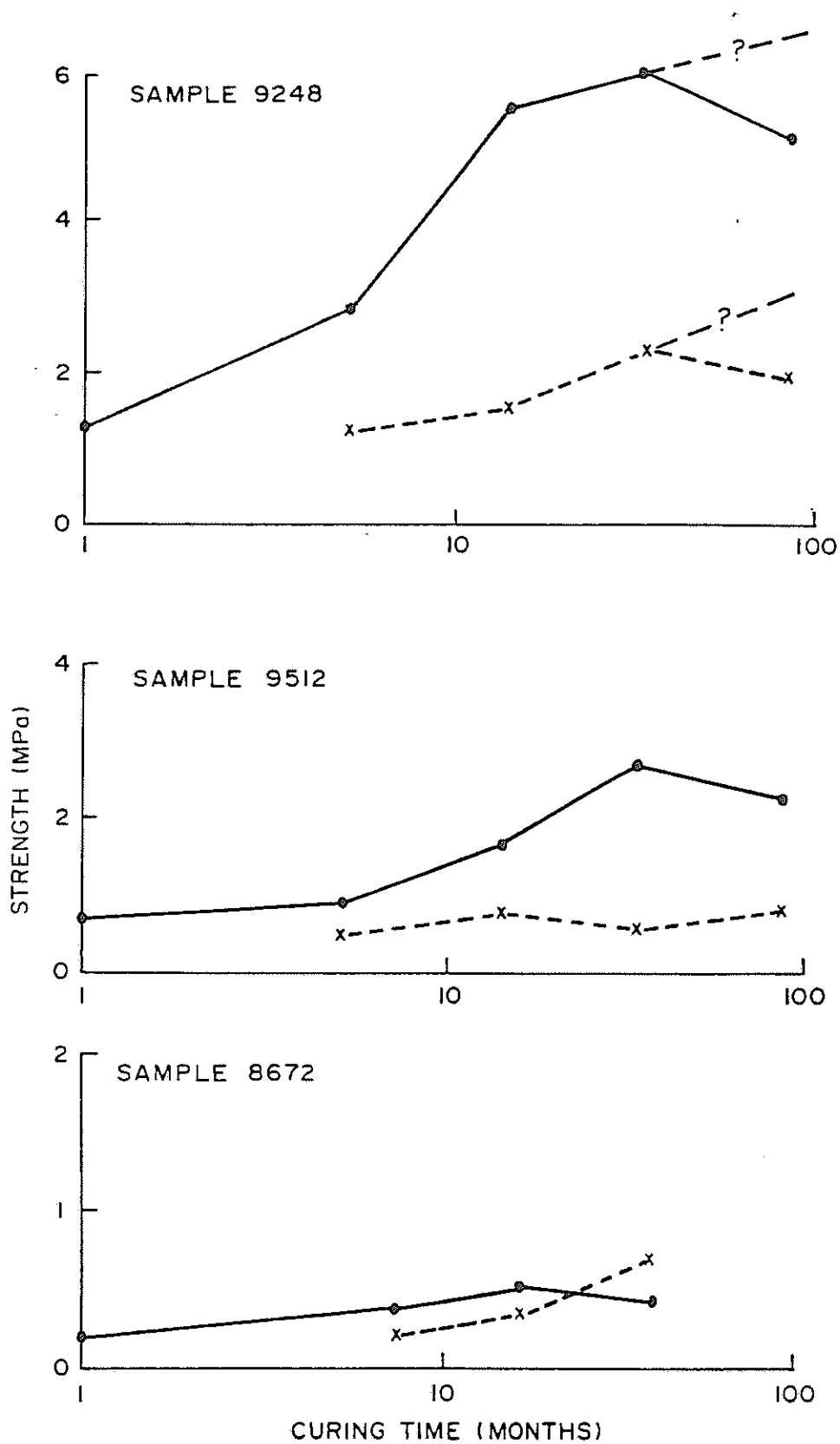


FIGURE 3
VARIATION OF STRENGTH (—) AND CARBONATED
STRENGTH (---) WITH TIME

Table 4: Change of phenolphthalein reaction with wet/dry brushing cycles

Material	Cement content (%)	Position ¹	Phenolphthalein reaction after number of wet/dry cycles ²					
			0	1	3	6	9	12
Calcrete (11617)	2	S	3	2	0	0	0	0
	2	I	3	3	2	2	2	1
	4	S	3	3	1	0	0	0
	4	I	3	3	3	2	2	2
	6	S	3	2	2	1	0	0
	6	I	3	3	3	3	3	3
Quartzite (11616)	2	S	3	2	1	0	0	0
	2	I	3	3	3	3	3	3
	4	S	2	1	1	0	0	0
	4	I	3	3	3	2	1	1
	6	S	2	1	1	0	0	0
	6	I	3	2	2	2	2	1
Dolerite (11615)	2	S	2	1	0	0	0	0
	2	I	3	2	1	1	1	1
	4	S	1	1	1	0	0	0
	4	I	2	1	1	1	1	1
	6	S	1	1	1	0	0	0
	6	I	2	2	2	1	1	1

¹ S = surface of specimen; I = interior of specimen.

² Phenolphthalein reaction as follows:

0 = colourless (ie pH below 10)

1 = light pink (pH approximately between 10 and 11)

2 = red (pH approximately between 11 and 12)

3 = dark red (pH above 12)

Qualitative pH and acid tests are the most convenient for field use and quantitative measurements of the same parameters for laboratory use. The in-situ (and laboratory) pH is strongly influenced by the partial pressure of CO₂ (P_{co2}) in the air with which it is in equilibrium and whether or not solid CO₃ is present. As a rough guide (Addis, 1986), in the absence of CaCO₃ water with a pH of less than about 7,5 would be regarded as being

aggressive towards concrete. If CaCO_3 is present a somewhat lower pH of down to about 6,0 could be tolerated. As the CO_2 content in the air in unstabilised pavements is invariably greater than atmospheric it is clear that the environment within a pavement is aggressive towards lime and cement. As soil pH is apparently the arithmetic mean pH of the individual soil minerals (Jackson, 1958), even a completely carbonated stabilised material may not necessarily have a pH equal to that of CaCO_3 (ie normally about 8,3). In addition, carbonation is seldom complete and only minute traces of Ca(OH)_2 , (solubility is about 0,159 per cent at 25 °C), are necessary (only 0,0159 per cent) to just saturate (and therefore to raise the pH to 12,4) the water phase of a soil with a moisture content of 10 %. The water phase of carbonated concrete has a pH of 8,5-9 (Moskvin et al, 1983).

The pH and solubility of lime decrease with increasing temperature. For example, the pH of a saturated solution of Ca(OH)_2 decreases from 13,4 at 0 °C through 13,0 at 10 °C; 12,4 at 25 °C; 12,0 at 40 °C to 11,5 at 60 °C. At temperatures of ≥ 80 °C it is $\leq 11,0$. The solubility decreases from 0,185 per cent at 0 °C to 0,077 per cent at 100 °C. This may exacerbate the carbonation problem in very hot areas where high temperatures in the road (under the black surfacing) result in both decreased solubility and pH. The stability of the stabilisation reactions could be adversely affected. The influence of hydrogenesis (condensation of moisture beneath the surfacing due to diurnal temperature variations) too could influence the pH and possibly cause dilution of the available lime.

As pH varies with temperature, the temperature during measurement must always be stated in accurate laboratory work (as well as applying the temperature compensator of the electrode).

As the reaction products are only stable at a pH of 11 - 12,5 further work is necessary to find a reliable field method of measuring such pHs. Of all the indicators tried, only phenolphthalein appears to be reliable in all cases. However, this can only indicate when the pH is in excess of about 10 (and less than about 8,4).

6 EFFECTS OF CARBONATION

Carbonation has a number of deleterious effects on the stabilisation process. Theoretically a number of effects are possible, but it is difficult to substantiate all of these in practice.

Theoretical effects

- 1 Destruction of Ca(OH)_2 and Mg(OH)_2 and production of CaCO_3 and MgCO_3 .
- 2 Destruction of cementing CSH and CAH compounds.
- 3 Expansion of lime (Lea, 1970) and shrinkage of hydrated calcium silicates (Goodbrake, 1979b) on carbonation.
- 4 Decrease in pH, from about 12,4 (that for lime) or higher (some cements) ultimately (on complete carbonation) to about 8,3 (that of CaCO_3).
- 5 Decreased solubility.
- 6 Reduced relative compaction (stabilised materials have an MDD and OMC less than the equivalent unstabilised material (Little et al, 1987).

Observed effects

- 1 Destruction of Ca(OH)_2 and production of CaCO_3 .
- 2 Decrease in UCS or CBR (rarely no change or increase).
- 3 Rutting (due to loss of density?).
- 4 Microcracking.
- 5 Increase of PI.
- 6 Decrease in pH to 8,3 - 10.
- 7 Decrease in paste electrical conductivity (EC), indicating a decrease in solubility.

On average, soils lose about 40 per cent of their uniaxial compressive strength on carbonation, whether stabilised with lime, cement, or lime-slag (Sampson, in preparation). Lime stabilised samples, however, cured for up to 7 years under controlled laboratory conditions, have shown decreases in their unconfined compressive strength of between 45 and 75 per cent on carbonation (Paige-Green, 1990). However, some materials may disintegrate, whereas others might show a moderate increase in strength. Plain Ordinary Portland Cement (OPC) concrete normally increases in strength on carbonation (Roberts, 1981). However, it is recognised in concrete technology that carbonation is seldom, if ever, complete and is probably partly reduced by the carefully controlled curing conditions applied to structural concrete.

A return of the plasticity of stabilised soils to the pre-stabilisation plasticity on carbonation has been reported by a number of workers (Pinard, 1985; Bagonza et al, 1987). It should not be possible for the PI to increase within the life of the road if the clay has been destroyed by the lime. Those cases where the PI has returned are probably due to insufficient stabiliser,

insufficient pulverization, inefficient mixing, or destruction of the stabiliser before it had time to react with all the clay. In the former two cases mechanical action and/or carbonation probably destroyed the weak stabilisation cementing bonds forming the clay particles into aggregations, freeing them and allowing the PI to increase. The investigation of a failed section of decomposed basalt modified with 2 per cent lime illustrates this clearly. The plasticity index of a sample of the failed outer wheel track of the road yielded a PI of 10. However, after treating the sample in a Durability Mill (Sampson and Netterberg, 1989), wet abrasion (Treatment D) resulted in an increase in PI to 17, the PI of the original unstabilised material. Larger aggregate particles which contain clay often result in reaction with lime only at the surface. Should they disintegrate after carbonation of the lime or release sufficient clay to consume the remaining lime, it is highly likely that the plasticity index of the material will return. It is plain that the standard PI test is an unsatisfactory measure of the clay content and activity of a stabilised material, and research is needed to standardise a more suitable soil preparation method. Pinard (1987) showed that the plasticity index of a weathered basalt (PI = 17; the same material discussed previously) was generally a minimum after 2 days curing with 2 per cent lime (PI = 6) but increased to 10 after 6 days. It may therefore be necessary when modifying or weakly cementing marginal materials to carry out time delayed Atterberg tests. Laboratory investigations have, however, shown that there is no significant difference between the plasticity of replicate samples treated in the Durability mill after 12 wet/dry cycles, one being tested immediately and the other after accelerated carbonation. This apparently occurs when the material is cemented and the ICL is satisfied. However, in the case of materials which are modified and cementing does not develop fully, an increased importance should be placed on testing of the unstabilised material durability in terms of more drastic disaggregation as simulated for example, by the durability mill.

Atmospheric air contains about 0,03 per cent CO_2 and the air in soils and unstabilised pavement layers 0,1-14 per cent CO_2 (usually a few percent (Sampson et al, 1987)). The CO_2 content of the air in hard, "uncarbonated" layers is much less than 0,03 per cent and that in carbonated layers 0,03 - more than 0,5 per cent. Like concrete therefore, "stabilised" materials are essentially unstable in the normal environment and carbonation is inevitable if CO_2 can gain access to the material. Whether or not the material is significantly weakened as a result of this and whether or not the road fails as a result are other matters entirely. In the absence of solid CaCO_3 the calculated pH of water in equilibrium with 3 per cent CO_2 (a typical value under stabilised layers) is about 4,7. This is well below the pH of 6,0 often recommended as the lower limit for ground-water in contact with Ordinary Portland Cement concrete. The situation is similar to that involved in the attack on concrete by aggressive

CO₂, which is aggravated by a low temporary hardness (low content of CaHCO₃). Although the presence of calcium carbonate in the soil below the stabilised layer should therefore be beneficial (eg as in a calcrete), the reservoir of CO₂ is so great (probably extending down to the water table) that it does not have much effect. In practice, calcrete pavements appear to carbonate just as fast (or faster) than others.

CO₂ appears to accumulate under compacted layers in pavements (effectively acting as an impermeable membrane) to higher values than in the adjacent veld and to reach an equilibrium condition analogous to that of moisture content in sealed roads (Sampson et al, 1987). Isotopic measurements indicate that this CO₂ is a mixture of the local plant-derived soil CO₂ and atmospheric CO₂.

Experience has shown that the addition of lime (both slaked and unslaked) to a soil results in an increase in the optimum moisture content and a decrease in the maximum dry density. Values for these changes can be up to 25 per cent for the optimum moisture content and between 3 and 10 per cent for density. Rapid carbonation of marginal materials thus may have significant implications. Marginal materials are particularly moisture sensitive and the loss of stabilisation may result in a material compacted to perhaps 5 or 10 per cent less than its maximum dry density and at a moisture content significantly higher than its natural optimum. The material is thus both significantly weaker and prone to densification (and rutting) under traffic.

7 IDENTIFICATION OF POTENTIAL CARBONATION SITUATIONS

The occurrence of carbonation may have serious implications:

- Premature rehabilitation or reconstruction may be necessary.
- Disputes, delays and expensive remedial measures may be caused during construction.

It is therefore important to recognise those materials prone to carbonation at an early stage of a project in order that suitable preventative measures can be planned. Similarly, certain construction procedures may result in an increased tendency of the stabilised materials to carbonate. The following aspects should thus be considered prior to the use of stabilisation.

7.1 General

Unless proven by experience or durability testing an attempt should not be made to improve the material too much. For example, it is inadvisable to stabilise a material for base that would not have been used unstabilised as subbase (J H M Williamson, 1965; pers. comm.). It is also suggested that any increase in strength of more than 100 per cent caused by stabilisation should be discounted. Texas for example (at least in 1972) apparently discounts any improvement in triaxial classification after lime stabilisation (modification?), although in McDowell's (1972) method of pavement design a reduction in cover thickness is permitted if the stabilised material can be demonstrated to possess significant tensile strength. TRH 13 (NITRR, 1986a) and TRH 14 (NITRR, 1985b) also give guidelines as to the minimum quality before stabilisation to a particular class of material. It is useful to imagine what the material would be like if it lost half its strength and/or the PI increased or if it even became completely destabilised. The aggregate hardness and durability should not be neglected as soft or non-durable aggregate will disintegrate releasing additional fines and consuming additional lime which may not have been accounted for in the initial consumption of lime test.

7.2 Soil composition

Any significant quantities of salts (particularly sulphates) may be harmful to stabilisation in general (Netterberg, 1979). The quantity of organic matter should be limited to less than 2 000 ppm by ASTM C-40, although the BS 1924 pH test for reactive organic matter (using the proposed stabiliser type and content) is probably more reliable. Roots, twigs, coal and paper are probably not harmful. Free, reactive Si, Al and Fe oxides in the soil stabilised may be beneficial or troublesome, whereas CaCO_3 is not harmful. Some clays do not react with lime, particularly those with low plasticity indices (< 10).

The early PCA work showed that tests for grading, surface area, Atterbergs, UCS, organic content, pH, density and cement-void ratio were unsatisfactory indicators of cement requirements and the wet/dry and freeze/thaw tests were therefore developed. These appear to take most or all likely factors into account, probably even carbonation indirectly.

An analysis of almost 300 results from a severely distressed road in Namibia constructed from a marginal quality calcareous sand indicated that the grading modulus in the failed areas was generally less than 1,80. Although the evidence is not strong enough to use this as a definitive indicator, it is recommended that where marginal materials are being used and the grading moduli are typically low, increased attention should be paid to durability

testing as discussed later ie grading modulus can be used as a first approximation of potential problems.

The properties of the soil strongly dictate the stabiliser content necessary for modification and cementation. It is thus important to determine the Initial Consumption of Lime (ICL) on the bulk of the material and not just the fines according to the method of Clauss and Loudon, 1971). The so-called "gravel ICL" (NITRR, 1984) should be carefully carried out to identify the minimum lime necessary to completely modify the material. It should be noted that additional lime is necessary to fulfil the cementation requirements. In very soft materials or low durability basic igneous rocks which may disintegrate in service, it may be necessary to grind the sample to less than say 0,425 mm in order to identify the worst possible ICL which may need to be satisfied. Although it is generally considered to be uneconomic to stabilise materials with an ICL greater than three per cent (Ballantine and Rossouw, 1989), if materials with a high ICL are to be used, the stabiliser content should at least exceed the ICL by one per cent. If no alternative materials are available, the use of higher stabiliser contents could still prove economic although the pavement may develop undesirable shrinkage cracking.

Further work is necessary to develop reliable chemical test methods and local criteria for the evaluation of the suitability of soils for stabilisation. Although most of these aspects can be evaluated by means of physical tests for strength and durability, chemical methods (including pH and Electrical Conductivity (EC)) are usually much cheaper and faster, and require much less soil.

7.3 Construction control

The following construction procedures should be carefully controlled to limit carbonation of marginal materials:

- a) Unnecessary delays between completion of a stabilised layer and application of the next layer should be avoided. This is particularly true of stabilised bases which should be primed and sealed as soon as possible after construction. During the project planning stage this should be taken into account.
- b) The layers should be compacted (without unnecessarily vigorous mixing) as quickly as possible so as to complete the compaction process before any excessive cementation and/or carbonation reactions occur. Certain materials, eg some

calcretes with a high amorphous silica content set quickly and compaction can not achieve the required densities without breaking cementing bonds.

- c) The stabiliser should be as fresh as possible and not stored for any length of time.
- d) If possible the bottom few centimetres of the stabilised layer should be compacted separately to ensure a high density is achieved in this area.
- e) The use of vibrating rollers for compaction may exacerbate the formation of shrinkage cracks (Bofinger et al, 1978) allowing the ready access of carbon dioxide from above and exposing a larger area of the stabilised layer to potential carbonation. Similarly high densities also significantly increase the potential shrinkage and subsequent cracking. Materials moulded at the British Standard light density exhibited shrinkage strains up to 75 per cent less than those compacted at BS heavy density (Bofinger et al, 1978).

8 SYMPTOMS OF CARBONATION

The likely symptoms of a carbonation problem include the following:

- 1 Surface weakening or disintegration of stabilised layer before or after surfacing. This may lead to punching of the chippings, bleeding, surfacing and shallow base failures, and scabbing of the surfacing.
- 2 Progressive in-service loss of cementation of whole layers, usually mostly from the bottom upwards, but also from all faces exposed to atmospheric or soil air. This may lead to rutting and/or shearing.
- 3 Return of plasticity during construction or in-service. This may lead to shearing.
- 4 Expansion (cracking, decrease in density). This may lead to rutting in the wheeltracks.
- 5 Shrinkage (cracking, increase in density). This may lead to pumping and secondary traffic-associated cracking.

The usual primary, non-traffic-associated block cracking (mostly shrinkage) does not always lead to distress. In itself it is not considered as evidence for a loss of stabilisation, but rather the reverse.

9 DURABILITY TESTING OF STABILISED MATERIAL

It is important to carry out durability testing of stabilised materials prior to their use. Numerous tests and variations within these tests are available. The most popular and useful tests discussed in the literature are apparently the wet/dry (Test T135 and D559) and freeze/thaw tests (T136 and D560) originally devised by the Portland Cement Association and now specified by both the American Association of State Highway and Transportation Officials (AASHTO, 1982) and the American Society for Testing Materials (ASTM, 1987). The former test is incorporated in the local testing manual, TMH 1 (NITRR, 1986b). However, the specification limits recommended by PCA (1971) are based on samples compacted at Proctor density and moisture content while local construction practices generally require higher Mod AASHTO densities. A major problem existed with the operator susceptibility of the brushing procedure in both the wet/dry and freeze/thaw tests. This has been overcome locally for the wet/dry brushing test with the development of a mechanical brushing device (Sampson, 1988).

The Portland Cement Association (PCA, 1971) durability criteria for the whole USA for soil-cement mixtures are as follows:

1. Loss after 12 cycles of wet/dry or freeze/thaw test not to exceed following limits:
 14 % for soil groups A-1, A-2-4, A-2-5 and A-3
 10 % for soil groups A-2-6, A-2-7, A-4 and A-5
 7 % for soil groups A-6 and A-7.

This requires one specimen to be prepared in a Proctor mould at Proctor effort and OMC at each cement content evaluated and is regarded as adequate for routine work on normal soils.

2. Compressive strength should increase both with age and with increase in cement content in the range producing results that meet (1) above. Research is needed to develop criteria for the reliable identification of materials suitable for stabilisation for a specific purpose. For example, fine-grained materials can be used for bases; their limitations just need to be ascertained. The Federal Highways Authority (FHWA, 1979) guide also recommends, on the basis of the PCA work, in addition:
3. Maximum volume changes (swell) during durability tests < 2 % of initial volume.

4. Maximum water content during durability tests less than saturation when moulded.

These latter two tests are included in ASTM D559 (ASTM, 1987) and AASHTO T135 (AASHTO, 1982) and only require one additional specimen. This testing is apparently only done on unusual soils and for research purposes. However, it could also possibly be used instead of the brushing test.

Both the wet/dry and freeze/thaw tests are regarded as more important than strength tests by the PCA and applicable as durability tests everywhere. Due to the possibility of accelerated strength gain and the partial cancellation of clay shrinkage by expansion at the high temperature (71 °C) of the wet/dry test, the PCA maintain that both the wet/dry and freeze/thaw tests should always be used. However, the wet/dry test is apparently less severe for most soils than the freeze/thaw test and in some southern areas of the USA only it is used. The same has been found for tropical areas (Ola, 1974; Osula, 1989) and is probably pertinent to sub-tropical areas as well.

Several states in the USA apparently now only specify a UCS. The maximum freeze/thaw loss requirement of 14 % when the UCS is less than 600 psi (4,2 MPa) previously in ASTM C 593 has now been dropped in favour of a minimum UCS of 2,8 MPa after 7 days curing at 38 °C and vacuum saturation. This is believed to correlate well with the UCS after 5 cycles of the Dempsey and Thompson (1973) freeze/thaw test for both lime and cement.

These PCA durability tests (only the local equivalent of the wet/dry test is covered by TMH 1 (NITRR, 1986b)) have apparently been very successful in the USA. The PCA emphasizes that these are the "proof" tests and that anything else such as UCS is a "short cut" procedure. Several other versions of these durability tests are in use and in some cases strength tests are carried out on the specimens instead of brushing. However, the brushing should not be omitted as the progressive loss of surface strength is not identified to the same extent by strength testing.

The PCA (1971) also list "short-cut" procedures (Leadabrand and Norling, 1953; Norling and Packard, 1958) for sandy soils (< 50 % passing 0,05 mm and < 45 % coarser than 4,75 mm, etc.) and a rapid "pick and click" procedure for emergency or very small projects. The short-cut method was said not to always indicate the minimum cement content required, but to almost always be safe, and generally close to that indicated by the ASTM-AASHTO freeze-thaw and wet-dry tests. They are apparently widely used in the United States. As an interim local procedure it may be possible to check the cement content indicated by the usual

TMH 1 UCS testing to satisfy local requirements using the appropriate PCA charts. If it is too low the wet-dry brushing durability test should be carried out and the cement content increased if necessary. Alternatively, the use of the short cut procedure as it should be safe in most cases, although possibly unnecessarily conservative for local use. Insufficient information is available to construct similar charts for local use. It is suggested that the UCS at the minimum field compaction be used. It appears as if a TMH 1 UCS is approximately the same as a PCA UCS at the same density. According to the PCA strength charts a minimum UCS of 1,4-2,1 MPa is required (depending on grading) at the cement content indicated by other charts (depending on grading and density).

The cement contents required can also be estimated from the pedological soil classification of the material, thus cutting down on the amount of laboratory testing required (PCA, 1971). Soils of the same soil series and horizon and of similar texture require the same amount of cement (PCA, 1979). This finding should also be applicable here. However, pedological soil surveys have limited usefulness in engineering as local borrow pits are often deeper than their surveys.

The use of non-destructive length change and pulse velocity tests on the durability specimens (in place of UCS tests) as a means of monitoring strength changes appeared promising in research carried out in the United States but does not seem to have been applied in practice.

The PCA durability tests (now called the ASTM-AASHTO even by the PCA) are designed to ascertain the minimum cement content required to produce a durable, hardened material called soil-cement. At lesser cement contents only a cement-modified soil is produced. The PCA (1971), although stating that modification is permanent, also present data which indicate that the PI can increase after freeze-thaw cycling a cement-modified chert (1 % cement by volume) and that, depending on the severity of weathering the "semihardening" produced in modification (apparently at least in the case of silt-clay soils) may only be temporary. At cement contents of 3 and 5 % the PI of the cement-treated chert did not increase after cycling. It is clear that the usual cement contents required for soil-cement (PCA, 1971) are mostly higher than those used in southern Africa. Most of our cement-treated materials are therefore only cement-modified. It is also clear from McDowell (1972) that the possibility of lime modification being only temporary is accepted by AASHTO T220-66.

The British Standard considers the PCA durability tests to be too harsh and classifies the material as durable if the specified UCS values are retained after 7 days curing and immersion in water (Osula, 1989). The specified UCS value for a cylindrical specimen is 2,8 MPa compacted according to BS 1924 (Croney, 1977; Department of Transport, 1970) which is very high: thus a material satisfying this is highly likely to be durable (Osula, 1989).

No widely applicable local limits for durability tests have so far been established. All that can be said with reasonable certainty at this stage is that stabilised materials which comply with the usual PCA requirements for the wet/dry brushing test of 7-14 % depending on their AASHTO soil classification will give good performance, and that materials which fail them badly will probably give poor performance. (It should be remembered that the in situ density will usually be higher than that at which the wet/dry loss is obtained in the laboratory (ie Proctor compaction) and an inherent conservatism is included in these values.) Evidence from Natal suggests that a loss of up to 40 % may be allowable for fine grained subbases under moderately heavy traffic (Department of Transport, 1984). It is important not to omit the brushing and to do the test correctly! The volume (or length) and moisture content changes on a separate specimen should be measured according to the ASTM procedure.

A summary of available possible test methods and criteria (Table 5) indicates that numerous researchers and authorities have developed test methods and acceptance criteria. However, it appears that very few can be related to actual performance in service.

The essential purpose of all the tests as well as those for strength, is basically to determine the correct type and amount of stabiliser to be added for the purpose intended. If the test shows the material to be non-durable or of inadequate strength, either:

- the stabiliser must be changed
- the amount of stabiliser increased
- or the material should be rejected and a better one located.

More stabiliser may have to be added to satisfy durability requirements than that for strength alone: the whole idea is to ensure that the strength measured presently does not decrease to some unacceptable value in the future. This may be estimated by either a carbonation type test or a wetting/drying test which appears to effectively incorporate surface carbonation during the wet/dry cycles. This is clearly shown in Table 4 where the carbonation penetration is significantly less as the stabiliser content increases. The influence of the initial consumption of lime (1, 3 and 4 per cent for samples 11617, 11616

Table 5: Potential stabilisation durability tests and criteria

Test	Possible local limit	Foreign Limit
ICL ("gravel") (NITRR, 1984)	Use \geq lime indicated by ICL	-
ICC ("gravel")	Use \geq cement indicated by ICC?	-
pH (BS 1924 paste with 10 % cement)	$\geq 12,1$	12,1 (U.K.)
pH (paste)	$\geq 12,1$ both after 1 h and after 7 days curing at stabiliser content used	-
Surface area (glycerol retention)	? m/g	≤ 10 ? (USA)
Freeze/thaw brushing	?	7-14 % (USA)
Wet/dry brushing ⁽¹⁾		
(a) Standard 12 cycles at Proctor compaction	≤ 20 % for bases under bitumen? $\leq 7-14$ % for subbases under concrete? (standard PCA requirements) TRH13 says 14 % for all.	7-14 % (USA) 7-14 % (USA)
(b) Standard 12 cycles at MAASHO or NRB compaction or at min. % MAASHO for that layer	≤ 40 % for other subbases? $\leq 7-14$ % ? (normal PCA requirements for Proctor compaction)	7-14 % (USA) -
(c) Four cycles at Proctor compaction	?	-
Erosion Index (De Beer, 1986))	1 mm base? (E0-E4 traffic) 3 mm subbase? (E3-E4) 5 mm subbase? (E0-E3)	- - -
Erosion Index after accelerated carbonation	As above?	-
UCS soaked 7d after 7d cure/UCS cured 14d (BS 1924)	≥ 80 %?	≥ 80 % (UK)
UCS vacuum soak/UCS normal	≥ 80 %?	-
UCS carbonated/UCS normal (eg VKE CADS test)	$\geq 50-75$ %? (De Wet & Taute, 1985)	-
UCS dried & soaked/UCS normal (eg VKE CADS test)	$\geq 50-75$ %? (De Wet & Taute, 1985)	-
UCS or CBR after 12 wet/dry cycles	\geq normal UCS or CBR requirements?	-
UCS or CBR after 12 freeze/thaw cycles	$> ?$ % of normal UCS/CBR at same age.	50 % ? (USA)
UCS vacuum soaked (Dempsey & Thompson, 1973)	\geq normal UCS or CBR requirements? ASTM C593 says 2,8 MPa	- $\geq 2,8$ MPa (USA)

(1) If the test is carried out on 127 x 152 mm specimens prepared in CBR moulds as if for a UCS the percentage loss should probably be multiplied by 1,33 to correct them to the same surface area/mass ratio as the standard 117 x 102 mm specimens

Table 5 (continued): Potential stabilisation durability tests and criteria

Test	Possible local limit	Foreign Limit
UCS or CBR after accelerated carbonation	\geq normal UCS or CBR requirements?	-
UCS or CBR after accelerated conversion (24h at 70-75°C ?)	\geq normal UCS or CBR requirements?	-
Vacuum saturation m/c (Dempsey & Thompson, 1973)	?	?
PI on normal UCS or CBR specimens	\leq SP for base, 5 for subbase?	-
DMI for normal UCS or CBR specimens	\leq 100 for base?	-
TRRL frost heave (BS 1924)	\leq 13 mm ? (when frost problem expected)	\leq 13 mm (UK)
Brand (1960) frost heave (in CBR mould)	?	? (Germany)
PI on brushings in wet/dry brushing test and on UCS or CBR specimens after 12 wet/dry cycles or accelerated carbonation	\leq normal requirements for unstabilised layers?	-
DMI on UCS or CBR specimens after 12 wet/dry cycles or accelerated carbonation	\leq normal requirements for unstabilised layers?	-
PCA short-cut procedure involving Proctor MDD, estimation of cement content from charts and verification by UCS	?	Cement content \geq that indicated by charts
Similar short-cut procedure at MAASHO MDD (or at min. % MAASHO for that layer)	Cement content \geq that indicated by appropriate chart.	-

and 11615 respectively) is also clearly demonstrated. Sample 11617 with the lowest ICL (3 per cent of the stabiliser is available for cementation after adding 4 per cent cement) retains much of its stabilisation away from the surface whereas the other samples with only 1 and 0 per cent cement available for cementation carbonate rapidly.

It is important to determine how the specified strength criteria originated. It has not been recorded for example, whether it is implicitly assumed in the design that the laboratory strength which is determined after 7 days for convenience, will continue to rise with time and that the actual strength in service will be higher. Perhaps the 7 day strengths specified fulfill the design requirements in the long term. Relating the UCS to CBR the latter would appear to be the case as even the UCS of C4 materials is equivalent to a CBR in excess of 80, the value typically specified for unstabilised bases.

The percentage loss in strength in the durability tests is really quite arbitrary at this stage and the guiding principle should probably be that the residual strength (and the PI) should be adequate for the job. The problem is that no one really knows what is adequate since the UCS, CBR and PI requirements are not well-founded and the extent to which they deliberately or accidentally take durability into account is not clear. What UCS is required if the pavement spends three-quarters of its life in the post-cracked phase? In short, because of the local cookbook approach to TRHs and other specifications, the source, basis and application of the criteria they contain may soon be forgotten, as well as the consequences of deviating from them. (Not everything in a TRH or other document is necessarily valid indefinitely: it only represents the state-of-the-art or else the best knowledge available at that time.) As an interim measure the normal TRH 14 (NITRR, 1985b) and 13 (NITRR, 1986a) requirements should be applied to the residual values. Another problem is that the durability tests do not all measure the same thing, and no certainty exists yet as to what is being measured. For example, the BS 1924 immersion test is intended to evaluate the effect of expansive clays or sulphates, the BS 1924 pH test the effect of organic matter, the wet/dry test primarily to simulate shrinkage forces, the freeze/thaw test expansive forces, and accelerated carbonation tests the strength and PI after carbonation. Curing at high temperatures may be of value as an accelerated conversion test for stabilised soils containing reactive Al or when using high alumina cement (HAC). The ICL test was originally intended as a quick indicator of the amount of lime required and not strictly as a durability test. The ICL increases with curing time, as does the optimum lime content for maximum strength. There is no optimum cement content although Bofinger et al (1978) showed that the shrinkage strain was often minimal at a cement content of 6 per cent for a number of soils and cracking would thus be least at this cement content. A soil surface area of more

than 10 m²/g appears to indicate that more than 5 per cent cement would be required to keep the loss in the freeze/thaw test below 10 per cent. The wet/dry brushing test does, however, simulate the most common mode of failure (ie surface disintegration) whether it arises from carbonation, wetting/drying, return of plasticity or combinations of these.

Durability appears to normally increase with stabiliser content by mass, age, density, and initial quality of raw material (including degree of pulverization of clay soils).

It is recommended that a combination of the gravel ICL, the wet/dry brushing test and the soaked UCS for residual strength are carried out in order to evaluate durability. The PCA short cut procedure for soil-cement and other overseas guides such as those for lime in AASHTO T220 and ASTM C593 should perhaps be evaluated further for possible local use. The method of de Wet and Taute (1985) also shows promise in identifying carbonation problems but lacks the necessary action of brushing. Their proposed limits have, however, not been related to the performance of stabilised layers within existing pavements.

The following tests and acceptability criteria are thus recommended as interim measures:

- a) Gravel ICL - the gravel ICL should be satisfied and at least one per cent additional stabiliser added if cementation is required.
- b) Wet/dry brushing test - A maximum 12 cycle loss in the wet/dry brushing test on specimens compacted at Mod AASHTO density and OMC and brushed with the mechanical device of 8 per cent for base material and 13 per cent for subbase should be permitted (Sampson and Paige-Green, 1990). This should not exceed 5 per cent for stabilised material under concrete pavements.
- c) The residual UCS after curing and carbonation should not be less than 750 kPa for so-called C4 materials and 1,5 Mpa for C3 materials.

It should be noted that the C3 and C4 nomenclature of TRH 4 and 14 (NITRR, 1985a and b) now need re-definition.

10 MEANS OF PREVENTING CARBONATION

The most important counter-measure against carbonation is to be able to diagnose the potential of a material to carbonate. Before any preventative action can be planned, a knowledge of the degree of carbonation expected, the speed with which it will occur and the effect on the material is necessary. These are, however, not easily determined in the

laboratory prior to construction. It is thus recommended that the appropriate tests described in section 9 are carried out in order to get a feel for the material. Should the material indicate a high propensity to carbonate and lose strength, even after increasing the stabiliser content and no alternative material is available the following precautions should be taken:

- 1 Keep the material moist during curing and avoid wet/dry cycles.
- 2 Keep out CO₂ by minimising the exposure of the layer to the atmosphere, ie **seal** as soon as possible, and encapsulate in potentially severe cases.
- 3 Improve construction practice and control, eg compact sooner with appropriate equipment to get higher density and lower permeability.
- 4 Compact to < 5 % (?) air voids, not just a prescribed percentage Mod AASHO.
- 5 Improve curing procedures and operations.

It should be noted that although all geological materials appear to be susceptible to carbonation, the poorer the material stabilised the greater the likelihood of it failing after carbonation ie the residual strength is probably inadequate for the prevailing conditions.

It would appear that generally in southern Africa, too little stabiliser is added for effective cementation. In the USA, recommended lime contents are generally between 3 and 10 per cent by mass whilst recommended cement contents range between 3 and 16 per cent by mass (Little et al, 1987). Those pavements in southern Africa incorporating stabilised layers which have performed exceptionally well (eg Fisantekraal and Langebaanweg airfields) have stabiliser contents well above the average 2 to 4 per cent used locally. Should carbonation occur the residual strength is still adequate for the layer to perform satisfactorily. TRH 4 (NITRR, 1985a) in fact recommends that C1 and C2 materials (7 day strength more than 3 MPa) are not used because of the cracking associated with them. Should carbonation reduce the strength of a C3 or C4 material by 60 or 70 per cent it is likely that the material will not function as desired, behaving in fact as materials of poor C4 and not even C4 (approximately a G5 natural gravel) respectively.

Surface carbonation during construction and upward and lateral carbonation as well as carbonation adjacent to cracks have been studied on a number of roads. Practical methods of preventing surface carbonation by improved curing techniques have been described by Netterberg et al (1987) and the value of a bitumen emulsion membrane in preventing carbonation from below shown by Sampson et al (1987).

10.1 Curling and surface protection

There are several conflicts here between the requirements for prevention of carbonation and water damage and between the requirements of a prime and a curing membrane and some degree of compromise seems inevitable.

For adequate curing the layer must be kept moist. For prevention of carbonation the permeability to air must be kept low. In practice this may mean that the interconnected voids must be filled with water and not air and carbonation can probably be expected to start as soon as the moisture content drops below about optimum. (Concrete apparently remains permanently saturated with water except at the surface.) However, it is undesirable to prime or seal a wet base due to the risk of water vapour blistering and even more undesirable to open it to traffic due to the risk of positive pore pressures causing it to shear under traffic. A primer with good (5-10 mm) penetrating properties is required to bind any excess fines present on the base. However, such penetration prevents cement and lime stabilisation if applied too early as well as permitting both moisture loss by evaporation and carbonation to take place as soon as it dries and becomes permeable. The best means of curing and carbonation protection appears to be an impermeable membrane applied for example as a bitumen emulsion. However, it cannot penetrate to any great extent and bind the "dust" on the base, it develops a bond with the base only slowly, penetration is very slow taking weeks or even months to reach 3-5 mm, it is inconvenient to work on, and it cannot be opened to traffic for some time unless it is sanded or chipped.

Carbonation attacks the surface of all exposed layers and even the stabiliser during construction. (For this reason, as well as to prevent drying out, in the USA clay soils mixed with lime and left to "mellow" for several days are always first surface sealed by light compaction.) It is aggravated by the wetting and drying cycles to which the average stabilised layer is subjected when "cured" by water spraying. Testing for carbonation using phenolphthalein should be carried out whilst curing and the layer covered or sealed without delay before significant (say 2 mm) carbonation is detected. A finely graded base which has experienced significant surface weakening due to carbonation or any other cause probably should not be surfaced. In such cases the damaged upper surface should be removed by brooming and/or cutting by grader and a prime should probably always be applied. It is not always easy to evaluate visually whether the upper base has been significantly weakened. If carbonation is shown by simple tests (eg phenolphthalein), it probably has. Tests such as the Ball Penetration test and the Clegg Hammer could probably be usefully applied when the

suspected weakening is shallow and the DCP when it is thicker (perhaps more than 10-20 mm), in all cases recording the penetration after every blow.

Carbonation and drying must be prevented in those cases in which a stabilised material has given an unacceptable performance in the wet/dry brushing test or after an accelerated carbonation or drying test, but is used anyway.

10.2 General rating of curing methods

Table 6 shows a summary of the results of the field and relevant laboratory experiments arranged in order of merit solely according to the minimum degree of protection afforded against downward carbonation under the conditions of the experiments. Although the data are incomplete and results are few, they do appear to rate the different methods in a reasonable order. In general, the traditional approach of maintaining the moisture content is also the best for the prevention of carbonation, although limiting the access to air is probably also desirable. The usual techniques of watering for 7 days or spraying with a fluid primer at a rate of about 0,7 litres/m² may not be much better than doing nothing. The performance of the watering method probably depends greatly upon the material type and the ambient humidity at the time as well as the quality of the supervision. Although some contradictory results were obtained, the performance of a fluid primer probably depends upon its application rate and the depth to which it penetrates. Of particular interest is the value of a cover of loose sand or gravel, whether or not it is watered.

Although this table can be used as a guide to the selection of the curing method most appropriate to a particular situation, the construction personnel must still apply sound judgement. In particular, it is suggested that the phenolphthalein test should be applied during construction and the appropriate steps taken. As already discussed, the actual length of time over which a particular method can be applied depends both upon the rate and the permissible depth of carbonation applicable to any given situation. The rate of carbonation can apparently be estimated fairly well in advance from model experiments using phenolphthalein reaction depth measurements on material compacted into CBR moulds at the expected field density and cured appropriately. As a guide, however, an approximate rate of surface carbonation of 1 mm/day can be assumed for material left to dry in the sun or which is watered or sprayed with conventional primers such as MC-30, MC-70, tar or invert emulsion.

Table 6: Carbonation resistance of curing methods evaluated

Apparent order of merit	Curing method	Time to 5 mm PHTH reaction depth Days ⁽¹⁾	Remarks
1	Surface treatment bitumen emulsion	>> 18-150	
2	150/200 pen. on MC-30	>> 52?	150/200 applied within 24h
3	MC-3000 on MC-30	>> 52	MC-3000 applied within 24h
4	Plastic sheet	46-150	
5	100 % relative humidity	> 30-90?	Continuously 100 %
6	40-50 mm loose sand watered once daily	55	
7	MC-800	29-32	
8	MC-70	35	
9	Overlapping empty lime bags	27	
10	Watered four times daily	21	
11	40-50 mm loose dry sand	20?-24	
12	40-50 mm loose sand watered once daily for 7 days	18-45	
13	150-200 mm loose gravel or graded crushed stone	15-> 97	
14	Watered twice daily	15	
15	Invert emulsion primer (MSP-1 with diesoline flux)	13	
16	RTH 3/12	10-> 97?	More than 22 days unlikely
17	RC-70	10-20	
18	Watered twice daily for 7 days followed by MC-30	10	
19	Watered every second day	10	
20	RTL 3/12	8-10	
21	Watered twice daily for 7 days	7-9	
22	MC-30	4-35	
23	None (allowed to dry)	4-31	

(1) From completion of stabilised layer.

The advantages and disadvantages of each technique are not discussed further here but are fully described in Netterberg et al (1987). However, the application of a penetrating coal tar primer sooner than about 10 days is not recommended (NITRR, 1986a). In this respect a penetrating bituminous primer such as MC-30 appears to be less harmful but still does weaken the material if applied before it has hardened. Watering the layer beforehand will minimise penetration, but may lead to water vapour blistering in hot weather. A material which has been cured in such a way (eg by water spraying) so as to lead to some carbonation (limited to a few millimetres at most), but which is judged to be acceptable, should probably always be primed to ensure an adequate surfacing bond, preferably after heavy brooming. A prime is always desirable on a base which cannot be swept to expose a good mosaic of aggregate. If an emulsion tack coat is used without a prime it may be necessary to delay opening to traffic for as long as is practicable in order to allow more time for the surfacing to cure and for the bond with the base to develop.

There appear to be a limited number of options which both permit adequate moist curing for the usual 7-14 days and also minimise or prevent carbonation.

10.3 Treatment of Bases

- 1 Keep continuously moist (using potable water with electrical conductivity $\leq 0,30$ S/m) for desired period (≥ 7 days if cementation required), preferably using 40-50 mm wet sand or plastic (polythene) sheet (Odier et al, 1971). Use light, fog-type water spraying of a bare layer (ie no sand cover) only under ideal conditions (when humidity is high, and when wet/dry cycles due to infrequent applications and leaching due to over-application of water can be avoided). Prime, using bituminous primers not before 7 days and tars not before 10 days. Surface as soon as the prime has dried sufficiently and within the time indicated by the depth of carbonation considered tolerable, or within one week of compaction, whichever is the longer. This is theoretically the best solution.

OR

- 2 Prime as soon as possible (preferably less than 48 hours after compaction), keeping continuously moist in the interim as above, but use a primer which keeps penetration to the minimum desirable, eg use MC-70 in preference to MC-30 and/or water the layer sufficiently to prevent penetration of the primer. (There is a risk of water vapour blistering if this is then followed by hot weather.) The best is probably to match the

primer to the material so as to eventually get just adequate penetration. This may require MC-250 or RC-250 in some cases. Do not use tar. Surface as soon as the prime has dried sufficiently and within the time indicated by the depth of carbonation considered tolerable, or within one week of compaction, whichever is the longer. The preferred method in the United States is membrane curing (Little et al, 1987) where the stabilised layer is either "sealed with one shot of cutback asphalt within one day of final rolling or primed with increments of asphalt emulsion applied several times during the curing period".

OR

- 3 Primerseal (1 or 2 coats MC-70 + 5 mm graded sand or aggregate) and surface within the time indicated by the depth of carbonation considered tolerable or within four weeks, whichever is the longer.

OR

- 4 Apply spray grade emulsion and chip immediately, or surface when convenient. Do not apply emulsion to a base cured by water spraying unless carbonation is absent, and excess fines have been removed to expose a good mosaic. Delay traffic as long as possible (≥ 1 month) on bends, superelevation and roundabouts.

10.4 Treatment of subbase and lower layers

The requirements for subbase and lower layers are somewhat less critical. However, weak layers at the top of such layers probably caused by carbonation during poor curing have definitely contributed to the distress observed on some heavily trafficked pavements. Carbonation-induced weakening of the top of the subbase during curing and before covering with the base, as well as the sides of the almost inevitable "cracks", may well be a factor contributing towards loss of load transfer across the cracks and pumping. The following precautions are recommended:

- 1 Any of above for base, preferably covering with the next layer within the time constraints given above for surfacing. It is important to ensure that minimal rutting and no shearing of the subbase occurs during dumping of the next layer on the uncured material. It is recommended in the United States (Transportation Research

Board, 1976) that the material is lightly rolled after sprinkling with water in order to knit the surface together and assist the curing.

- 2 Dump and spread the next layer, preferably within 48 hours of compaction and with due attention to shearing or rutting.

These suggestions are essentially intended to prevent carbonation damage. However, they should also prevent possible salt damage by crystallization in most cases where an excessively saline material is used in the structure.

10.5 Prevention of carbonation from below

- 1 Use durable stabilised material.

OR

- 2 Apply membrane overlay and/or underlay to stabilised layer, extending at least 1,0 metre wider than the area to be protected. Use plastic sheet (only practical over small areas), emulsion, penetration grade bitumen, heavy cutback or heavy tar at 0,7-1,2 l/m² as necessary or a plastic sheet, and prevent damage during construction. It is unlikely that a primer such as MC-30 or 3/12° EVT tar would be adequate. In severe cases it may be necessary to consider completely encapsulating the layer.

In addition, carbonation can probably be minimised or even prevented by compacting to the highest absolute density possible and thus minimising the permeability. It may be necessary to compact the lower 50 mm or so separately in order to achieve an adequate density (and suitably low permeability) and hence working platform, before the remainder of the layer is compacted. The former British requirement of a maximum compacted air voids content of 5 per cent may have helped to prevent carbonation in that country. This may be a better requirement than just a percentage of Mod AASHO. Similarly minimising the permeability of adjacent layers should also be helpful.

Limited measurements of CO₂ beneath pavements suggest that it makes no difference to the CO₂ contents in the pavement whether or not the grass and topsoil

are removed before construction. However, except possibly on wet clayey soils it is usually regarded as good practice to do so.

11 REHABILITATION OF CARBONATION DAMAGE

The rehabilitation methods for layers damaged by carbonation depend on whether carbonation occurred from above, below or anywhere else and the extent of the carbonation.

11.1 Carbonated from above

Carbonation from above may affect any stabilised layer (ie base, subbase, selected layers or stabilised shoulder) and is usually the result of poor curing techniques or extended periods of exposure before covering or sealing. If less than 10 millimetres of the surface of any layer has carbonated the layer should be heavily broomed before the next layer is added. If the layer is the base or a sealed shoulder, or the carbonation extends to a depth greater than 10 mm the carbonated layer should be cut with a grader and immediately replaced with a convenient material (usually a thick slurry is easiest).

11.2 Carbonation from below

Only the bottom of the lowest stabilised layer is prone to carbonation from below. This usually starts after construction and no evidence is visible until the whole layer has carbonated and distress occurs. Where significant carbonation from below occurs and the structural capacity of the road becomes inadequate, the road will usually require rebuilding (Paige-Green, 1986).

11.3 Carbonation from the side or from cracks in the layer

Carbonation may occur from the edge of the seal or horizontally into the layer from vertical cracks (usually block or longitudinal cracking caused by the stabilisation or active subgrade materials). Carbonation from the side usually presents no problem unless heavy traffic drives on the edge of the seal. Cracks which are affected by carbonation and could influence the performance of the road are usually reflected at the surface to allow ready access of carbon dioxide. Although carbonation adjacent to these cracks has been observed in practice, no distress directly attributable to this mode of carbonation has been reported. It is advisable, however, to seal these cracks in order to minimise the possibility of problems occurring (as well as to minimise the ingress of water).

12 CONCLUSIONS

Carbonation of stabilised layers (particularly consisting of marginal quality materials) has become a significant problem in southern Africa with at least 44 known cases of distress due to carbonation of stabilised layers. The literature on cement and concrete technology notes the possibility of carbonation occurring but records that it is usually more beneficial than disadvantageous. However, other than a few brief notes on the potential of lime to carbonate, very little attention has been paid to the problem in the road stabilisation context.

The existing requirements and specifications for stabilised materials are based almost entirely on the strength requirement after 7 days curing, with little attention being paid to the long-term durability of the material. Research has shown little correlation between durability and strength and has identified the ability of a stabilised material to lose strength under certain environmental conditions, namely, wetting/drying and carbonation. It is important that this strength reduction, in terms of the residual unconfined compressive strength, is not such that the structural capacity of the layer becomes inadequate for the applied loads.

The causes and effects of stabilisation are discussed in this report but it is clear that the mechanics and chemistry of soil stabilisation is still very poorly understood. The field investigation of carbonation problems indicated that the concentration of carbon dioxide beneath pavements can be extremely high (up to 450 times more than the natural concentration of carbon dioxide) and carbonation is much more rapid than was previously considered possible.

The main modes of distress caused by carbonation of stabilised layers are loosening of unsurfaced or primed layers, loss of bond between surfacing and base, increase in plasticity and eventually rutting and potholing as the road structure becomes unable to support the applied loads without permanent deformation.

Numerous tests have been developed, adapted and recommended for the identification of possible durability problems with stabilised materials. Very few, however, have performance-related limiting criteria for differentiating good from bad materials and those which do have been developed overseas and are generally not applicable to local conditions. Recent local research has resulted in an improvement in the results obtained from the wet/dry brushing test and recommended performance-related acceptability limits. These require that the gravel ICL is at least exceeded by one per cent, the mechanical wet/dry brushing loss on

samples compacted at Mod AASHO density does not exceed 8 per cent for base and 13 per cent for subbase and the residual carbonated UCS complies with the C4 and C3 requirements of TRH 4 and 14.

It is not always possible to locate materials which are unlikely to carbonate if stabilised and marginal materials with a high propensity to carbonate are of necessity utilised (especially in rural and remote areas). In these cases means of preventing or at least limiting carbonation should be planned into the construction procedures.

The rehabilitation of carbonation-induced distress is usually a problem. By the time distress caused by carbonation of stabilised bases or subbases is observed the stabilised layer is usually beyond repair. However, distress due to poor curing or delayed construction of stabilised bases or shoulders can be treated before construction of the surfacings.

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