Study of the Occurrence and Effects of Organic Matter in Relation to the Stabilization of Soils with Cement

Étude de la présence et des effets des matières organiques dans la stabilisation du sol par les ciments

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Summary

A study has been made of the occurence and effects of soil organic matter that interferes with the hardening of cement-stabilized soil. It is shown that the pedological classification can be used for estimating the depth of surface soil that contains active organic matter ; this depth ranges from 0 to 5 feet in Great Britain depending to which one of the five major soil groups the soil profile belongs. A " transition zone " was found to occur between zones of high and low organic activity, and calcium chloride was effective in overcoming the effect of organic matter only with soils from this zone.

By extracting and fractionating the organic matter in a soil from a podzol profile it was shown that the organic compounds that effect the hardening of cement were concentrated in a fraction that was rich in compounds of the hydroxy-quinone type.

The effect of organic matter is known to be closely associated with its ability to combine with calcium ions liberated by the hydrating cement. A test based on the measurement of the amount of calcium absorbed by the soil was found to be satisfactory for sandy soils but unsuitable for clay soils due to the difficulty of separating the amounts of calcium removed respectively by the clay fraction and by the organic matter. A generally satisfactory test for detecting the presence of active organic matter was found to be to measure the pH of a soil-cement paste during the early stages of hydration. The normal hardening of soil-cement did not occur when the pH of the soil-cement paste fell below 12·0 in the conditions of test. This effect was reflected in a relation that was obtained between the pH of the soil and the unconfined compressive strength of the cement-stabilized soil. Alkaline soils were found to be generally more suitable for stabilization than acidic soils.

Sommaire

Les auteurs ont étudié les conditions dans lesquelles on rencontre dans le sol des matières organiques qui s'opposent au durcissement du sol stabilisé au ciment et les effets qu'elles produisent. Ils démontrent qu'on peut se servir de la classification pédologique pour estimer la profondeur du sol superficiel qui contient des matières organiques actives ; en Grande-Bretagne cette profondeur varie entre 0 et 5 ft., selon le groupe auquel appartient le profil du sol (il y a cinq groupes principaux).

Il y a entre les zones de haute et basse activité organique une " zone de transition " ; le chlorure de calcium peut s'opposer à l'effet des matières organiques dans les sols de cette zone seulement.

L'extraction et le fractionnement des matières organiques d'un sol avec un profil podzolique ont démontré que les composés organiques qui influencent le durcissement du ciment étaient concentrés dans la fraction riche en composés du type hydroxy-quinone.

On sait que l'effet des matières organiques est intimement lié avec leur capacité de se combiner avec les ions de calcium, libérés par le ciment en cours de prise. On a constaté qu'un essai basé sur la mesure de la quantité de calcium absorbé par le sol convient aux sols sableux mais pas aux sols argileux, à cause de la difficulté de séparer la quantité du calcium enlevé par l'argile et par les matières organiques lors du fractionnement.

Un essai en général satisfaisant pour démontrer la présence des matières organiques actives, est la mesure du pH d'une pâte de sol-ciment pendant les premières phases de l'hydratation. Le durcissement normal du sol-ciment ne se produit pas quand le pH de la pâte de sol-ciment descend au-dessous de 12,0 dans les conditions de l'essai. Cet effet se reflète dans le rapport obtenu entre le pH du sol et la résistance à la compression sans contrainte latérale du sol-ciment. On a constaté que, en général, la stabilisation convient mieux aux sols alcalins qu'aux sols acides.

Introduction

In Great Britain the suitability of soils for stabilization with Portland cement is determined largely by their freedom from those forms of organic matter that are able to interfere with the normal hydration of the cement. Research has been carried out :

(*i*) To determine the soil conditions and depths below the ground surface in which such active organic matter is present.

(*ii*) To obtain a better understanding of the chemical reaction by which organic matter interferes with the hydration of cement and to identify the types of compound responsible.

(*iii*) To develop a diagnostic test for soils containing active organic matter.

Investigation of soil conditions in which active organic matter is present

Since the pedological classification of a soil profile is related to the chemical composition of the surface layers of soil, an investigation was made to determine whether the interference with the hardening of soil-cement was related to the type of profile from which the soil was obtained. In Great Britain the principal pedological groups are the pozdols, brown earths, gleys and calcareous. A number of profile samples were obtained from each group and the compressive strengths of cylindrical specimens at an age of 7 days were determined according to the British Standard procedure (BRITISH STANDARDS INSTITUTION, 1957) for each sample when stabilized with 10 per cent of ordinary Portland cement. The results of this investigation showed that although the relations between the strength of soil-cement and sample depth differed as between different profiles in the same pedological group, the differences were much greater for the relations for profiles taken from the different pedological groups. Typical results for each group (Fig. 1) show that satisfactory hardening of the soil-cement (defined here as a minimum compressive strength at 7 days of 250 lb./sq. in.) was obtained for all depths in the high base status (alkaline) brown earth profile and at all except at the surface for the calcareous profile. With the low base status (acidic) brown earth, gley and podzol profiles, on the other hand, satisfactory soil-cement strengths were obtained only with samples taken from some depth ranging from 15 in. with the gley to as much as 3 ft. or more with the low base status brown earth and podzol. These results are of particular significance in relation to mix-in-place construction, for which it is possible to predict from a knowledge of the pedological group the probable depth at which soil chemically suitable for cement stabilization will be located.

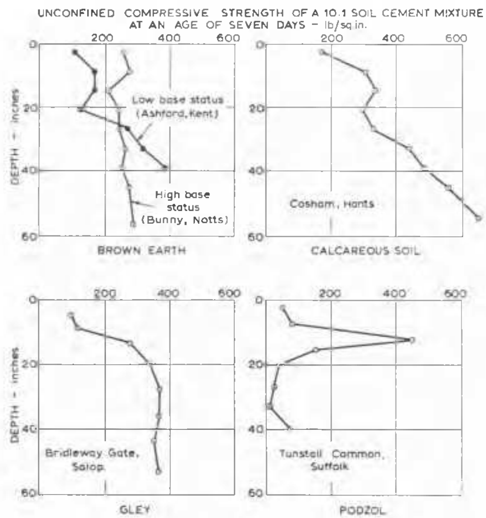

Fig. 1 Typical strength-depth relations for soils from four major soil groups.

Relation type entre la résistance et la profondeur du sol pour quatre des principaux groupes de sols.

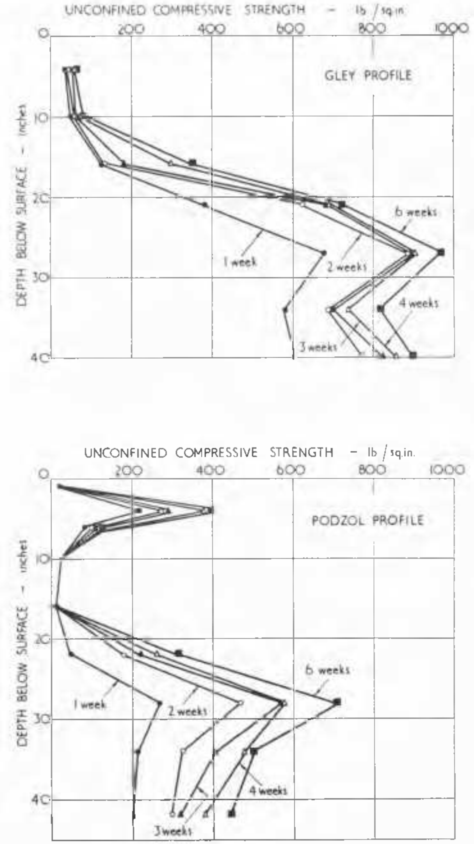

Fig. 2 Strength-depth relation after different intervals of time for soils from podzol and gley profiles stabilized with 10 per cent of ordinary Portland cement.

Rapport entre la profondeur et la résistance à des intervalles de temps différents pour des sols des groupes podzol et gley stabilisés avec 10 pour cent de ciment Portland.

Further tests were made in which the strength/age relations were determined for cylindrical specimens made by stabilizing with 10 per cent of Portland cement soil samples taken from different depths in a number of soil profiles. Fig. 2, which shows the results for a gley and a podzol profile, illustrates two general findings. First, soils which failed to harden after 7 days also failed to reach a satisfactory strength after 42 days and secondly, soils immediately above those which gave satisfactory soil-cement strengths, had low soil-cement strengths at 7 days but satisfactory strengths at 42 days. In Fig. 2 this " transition zone ", as it has been described, occurred around a depth of 16 in. in the gley profile and a depth of 22 in. in the podzol profile.

It is well known that the use of calcium chloride as a secondary additive with Portland cement sometimes enables satisfactory hardening to be obtained with organic soils. Experiments were made to determine the effect on the strength of specimens made from profile samples stabilized with 10 per cent of Portland cement of admixing 1 and 2 per cent of calcium chloride. Other tests were made to determine the effect of increasing the cement content from 10 to 15 per cent. For soils which did not harden with 10 per cent of cement even after 42 days no increase in strength was obtained with either treatment, but for soils from the transition zone adequate soil-cement strength at 7 days were obtained by either the addition of the calcium chloride or the increase in cement content. This indicates that calcium chloride is of value for improving the cement stabilization of organic soils only from a limited part of the soil profile, where its function appears to be that of accelerating the reaction of the cement. In another experiment it was shown that 2 per cent of hydrated lime had no effect in improving the strength of soil-cement irrespective of the position in the profile from which the soil was taken.

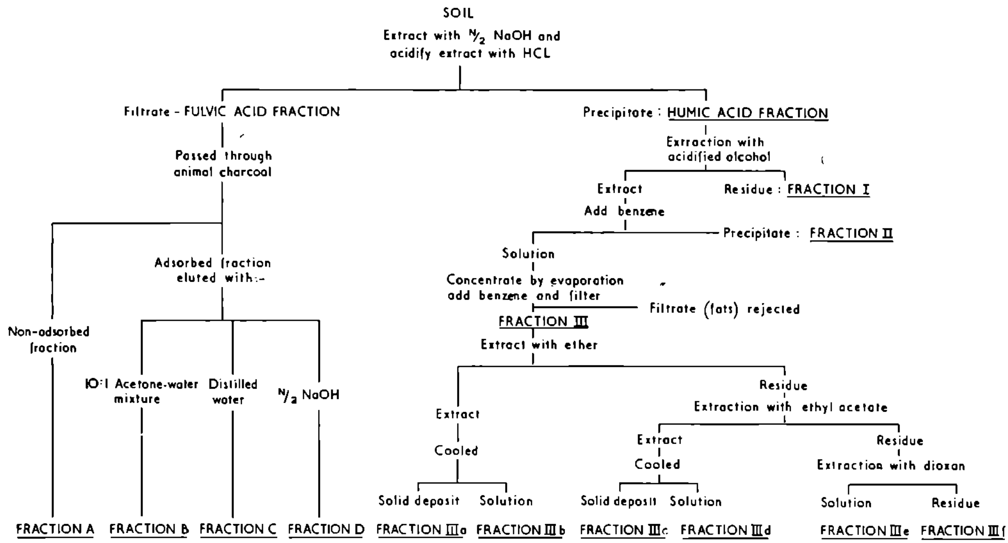

Fig. 3 Procedure for extracting and fractionating soil organic matter.
Processus d'extraction et de fractionnement de la matière organique contenue dans le sol.

Requirement for diagnostic test for active component of soil organic matter

In spite of the usefulness of the pedological system of classification it would clearly be an advantage to have a reliable diagnostic test for soils containing active organic matter.

Measurement of the total amount of organic matter present is only of limited value since, although it has been shown that British soils that have organic contents in excess of 2 per cent are unsuitable for stabilization with soils containing less than 2 per cent of organic matter it is the type rather than the total amount of organic matter which influences the hydration of cement. Research has therefore been directed to the development of such a test, and this has involved investigations to determine the types of organic compound responsible for the interference with the hardening of cement and to obtain a better understanding of the chemical reactions that take place. It was anticipated that a knowledge either of the type of compound or of the type of reaction would provide a good indication of the form of diagnostic test required.

Separation and identification of the active component of soil organic matter

The first line of approach was studied by Dr. V. Arkley at the Road Research Laboratory. He used the procedure for extracting and fractionating soil organic matter shown schematically in Fig. 3. The soil chosen for study was taken from the horizon in a podzol profile known to contain the highest proportion of active organic matter. The proportions of each fraction of the organic matter separated from this soil are shown in Table I.

It was found that extraction with sodium hydroxide removed approximately 60 per cent of the organic matter present and that the organic matter remaining had no effect on the hardening of the cement. The effect of the fractions isolated from the soil on the setting of ordinary Portland cement was established by mixing them with an organic-free sand, and determining the compressive strength at an age of 7 days of the organic-contaminated sand when stabilized with 10 per cent of Portland cement.

The results (Table I) showed that most of the fractions extracted reduced the strength obtained with cement-stabilized organic-free sand but that fraction III *d* was the most active and was probably chiefly responsible for the low strengths obtained when this soil was stabilized with cement. It is of course probable that in the organic matter contained in other soils some of the other fractions might be more active.

A more detailed study was made of this fraction with the object of determining its chemical constitution. The main conclusion was that the active component comprised a group of chemical compounds having molecular structures which had one portion of their structure that was common to all, but which differed structually in other respects. Evidence was obtained that the common portion of the structure resembled that of compounds of the hydroxy-quinone type. Hydroxy-quinones are known to form complexes with metal ions such as those of calcium and, as is discussed later in this paper, this property is believed to be the cause of the activity of certain organic compounds towards cement.

Investigation of the calcium absorption capacity of soils

In the search for a diagnostic test based on the reaction between organic matter and cement, consideration was first given to the fact that interference with the hardening of cement is invariably associated with the ability of the active organic matter to form compounds with calcium. An investigation was therefore carried out to determine whether it was possible to detect the presence of active organic matter by measuring the amount of calcium that is immobilized by soil. This is called here the calcium absorption capacity and is determined by finding the amount of calcium removed fom a saturated solution of calcium hydroxide when it is shaken up with the soil.

Table I

Composition of organic matter extracted from the soil and the effect of the fractions on the hardening of cement

Fraction	*Percentage of each fraction present*		*Amount added to 10:1 sand-cement mixtures (%)*	*Compressive strength reduction of sand-cement mixtures (%)*
	in soil organic matter extracted	*in the soil*		
Total fulvic acid	25	0·3	0·5	30
A	5	0·06	0·2	20
			1·0	50
B	5	0·07	0·1	10
			1·0	100
C	2	0·02	0·05	15
			1·0	100
D	13	0·15	0·1	20
			1·0	100
Total humic acid	75	0·9	1·3	75
I	26	0·32	1·0	12
II	25	0·30	1·0	10
III a-f	24	0·28	1·0	80
III a	2	0·02	0·25	0
			0·5	100
III b	1	0·01	0·25	0
			0·5	100
III c	2	0·02	0·25	30
			0·5	100
III d	7	0·09	0·25	85
			0·5	100
III e	9	0·10	0·25	20
			0·5	100
III f	3	0·03	0·25	15
			0·5	15

The calcium absorption capacity of 45 soils was measured and plotted against the unconfined compressive strengths at an age of 7 days of the soils when stabilized with 10 per cent of ordinary Portland cement. The results (Fig. 4) for the 25 sandy soils (containing less than 10 per cent of material passing the B.S. No. 200 sieve) that were studied showed that, where the calcium absorption capacity of the soil exceeded 80 mg of calcium/100 g of soil, the unconfined compressive strength at 7 days was invariably below 100 lb./sq. in. When the calcium absorption capacity was lower than 80 most of the samples gave strengths greater than 250 lb./sq. in. at 7 days. However, with the soils having a significant clay fraction no relation was found between the calcium absorption value and the strength of soil-cement. It was clear that in this case absorption of calcium by the clay fraction due to a base-exchange reaction was taking place in addition to the absorption of calcium by organic matter and that the former effect had no significant effect on the hardening of the soil-cement. An attempt was therefore made to develop a method for correcting for the absorption of calcium by the clay fraction. This consisted of measuring the calcium absorption capacity of the soil before and after treatment with 30 per cent hydrogen peroxide. Treatment of the soil with hydrogen peroxide followed by washing with water removes most of the organic matter and it was hoped that the difference in the calcium absorption before and after this treatment would give a measure of the absorption capacity of the organic matter alone.

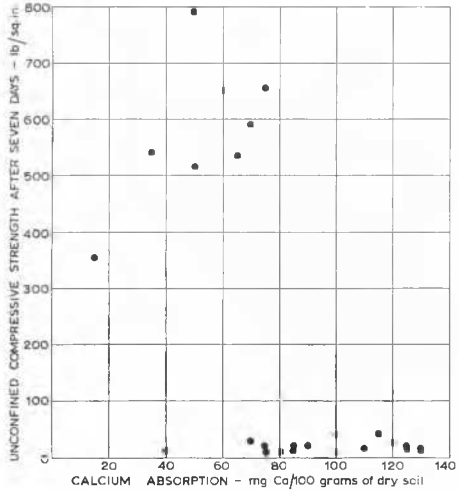

Fig. 4 Variation of unconfined compressive strength of sand-cement with the calcium absorption of the sand (cement content = 10 per cent).

Variation de la résistance à la compression simple du sable-ciment avec le taux d'absorption du calcium par le sable.

Investigations showed, however, that the calcium absorption capacity of some soils after treatment with peroxide was greater than it had been before pretreatment. The results obtained for the soils from one soil profile are given in Fig. 5, while Fig. 6 gives the clay contents and organic contents of the same soils. This increase in calcium absorption capacity after pretreatment with peroxide could be due partly to the incomplete removal of organic compounds by treatment with hydrogen peroxide followed by washing with water. The organic matter remaining could acquire, as a result of oxidation, a greater affinity for calcium than before treatment with peroxide. Another possibility is that the clay and organic matter present in a soil are partially bound, and removal

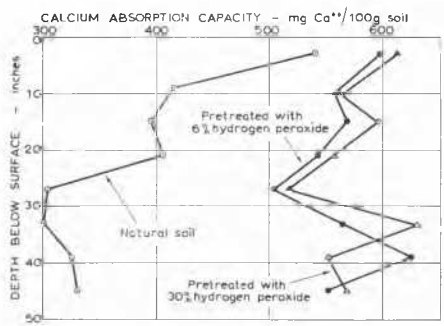

Fig. 5 Variation with depth of the calcium absorption capacity of soils from a podzol profile.

Variation du pouvoir d'absorption du calcaire par un sol du groupe podzol.

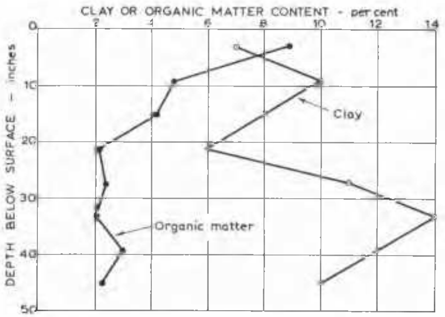

Fig. 6 Variation with depth of the clay content and organic content of soils from a podzol profile.
Variation avec la profondeur de la teneur en argile et en matières organiques de sols du groupe podzol.

of the organic part of the clay-organic matter complex would leave positions on the clay particle that are able to accommodate extra cations. It is possible that both these effects combine together to produce the increase in calcium absorption capacity that was noted.

It was concluded that a test for the calcium absorption capacity of soil was limited in its application as a diagnostic test for the presence of active organic matter to soils free from a clay fraction.

Effect of the pH of soil-cement mixtures on the unconfined compressive strength

Because of the serious limitation of the calcium absorption test, attention was directed to the possibility of using, as the basis for a diagnostic test, the reduction in the pH of soil-cement due to the presence of organic matter in soil. The reduction in pH results from the absorption of calcium from the cement by the active organic matter. According to FORSEN (1938) the effect of the reduced pH is to cause the precipitation of an alumina-silica gel over the cement particles, thus inhibiting the normal hardening process.

To find whether there was a relation between the unconfined compressive strength of a cement-stabilized soil and its pH value, samples of cement-stabilized soil were taken immediately after the determination of the unconfined compressive strength test, and the pH values of suspensions of the soil-cement in water were measured. The relation obtained showed that soil-cement with strengths greater than 250 lb./sq. in. at an age of 7 days invariably had pH values above 12·0, while soil-cement not attaining a pH value of 12·0 had strengths below this value. However, the relation was limited by the fact that several soil-cement mixtures with strengths below 250 lb./sq. also had pH values above 1·20.

The determination of the pH of a hardened soil-cement does not afford a rapid means of determining the presence of active organic matter in soils, and further investigations were therefore made to determine whether the pH of the soil-cement in the early stages of hydration was related to the hardening process.

The factors, apart from those under investigation, which might be expected to affect the pH a soil-cement mixture are :

(a) Time ;
(b) Temperature ;
(c) Moisture content ;
(d) Cement content.

The effect of each of these factors was investigated and it was found that the pH decreased with increase in temperature and moisture content and increased with increase in time and cement content (Figs. 7 *a-d*).

The effect of variations in temperature in the range 15-25° C in which pH measurements are normally made was sufficiently small to be ignored and the effect of moisture content was also unimportant provided there was not too much water present to cause dilution effects. The most consistent results were obtained when sufficient water was added to the soil-cement to form a thick paste.

The effect of cement content and time were too large to be ignored and these factors were standardized by using a cement content of 10 per cent throughout and by taking the pH value of the soil-cement paste exactly 15 minutes after the addition of water.

To determine whether a relation existed between the pH of a soil-cement paste at an age of 15 minutes and the unconfined compressive strength at an age of 7 days, 80 soils were taken representing all the major pedological soil groups found in Great Britain and varying in texture from sands to heavy clays. The unconfined compressive strengths of these soils when stabilized with 10 per cent of ordinary Portland cement were determined and the pH of the soil-cement mixtures (prepared by weighing out 25 g of soil, 2·5 g of cement and adding

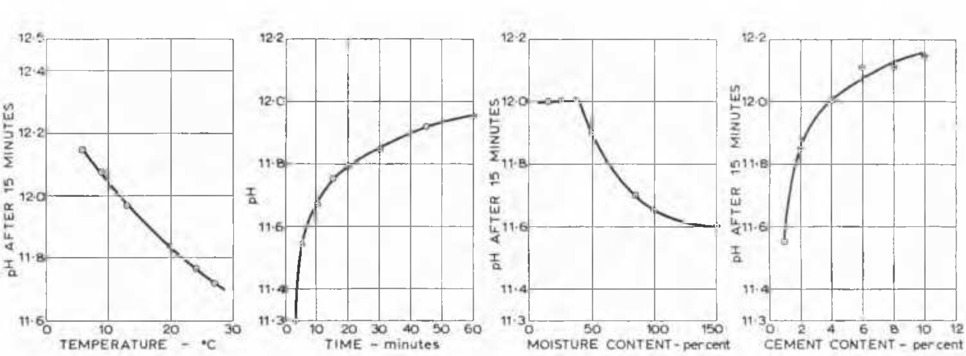

Fig. 7 Effect of temperature, time, moisture content and cement content on the pH of soil cement mixtures.
Effet de la température, du temps, de la teneur en eau et en ciment sur le pH de mélanges sol-ciment.

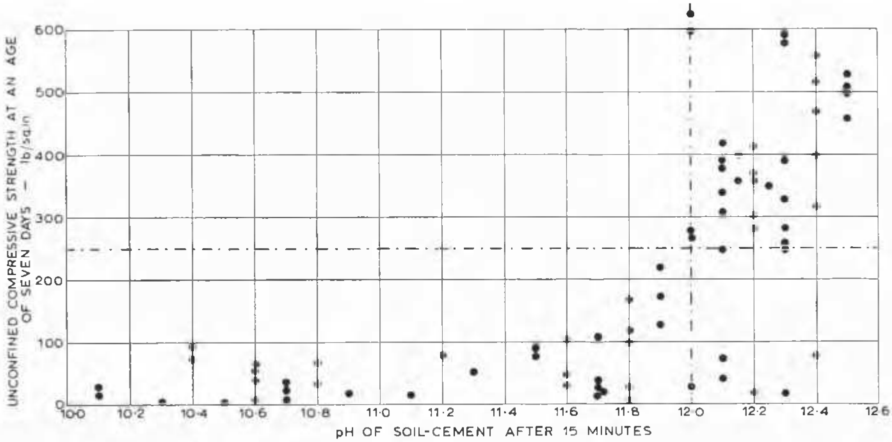

Fig. 8 Relation between pH and un confined compressive strength of 10/1 soil cement mixtures.
Relation entre le pH et la résistance à la compression simple de mélanges sol-ciment à 10 pour cent de ciment.

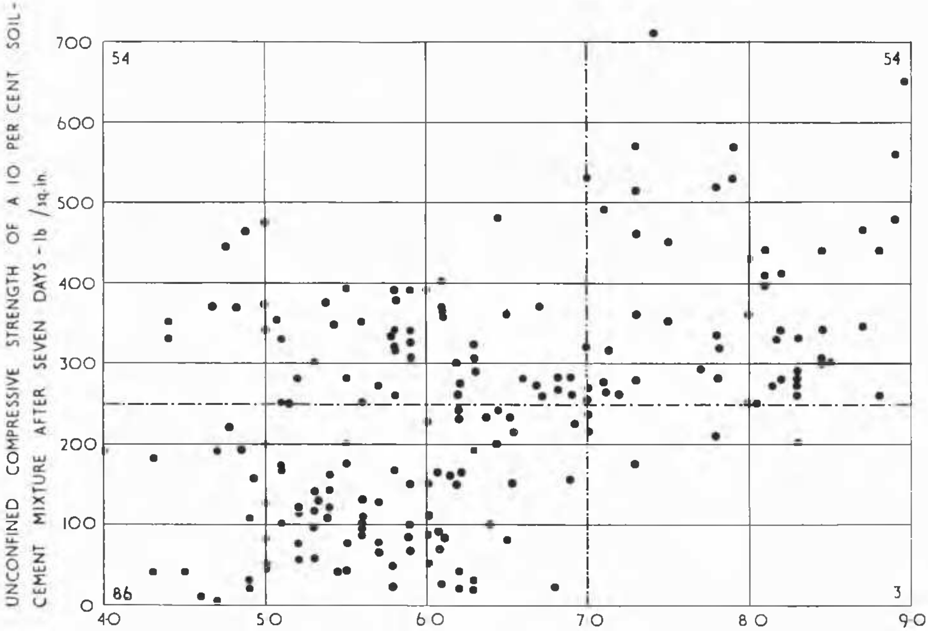

Fig. 9 Effect of soil pH value on the unconfined compressive strength of soil-cement mixtures containing 10 per cent of ordinary Portland cement.
Effet du pH du sol sur la résistance à la compression simple de mélanges sol-ciment à 10 pour cent de ciment Portland.

sufficient water to make a thick paste) was measured exactly 15 minutes after the addition of the water. The relation obtained between the unconfined compressive strength of the soil-cement and the pH value is shown in Fig. 8.

Assuming that adequate hardening of cement-stabilized soil is defined by a minimum unconfined compressive strength of 250 lb./sq. in. at an age of 7 days, then Fig. 8 shows that of the 40 soil-cement mixtures with pH values after 15 minutes of 12·0 or greater, 34 would be considered as satisfactory for use. The remaining six were mixtures made from soils from the transition zone, and these hardened satisfactorily after 42 days. All soil-cement mixtures with pH values below 12·0 would be regarded as unsatisfactory since they all had unconfined compressive strengths of less than 250 lb./sq. in. at 7 days and, in fact, 80 per cent of them had strengths of less than 100 lb./sq. in. The test seems therefore to be a reliable method of indicating whether or not the soil contains active organic matter that interferes with the normal hardening of the cement.

The pH ultimately reached by soil-cement is influenced by the pH and the buffering capacity of the natural soil. A relation should therefore exist between the pH of the soil and the unconfined compressive strength of the cement-stabilized soil mixture. In Fig. 9 the pH — unconfined compressive strength relation for nearly 200 soils shows this to be the case. Soil pH values above 7·0 were nearly always associated with unconfined compressive strengths of soil-cement greater than 250 lb./sq. in., but for soil pH values below 7·0, however, both high and low soil-cement strengths were obtained.

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