THE DURABILITY OF CEMENT BOUND MINESTONE

BY

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Summary

The demand for road making materials continues to pressurise the supply of traditional good quality aggregates. Over the years, therefore, consideration has been given to alternative materials including industrial wastes. This thesis is concerned with the potential use of Minestone, the by-product of coal mining, for the lower structural layers of pavement construction. Because of their clay like nature, Minestones do not merit consideration for such applications in an unbound state and, therefore, some form of stabilisation is necessary.

Previous research has demonstrated that certain cement bound minestones, containing between 5 and 10 per cent cement, satisfy current Department of Transport requirements for use in pavement construction and, furthermore, they are not frost susceptible. However, doubts concerning the durability of cement bound minestones still remain.

The thesis includes a review of both the cement and lime stabilisation techniques and also traces the origin and development of the methods used to assess the quality and durability of stabilised materials. An experimental study is described in which cement bound minestone specimens were subjected to a programme of tests which examined compressive strength, resistance to immersion, and resistance to freezing and thawing. The results of the tests were related to the properties of the raw materials. It was discovered that the response to cement stabilisation was governed mainly by the source of the minestone and, to a lesser degree, the cement content. It was also found that resistance in the durability tests was generally improved when the initial moisture content was raised above the optimum value.

The results suggest that current methods for assessing cement stabilised materials are not appropriate to cement bound minestones. Alternative methods and criteria, based on volume change and retained strength following immersion and freeze-thaw tests, have been proposed. It is believed that these methods and criteria should also apply to other cement bound materials.

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Keywords

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CHAPTER 1 INTRODUCTION

The supply of traditional road making materials is continually being pressurised by the demands of the construction industry. In certain parts of the country, notably the south eastern counties, naturally occurring rock is in short supply and good quality aggregates for road construction have to be imported from areas many miles away. Over the years, consideration has been given to the use of alternative materials including industrial wastes. This approach has the advantages of:-

(a) conserving the supply of good quality aggregate for particularly demanding applications;

(b) complying with the environmental need to identify methods of disposal for industrial waste.

The Minestone Executive was established by the National Coal Board in 1971, its aim being to "locate, identify, and exploit the non-coal mineral" (1) associated with the mining operation. Its responsibilities include the exploitation of colliery wastes, which comprises burnt colliery spoil and unburnt colliery spoil, the latter having been renamed Minestone. The efforts of the Minestone Executive have been encouraged by the report of The Advisory Committee on Aggregates (2) which included the following recommendations:-

1. To discourage unnecessarily high standards, The Department of the Environment should publicise the results of studies on use of fine materials and

poor quality aggregates and draw attention to the benefits to be gained from using such materials.

2. While there should be no reduction in the standard of safety and performance required, British Standards, Codes of Practice and Departmental Specifications should actively encourage the use of new and tested waste materials.
3. The Department of the Environment should alter the Road and Bridge Specification to acknowledge that in many cases locally available alternative materials, which may not be nationally recognised, can be suitable for use in road construction.

Minestone and burnt spoil have many applications within the engineering and construction industries (1), however, the major outlet has been, and continues to be, the highway engineering industry. Burnt spoil has been used extensively for many years but, because of changes in spoil disposal practices, stocks are limited. Thus, it is Minestone that the Minestone Executive can offer as an abundant supply for construction schemes.

This material is now accepted as a suitable fill material for the construction of road embankments. Whilst the bulk outlet will continue to be as fill, a more limited and more demanding application is in the pavement sub-base and, possibly, road base. It is generally acknowledged that minestones, because of their clay like nature, do not merit detailed consideration in an unbound state and that cement stabilisation is essential. Proven durability is of fundamental importance if cement bound minestones are to be accepted for pavement construction. Consequently it is this aspect of the material which has been studied in this particular investigation.

CHAPTER 2 COLLIERY SPOILS AND THEIR USE IN HIGHWAY CONSTRUCTION

2.1 Minestone and Burnt Spoil

Minestone, often referred to as unburnt colliery spoil or shale, is an unavoidable by-product of coal mining. The material comprises (3) the following fractions:

(a) Coarse discard from the washery, where the coal is separated from the accompanying rock.

(b) Fine discard, produced either by degradation during mining and transport of coal and non-coal materials, or as tailings from the final process to separate the fine coal. This fine material often contains appreciable quantities of coal, although modern washeries now remove much of this fine coal.

(c) Materials removed from shafts and drifts to provide access to the coal seams.

Minestone is grey or black in colour and is composed of the rocks surrounding the seams, mainly siltstone, mudstone, shale and seatearths, although it may include sandstone, limestone, and other rocks (4). The bulk of this material is tipped onto spoil heaps which cause widespread dereliction of the landscape. Although the composition of minestone is largely governed by the geographical location of the mine, variation within a particular spoil heap is inevitable.

Older spoil heaps were loosely compacted and often contained significant quantities of coals. These heaps were often used for tipping other industrial wastes such as ashes, and so they could become readily ignited with the burning being sustained by the free flow of air throughout the heap (5). This resulted in the formation of burnt spoil with its characteristic red colour. The burning process changes the physical and chemical composition of the spoils and hence the engineering properties of burnt spoil differ from those of minestone (6). The stocks of burnt spoil are, generally, of pre-war origin and, because of changes in spoil disposal practices, will not be added to (7).

It is estimated that approximately 2000 million tonnes of minestone have been deposited on spoil heaps throughout Great Britain and that the stocks are increasing by 50 million tonnes per year (8). Annual usage of minestone has been as high as 7 million tonnes (3). The major user has been, and remains, the highway engineering industry.

2.2 The Present Use of Colliery Spoils in Highway Construction

2.2.1 <u>Burnt Spoil.</u> The Department of Transport Specification (9) permits the use of burnt spoil, complying with the relevant grading, physical and chemical requirements, as fill, granular sub-base material, and as cement stabilised material. This implies that it may be used at all levels of road construction up to, and including, the road base, though further restrictions apply to cement bound spoil on heavily trafficked roads (10).

Burnt spoil is also classed as a selected fill for use in "capping layers" as defined in Technical Memorandum No H6/78 (11).

2.2.2 Minestone. The Specification (9) permits the use of minestone as fill. In the past its use had been restricted because of fears that it was susceptible to spontaneous combustion but experience has shown (12) that minestone, compacted in accordance with the Specification (9) is not liable to such burning.

Minestone is not included in the Specification (9) list of permitted granular sub-base materials. Furthermore, it is generally acknowledged that, because of the moderate to high plasticity of the fine fraction, minestones do not merit consideration for such applications in an unbound state.

2.3 The Use of Cement Bound Colliery Spoil in Highway Construction

2.3.1 Burnt Spoil. Many burnt spoils have sufficient strength in themselves to be employed as granular sub-base materials provided they comply with grading requirements. However, material within 450 mm of the surface is required to be resistant to frost, as defined by the Transport and Road Research Laboratory Frost Heave Test (13), and since many burnt spoils are not frost resistant (14), they are excluded from such applications. The addition of small quantities of cement reduces heave to acceptable limits (15) and it has been suggested that such cement modified materials are suitable for incorporation at sub-base level (14). It should be noted, however, that the present specification (9) requires cement bound materials to meet a minimum strength requirement which is not necessarily achieved at the cement content that produces a satisfactory level of resistance.

As already mentioned, burnt spoil may be used as an unbound material in a Type 2, granular sub-base (9). However, it is usually considered to be frost susceptible and so may be stabilised with cement for use as sub-base material. Similar treatment may be considered when the grading curve of the burnt spoil does not satisfy the requirement for Type 2 material given in the Department of Transport Specification (9). Indeed the Specification (9) only permits the use of burnt spoil at road base level when stabilised with cement.

Clause 805 of the Department of Transport Specification (9), which details the requirements for soil-cement, specifically mentions well burnt shale. Clause 806, relating to cement bound granular material, makes no mention of burnt spoil.

2.3.2 <u>Minestone.</u> A number of investigations (16,17,18) have demonstrated that selected minestones, stabilised with 5-10 per cent cement, satisfy the current strength requirements (9) for cement stabilised materials. Furthermore, these cement bound minestones were not frost susceptible (16) as defined by the Frost Heave

Test (13) which suggests that they may be used within 450 mm of the road surface. However, neither Clause 805, soil-cement, or Clause 806, cement bound granular material, make any mention of minestone or unburnt spoil. It would, therefore, appear that cement bound minestone is excluded from the sub-base and road-base of pavements constructed to the Department of Transport Specification (9).

The National Coal Board has considerable experience both in the production of cement bound minestone, and the construction of pavements incorporating the material (19). Cement bound minestone has been used, satisfactorily, both by the National Coal Board, for in-house construction (20) and by commercial organisations, for the construction of car parks, hard standings, sub-bases and road-bases (1). However, attempts to gain the confidence of the highway engineering fraternity have been severely frustrated by the rapid deterioration of the cement bound minestone sub-base during the construction of the Canterbury By-Pass (21). This contract would have represented the first major permanent road pavement to incorporate cement bound minestone, however, following a period of heavy rainfall, the construction joints were observed to "blow-up". The disruption was attributed to swelling which appeared to be associated with an increase in the moisture content (21). In order to complete the contract, it was decided to abandon cement bound minestone and revert to Type 1 granular material.

2.4 The Future Utilisation of Colliery Spoils in Highway Construction

2.4.1 Burnt Spoil. Burnt spoil is a well established road making material which satisfies the requirements for inclusion up to road-base level. As the stocks of burnt spoil are limited, it is the policy of the National Coal Board that, so far as possible, the remaining stocks of quality burnt spoil should be conserved for premium use, ie. sub-base etc. rather than for fill (7).

2.4.2 Minestone. The bulk outlet for minestone will continue to be as fill material. Despite the set-back at Canterbury, the National Coal Board continues to actively promote the use of cement bound minestone in the sub-base and road-base of in-house haul roads, as well as in the structural layers of hard standings, and car parks (1,20). It is hoped that the increased use of the material within the mining industry will encourage wider interest from outside authorities (20). However, in the light of Canterbury, doubts concerning the durability of cement bound minestone still present a major hurdle to its acceptance as sub-base and, possibly, road-base material for the construction of public highways.

CHAPTER 3 CEMENT AND LIME STABILISATION

3.1 Introduction

The purpose of adding cement or lime to a material to be incorporated in a pavement is to improve its load bearing quality and ensure that this improvement provides a durable material that will be resistant to the traffic and the local environment. This technique of soil improvement is termed cement or lime stabilisation. To gain an understanding of the mechanisms and chemical reactions involved in the two processes it is necessary to review the techniques.

3.2 Cement Stabilisation

The original concept of stabilising soils with cement is attributed to trials on Salisbury Plain in 1917 (22) but it was not until 1932, when the South Carolina State Highway Department began investigations into the mixing of soil and cement for "low cost, all weather roads" (23), that the technique was used on a substantial scale. The demand for airfields during the Second World War, followed by a vigorous highway construction programme in the post war years, stimulated interest in Great Britain and promoted research into the stabilisation of soil, mainly with cement.

Early applications in Great Britain were with gravels, sands, and light clays (24). It was intended that the process should be employed in situations where stabilisation of the in situ material would economise on borrow material and haulage costs (22). The plant, which was derived from agricultural machinery such as rotavators (25), was inefficient and could not keep pace with the rapid expansion of the highway network. With the development of lean concrete, which offered ease of construction, the use of traditional plant, and good quality control (26), the use of cement stabilised soil declined. The rapid failure of the cement stabilised sand road-base used in the experimental pavement constructed in 1957 at Alconbury Hill (27) did nothing to encourage the development of cement stabilisation. Recently, however, interest has been regenerated. The increasing cost of haulage, the price of processed crushed rock and graded materials, the increased awareness of the public to the environmental damage of borrow pits, quarries etc. has made cement stabilisation a more competitive alternative (28).

Many different materials may be stabilised with cement and, consequently, the mode and extent of the improvement varies considerably. However, three major groups may be defined into which the various materials may be divided.

3.2.1 Non-cohesive Materials. This group comprises the clean granular materials such as sands and gravels. The size of the individual grains is larger than that of the cement grains, and so it is possible to, at least, partially coat them with cement paste. In the compacted pavement layer the coated particles are forced into close contact and bonded by the hydration products. The apparent cohesion of the material contributes to its ability to resist deformation by traffic or environmental stresses. The improvement of these materials is a function of the strength of the bonding compounds, or mortar, and the overall area of bonding. It is, therefore, dependent upon the grading of the material (26). This structure is acknowledged by the current specification (9) which defines the grading limits for "cement-bound granular materials".

3.2.2 Mixtures of Non-cohesive and cohesive. This group contains most natural soils which are invariably mixtures of clay, silt and sand possibly with gravel. At one end of the group are the gravel-sands and sands containing small quantities of silts and clay, their grading often resulting in a better stabilised material than a clean sand (26). As the clay content increases its influence on the reaction of soil with cement becomes increasingly important. When mixed with cement, materials with significant amounts of clay react more like cohesive soils.

3.2.3 Cohesive Materials. The cement stabilisation of cohesive soils is more complex than that of clean materials and, as such, has attracted considerable attention from researchers. The particles in the finer fraction of these soils are smaller than the individual cement grains and so the structure of clay-cement is considerably different from that of sand-cement mixtures.

Lilley (29) has described a clay cement structure as consisting of aggregations coated with cement and, with compaction, the adjacent aggregations are compressed together to form a skeleton of cement paste. Following hydration the strength of the structure is dependent upon the strength and extent of this skeleton and the size of the aggregations. Another skeleton matrix structure has been described by Herzog (30), in which the cement grains are suspended in the clay and hydrate individually. As hydration proceeds, needles develop from the grains and consist of secondary cementitious products derived from reaction between liberated lime and clay minerals. Providing the concentration of cement grains is sufficiently high, the protruding arms will intermesh to form the skeleton.

Both hypotheses suggest that the strength of clay-cement mixtures increases with decreasing aggregation size or increased pulverisation. During an investigation of the effect of pulverisation on the quality of clay cement, Grimer and Ross (31) observed that both strength and resistance to immersion increased with decrease in percentage of aggregation less than 5 mm. The nature of the clay-cement structures, proposed by Lilley and Herzog, envisage a continuous skeleton surrounding aggregations or "domains" of clay. They will, therefore, require higher cement contents than required to stabilise a well-graded sand where the cement matrix is not necessarily continuous.

3.3 The Influence of Soil Mineralogical Composition on Cement Stabilisation

The mineralogical composition of a given soil is an important factor in its effective stabilisation with cement. Extensive investigations (32,33,34), using the electron microscope, x-ray diffraction, and differential thermal analysis, to study pozzolanic-cement reactions, have identified the more important mineral cement reactions. Herzog and Mitchell (32) and later Mitchell and El Jack (33) demonstrated that, under certain conditions, secondary reaction products are formed from the reaction between lime, released during hydration of the cement, and the clay minerals kaolinite and montmorillonite. Moh (34) has presented evidence of the secondary reaction products formed from the reaction of cement with quartz, although the extent of this reaction is negligible when compared to that of kaolinite and montmorillonite.

A study of the hydration process of clay cement and the resulting changes in physical properties (35) has provided valuable information on the role and effectiveness of various clay minerals in cement stabilisation. The conclusions drawn from this study are summarised in section 3.3.1 and 3.3.2 below.

3.3.1 Mineralogical Changes in Clay-Cement Mixtures. The reaction between kaolinite and lime is slow and consequently hydration and hardening proceed irrespective of the presence of the clay mineral. After long curing periods, all liberated lime is consumed either to form secondary cements or by the hydration process. Well crystallised illite can be assumed to be inert to lime, with the process of hydration and hardening being unaffected by its presence. Poorly crystallised and degraded illite will behave similar to mix layered clays.

Expansive clay minerals such as montmonillonite are highly reactive with the liberated lime and will result in a lime depleted environment. The result is a reduction in the pH of the aqueous phase, consequently the hardening of gelatinous material and the crystallisation of new minerals is retarded. Both the primary and secondary cementitious products in montmorillonite-cement mixtures are inferior to those produced by mixtures containing non-expansive minerals.

The action of mixed layered minerals is dependent upon their activity. Highly expansive clay minerals behaving similar to montmorillonite, with the less expansive being more like kaolinite and illite.

3.3.2 <u>Changes in the Physical Properties of Clay-Cement Mixtures.</u> The addition of cement to clays has been shown (35) to produce only slight changes in the compaction characteristics. With kaolinite and illite clays the addition of cement results in a small increase in the maximum dry density and a correspondingly small decrease in optimum moisture content, whilst with montmorillonite the opposite effects are observed leading to slightly lower density and slightly higher optimum moisture content.

The strength development of clay-cement mixtures may be predicted from the mineralogical changes described above. The slow lime-kaolinite reaction ensures that sufficient lime is available in the early stages of hydration to promote hardening and thus give higher strength than cement stabilised expansive clays. Cement illite mixtures also give higher strengths than cement stabilised expansive clays. The inferior cementitious compounds produced by the cement-montmorillonite mixtures, results in lower strength values. The quantities of cement required to supply sufficient liberated lime for the secondary cementation and hardening may make cement stabilisation of such soils uneconomic. Lime stabilisation has been shown (36) to be more suitable for such heavier expansive clays.

The action of cement on clay minerals reduces plasticity indices and increases shrinkage limits. This reduction in the activity of clay soils has been attributed (37) to:

(a) a reduction in the electrostatic repulsive charges between adjacent clay particles,
(b) precipitation of gelatinous reaction products on the particle surfaces,
(c) increased effective particle size as the individual particles are cemented together.

After short curing periods all the clays react similarly but, after prolonged curing, the greatest changes have been obtained with the less active kaolinite and illite clays.

This is probably due to the uninhibited hardening of the cement.

3.4 Lime Stabilisation

Lime stabilisation for pavement construction originated in the USA. The earliest trials are believed to have been conducted in 1924 by the University of Missouri (36), although large scale uses were reported (38) during the 1940's when lime was used to modify a caliche gravel during construction of an airfield base. In Africa, lime has been extensively used (38) to stabilise clayey gravels and lateritic soils, and in Germany quicklime has been used (39) as a construction expedient when haul roads through clay bearing loess could not be trafficked due to rain. Although there have been instances in Britain of lime stabilisation, one being the stabilisation of a keuper marl with 8 per cent hydrated lime in 1945 (22), the technique has not been extensively applied.

The fundamental difference between lime and cement stabilisation of soils lies in the production of the cementing compounds. Cement, when added to water, hydrates to produce primary cementitious compounds, independent of the soil. The lime liberated during this hydration may react with constituents in the soil to produce secondary cementitious compounds. With the addition of lime to soil there is no source of primary cementation and, therefore, the strength is derived from reactions between the lime and minerals in the clay fraction of the soil. These reactions may be divided into two groups according to the time elapsed before their benefits are realised.

3.4.1 Rapid Reactions. Most clay soils exhibit cation exchange and flocculation when treated with lime. Multivalent calcium cations, Ca^{++}, introduced as lime, replace monovalent potassium, K^{+}, and sodium, Na^{+}, cations (40). In calcium bearing clays calcium cations are crowded onto clay particles (41). The flocculation is attributed (37) to changes in the electrostatic charges surrounding clay particles. These highly repulsive charges are reduced by high electrolyte contents in the pore water following the addition of lime to a soil. The result is a net attraction, especially between the negatively charged faces and the positively charged edges of the clay particles. This produces flocs which possess a "cardhouse" or "double-T" structure (37). If the electrolyte is removed the repulsive charges are restored so the flocs weaken and are reduced in size. However, it has been observed (37) that when the restoration of low electrolyte levels, ie the removal of lime from the pore solution, is delayed for a few hours, the flocs do not weaken. It was suggested that the immediate reactions at the edge of the particles, between lime and hydrous alumina, to form tricalcium aluminate hydrate, a cementing compound, are sufficient to stabilise the flocs and hold the particles together. This initial reaction will not provide sufficient strength to produce a material for incorporation in a pavement structure.

3.4.2 Long-Term Reactions. Long term reactions between lime and the alumina-silicates, present in the clay, are termed pozzolanic reactions (42). They proceed beyond completion of the rapid reactions described in 3.4.1. The addition of lime to a soil raises the pH of the pore water to approximately 12.4. At pH values in excess of 10.5 the solubility of silica and alumina is increased (43) and so they become available for combination with calcium to form calcium silicate and calcium aluminate hydrates (37). These compounds are poorly crystalline and of inferior quality to the cementing compounds produced by the hydration of cement (37). Calcium aluminate hydrate provides early strength whilst calcium silicate hydrate forms more slowly giving long term strength development (44). Diamond and Kinter (37) have reviewed the various phases and formulations of the cementitious compounds. Laboratory experiments suggest that these reactions require warm rather than temperate climate to ensure that they proceed at an acceptable rate (38).

3.5 Changes in the Physical Properties of Soil-Lime Mixtures

Physical changes in soil-lime mixtures are well documented (38-47). As has been inferred by the groupings of the reactions, two levels of physical change occur, often termed "modification" and "stabilisation".

Modification occurs within a few hours (37) and includes:

(a) increased plastic limit and reduced plasticity indices (41),

(b) apparent reduction in the clay sized fraction of the soil due to flocculation (37),

(c) increased moisture content and increased compactive effort to achieve a given dry density (38),

(d) reduced swell pressure and volume change (45).

The literature is divided on the effect modification has on the permeability of compacted soil-lime mixtures. The permeability of mixtures compacted immediately after the addition of lime is less than that of the compacted soil alone (47). The permeability of mixtures which are allowed to cure in the uncompacted state for up to one week before compaction is greater than that of the compacted soil alone (48,49). This increase in permeability is probably due to the apparent increase in the minimum particle size as flocs, formed within a few hours of the initial mixing, are cemented together by pozzolanic reactions (37) during the curing period preceding compaction.

Hilt and Davidson (41) have defined a "lime-fixation point" as "the amount of lime required to produce maximum modification effects without providing lime for other reactions". They related the lime fixation point to the change in plastic limit and confirmed this with compressive strength tests. The lime fixation point of montmorillonite soils, with their high proportion of exchangeable cations is higher than that of kaolinite or illite soils (41). A similar relationship between optimum lime content for modification and plastic limit was observed by De Sousa, Davids on and Laguros (50).

The stabilisation of a soil by lime relies on the long term reaction which produce cementitious compounds. The reactivity of a soil to long term stabilisation can again be related to the predominant minerals in the clay fraction. Hilt and Davidson (41) observed that increases in strength are greater for montmorillonitic soils than for kaolinitic or illitic soils. In their research the kaolinitic and illitic soils showed evidence of a maximum lime content above which no further strength increase occurs. No such maximum was observed for the montmorillonitic within the range of lime contents investigated. Dumbleton (38) investigated the lime stabilisation of a variety of soils and confirmed the importance of the clay fraction in soil-lime stabilisation. He concluded that gravels, sands, and silts can not be effectively stabilised with lime.

3.6 Lime Stabilisation in the United Kingdom

There are very few reported instances of the use of lime for soil stabilisation in the UK. In the 1940's, in Worcestershire, a car park was constructed by stabilising keuper marl with 8 per cent lime (22). This is the only reported case of lime being used alone to stabilise a soil. For the most part lime appears to have been used in small quantities, typically 2 per cent by dry weight of soil, as a supplement to cement. One instance (22) was at Droitwich, in 1951, where the base of section of carriageway was constructed by stabilising the in situ sodium clay with 20 per cent chippings, 8 per cent cement, and 2 per cent lime.

An investigation (38) to assess the potentialities of lime stabilisation in the UK concluded that clay and clayey gravels may be stabilised with lime. However, it was acknowledged that the success of lime stabilisation in Africa, and in Texas and Louisiana in the USA, was principally due to their hotter climates which ensured a satisfactory rate of mineral-cement reaction. Others (51) also believe that the limited practical utilisation of lime in the UK is associated with the very limited construction season during which the temperature is sufficient to promote an acceptable rate of reaction. The future use of lime for pavement construction in the UK is, therefore, limited to lime modifications. This has proved successful in the past (22,24) and currently the technique is gaining in popularity (52) for improving unsuitable subgrade soils. It is in this role that lime can be further exploited to improve the performance of cement stabilised clayey materials for highway construction.

3.7 Pre-treatment of Clay Soils with Lime

The lime liberated by cement during hydration plays an important role in the stabilisation of clay soils. Croft (35) noted that the addition of small quantities of lime to clay cement mixtures improved the quality of the resulting compacted material. The unconfined compressive strength of lime cement-kaolinite was further enhanced by pre-conditioning with lime for 24 hours prior to the addition of the cement. De Sousa, Davidson and Larguros (50), observed that the addition of lime to a montmorillonite soil, facilitated mixing, minimised the reduction of both compacted density and strength with the time elapsed between addition of the cement and compaction, and improved the compressive strength. These improvements can be explained in terms of the lime-mineral reaction. The additional lime satisfies the requirements of the mineral, leaving the lime, liberated by the cement, free to assist the hardening and hydration process (35). The flocculation, caused by the reduction of the electrostatic charges and subsequent cementing (37), results in a material that appears drier and has improved tilth (38). Pulverisation and mixing are made easier and, therefore, an improved and stronger skeleton structure is produced (31).

An investigation by Maclean, Robinson and Webb (24), confirms the benefits described above. In this case 2 per cent lime increased the 7 day strength of a heavy clay, stabilised with 15 per cent cement. Brooke-Bradely (22) cites examples in which clay soils were stabilised with 2 per cent lime and 8 per cent cement.

The optimum lime content for improving cement stabilised soils is dependent upon the predominant clay mineral and the clay content (41). The "lime fixation point" method, proposed by Hilt and Davidson (41), may be used to determine the optimum lime content for improvement.

CHAPTER 4. DURABILITY OF STABILISED MATERIALS.

4.1 Introduction

The most important property of a cement or lime stabilised material is, probably, its ability to retain a state of stability over a long period of exposure to the destructive forces of weather. This property is termed durability and it involves the definition of the minimum quality of the material together with the objectives of the method used to design the mix.

Throughout the world there are many mix design procedures but the requirements for proven durability have significantly influenced the development of the individual methods.

During the 1930's the Portland Cement Association of USA developed a series of testing procedures, their objective being the selection of moisture content, density, and cement content values that would produce a material of proven durability. Over the subsequent 50 years, many pavements have been laid incorporating cement stabilised materials using control factors obtained by these test procedures. The favourable performance of these pavements demonstrates the dependability of these test methods. Indeed, they have only undergone minor revisions since they were first approved by the AASHO in 1945.

In 1939 the Road Research Laboratory of Great Britain followed up American experience by conducting a programme of laboratory tests and small scale field trials on soil cement mixtures. From the outset it was decided to depart from American practice and use the unconfined compressive strength test to evaluate soil cement mixtures, rather than follow the lengthy American durability test procedures. Current British practice still relies on unconfined compressive strength as the major criterion for evaluating cement stabilised materials in pavement construction. Experience has shown that certain soils may be susceptible to frost or sensitive to changes in moisture content and so a series of tests, related to durability assessment, have been developed.

In the following paragraphs major developments in the testing of cement stabilised soils are reviewed. A number of investigations are described which illustrate the use of various test procedures.

4.2 The Origin of Test Procedures in the USA

One of the earliest findings of research conducted in the 1930 by the Portland Cement Association (53) was that the moisture content-density relationship for soils, discovered by Proctor in 1929 (54), were also valid for mixtures of soil and cement when compacted immediately after mixing. It was found that optimum moisture content not only produced the highest density but also provided sufficient water for cement hydration and produced maximum strength. The standard procedure for determining the moisture-density relationship of soil cement mixtures (55) was described at the same time as that for soils.

In considering the effects of cement content, moisture content, and density on the properties of compacted soil-cement mixtures it was recognised that the degree of hydration would have a significant influence on the results. A curing period of 7 days was selected to ensure that a significant amount of hydration had occurred prior to testing.

The main cause of the deterioration of compacted soil cement mixtures was thought to be associated with changes in moisture content. These changes may occur on a macro-scale, due to seasonal variations, or on a micro-scale, due to local migration of moisture, for example during freezing and thawing. The various compressive and tensile tests used for assessing soil and concrete were considered unsuitable as they did not simulate the forces induced by changes in moisture content (23). However, it was discovered (53) that subjecting specimens of compacted soil cement to repeated cycles of wetting and drying, or freezing and thawing, could induce forces of the desired nature and magnitude, and so simulate the level of disruption observed in-situ. Following a series of exploratory experiments (53), two test procedures were developed to simulate these phenomena of moisture changes. The wet-dry test (56) was designed, primarily, to simulate shrinkage forces, whereas the freeze-thaw test (57) was designed to reproduce the internal expansive forces associated with moisture changes in fine grained soils (58). Moisture movements are important in both tests. In the wet-dry test, the specimens are submerged during the wetting period of each cycle and in the freeze-thaw test they are allowed to absorb water by capillarity during the thawing period of each cycle.

The test procedures may be summarized as follows:

4.2.1 Wet-dry Test (56). At least two cylindrical specimens of compacted material are cured for 7 days in a moist room operating at 21°C and having a relative humidity of 100 per cent. On removal from the curing environment each specimen is submerged in water, maintained at a temperature of 21°C, for a period of five hours. They are removed from the water and placed in a thermostatically controlled oven, set to 71°C, for a period of 42 hours. At the end of each wetting and drying period one of the specimens is weighed and measured. At the end of each drying period the faces of the second specimen are wire brushed in a specified manner to remove loosened material. On completion of 12 cycles the brushed specimens are weighed to determine the weight loss during the test and the moisture content of each specimen is determined.

4.2.2 Freeze-thaw Test (57). At least two cylindrical specimens are produced and cured in the same way as the wet-dry specimen. After 7 days curing, one is placed in a thermostatically controlled room, set to -23°C, for 24 hours. On removal from the cold room the specimen is placed on an absorbent pad in a thermostatically controlled moist room, set to 21°C and having a relative humidity of 100 per cent. The absorbent pad is partially submerged in water to allow the specimen to absorb water by capillary action during thawing. The freezing and thawing is continued for 12 cycles. The freeze-thaw specimens undergo similar treatment to the wet-dry, the faces of one being brushed at the end of the thaw cycle, and the length changes, mass changes, and final moisture content, also being determined

4.2.3 <u>Test Criteria.</u> A comparison of laboratory data and the field exposure of laboratory specimens, together with an investigation of field performance, resulted in the development of test criteria. These enable a minimum cement content to be established which will ensure that the stabilised mixture will have sufficient long term stability to resist the shrinkage and expansive forces that occur in the field. The criteria (23) established in this initial research are as follows:

(i)	Volume Change:	The volume of the specimen shall not increase by more than 2 per cent.
(ii)	Moisture Content:	The maximum moisture content shall not exceed that required to fill the voids at the time of moulding.

(iii) Weight Loss: The maximum weight loss of the brushed specimens shall not exceed the following:

Soil Group (59)	Maximum Permitted Weight Loss
A-1, A-2, A-2-5, and A3	14 °/o
A-2-6, A-2-7, A-4, and A-5	10 °/o
A-6 and A-7	7 °/o

(iv) Compressive Strength: Compressive Strength shall increase with both age and cement content.

The wet-dry and freeze-thaw procedures were approved as standards (56,57) by the American Society for Testing and Materials (ASTM) in 1944. In 1957, following the successful stabilisation of many pavements designed using the standard tests, the procedures were refined to reduce the time, manpower, and material required, although the basic procedures and test criteria were as originally described.

4.3 The Origin of Test Procedures in the UK

The systematic evaluation of cement stabilised materials in the United Kingdom is believed (60) to date from research studies carried out at the Road Research Laboratory in 1939. These followed from the American experience and included a programme of laboratory tests together with small scale field trials.

A review of British practice, published in 1963 (60), states that this original research "showed that a minimum compressive strength of 1.75 N/mm^2 at 7 days was required to ensure that cement stabilised soils satisfied the requirements of the American wet-dry and freeze-thaw tests". The review suggests that the departure from American practice arose partly from doubts as to the relevance of the American tests in the less severe climate of Great Britain, and partly from a desire to adopt equipment and testing procedures used for concrete testing. Williams (61) refers to a paper, published in 1942 by Markwick and Keep (62), in which it is suggested that a preliminary indication of the suitability of a soil could be obtained by determining the compressive strength of the mixed material. The 1964 edition of "Soil Mechanics for Road Engineers" (63) also suggests that there is a correlation between durability, as determined by the American durability tests, and a compressive strength of 1.75 N/mm^2. Thus, during the earliest years of British soil-cement technology, the compressive strength test was adopted as the primary method of assessing the performance of the mixtures.

The importance of adequate compaction has long been acknowledged. Maclean, Robinson and Webb (24), reporting in 1952 on the study of a cement stabilised heavy clay, state that "it would be essential to obtain a state of compaction... equivalent to an air content of not more than 5 per cent to ensure satisfactory performance". The earliest forms of specification usually defined the minimum state of compaction as that required to ensure 5 per cent air voids at the moisture content at the time of compaction. This approximates to the state of compaction achieved in the British Standard Compaction Test (64), which is almost identical to the American Standard Compaction Test (65).

An interesting feature of the study, by MaClean et al. (24), was the assessment of the susceptibility of a cement stabilised material to water. This was achieved by comparing the average compressive strength of specimens cured in a humid atmosphere for 7 days with that of specimens which had been immersed in water for the latter 5 days of the curing period. This method of test, including a period of immersion, was considered (24) to be a particularly satisfactory measure of the strength of cement stabilised clay.

From these early experiences a method evolved for selecting the moisture content and cement content necessary to ensure adequate field performance. These procedures were formalised in 1953 with the publication of the first edition (66) of BS 1924, "Methods of Test for Stabilised Soils".

The moisture content adopted was the optimum value as determined by conducting a British Standard compaction test (64) on mixtures of soil and cement. The cement content was selected by determining the strength of soil cement mixtures. The second edition (67) of BS 1924:1957 described strength tests which employed cylindrical specimens with a height:diameter of 2:1. The soils were divided into three groups, based on their grading and the dimensions of the specimens were related to these groups. For fine grained soils, 50 mm diameter specimens were used, whilst for medium and coarsely grained soils the specimen diameters were 100 mm and 150 mm respectively. The 150 mm diameter cylinders proved too cumbersome and heavy to handle and were replaced with 150 mm cubical specimens in the 1967 edition (68) of BS 1924:1967. A number of specimens were produced at optimum moisture content to cover a range of cement contents. The specimens were cured for 7 days at constant moisture content before being tested for compressive strength, with a minimum strength of the order of 1.75 N/mm^2 usually being required.

The method of evaluating cement stabilised materials in the United Kingdom has remained essentially the same up to the present day. The most recent edition of the Department of Transport specification (9) reflects improvements in the production of stabilised soils. The optimum moisture content is determined using the vibrating hammer test which reproduces the state of compaction achieved by modern compaction plant. The minimum strength requirements have increased to 2.8 N/mm^2 for cylindrical specimens and 3.5 N/mm^2 for cubical specimens. These strengths are for specimens compacted to field density.

The 1957 edition (67) described three tests for assessing the durability of soil cement. The tests assessed the deleterious effects of immersion in water, cyclic freezing and thawing, and water absorption. These tests were not intended as alternatives to the compressive strength test and were only required if the durability of the material was suspect.

The resistance to immersion was determined by measuring the unconfined compressive strength of a cylindrical specimen cured at constant moisture content for 7 days and then immersed in water for 7 days. The average strength of these specimens was expressed as a percentage of that of a similar specimen cured at constant moisture content for 14 days. The purpose of this test was to reveal the adverse effects that the presence of certain constituents, such as expansive clay minerals or sulphates, may have on the stabilised soil when it comes into contact with excess water (69). Although this test is included in the current edition of BS 1924:1975 (69), an acceptance criterion has never been specified. In a forthcoming revision of the Department of Transport Specification (9) it seems likely that the resistance to immersion will also be included in the testing regime. In a review of British practice, published in 1960, Sharp (70) suggested that the average strength of the immersed specimens should not be less than 80 per cent of the average strength of those cured at constant moisture content. Reviewing the practice in 1963, Maclean and Lewis (60) suggested that 90 per cent of the strength should be retained following immersion.

In the 1957 edition (67) of BS 1924:1957 a test was described for measuring the resistance to cycles of freezing and thawing. This test was required when the material was liable to be exposed to severe frost or the material was suspected of being frost susceptible (70). A number of specimens were produced from the given mix. All the specimens were cured for 7 days at a constant temperature and moisture content. Half the total number of specimens were removed from the curing environment and subjected to 14 cycles of freezing, for 16 hours at $-5^{o}C$, and thawing, for 8 hours at $25^{o}C$. The freezing temperature was applied to the top face of the specimen, the bottom face being submerged to a depth of 6 mm in water at $8^{o}C$. The remaining control specimens were immersed in water for the duration of the freeze-thaw test. At the end of the 14th cycle the compressive strengths of the specimens were determined. The average strength of the freeze-thaw specimens was expressed as a percentage of the strength of the control specimens. A criterion for interpreting the results was never established, although Sharp (70) suggested that the average strength of the freeze-thaw specimens should be not less than 80 per cent of the average strength of the control specimens. The test was seldom used because of difficulties interpreting the results (68) and it was deleted from the Standard in 1967.

A water absorption test appeared in the 1957 edition of BS 1924 but it was rarely used because of the lack of correlation with field results (70). It, also, was deleted from the Standard in 1967.

4.4 Alternative Methods for Assessing Performance in the US Tests.

Following the introduction of the PCA durability tests (56,57), alternative methods were considered for assessing performance in the wet-dry and freeze-thaw tests. During the early 1960s the Portland Cement Association conducted an investigation (71) to determine the ability of these alternative methods to select a cement content compatible with the proven weight loss criterion. The following methods were used with the standard wet-dry and freeze-thaw procedures.

1. Weight loss measured after brushing the specimen at the end of each cycle.
2. Moisture changes determined by weighing the unbrushed specimen at the end of each wet, dry, freeze or thaw period.
3. Length changes determined by measuring the unbrushed specimen in a length comparator, equipped with a dial gauge graduated to 0.0025 mm, at the end of each wet, dry, freeze or thaw period.
4. Compressive Strength of:-
   (a) Specimens cured for 7 days at a constant temperature and 100 per cent relative humidity.
   (b) Specimens cured for 39 days as in (a).
   (c) Specimens subjected to standard wet-dry and freeze-thaw procedures.
5. Pulse velocity measurements at the end of each cycle.

Methods 1 and 2 are the standard techniques described in the standard methods (56,57). The measurement of length change is a standard measure of resistance but for the purpose of this investigation the accuracy of the measuring device was increased from 0.2 mm. Methods 4 and 5 are non-standard techniques.

The initial study (70) was concerned with examining the accuracy and sensitivity of the different methods. Four soils, representing a range of textural types were selected and subjected to wet-dry and freeze-thaw tests. The conclusions drawn from this study are summarised below.

4.4.1 Weight Loss. The absolute value of the final weight loss was directly related to the degree of deterioration. A durable specimen maintains a constant degree of "hardness" during the test and so the rate of weight loss will be uniform. The rate will depend on the degree of "hardness" and the abrasive characteristics of the soil-cement mixture. Specimens passing the weight loss criteria had varying degrees of "hardness" as measured by the compressive strength. The abrasive variable was recognised during development of the test procedures and as a result three separate weight loss criteria (72) have been produced for different soil types. These criteria are given in 4.2.3 above.

Specimens made from silty soils and fine sands may form a surface shell when subjected to the freeze-thaw test. The existence of a surface shell did not necessarily indicate deterioration but scaling occurred on specimens failing the weight loss criteria.

4.4.2 Moisture Gain. Moisture gain was not a sensitive measure of deterioration. The moisture content only exceeded that required to fill the voids at the time of moulding when the physical deterioration was at a very advanced stage and so this is not a particularly useful criterion. Many specimens failed the weight loss criteria although their moisture gains were less than those required for saturation.

In the freeze-thaw test, moisture gains below the saturation limit were not necessarily indicative of deterioration. Fine textured mixtures, when subjected to the wet-dry test, underwent a progressive drying that was independent of deterioration except in the most advanced stages. Deviation from the moisture change patterns, established by non-deteriorating specimens, may indicate failure of specimens produced from the same soil but having lower cement content.

4.4.3 Dimensional Stability. The volume change technique described in the standards (56,57) was not found to be a sensitive measure of deterioration. The criterion of a 2 per cent volume increase only reflected deterioration in very advanced stages. Many specimens failed the weight loss criteria with very low accompanying increase in volume.

Accurate measurement of length changes during durability tests was found to be a very sensitive measure of deterioration. The amounts of shrinkage, on freezing or drying, and expansion, on thawing or wetting, were dependent upon soil type, moisture content, and to a lesser degree, cement content. The expansion accompanying deterioration was, however, too great to be masked by such extraneous factors.

4.4.4 Strength. The strength of a soil-cement mixture is greatly dependent upon the soil's reaction with cement (71). It is, therefore, not possible to have a single value strength criterion that will ensure durability for all soil types. The change in strength which occurred during a durability test, rather than the absolute value at the start or end of the test, seemed to be a useful measure of deterioration. The strengths of specimens after wet-dry and freeze-thaw tests were highly variable and so replicate specimens should be tested to ensure that accurate strength change data are produced.

4.4.5 Pulse Velocity. The pulse velocity technique was not a sensitive measure of deterioration. Advanced deterioration was reflected in major decreases in velocity. The technique was subject to large experimental variation between both replicate specimens and repeated measurements on a single specimen.

4.5 Specimen Behaviour in the US Tests

Packard and Chapman (73) presented the results of an investigation which extended the initial study (71), summarised in Section 4.4 above, to over one hundred soils of various types. The criteria used to assess performance during the tests were weight loss, moisture change, precise length change, and compressive strength. The observations made by the authors are summarised below.

4.5.1 Freeze-thaw Test. The pattern of length change observed for stable specimens is shown in Figure 4.1a. Specimens contracted during freezing and expanded during thawing. The length of such specimens at the end of the thawing period normally did not significantly exceed the original length, an exception being where considerable moisture was absorbed during thawing. Although during the course of the test there may have been an overall increase in length, the amplitude of the length changes remained approximately constant. The amplitude of these length changes appeared to be dependent upon soil type. Shrinkage on freezing varied from 0.05 per cent for gravel and well graded sands, to 0.6 per cent for clayey soils.

The pattern of length changes for deteriorating specimens is shown in Figure 4.1b. The onset of deterioration is indicated by successive reductions in shrinkage on freezing. This may be observed from the figure which shows a rise in the envelope of the frozen lengths. The thawed length envelope also changes but to a lesser degree. As deterioration progressed both the thawed and frozen lengths exceeded the original length. At severe deterioration the specimen expanded on freezing and contracted on thawing.

The shrinkage of specimens containing appreciable quantities of fine material exceeds that which may be attributed to thermal effects. The authors suggested that the additional shrinkage was due to drying of the fines as moisture migrated towards the ice forming in the larger voids. This mechanism, which is similar to that reported to occur in concrete (44) was considered an acceptable explanation of the direct relationship between freezing shrinkage and silt, or clay content.

The change in moisture content during the freeze-thaw test was also dependent upon soil type. Stable specimens, produced from gravels and well graded sands, gained little moisture during the test, with maximum increases of 2 per cent being reported. Fine sands also gained little moisture although increases of up to 4 per cent were observed. Silty soils absorbed considerable amounts of moisture during the test, with poorly graded silts experiencing moisture gains of as much as 7 per cent. Clayey soils absorbed only small amounts of moisture. A shell, which was observed to form on the surface of specimens containing fine sands and silts, was thought to be associated with large moisture gains.

For most types of soils the air content of stable specimens remained reasonably constant during freezing and thawing. The only exceptions were the silty soils which, regardless of hardness or state of quality, absorbed considerable amounts of moisture.

The rate of hydration of cement varies with temperature. Once a cemented material has set and started to harden, further hydration will continue in all the unfrozen cavities down to approximately -4°C. Indeed hydration may continue down to temperatures as low as -11°C. When the temperature rises above freezing normal hydration is resumed according to the maturity rules governing hydration of cement (44). During the freeze-thaw test, there is an opportunity for strength gain equivalent to about 10 days of additional curing at constant moisture content. It would, therefore, be expected that the strength of specimens satisfactorily completing the freeze-thaw test should be similar to that of specimens cured normally for 17 days. In the initial study (71) Packard observed that the strength of specimens possessing adequate freeze-thaw resistance increased from 1.5 to 5.4 N/mm^2 at 7 days, to 2.5 to 7.0 N/mm^2 at the completion of the test, the individual values depending on soil type. The compressive strength retained by acceptable specimens was in excess of that developed at 20 days by normally cured specimens. The strength retained by failed specimens was less than this 20 day value whilst the badly deteriorated specimens had residual strengths below the 7 day value.

4.5.2 Wet-Dry Test. The length change pattern displayed by a soil cement specimen during wetting and drying depends largely on soil type. Shrinkage during the first drying period varied from 0.5 per cent for granular soils to 10 per cent for clayey soils. The total drying shrinkage was almost twice the freezing shrinkage that occurred in the freeze-thaw test. The stable specimens exhibited a regular pattern of length changes, whilst a net increase in length was indicative of deterioration.

The moisture content of specimens during wetting and drying depended upon soil type. On drying, moisture contents varied between 2 and 6 per cent, the drier for soils containing less clay. On wetting, the moisture content varied between saturation and several percentage points below the moulded moisture content, the wetter for soils containing less clay. Heavy clays were progressively dried during the standard test and in the latter cycles did not absorb sufficient water to reveal their sensitivity to changing moisture content. During the development of the wet-dry test it was recognised (23) that the 71°C oven drying condition may be unduly beneficial to specimens that might not otherwise be resistant to wetting and drying. At the completion of the test the compressive strength of satisfactory specimens was reported to be as high as twice the strength of specimens cured normally for a similar period.

The influence of thermal expansion and contraction on the length change pattern during wetting and drying is shown in Figure 4.2. During drying the shrinkage is counterbalanced by thermal expansion and similarly the expansion during wetting is opposed by thermal shrinkage. The degree to which moisture effects are counteracted by thermal effects amounts to about 16 per cent for clayey soils rising to 100 per cent for soil containing less than

10 per cent clay (73).

Packard and Chapman (73) concluded that the wet-dry test should not be used alone to measure durability. They suggested modification of the moisture and temperature conditions but did not give specific guidance.

4.6 Failure Mechanism in the Freeze-Thaw Test

Packard and Chapman (73) proposed a number of failure mechanisms based on their observations of soil cement specimens subjected to the standard freeze-thaw test. In soil-cement, as in concrete, hydraulic pressure develops as ice, forming in the voids, expels excess water. The magnitude of these hydraulic forces is dependent upon the freezing rate and the resistance to flow encountered by the excess water (44). Damage caused by differential volume changes is likely to be more significant for soil-cement mixtures than for concrete because of the wide variation in the volume change characteristics of soil constituents. Fine grained soils undergo freezing shrinkages several times greater than concrete and are, therefore, particularly susceptible to differential volume changes. Prior to the onset of deterioration, represented by the regular pattern of length changes in Figure 4.2, the hydraulic pressure and differential volume change mechanisms predominate. Once sufficient internal damage has occurred, ice growth begins to contribute to the deterioration. The onset of such deterioration was reflected in the length change pattern by a rise in the frozen length envelope. At the terminal condition, with expansion on freezing and contraction on thawing the ice growth mechanism became predominant.

Packard and Chapman observed that for the majority of soils, failure occurred in the matrix between aggregate particles. In the case of soils which were composed of soft or unstable aggregates, such as shales, aggregate failure may also occur.

4.7 Alternative Criteria for ASTM Freeze-thaw Test

Packard and Chapman (73) have proposed the following alternative criteria for assessing a soil-cement specimen's resistance to the standard freeze-thaw test (57).

1. Length Change: Twelfth cycle frozen length must not exceed the first cycle frozen length by more than 0.1 per cent.
2. Residual compressive strength after test:
   a) >145 per cent of 7 days strength - highly resistant.
   b) 90 to 145 per cent of 7 day strength - may or may not be resistant
   c) <90 per cent of 7 day strength - not resistant.

The method of accurately measuring length changes was considered to be an improved, alternative technique to the brushing procedure used in the standard test.

4.8 The Illinois Procedure

Thompson and Dempsey (74) recognised the empirical nature of the standard American freeze-thaw test and considered it to be unsuitable for the conditions prevailing in the northern areas of the United States.

They proposed a more rational approach to freeze-thaw testing. A heat transfer model was developed (74) which could simulate the temperature regime in a pavement system. The model was used (74) to establish relevant quantitative frost action parameters pertinent to stabilised pavement systems for various locations in Illinois. The resulting idealised freeze-thaw cycle, representing field conditions in the most severe environments of Illinois was programmed into a specially developed (75) freeze-thaw test unit. In addition to the control of temperature, the unit was capable of controlling the rate of freezing and thawing. A laboratory programme examined a wide range of typical Illinois stabilised materials. The strength of specimens, subjected to 5 and 10 cycles of freezing and thawing, were related to the strength of specimens immediately prior to freezing and thawing. A durability criterion was developed (76) based on residual strength following exposure to the equivalent of the first winter of freeze-thaw cycles. Minimum tolerable strengths, defined (76) as the strength of the material during spring of the first winter of exposure to freezing and thawing, have been established for the state of Illinois. The design strength of laboratory and field cured specimens is determined from the relationships established in the laboratory programme.

4.9 Experience with the UK Freeze-thaw Test

Following the experience in North America and the formulation of standard procedures (56,57), considerable experience was gained in testing various soil-cement mixtures. However, many investigations considered modifying the procedure to suit local variations in climate and exposure. In the United Kingdom, the testing regime was modified to simulate the freeze-thaw conditions likely to occur in a pavement in a severe winter. This lead to the publication, in 1957, of a standard procedure for the UK test (67). This procedure is described in Section 4.3 above. During the 1960's various agencies adopted this procedure to measure the durability of both cement stabilised and lime stabilised materials. Some of these studies are outlined in the following paragraphs.

4.9.1 <u>Review.</u> George and Davidson described (77) how the standard test (67) as modified to simulate the conditions likely to occur in Iowa. Townsend and Klym (49) adopted the British test but modified the procedure to account for climatic conditions in central Canada. In the UK Oflaherty and Andrews (36) used a similar procedure to that adopted by George and Davidson, but with minor changes designed to simulate the freeze-thaw conditions likely to occur in Yorkshire. In Table 4.1 these procedures are compared to the standard procedure specified in BS 1924:1957 (67).

The duration of the freezing and thawing periods are the same for all procedures, although local climatic conditions have been simulated by varying the freezing and thawing temperatures. These differences in temperature have resulted in each test having different rates of freezing. The low freezing temperature, −11°C, adopted

Table 4.1 Some Variations on the British Freeze-Thaw Test

AGENCY	BRITISH STANDARD INSTITUTE	IOWA STATE UNIVERSITY	QUEENS UNIVERSITY, ONTARIO	CENTRE FOR TRANSPORT STUDIES - LEEDS UNIVERSITY
	BS 1924:1957	George & Davidson (77)	Townsend & Klym (49)	Oflaherty & Andrews (36)
Specimen Type	Cylinder	Cylinder	Cylinder	Cylinder
Specimen Size (mm)	100 x 50 0	50 x 50 0	75 x 50 0	50 x 50 0
Curing Period (days)	7	7	varied	28
Curing Temp (°C)	25	25	22	25
Freezing Temp[a] (°C)	-5	-5	-11	-5
Water Temp[b] (°C)	8	2	2	2
Thawing Temp (°C)	25	25	22	7
Freeze Duration (hrs)	16	16	16	16
Thaw Duration (hrs)	8	8	8	8
Total No of Cycles	14	10	7	10
Mixtures Tested	-	Soil-Cement	Soil-Lime	Soil-Cement and Soil-Lime

a. Applied to top face only
b. Bottom face immersed in water to a depth of 6 mm.

by Townsend and Klym resulted in a shortening of the effective curing period. The low thaw temperature, 7°C, employed by Oflaherty and Andrews is considered to be unduly rigorous. It reduces the effectiveness of additional curing during the thaw period when soil-lime mixtures may benefit from autogenous healing (78). The differences between the procedures make comparison of the results difficult and so they are discussed independently.

4.9.2 <u>Iowa Investigation.</u> George and Davidson (77) conducted an investigation of the durability of a silty clay loam mixed with cement. An experimental pavement was constructed and laboratory data were correlated with field performance. The durability of the material was judged in terms of the following parameters:

1. The unconfined compressive strength of specimens cured for 8 days.
2. The unconfined compressive strength of specimens subjected to the "Iowa" freeze-thaw test.
3. The "Index of Resistance to Freezing" determined by expressing the unconfined compressive strength determined in 2 as a percentage of the unconfined compressive of the control specimen.

The following durability criteria were proposed on the basis of satisfactory performance in the experimental pavement:

1. A minimum unconfined compressive strength at 8 day of 3 N/mm^2.
2. A minimum unconfined compressive strength after testing in the "Iowa" freeze-thaw test of 3.2 N/mm^2.

3. Laboratory specimens shall give a minimum "Index of Resistance to Freezing" of 80 per cent.

The 8 day strength is compatible with current British practice (9) which requires that cylindrical specimens achieve a minimum 7 day strength of 2.8 N/mm^2.

The strength criteria, 1 and 2, above, should only be applied to soils possessing similar characteristics to the silty clay loam used in the investigation. George and Davidson considered the "Index of Resistance to Freezing" to be a universal measure of resistance which may be applied to soils other than silty clay loam.

4.9.3 Ontario Investigation. The investigation by Townsend and Klym (49) was concerned with the durability of soil-lime mixtures. A variety of clayey soils were selected to represent the range of mineralogical and textural types likely to be found in Canada. Unlike the majority of studies, which vary the stabilizer content and employ a common curing period, Townsend and Klym used a single lime content, selected for each soil by the method of "lime fixation capacity" (41), and varied the curing period. Durability was assessed by measuring the heave of a specimen after 7 cycles of freezing and thawing. Specimens which exhibited less than 1 per cent heave were considered to be durable. Durability was correlated against compressive strength, permeability, and suction, and degree of saturation.

It was observed that the durability of soil-lime mixtures increased as the curing period was extended and a satisfactory performance could be related to the strength prior to testing. The results suggested that heavy and silty clays should develop a minimum compressive strength of 1.4 N/mm^2, before being subjected to the freeze-thaw test, and that clayey silts should develop a minimum strength of 2.1 N/mm^2. Heavy and silty clays required considerably less curing time than clayey silts to develop adequate resistance to the freeze-thaw test.

Disruption of the soil due to the creation of hydraulic pressures is resisted by the strength of the pore structure. Providing the pore structure has sufficient strength, when the pore space becomes filled with ice, the excess water is dispersed to other parts of the system, and so the hydraulic pressures cease as the ice-water interface is destroyed (49). Thus, as the larger pores fill with ice, the dissipation of the hydraulic pressures will depend on continuity of the pore system ie. the permeability of the compacted mixture. Townsend and Klym (49) observed that heavy and silty clays exhibited substantial increases in permeability when mixed with lime. They also observed that the permeability of the clayey silts did not change significantly through lime addition. The increase in permeability arising when clay is mixed with lime was attributed to flocculation of the clay particles as their mutually repulsive electrostatic charges were depressed by the calcium ions. The lack of response from the clayey silts emphasised the importance of the clay fraction in lime stabilisation. The results of the unconfined compressive strength tests suggested that the strength (of the pore structure) of the heavy clay - and silty clay - lime mixtures developed more rapidly than that of the clayey silt-lime mixtures. The substantial improvements in permeability and pore structure strength, accompanying the addition of lime to heavy and silty clays, combined to produce a satisfactory durable material. Clayey silts were not so responsive and did not develop satisfactory durability unless cured for extended periods.

The results of drying curve suction tests suggested that the addition of lime increases the size and number of larger pores and are, therefore, consistent with the results of the permeability tests. Air voids in the soil provide reservoirs into which the excess hydraulic pressures may dissipate thus reducing heaving pressures created by ice formation. Townsend and Klym suggested that lime stabilised soils having an initial degree of saturation less than 95 per cent and possessing adequate strength will not be susceptible to the freeze-thaw test.

4.9.4 <u>Leeds Investigation.</u> Oflaherty and Andrews (36) examined the resistance of selected soil-cement and soil-lime mixtures to freezing and thawing. The six soils, a heavy clay, a silty loam, a natural clay loam, and three laboratory blended clay loams, were mixed with:-

a) 4 per cent cement.

b) 4 per cent lime.

The blended clay loams had particle gradings similar to the natural clay loam but contained a different predominant clay mineral. Durability was evaluated by the "Index of Resistance to Freezing", as defined by George and Davidson (77), and also by measuring heave during freezing and thawing. The study was a laboratory investigation and no attempt was made to correlate the results with proven durability criteria or field exposure.

It was observed that clayey soils, in which the predominant clay mineral was kaolinite, were more effectively stabilised by cement than by lime. The "Index of Resistance" for these soil-cement mixtures exceeded 80 per cent and the measured heave was less than 3 per cent. The addition of lime to these kaolinitic clays did not give satisfactory results, the "Index of Resistance" being less than 25 per cent with heave of individual specimens exceeding 20 per cent. However, for clayey soils with montmorillonite as the predominant clay mineral, stabilisation by 4 per cent lime was more effective than 4 per cent cement. The "Index of Resistance" for these soil-lime mixtures exceeded 90 per cent and the measured heave was less than 2 per cent. Cement did not effectively stabilise these clays for the "Index of Resistance" was less than 40 per cent and heave exceeded 10 per cent. It was not possible to satisfactorily stabilise the silty clay loam with either 4 per cent cement or 4 per cent lime. The specimens began to heave rapidly shortly after the test commenced. Many of the specimens disintegrated before the completion of the test. The "Index of Resistance" of those specimens which completed the test was very low and all heaved more than 3 per cent.

Oflaherty and Andrews do not report 7 day strengths and the control specimens, which were immersed in water for the duration of the freeze-thaw test, do not provide a realistic measure of the strength of specimens cured at constant moisture content. In their concluding remarks it is stated that "the ability of a soil-additive mixture to withstand frost action involves an examination of its frost heaving history and calculation of its resistance index value". They do not, therefore, consider absolute strength alone to be a satisfactory measure of resistance to freezing and thawing.

<u>4.9.5 Conclusions.</u> Although direct comparison of the results from the three investigations is complicated by the differences in the procedures used, their similarity permits a number of general conclusions to be made.

1. The measurement of the heave and the heave pattern exhibited by a stabilised specimen, undergoing freezing and thawing, is a reliable measure of durability.
2. The ratio of the unconfined compressive strength of the freeze-thaw specimen to that of a control specimen is a measure of the strength development of soil-additive mixtures during cycles of freezing and thawing.
3. The absolute strength of specimens, as determined by the unconfined compression test, is not a measure of durability unless it has been correlated with heave and/or residual strength after freezing and thawing.

These conclusions are remarkably similar to those of Packard and Chapman (73) (see Section 4.7 above).

With the exception of the limited study by George and Davidson (77), the behaviour of specimens subjected to the British freeze-thaw test has not been correlated with field performance. The test was criticised because of the lack of guidance on how to interpret the results (68) and also because of insufficient control of the temperature conditions during freezing and thawing (60). There is no reported use of the test since its deletion from the standard in 1967.

4.10 Experience with the UK Immersion Test

In a paper (24) published in 1952, Maclean et al described a test to determine the susceptibility of a stabilised clay to softening by water. Stabilised specimens were cured in a humid atmosphere for 1 day, immersed under water for 5 days, and then allowed to drain, in a humid atmosphere for 1 day before testing in unconfined compression. The strength were compared with strength of specimens which had been cured in a humid atmosphere for 7 days. It was found that the strength of specimens containing 20 per cent cement was increased by immersion whilst specimens containing 15 per cent cement was reduced. Specimens containing 10 per cent cement disintegrated when immersed under water. This is the first reported use, in the UK, of a test specifically aimed at measuring the adverse effects of prolonged immersion on soil-cement mixtures. Although the current test for resistance to immersion (69) has changed little since its introduction in the 1950's an acceptance criterion has never been clearly established. Performance is expressed as an Immersion Ratio which expresses the strength of the immersed specimen as a percentage of similar specimen cured at a constant moisture content for 14 days. There is only a limited amount published on the use of the immersion test. Sherwood (79,80,81) has conducted a number of investigations in which the immersion test was used to determine the effect of sulphates on cement and lime stabilised soils in contact with excessive amounts of moisture. Kettle and Williams (82) have described the effects of immersion on the strength of specimens produced from cement stabilised colliery spoils.

In an investigation (79) of the effects of sulphates on the resistance to immersion of cement stabilised clays, Sherwood found that the resistance was reduced when sulphates were added to the clay. The addition of calcium sulphate, expressed as 2 per cent SO_3, reduced the strength of immersed specimens to only 34 per cent of the strength of specimens cured at constant moisture content. In comparison, similar specimens immersed in sulphate free water retained 80 per cent of the strength of normally cured specimens. Furthermore, substitution of sulphate resisting cement for ordinary portland cement did not reduce the disintegrating effect of the calcium sulphate. Immersion of similar specimens in a magnesium sulphate solution also resulted in a significant reduction in strength.

Another investigation (80) examined the role of the clay fraction in the disruption of soils-cement by sulphates. Experiments on five different soils, produced by blending varying proportions of clay and pure silica sand, showed that the degree of disruption, as measured by the immersion test, was dependent upon the clay content, rather than the sulphate content of the soil. The disintegration of the stabilised clay specimens was attributed to cracking brought about by a large increase in volume. Additional tests, which incorporated an extended immersion period, showed that the onset of deterioration occurs within a short period after immersion.

The results of tests on cement stabilised clays led to the hypothesis that, in the presence of excess moisture, lime, liberated during hydration of the cement, and sulphates combine directly with clay minerals to form ettringite, a highly disruptive mineral. Further experiments were conducted on specimens produced from London clay mixed with 10 per cent lime. These results showed that, provided the specimens remained at constant moisture content, the presence of sulphates was not necessarily detrimental. However, a considerable loss of strength, due to expansion and cracking, occurred when the specimens were immersed in water. It was concluded that the disruption of the soil-cement by sulphate ions was probably caused by the formation of ettringite resulting from a reaction between sulphate and clay mineral. In a more recent investigation (81) Sherwood and Ryley examined the influence of sulphate content on the resistance to immersion of cement stabilised (well burnt) colliery spoil. The specimens were subjected to a modified immersion test in which specimens were cured at constant moisture content for 7 days and then immersed under water for 84, 175, and 357 days. The strengths of the specimens, following immersion, was expressed as a percentage of similar specimens cured at constant moisture content for 91, 182 and 364 days respectively. The strength ratio of satisfactory specimens ranged from 92 to 108 per cent whilst that of unsatisfactory specimens ranged from 66 to 102 per cent. Performance was judged on visual examination rather than strength loss. Specimens containing more than 1 per cent total sulphate (as SO_3) underwent varying degrees of disintegration, observed as cracking. Specimens containing less than 1 per cent total sulphate showed no visible signs of disintegration. It was concluded that the maximum total sulphate content of 1 per cent for granular materials, specified in the Department of Transport Specification (9) also applied to (well burnt) colliery spoil.

Kettle and Williams have reported (82) the results of a limited study in which immersion tests were conducted on a number of cement stabilised unburnt colliery spoils. Performance was judged on the basis of strength following immersion. Strength ratios exceeding 80 per cent were considered to indicate satisfactory resistance. The results were related to sulphate content and showed that spoils containing less than 0.05 per cent achieved satisfactory resistance to immersion. One sample, containing 0.21 per cent sulphate, had very poor resistance to immersion which was attributed in part to its high absorption characteristics.

In a forthcoming revision of the Department of Transport Specification (9) it seems likely that the resistance to immersion will be included in the testing regime. Perhaps for the first time there will be specific guidance to assist interpretation of the results from the immersion test.

4.11 The Road Research Laboratory Frost Heave Test

At about the same time that the British freeze-thaw test was deleted from the British Standard (68), a report (13) was published by the Road Research Laboratory which described a research test for measuring the frost susceptibility of soils and road materials. In the test, developed from original work by Taber (83) in the USA, the upper surface of a cylindrical specimen, 152.4 mm high and 101.6 mm in diameter, is exposed to temperature of -17°C whilst the lower surface is in contact with water at +4°C. The specimen is subjected to this condition for a period of 250 hours and the total heave is measured at the end of the test. The conditions imposed by the test were originally correlated (13) with the field behaviour of sub-grade soils and subsequently applied to sub-base and road base materials. Tests conducted on sub-base materials damaged by the severe frosts of 1940 and 1947, and on other materials which had apparently been satisfactory under similar conditions, have enabled criteria to be chosen. Materials having less than 12.7 mm were judged to be satisfactory, materials heaving 12.7 to 17.8 mm were considered marginally frost susceptible whilst materials heaving more than 17.8 mm were classified as very frost susceptible.

The report includes results of tests conducted on a variety of materials comprising: cohesive and non-cohesive soils, chalks, limestones, granites, burnt colliery spoils, slags, and pulverised fuel ashes. Cement was one among a number of additives which were added to frost susceptible, unbound materials, to reduce heave to less than 12.7 mm. This approach has been extended, perhaps unwittingly, so that frost susceptibility of cement stabilised materials is assessed by the frost heave test (13). Current editions of both the Department of Transport Specification (9) and Road Note 29 (10) require that all materials, including cement stabilised materials, within 450 mm of the running surface should be frost resistant as defined by the frost heave test (13).

Kettle and Williams (15) conducted frost heave tests on both burnt and unburnt colliery spoils in both the unbound and cement stabilised states. The results for the unbound spoils showed that whilst unburnt spoils are non-frost susceptible, burnt spoils may be very frost susceptible. It was found that the addition of only 5 per cent cement to the frost susceptible burnt spoils was sufficient to reduce the heave to less than 12.7 mm. The tests on cement stabilised spoils included measurements of residual tensile and compressive strength after freezing. The results of these tests showed that "positive movement during the test is evidence of a non-recoverable breakdown within the specimen". Furthermore, a total heave of more than 1 per cent resulted in a complete loss of tensile strength. It was concluded that the frost heave criteria for soils and road materials was inappropriate for soil-cement.

a. Durable Specimen

b. Deteriorating Specimen

Figure 4.1 Length changes during Freeze-Thaw Test (After Packard & Chapman (73))

a. Thermal Effect

b. Effect of Moisture Change

c. Combined Effect

(expansion on drying, shrinkage on wetting)

Figure 4.2 Effect of Temperature and Moisture Change on lengths of specimens in the Wet-Dry Test (After Packard (71))

CHAPTER 5 SCOPE OF THE LABORATORY INVESTIGATION

5.1 Introduction

The broad objectives of this study were to assess the quality and durability of cement bound minestones, with a view to their use as an alternative to the traditional materials used for the lower layers of highway pavements.

The study comprised the following investigations:

(a) Classification and properties of the minestone samples.

(b) Compressive strength of the cement bound minestones.

(c) Experimental study of the durability of the cement bound minestones.

5.2 Classification and Properties of Minestone Samples

The classification and properties of each minestone sample were established before commencing tests on the cement bound minestones. The physical, chemical and mineralogical composition of minestones vary considerably, not only from colliery to colliery, but also within the spoil heap at a given colliery. Since all these properties may influence the response to the addition of cement, their identification is necessary to assist in the interpretation of the results obtained during the investigations of compressive strength and durability. Each minestone was subjected to a series of classification tests, described in BS 1377:1975 (64), which provided a description of the sample in terms of its physical characteristics. The presence of excessive amounts of sulphate can be highly disruptive to cemented materials and, therefore, tests were conducted to determine the sulphate content of each sample. The mineralogical and ion exchange characteristics of clay particles can significantly influence the hydration of cement and also lead to an unacceptable degree of swelling due to the adsorption of water. Tests were, therefore, conducted to:

(a) identify the predominant clay mineral, and,

(b) determine the cation-exchange capacity of the fine fraction.

The relationship between the moisture content of a soil and the density to which it can be compacted with a controlled compactive effort also applies to soil-cement mixtures. The subsequent investigations of compressive strength and durability were conducted on compacted specimens and so it was necessary to establish the moisture density relationships of the various minestone-cement mixtures.

The classification and properties of each minestone sample are reported and discussed in Chapter 6.

5.3 Compressive Strength

Compressive strength is an important measure of the quality of cement stabilised materials. The Department of Transport employs compressive strength to specify (9) the minimum quality of cement stabilised materials permitted in the base courses of road pavements. It is also the single property reported in the great majority of investigations dealing with cement stabilised materials. The investigation was initially concerned with the effect of cement content and age on the compressive strength of the various mixtures, particularly with regard to the current requirements of the Department of Transport Specification (9). This specification permits the moisture content of cement stabilised materials to be in the range of optimum to optimum + 2 per cent, and so it was decided to examine the effect of initial moisture content on a number of the cement bound minestones. The compressive strength results are presented and discussed in Chapter 7.

5.4 Experimental Study of Durability

There are currently two British tests that may be used for assessing the durability of cement stabilised materials. They are the British Standard Institution Immersion Test (69) and the Transport and Road Research Laboratory's Frost Heave Test (13). The immersion test is considered to reveal the adverse effects that sulphates and expansive clay minerals may have on the performance of a cement stabilised soil when exposed to excess moisture. This test is not mandatory and there is no universal criterion by which the performance can be judged. It seems very likely, however, that it will be included in a forthcoming revision of the Department of Transport Specification for Road and Bridge Works (9). The test was, therefore, selected for the experimental study of durability. The frost heave test was originally devised to assess the susceptibility of sub-grade soils exposed to excessive and prolonged freezing (13). The Department of Transport Specification (9) requires that materials used within 450 mm of the road surface are not frost susceptibility as defined by the frost heave test. For the majority of cement bound minestones the cement content required to satisfy the 7 day strength criterion normally exceeds that required to satisfy the frost heave criterion (15). It was suggested in Section 4.11 that the heave criterion developed for soils and unbound materials, may not be an appropriate measure for classifying the frost susceptibility of cement stabilised materials and so the test was excluded from the study of durability.

The current strength criterion (9) used to assess cement stabilised materials has been devised from an earlier criterion (60) which was considered to correlate with satisfactory performance in the ASTM wet-dry and freeze-thaw tests. The procedures used in these durability tests (56,57) were developed to simulate, in magnitude and nature, the forces generated in compacted soil-cement associated with a change in moisture content or with freezing (23). By adopting these procedures it was considered that the performance of cement bound minestones could be compared with established and proven durability criteria.

It was originally intended to include both the wet-dry and freeze-thaw test in the experimental study, however, a review of the literature revealed that the elevated temperature used in the wet-dry test was unduly severe on materials containing an appreciable amount of silt or clay sized materials (73). It was felt that the test would also be unduly severe on cement bound minestones, not only because many contain a high proportion of fine material, but also, because of the fissile nature of the shale particles present in minestones. It was, therefore, decided to include only the freeze-thaw test in the study of durability.

The experimental study of durability examined the effects of cement content and initial moisture content. The results are presented in chapter 8 and discussed in chapter 9.

CHAPTER 6 MATERIAL CLASSIFICATION AND PROPERTIES

6.1 Introduction

It is evident from the literature review that the response of a soil to the addition of chemical stabilisers is greatly influenced by the physical, chemical and mineralogical characteristics of the soil. Seven minestones were selected from separate collieries so as to provide samples with a wide range of properties. The geographical locations of the minestones are shown in figure 6.1.

This chapter is concerned with the determination of those properties of minestones which could assist in the interpretation of the results of the durability investigation. In addition, they would enable the minestones to be described. The properties examined were, particle size distribution, plasticity, sulphate content, specific gravity, clay mineralogy and cation exchange capacity. The moisture-density relationships of the compacted minestone-cement mixtures were also determined for subsequent mix designs to be used in the strength and durability investigations.

Particle size distribution and plasticity are important properties which are used by practising engineers to classify soils. Freshly dug colliery spoil contains iron pyrites (FeS_2) which, when exposed to air and water, oxidise and, in the presence of alkali and alkaline-earth compounds, results in the formation of the sulphates of calcium, magnesium, sodium and potassium (81). The presence of excessive amounts of soluble sulphates in the raw material will usually result in the disintegration of compacted soil-cement mixtures when exposed to excessive amounts of moisture (84) In cohesive soils the problem is not necessarily solved by the use of sulphate resisting cement, since the disruptive mineral ettringite (calcium sulpho-aluminate) may be formed by the reaction of calcium sulphate and alumina present in the clay minerals (80). The Department of Transport Specification (9) includes clauses which define requirements for the particle size distribution, plasticity, and total sulphate content of raw materials for use in cement stabilised materials for pavement construction. The specific gravity of the raw material is necessary for calculating the air voids content of the compacted minestone-cement mixtures. A study of the clay mineralogy will help to identify those minerals which are sensitive to moisture changes or are highly reactive with lime liberated during hydration of cement. The cation exchange capacity may indicate the possibility of calcium uptake with resultant lime depletion and retardation of cement hydration (35).

In addition to the minestone samples, a laboratory blend of brickearth, sand and gravel was also investigated. This was produced as a reference material in the strength and durability studies and so was also included in this study of material properties.

6.2 Minestone Sampling Procedure

The minestone samples were provided by the National Coal Board and were obtained using well defined sampling procedures (85). Samples were taken from one specific location in the stockpile in an attempt to restrict the variation (in terms of particle size and lithology) between samples from the same source. No attempt was made to provide a sample, or samples, representative of the stockpile as a whole. All samples were taken from material at least 0.5 m below the surface so as to avoid the effects of surface weathering. Prior to despatch each sample was screened on a 50 mm sieve, and placed in polythene bags. All the samples were, therefore, kept at their natural moisture content until required for testing.

6.3 Material Preparation

In the main investigation, of strength and durability, it was decided to use 100 mm cubical specimens and so the maximum particle size was limited to 28 mm. Thus, the material was screened on a 28 mm sieve with the larger particles being discarded. This screening of the material, at the natural moisture content, proved difficult and time consuming, so it was decided to initially air dry the material to assist the process. One or two days of air drying was sufficient to permit screening. The screened material was then stored in air tight containers until required

6.4 Classification Tests

The standard procedures for testing soils, as described in BS 1377:1975 (64), are not directly applicable to minestones which may degrade if dried and re-wetted before testing (85). It was for this reason that only the minimum amount of air drying was used prior to screening. The degree of degradation depends on both the source of the individual minestone and its location within the tip. The National Coal Board has issued a Technical Memorandum (85) which modifies the standard procedures (64) to minimise pre-treatment of .the material, The NCB Technical Memorandum was based on the 1967 edition of BS 1377, however, a revised and metricated version, BS 1377:1975 (64), has now been issued and the test procedures were, generally, related to this version. It is considered that cement stabilisation will normally be performed on material from a tip and so the only form of pre-treatment likely to be employed is air drying. It was, therefore, decided that procedures would be used where applicable to minimise further drying of the materials.

6.4.1 Particle Size Distribution. The particle size distribution of the minestone was determined by the standard wet sieving method (64) modified (85) to avoid drying of the material prior to testing (85). The modification entails the sub-division of an undried sub-sample into two parts; one part is wet sieved using a procedure similar to the standard method, and the other is issued for a moisture content determination. The moisture content was used in the calculation for the weights of the various fractions.

The particle size distribution of the material passing the 75 um sieve was determined by the standard pipette method (64). The test has been modified since the publication of the Technical Memorandum (85) and so the following modifications were made to the procedure. The oven used to dry the sample following pre-treatment was set to 50°C so as to avoid exposure to high temperatures and possible aggregation or cementation of such fine materials. Prior to dispersal the sample was sub-divided into two parts; one for the normal pipette procedure and the other for a moisture content determination. The moisture content was used in the calculation for the weights of the various fractions. The dispersal time was reduced from 4 hours to 10 minutes, in line with similar reductions recommended in the Technical Memorandum (85), to avoid further breakdown of the minestone as received from the stockpile.

The particle size distribution of each minestone sample is given in Table 6.1 and presented graphically in figure 6.2.

The particle size distribution of the brickearth, sand and gravel, used to produce the laboratory blend, were determined using the standard wet sieve and standard pipette techniques (64). The brickearth, sand, and gravel were blended in the proportions of 1:2:5. The particle size distribution of the individual constituents and the resulting laboratory blend are given in Table 6.2. In figure 6.3 the laboratory blend is compared with the

Table 6.1 Results of the Partical Size Analysis - Minestones

Minestone	Percentage finer than.									
	mm.				:		um.			
	20	10	5	2.4	1.2	600	300	150	75	2
Snowdown-1	87	63	39	23	14	10	8	7	6	1.5
Snowdown-2	87	60	38	24	16	12	9	7	6	2
Hem Heath	91	73	52	36	24	16	10	8	6	2
Wardley	93	74	50	32	21	14	9	8	7	3
Parkside	97	84	70	59	51	42	34	28	25	6
Blidworth	97	82	64	49	39	33	28	25	23	10
Bagworth	92	76	61	51	43	39	35	33	31	0
Peckfield	91	79	69	60	53	46	41	36	35	0
Specified* (finer than)	45	35	25	-	-	8	5	-	0	-

* D.O.T. Specification - Clause 805 Soil-Cement(9).

Table 6.2 Results of the Partical Size Analysis - Lab. Blend.

	Percentage finer than.									
	mm.				:		um.			
	20	10	5	2.4	1.2	600	300	150	75	2
Gravel	100	40	15	5	3	3	0	0	0	0
Sand	100	100	97	82	70	57	14	3	2	0
Brickearth	100	100	100	100	100	100	98	95	87	14
Lab Blend*	100	62	46	36	32	29	17	10	8	1

* 62.5°/o Gravel + 25°/o Sand + 12.5°/o Brickearth.

Table 6.3 Results of the Plasticity Tests

(1) DOT Specification Clause 805 Soil Cement (9).

minestone samples.

6.4.2 Plasticity Limits. The plasticity limits were determined using the standard methods of testing (64). The Technical Memorandum does not require any modification of the plasticity tests. Although these tests are not normally required (85) for colliery spoils containing less than 10 per cent finer than 63 um, it was decided to test all the minestone samples. The results are then available for comparison with the clay mineralogy and for consideration with respect to the main study. The results are given in Table 6.3.

6.4.3 Sulphate Content. Both the water soluble and total acid soluble contents were determined using the standard methods described in tests 9 and 10 of BS 1377:1975 (64). Again no modifications of the procedures was necessary and the results are given in Table 6.4.

6.4.4 Specific Gravity. The specific gravities were determined using the standard method for medium and coarse grained soils (64). The values obtained are given in Table 6.5.

6.5 Mineralogical Composition

The mineralogical composition of the clay fraction of six of the minestone samples and the laboratory blend was determined by the x-ray diffraction technique. A description of the method used and the results obtained are given in Appendix A.2. The mineralogical composition of the samples analysed are given in Table 6.6. The value given for the proportion of illite in each sample includes the mixed layered minerals, illite-vermiculite or illite-montmorillonite. Where possible these mixed layered minerals have been identified and they are included in Table 6.6.

6.6 Cation Exchange Capacity

The cation exchange capacity (CEC) of the fine fraction of each minestone sample was investigated. Three size fractions were tested (1200-475 um, 475-63 um and sub 63 um) to investigate the variation in CEC with particle size. The tests were carried out using barium saturation, subsequently acid stripped and the CEC determined using atomic absorption spectrophotometry (the method is given in full in Appendix A.3). The results are expressed in millequivalents per 100 grammes (meq/100 g) and are given in Table 6.6.

6.7 Moisture Content-Density Relationships

The moisture content-density relationships of the stabilised mixtures (8^{o}/o cement content) were determined using the vibrating hammer compaction test described in test 5 of BS 1924:1975 (64). It was proposed that mixes for the durability and strength investigations would contain between 5 and 12 per cent cement. It seemed, therefore, appropriate to select a cement content of 8 per cent for the compaction study. It was further proposed that the optimum moisture content obtained from this study

Table 6.4 Results of the Sulphate Analyses

Sample	Acid Soluble ($^0/_0$ SO_3) BS 1377:Test 9	Water Soluble ($^0/_0$ SO_3) BS 1377:Test 10
Snowdown-2	0.45	0.13
Hem Heath	0.31	0.15
Wardley	0.21	0.14
Parkside	0.21	0.19
Blidworth	0.20	0.10
Bagworth	0.32	0.11
Peckfield	0.37	0.14

Table 6.5 Results of Specific Gravity Tests

Sample	Specific Gravity
Snowdown-1	2.52
Snowdown-2	2.60
Hem Heath	2.49
Wardley	2.45
Parkside	2.27
Blidworth	2.56
Bagworth	2.44
Peckfield	2.33
Lab Blend	2.69

Table 6.6 Mineralogical Composition and Cation Exchange Capacity

Sample	Approximate Proportion of Clay Minerals (°/o)				Mixed-Layered Minerals Detected	Nature of Minerals Ordering	Cation Exchange Capacity (meq/100 g)		
	Kaolinite	Illite*	Montmorillonite	Chlorite			1.2 mm-475 micron	475-63 micron	63-0 micron
Snowdown	36	64	0	0	-	Poor	17.5	13.9	10.4
Hem Heath	94	6	0	0	-	Good	12.1	12.5	12.6
Wardley	- NOT DETERMINED -				-	-	13.7	6.2	6.6
Parkside	10	90	0	-	I/V	Poor	20.6	45.1	48.3
Blidworth	36	54	0	10	I/V	Good	25.4	22.7	22.2
Bagworth	45	55	0	trace	I/V + I/M	Good	38.0	46.3	35.9
Peckfield	50	50	0	0	I/M	Good	4.8	0.8	2.2
Lab Blend	34	39	27	-	-	Poor	- NOT DETERMINED -		

* Including Mixed Layered Minerals

I/V = Illite/Vermiculite

I/M = Illite/Montmorillonite

could be used in the preparation of all the mixtures.

At least 7 days before the test was conducted the moisture content of the raw material was increased to approximately two thirds of the estimated optimum value. A minimum period of 7 days was allowed for the internal moisture content to stabilise so that the hygroscopic requirement of the sample was satisfied prior to final mixing. In the final mixing stage immediately before the compaction test, the cement and additional water was added.

The moisture content-density relationship of the compacted mixtures are presented graphically in figures 6.4a and 6.4b. The optimum moisture contents, and corresponding maximum dry densities, of the compacted mixtures, are given in Table 6.7 which also includes the air voids content at the optimum moisture content for each mixture.

6.8 Discussion of the Results

6.8.1 <u>Classification.</u> The grading curves given in figures 6.2 and 6.3 indicate that minestones contain between 40 and 80 per cent gravel sized material. The samples from Snowdown, Hem Heath, and Wardley collieries were well graded. They contained only limited amounts of material finer than 75 um, and are unquestionably granular materials. Although the Parkside sample contained 25 per cent of such fine material, the majority of this fraction was coarse silt sized particles rather than clay. The Parkside sample was also considered to be granular. The samples from Blidworth, Bagworth and Peckfield, had

Table 6.7 Results of Compaction Tests

Sample	Optimum Moisture Content (°/o)	Maximum Dry Density (g/ml)	Air Voids (°/o)
Snowdown-1	8	2.05	3.4
Snowdown-2	8	2.13	2.3
Hem Heath	8	2.06	2.0
Wardley	7	2.06	2.8
Parkside	13	1.76	1.1
Blidworth	11	1.99	1.5
Bagworth	14	1.82	1.1
Peckfield	19	1.63	0.7
Lab Blend	5	2.34	2.2

inferior gradings and contained 23, 31 and 35 per cent respectively of this fine material. The fine fraction of the Blidworth sample was composed of approximately equal amounts of clay sized and silt sized material. However, the fine fraction of both the Bagworth and Peckfield samples were composed almost entirely of silt sized material. During sedimentation of the Bagworth and Peckfield samples the sediments settled rapidly leaving a sharply defined layer of clear water above the suspension. This suggests high aggregation of the fine fraction (64) due, possibly, to the presence of calcium compounds (85). Hydrochloric acid may be used to disperse such soils (63) but its use was avoided to prevent modification of the clay mineral in the suspension which was required for the x-ray diffraction analysis. The apparent lack of clay-sized particles may also be attributed to the washing process undertaken at the colliery. The fine material (or tailings), containing the clay sized particles, are handled separately to the washing discard. The materials are not re-combined and so the discard, particularly from modern coal preparation plants, is likely to lack clay sized particles. The Blidworth, Bagworth and Peckfield minestones are classified as granular materials displaying some cohesion.

The plasticity characteristics of the fine fraction of the samples, given in Table 6.4, ranged from low, for the well graded Snowdown, Hem Heath and Wardley samples, through intermediate for the Parkside, Blidworth and Bagworth samples, to high for the Peckfield sample.

Maximum plastic and liquid limits are specified in clause 805 of the Department of Transport specification (9) to ensure that the material can be satisfactorily mixed with cement. The plasticity values given in the specification (9) may be considered too conservative for the modern mixing plant that is available for the processing of cement stabilised mixtures. The plastic limit values of some of the dominantly granular minestone samples marginally exceeded the specified maximum. However, it was considered unlikely that this would impair the mixing of these materials since the fine fraction represents only a relatively small proportion of the whole sample. The higher plasticity of the fine fraction of the Bagworth and Peckfield minestone may limit intimate mixing of the cement and minestone.

The grading of the laboratory blend resembled that of the well graded minestone samples, although there is a lack of particles in the 1 mm to 3 mm size range. The fine fraction was found to be non-plastic.

In Table 6.8 the samples have been classified according to the method recommended by Dumbleton (86). This method is based upon particle size distribution and plasticity of the fine fraction.

6.8.2 <u>Sulphate Content.</u> As can be seen in Table 6.4, the water soluble sulphate contents of the minestones are all very similar, with an average value of 0.14 per cent. Apart from the Parkside samples, the acid soluble, or total (9) sulphate content of the minestones was greater than the water soluble sulphate content by a factor of 2 or 3. The Snowdown sample at 0.45 per cent total sulphate content, had the highest concentration of sulphate ions. Clause 801 of the Department of Transport Specification (9) defines the permitted sulphate content for soils which are to be mixed with cement. Granular soils shall not contain more than 1.0 per cent total (acid soluble) sulphate whilst cohesive soils shall not contain more than 0.25 per cent. It has been suggested (81) that the more stringent limit of 0.25 per cent should apply to all soil containing clay minerals. The sulphate contents of the samples from Snowdown, Hem Heath, Bagworth and Peckfield would, therefore, appear to exceed the specified limit.

6.8.3 <u>Clay Mineralogy.</u> From the results in Table 6.6, it was clear that the clay minerals kaolinite and illite were observed in all the minestone samples. Chlorite was also positively identified in the Blidworth and Bagworth samples. The mixed layered mineral illite/vermiculite was positively identified in the Parkside and Bagworth samples and traces were also observed in the Blidworth sample. The mixed layered mineral illite/montmorillonite was positively identified in the Bagworth and Peckfield samples. The highly active and expansive clay mineral montmorillonite was not observed in any of the minestone samples. The type and nature of the clay minerals observed is consistent with other studies of the mineralogical composition of colliery spoils (4,5).

The minerals kaolinite, illite and chlorite are not

Table 6.8 Classification and Description of Samples

Sample	Classification Code (86)	Description
Snowdown	G, W, L	Well graded GRAVEL with *some low plastic fines.*
Hem Heath	G, W, L	- ditto -
Wardley	G, W, L	- ditto -
Parkside	G, C, I	GRAVEL with medium plastic fines containing "C" type clay.
Blidworth	G, C, I	- ditto -
Bagworth	G, C, I	- ditto -
Peckfield	G, M, H	GRAVEL with high plastic fines containing "M" type clay.
Lab Blend	G, P, N	Slightly gap graded GRAVEL with some non-plastic fines.

Table 6.9 Comparison of Mineralogical, Particle Size, and Plasticity Data

Sample	Predominant Mineral	BS 1377:1975 (69) Particle Size < 75 um	BS 1377:1975 (69) Particle Size < 2 um	Plasticity Index
Snowdown	Illite	7	< 2	10
Hem Heath	Kaolinite	8	2	8
Wardley	-	8	3	10
Parkside	Illite	28	6	14
Blidworth	Illite	25	10	16
Bagworth	Illite-Kaolinite	33	0	22
Peckfield	Illite-Kaolinite	36	0	25

expansive (35) and do not, therefore, swell significantly upon contact with water. Furthermore, their reaction with lime is negligible and will not, therefore, interfere with the normal hydration and hardening of the cement (35). The mixed layered minerals are poorly ordered and constitute only a minor proportion of the mineralogy of the minestones in which they were observed. They will not significantly influence the behaviour of the clay fraction.

The clay minerals kaolinite, illite and montmorillonite were identified in the laboratory blend. The clay sized fraction of the laboratory blend constitutes less than 1 per cent of the total material and, therefore, the montmorillonite is unlikely to have any major influence on its behaviour when mixed with cements and/or exposed to water.

Data from the mineralogical analyses, particle size analyses, and plasticity tests are compared in Table 6.9. The plasticity of pure clays generally increases in the order kaolinite, illite, chlorite, montmorillonite (35). The Table shows that the plasticity of minestones is a function of particle size grading rather than mineralogy. The data from the Snowdown and Hem Heath samples, which have similar grading, may suggest that the mineralogy has influenced the plasticity but both the grading and plasticity are within the range of experimental error and sample variation. The kaolinite rich Hem Heath sample has a lower plasticity than the illite rich Snowdown sample.

6.8.4 Cation Exchange Capacity. A comparison between the data in Tables 6.1 and 6.6 indicates that, with the exception of the sample from Peckfield, the cation exchange capacities (CEC's) of the coarser grained materials from Snowdown, Wardley and Hem Heath, with values of 6.2 to 17.5 meq/100 g, are lower than those of the finer grained materials from Parkside, Blidworth and Bagworth. The CEC's of the Peckfield samples were the lowest recorded and ranged from 0.8 to 4.8 meq/100 g. The CEC of clay minerals increases in the order kaolinite, illite, montmorillonite (87). Although the results indicate that the CEC of minestones is largely a function of particle size distribution, there is some evidence that the mineralogy may influence the value. Among the coarser grained materials the illite rich Snowdown sample has, on average, a higher CEC than the kaolinite rich Hem Heath sample. In addition, the highest values were recorded for the illite rich Parkside and Bagworth samples. The very low values of the Peckfield sample are, possibly, a result of the high aggregation implied by the sedimentation test. The Bagworth sample displayed the same characteristics during sedimentation analyses but gave a CEC in the region of 40 meq/100 g. This discrepancy has not been accounted for but the nature of the aggregation must differ between the two samples such that exchange sites are available for one and not the other.

The values of CEC given in Table 6.6 do not necessarily reflect the distribution of cation exchange sites in the fine fraction of the minestone samples because the size of the individual fractions has not been accounted for. In addition, since in cement stabilisation the cation available for exchange is calcium, it is convenient to express the capacities in terms of milligrams of calcium hydroxide per 100 grammes of minestone (mg $Ca(OH)_2$/100 g).

The following equation was used to convert the cation exchange capacity (meq/100 g) of each fraction to exchange capacity (mg $Ca(OH)_2$/100 g).

$$EC = \frac{P}{100} \times W \times CEC$$

where: EC = exchange capacity (mg $Ca(OH)_2$/100 g)

P = fraction size (°/o)

W = equivalent weight of $Ca(OH)_2$ = 37

CEC = cation exchange capacity (meq/100 g)

The exchange capacity of the minestone sample is expressed as the sum of the exchange capacities of the three fractions on the basis that the majority of the exchange sites are associated with the fine fraction. The exchange capacity values are given in Table 6.10.

Neville (44) implies that a typical ordinary portland cement, when fully hydrated, liberates a quantity of lime amounting to approximately 30 per cent of its own weight. A minestone-cement mixture containing 5 per cent cement, therefore, produces approximately 1.5 per cent lime, and a mixture containing 10 per cent cement ultimately produces 3

Table 6.10 Exchange Capacity of Minestones Expressed as Hydrated Lime ($Ca(OH)_2$)

Sample	Exchange Capacity of Sample Fractions						Exchange Capacity of Minestone Sample	
	1.2 mm-475 micron		475-63 micron		63-0 micron			
	Size (°/o)	$Ca(OH)_2$/100 g (mg)	Size (°/o)	$Ca(OH)_2$/100 g (mg)	Size (°/o)	$Ca(OH)_2$/100 g (mg)	$Ca(OH)_2$/100 g (mg)	$Ca(OH)_2$ (°/o)
Snowdown	5	32.4	4	20.5	5	19.2	72.1	0.07
Hem Heath	11	49.2	8	37.0	5	23.3	109.5	0.11
Wardley	9	45.7	6	13.8	6	14.7	74.2	0.07
Parkside	12	91.4	19	317.1	20	357.4	765.9	0.77
Blidworth	9	84.5	10	84.0	20	164.3	332.8	0.33
Bagworth	6	84.4	7	119.9	30	398.5	602.8	0.60
Peckfield	11	19.6	8	2.4	34	27.8	49.8	0.05

per cent lime. The lime demand of the coarser grained Snowdown, Wardley and Hem Heath minestones amounts to approximately 0.1 per cent and is unlikely to effect, significantly, the hydration of cement. The lime demand of the finer grained materials is higher. At a cement content of 5 per cent Parkside and Bagworth minestone demands in excess of 30 per cent of the total lime produced and it is considered likely that the hydration process may be retarded. At higher cement contents their lime demand will be less significant, but it may still retard the initial hydration and strength development.

6.8.5 <u>Moisture Content-Density Relationships.</u> The moisture content-density relationships of the minestone-cement mixtures are shown in figures 6.4a and 6.4b. The zero air voids relationships for each mixture, computed from the specific gravity data and modified (64) for cement content, are also included in the figures. The maximum dry density and optimum moisture content for each mixture is given in Table 6.7 and corresponding air voids content is given for comparison. Values are compatible (63) with the particle size distribution (Table 6.1) and specific gravity (Table 6.5) of the raw minestones. That is the coarser grained minestones, from Snowdown, Hem Heath, and Wardley, produce mixtures with higher maximum dry densities, and lower optimum moisture contents, than mixtures produced from the other finer grained minestones. The Peckfield sample, containing the highest proportion of fine material, produced a mixture which had the lowest maximum dry density and highest optimum moisture content of all the minestone-cement mixtures. The laboratory blend-cement mixture had the highest maximum dry density, and the lowest optimum moisture content, of all the mixtures tested.

The two batches from Snowdown were observed to have the same optimum moisture content, however, the second batch achieved higher dry densities. This result is consistent with the specific gravity measurements which gave a higher value for the second batch. These results are considered to be within the limits of the variability likely to be encountered when sampling from different locations on the stockpile.

The air voids content of the minestone-cement mixtures ranged from 3.4 to 0.7 per cent and appears to be a function of particle size distribution. The finer grained minestones, by their very nature, contain a higher proportion of fine material capable of filling the voids between larger particles and so these mixtures have a low air voids content. The air voids content of the Peckfield minestone-cement mixture was only 0.7 per cent and this may be an indication that the aggregated particles were crushed under the action of the hammer. The air voids content of the laboratory blend-cement mixture was similar to that of the mixtures produced from the coarser grained minestones.

Figure 6.1 Geographical locations of minestone samples

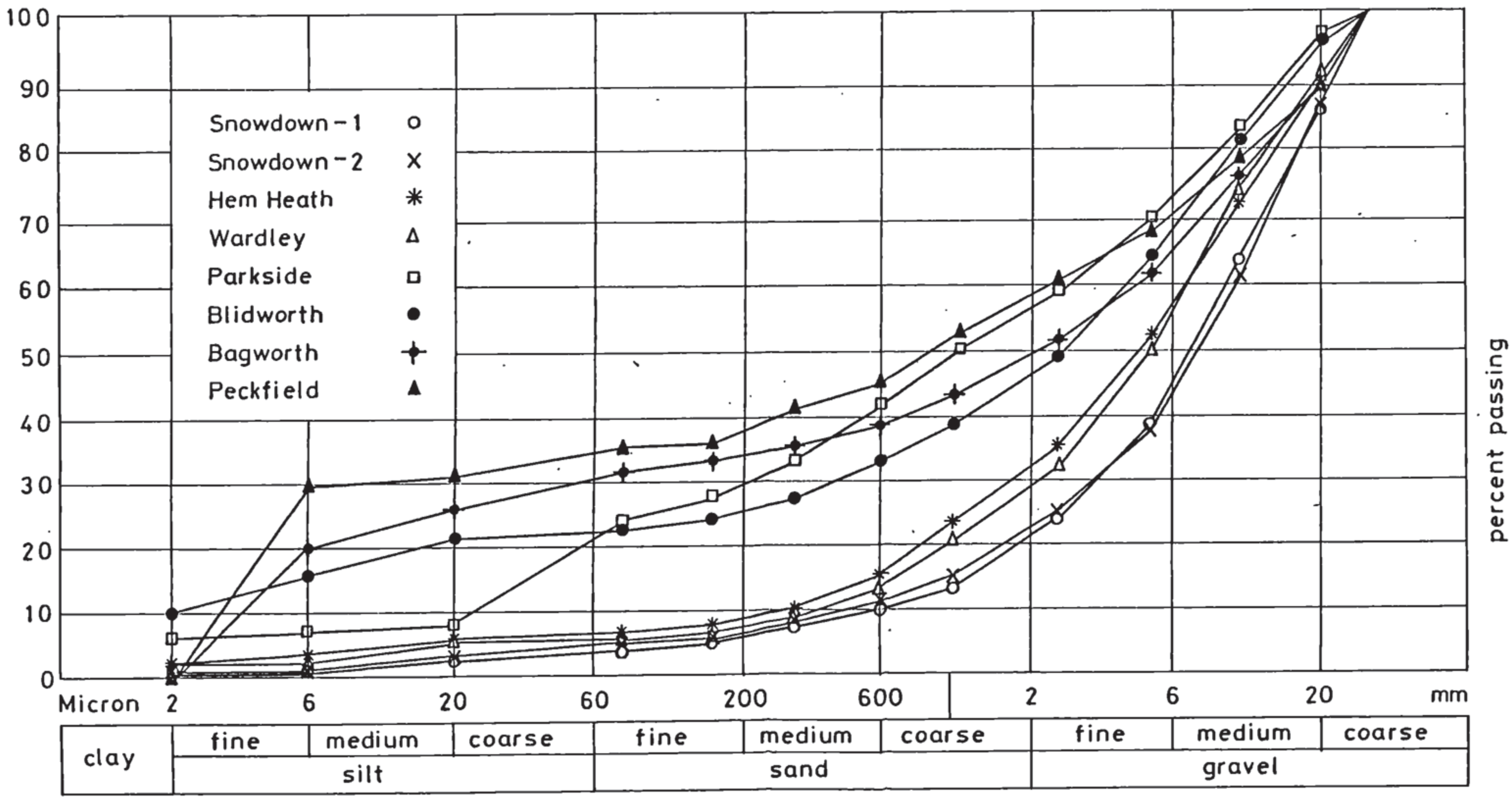


Figure 6.2 Particle size analysis of minestones

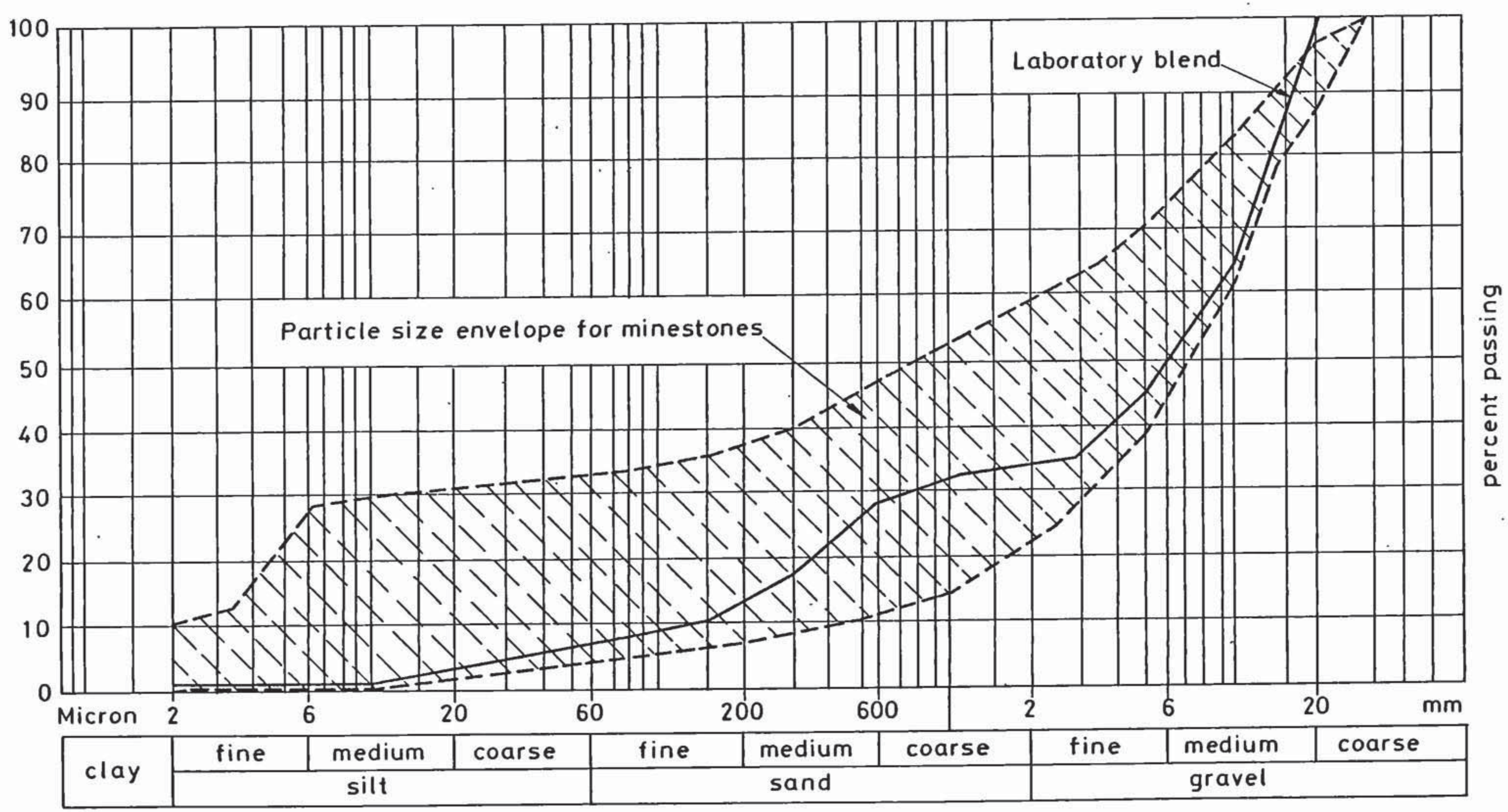


Figure 6.3 Particle size distribution of laboratory blend

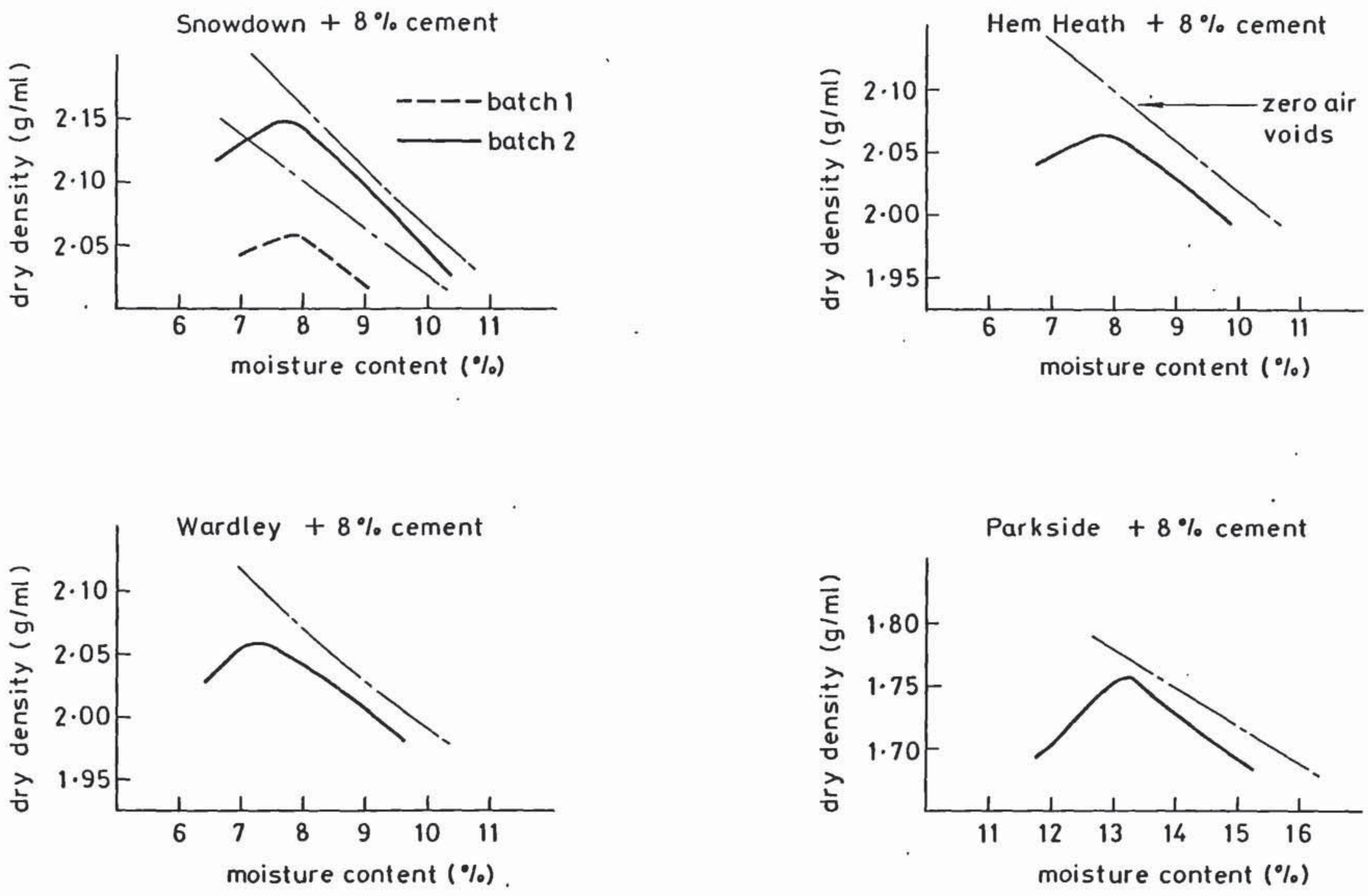


Figure 6.4a Moisture content / density relationships of compacted mixtures

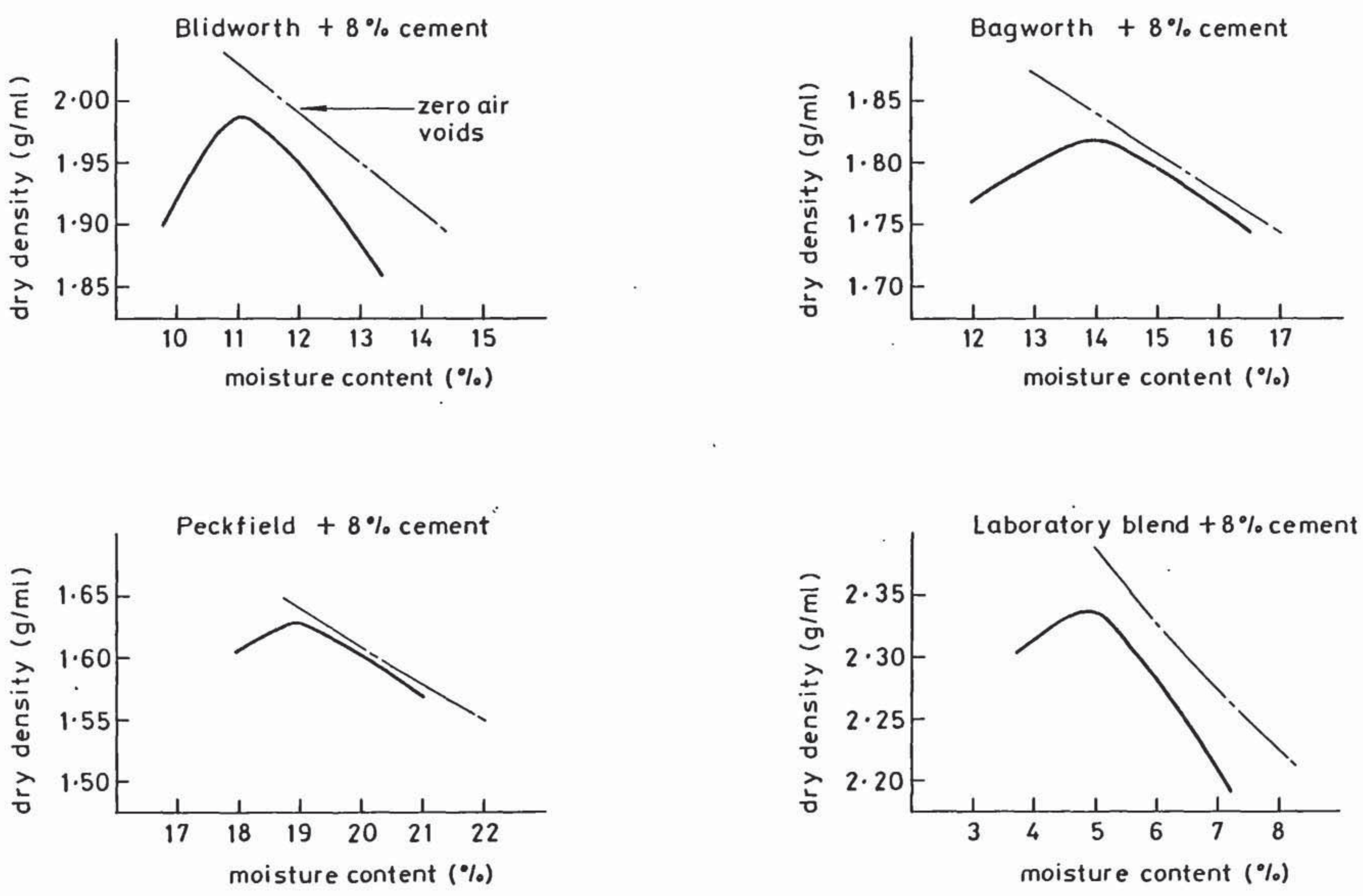


Figure 6.4b Moisture content / density relationships of compacted mixtures

CHAPTER 7 COMPRESSIVE STRENGTH.

7.1 Introduction

The compressive strength provides a measure of the ability of cement to improve the mechanical properties of a soil or, in this case, minestone. The Department of Transport Specification for Road and Bridge Works (9) uses compressive strength as the basis of compliance testing for cement stabilised materials. It is also a property reported in the great majority of investigations dealing with cement stabilised materials and hence provides a means for comparison. It therefore seemed appropriate that a study of cement stabilised minestone should include an investigation of the compressive strength.

The investigation was initially concerned with the effect of cement and age on the compressive strength of eight minestones and a laboratory blend. The cement contents were selected from within a range considered to be commercially viable and specimens were tested at various ages during the first month after moulding. The current specification (9) permits the moisture content of stabilised materials to be within the range - optimum to optimum plus 2 per cent. It was therefore decided to examine the effect of initial moisture content on the compressive strength of four minestone samples.

Prior to commencing the investigation it was necessary to establish procedures for mixing and preparing the materials, and also to select a suitable specimen size.

7.2 Mixer Selection

The details of the two mixers used in the investigations are given in Table 7.1. The final selection of the mixers was based on their performance and on consideration of the mixing procedure adopted. Briefly, the minestones were mixed with water and allowed to condition for 7 days in sealed drums before the final mixing with cement was undertaken.

The Hobart food mixer was selected for the initial mixing with moisture because:-

(a) during mixing the dough hook caused only minimal breakdown of the minestone particles;

(b) at the completion of the mixing, the bowl was removed and completely emptied with negligible loss of material, thus ensuring accurate batching weights for the final mixing stage.

The Winkworth mixer was selected for the final mixing stage as an earlier investigation (88) had shown it to be capable of producing consistent results with stabilised minestone mixtures. In addition its mixing action resembles that of pug mill mixers used in the full scale production of stabilised minestone (7).

7.3 Material Preparation

To ease handling the minestones were air dried at room temperature for two days. Artificial methods of drying were

Table 7.1 Details of Mixers Used

Mixer	Hobart AE125	Winkworth 11Z
Type	Positive Open Pan	Positive Pug Mill
Capacity (litres)	12	12
Blade Arrangement	1 eccentric powered "dough hook"	2 eccentric powered "Z" blades
Application	Prewetting Minestones. Blending standard material.	Final Mixing with cement and water.

avoided so as to minimise the risk of modifying the material (85). The air dried materials were screened on a 28 mm sieve and transferred to airtight containers for storage. At least 7 days before the material was required it was weighed into mix batches and the moisture content raised to approximately two thirds of the optimum value. Air dried minestones and water were mixed for one minute in the Hobart mixer and each batch was stored in individual airtight containers. A minimum period of 7 days was allowed for the internal moisture content to stabilise so that the hygroscopic requirement of the spoil was satisfied prior to final mixing. In the final mixing stage, the prewet minestone and cementwere mixed in the Winkworth mixer for one minute. Sufficient water was then added to raise the mixture to the desired moisture content and mixing continued for two more minutes.

The laboratory blend consisting of brickearth, sand, and gravel, was blended in batch weights for five minutes at a moisture content of 5 per cent and stored in airtight containers for at least seven days. The final mixing procedure was the same as that used for the minestone mixtures.

7.4 Test Specimen: Selection and Preparation

7.4.1 <u>Selection of Specimen Size</u> It is generally considered that the specimen size should be based on the grading of the material, and guidelines are given in BS 1924:1975 (69). All the minestones used in the investigation are medium grained materials for which there is a choice of two specimen types:

(i) 200 mm x 100 mm diameter cylinders requiring approximately 3.5 kg of material.

(ii) 150 mm cubes requiring approximately 7.5 kg of material.

Both specimen types were considered unsuitable for the investigation in view of the large numbers of specimens that were envisaged. The large quantity of material required to conduct a meaningful investigation would have resulted in some minestones being excluded as only relatively small supplies were available. The mixers were capable of containing only sufficient material to produce two cylindrical specimens and within the time available it would not have been possible to process all the material required. It was, therefore, decided to adopt 100 mm cube specimens for the investigation as they only required approximately 2 kg of material. The ratio between the mould dimension and the maximum particle size, of 28 mm, compares favourably with the ratio recommended in the standard (69), which permits a maximum particle size of 37.5 mm. An investigation conducted by Symons (89) on a material containing particles of up to 25 mm showed that the compressive strength of 150 mm cubes was the same as that of 100 mm cubes. Another reason favouring the use of the 100 mm cube was its suitability for use in the main study of durability.

7.4.2 <u>Preparation of Specimens.</u> The preparation of the specimens was in accordance with the procedure of test 11 in BS 1924:1975 (69). Each batch of material contained sufficient material to produce three 100 mm cubes. The specimens were compacted, using a vibrating hammer, to a predetermined density using the preferred method. One deviation was made from this method in that the 100 mm cubes were compacted in two layers rather than three. To minimise the risk of damage, the specimens were not demoulded for twenty four hours. Immediately after compaction the mould and specimen were placed in a polythene bag to minimise moisture loss and stored in a laboratory at 20°C. The following day the specimens were demoulded, weighed, wrapped in polythene film, and placed in polythene bags. They were stored in a thermostatically controlled room operating at 20°C until required for testing.

7.5 Determination of Compressive Strength

The procedure followed test 11 of BS 1924:1975 (69), "The Determination of the Unconfined Compressive Strength for Stabilised Medium and Coarse Grained Soils". Three determinations were conducted and the mean value calculated.

7.6 Test Programme

The initial programme was concerned with examining the effects of cement content and age on the strength of the stabilised mixtures. It has been suggested that a cement content of 10 per cent is the maximum for commercially viable production of cement stabilised minestone (18), however, Kennedy (90) believes that, in certain circumstances, cement contents as high as 15 per cent may be viable. The minestone samples were studied at three cement contents between 5 and 15 per cent. With some materials, however, this was reduced to only two cement contents due to the limited size of particular samples. The laboratory blend was mixed with 4, 8, and 12 per cent cement. All the specimens were produced at the appropriate optimum moisture content and the target dry density corresponded to the maximum value established in the compaction tests. Tests were performed at 7, 14, 21 and 28 days and the results of these tests are given in Table 7.2.

The current specification (9) permits the moisture content of the stabilised material to be within the range optimum to optimum plus 2 per cent. It was, therefore, considered necessary to assess the effect of the initial moisture content of the mixed material on the strength of the hardened material. For this study minestone from four collieries, Snowdown-2, Hem Heath, Wardley and Parkside, were investigated. Additional specimens were produced at moisture contents 1 and 2 per cent above the optimum value. The target densities were determined from the results of the compaction tests reported in Chapter 6. The results are given in Table 7.3.

7.7 Discussion of the Effect of Cement Content

7.7.1 Comments on Specimen Reliability. To permit useful correlation of the data, it must be established that the method of preparation produced specimens of consistent quality and characteristics. In this investigation the

Table 7.2 Compressive Strength of Specimens (N/mm^2)

Sample	Cement Content (°/o)	Dry Density (g/ml)	Moisture Content (°/o)	Age (days)			
				7	14	21	28
Snowdown-1	5	2.05	8	5.1	5.7	5.6	6.0
- do -	10	2.05	8	8.1	8.4	8.8	9.1
- do -	15	2.05	8	9.7	10.5	11.4	11.8
Snowdown-2	5	2.13	8	4.0	ND*	ND	ND
- do -	10	2.13	8	7.9	10.9	10.7	12.4
- do -	15	2.13	8	10.2	ND	ND	ND
Hem Heath	4	2.06	8	3.1	3.8	4.9	5.3
- do -	8	2.06	8	4.8	5.7	8.0	7.8
- do -	12	2.06	8	6.2	7.3	9.8	10.6
Wardley	6	2.06	7	5.8	6.7	7.0	7.4
- do -	12	2.06	7	9.4	10.2	9.9	11.6
Parkside	6	1.76	13	2.7	3.1	ND	ND
- do -	12	1.76	13	4.5	5.4	5.5	5.9
Blidworth	6	1.99	11	3.1	3.1	ND	3.4
- do -	12	1.99	11	4.6	4.6	5.7	5.2
Bagworth	6	1.82	14	2.6	3.0	ND	3.4
- do -	12	1.82	14	3.7	3.2	ND	4.3
Peckfield	6	1.52*	19	1.2	1.4	1.6	2.0
- do -	12	1.52	19	1.9	2.1	2.9	3.0
Lab Blend	4	2.34	5	10.3	11.6	12.2	14.0
- do -	8	2.34	5	21.1	21.6	23.5	25.3
- do -	12	2.34	5	28.2	28.4	33.6	31.4

* Reduced Target Density (see Sec. 7.7.1)

ND = Not Determined

Table 7.3 Compressive Strength of Specimens Produced at Various Moisture Contents

Sample	Cement Content (°/o)	Moisture Content (°/o)	Dry Density (g/ml)	Strength (N/mm^2)
Snowdown-2	10	Optimum	2.13	7.9
- do -	10	Opt + 1	2.08	9.2
- do -	10	Opt + 2	2.04	8.6
Hem Heath	8	Optimum	2.06	4.8
- do -	8	Opt + 1	2.03	5.0
- do -	8	Opt + 2	1.99	4.9
Wardley	6	Optimum	2.06	5.8
- do -	6	Opt + 1	2.04	5.2
- do -	6	Opt + 2	2.00	5.0
Parkside	12	Optimum	1.76	4.5
- do -	12	Opt + 1	1.73	4.3
- do -	12	Opt + 2	1.69	4.1

major variables of cement content, moisture content, age and curing temperature were easily and accurately controlled by careful batching, mixing and storage. One source of variability, however, concerned the compacted density of the specimens. A difference of 1 per cent in the dry density of two otherwise identical specimens can result in a difference in their compressive strength of 10 per cent (88). It was, therefore, vital that any variations in density were minimised.

Specimens produced at the optimum moisture content were difficult to compact to the target densities although, with the exception of the material from Peckfield, such densities could be achieved. With the Peckfield minestone-cement mixtures this proved to be impossible. The optimum moisture content of the mixed material appeared to be correct and so a number of trial specimens were produced at reduced density and, based on this experience, it was decided that the target density should be reduced to 93 per cent of the maximum value. Even these specimens were difficult to compact but the variability of the densities was minimised.

The author believes that the difficulties experienced during compaction arise from differences between the mode of compaction used in the vibrating hammer compaction test, and that used to produce the test cubes. In the compaction test the compacting foot compacted the whole of the exposed surface of the material so it is forced into the mould under an even pressure with no compactive effort being wasted. In the production of the cube specimen, however, the face area of the compacting foot was considerably less than the exposed surface of the material. Only part of the material was being forced into the mould at any one time with the result that adjacent material was disturbed and could be displaced rather than compacted. This displacement of the material adjacent to the compaction foot absorbed some of the compactive effort and so reduced that available to achieve the desired density. The degree of displacement, or flow, varied according to the material and appeared to be related to the plasticity of the material. Thus, the coarser grained minestone with low optimum moisture contents exhibited the least displacement. However, the Peckfield minestone-cement mixture, with an optimum moisture content of 19 per cent, and some 35 per cent of the material finer than 75 um, exhibited a considerable amount of displacement. From the experience with these plastic minstones, it would appear that the vibrating hammer may not be the most suitable equipment for compacting such mixtures.

In view of the difficulties encountered in compacting specimens, it was decided to examine the variation in density for each material. The densities were calculated using the nominal volume of the mould, this being judged acceptable in view of the method of moulding (69). These densities were subjected to a statistical analysis and the results are given in Table 7.4. The values reported in the table suggest that the densities for each material are consistent. Apart from the mixture produced from Blidworth

Table 7.4 Statistical Analysis of Densities of Specimens (Strength Development Data Only)

Sample	Snowdown 1	Snowdown 2	Hem Heath	Wardley	Parkside	Blidworth	Bagworth	Peckfield	Lab. Blend
n	36	18	36	24	18	21	18	18	36
D(kg/m^3)	2214	2300	2225	2204	1989	2209	2075	1810* (1940)	2457
D'(kg/m^3)	2211	2295	2225	2202	1993	2194	2062	1788	2455
D'/Dx100	99.9	99.8	100.0	100.0	100.0	99.3	99.4	98.8 (92.2)	100.0
S(kg/m^3)	6.45	5.76	3.96	4.64	5.38	14.29	2.38	2.84	6.30
c.v($^o/_o$)	0.29	0.25	0.18	0.21	0.27	0.65	0.12	0.16	0.26
max D (kg/m^3)	2224	2307	2231	2208	2000	2204	2067	1792	2467
min D' (kg/m^3)	2195	2284	2216	2192	1985	2169	2057	1782	2435
Range (kg/m^3)	29	23	15	16	15	35	10	10	32

n = number of specimens D = target bulk density D' = mean bulk density of cubes S = standard deviation
c.v = coeff of variation * reduced value following difficulties with compaction of specimens (see Sec. 7.7.1)

minestone, the coefficient of variation is generally less than 0.3 per cent. Other investigations of cement stabilised materials (91,92) have reported that the variability in density produced coefficients of variation in the range 0.45 to 1.75 per cent. This further demonstrates the low variability in specimen density that is shown in Table 7.4.

7.7.2 Visual Examination. All the specimens were visually inspected when stripped from their moulds. Evidence of the compaction plane was only observed on the faces of specimens produced from Peckfield and Bagworth minestones. The majority of specimens appeared to be uniformly compacted to their full depth. Occasionally areas deficient of fine material were observed on the faces of specimens produced from Snowdown and Wardley minestones. Similar areas were observed on specimens produced from the laboratory blend. These specimens did not prove to be significantly weaker or stronger than similar specimens which did not exhibit such areas.

7.7.3 7 day Strength of Minestones Specimens. The results given in Table 7.2 demonstrate that the compressive strength of cement stabilised minestones varies according to the source of the material and cement content. The minestones from Snowdown and Wardley collieries exhibited similar compressive strength values after 7 days curing. They had the highest strengths of the minestone-cement mixtures tested. The stabilised material from Hem Heath had a lower compressive strength after 7 days curing than the

Snowdown and the Wardley mixtures but it was stronger than the mixtures from Parkside and Blidworth, both of which achieved similar strengths. The mixtures produced from Bagworth and Peckfield minestones had the lowest compressive strengths after 7 days.

The mechanical properties of the raw minestones appeared to have a major influence on the strength of the minestone-cement mixtures. Minestone particles are, by the very nature of their laminated structure, prone to degradation. Minestones from Parkside, Blidworth, Bagworth and Peckfield are composed of weak particles, which are easily broken in the hand. They have undergone considerable degradation in the spoil heap and contain more than 20 per cent material finer than 75 um. It was apparent that these minestones underwent further degradation during mixing and compaction and the resulting minestone-cement mixtures had low compressive strengths. Minestones from Snowdown, Hem Heath and Wardley are composed of stronger particles. They have undergone less degradation in the spoil heap and contain less than 8 per cent material finer than 75 um. The minestone-cement mixtures produced from these materials had higher compressive strengths.

The nature of minestones containing a high proportion of fine material approaches that of a truly cohesive material. When mixed with cement the optimum moisture content of such materials is high with correspondingly low values of dry density. Although a degree of pulverization occurs during mixing and compaction, regions of aggregated fine material remain. Materials consisting of aggregation of fine particles are thought to be stabilised by the formation of a continuous skeleton of cement matrix which binds the aggregation together (29,30). These materials have a high voids content which, at the cement contents used in this investigation, inhibits the formation of a continuous skeleton structure. The regions of aggregated fine materials are, therefore, poorly stabilised. A minestone-cement mixture containing an appreciable quantity of fine material will have a low dry density and consists of weak coarse particles suspended in a poorly stabilised fine matrix.

Coarser grained minestone-cement mixtures have a lower optimum moisture content and higher dry density than the finer grained minestone-cement mixtures. The regions of poorly stabilised fine matrix are fewer and smaller. The majority of particles are coated with cement and a conventional structure predominates with the relativly stonger particles joined at the points of contact. The resulting hardened material has higher compressive strength than that produced from the finer grained minestones.

The Department of Transport specifies the quality of material permitted in a road pavement (9). The material is required to achieve a minimum 7 day compressive strength of 3.5 N/mm^2. This is based on the average strength of five cubes compacted to field density. Further constraints minimise variation in the quality of the laid material. It has been suggested that under condition of good site control an average 7 day compressive strength of 5.2 N/mm^2 is required to ensure that the specified minimum is satisfactorily exceeded in the field (61).

The results of the 7 day compressive strength tests are presented in figure 7.1. Minestones from Snowdown and Wardley exceeded the suggested average value with less than 10 per cent cement. Hem Heath minestone approximately equals the suggested average with 10 per cent cement. Minestones from Parkside and Blidworth mixed with 12 per cent cement exceed the specified minimum but do not achieve the suggested average value. Both minestones from Bagworth and Peckfield fall far short of achieving the suggested average strength with cement contents of 12 per cent although Bagworth, with 12 per cent cement, just achieves the specified minimum value.

A cement content of 10 per cent has been suggested as the maximum for commercially viable production of cement stabilised minestone (18) although Kennedy (90) has suggested that higher cement contents may still be economic. Snowdown, Hem Heath and Wardley minestones satisfy the 10 per cent constraint. Parkside and Blidworth minestones may satisfactorily exceed the suggested average value with a cement content of 15 per cent and may be viable if economic factors particularly favour their use (90). Cement stabilisation of Bagworth and Peckfield minestones is not considered to be commercially viable. This assessment of commercial viability of cement stabilised minestones is based solely on their ability to satisfy the strength requirement of the specification (9). The durability of the materials has not been considered at this stage.

In figure 7.2 the ability of minestone-cement mixtures to satisfy both the specification and economic constraints is related to their particle size distribution. The minestones included in this investigation and which satisfy the strength requirement within the economic constraints fall into a relatively narrow grading zone, Zone A in figure 7.2. These minestones are composed of stronger particles and have undergone less degradation than the finer grained materials. The minestones studied which failed to satisfy the strength requirements within the economics constraints fall into a distinctly different grading zone, Zone B in figure 7.2. A possible index for assessing the ability of a material to economically achieve satisfactory strength is the amount of material finer than 75 um. From this study it would appear that minestones containing in excess of 20 per cent material finer than 75 um are unlikely to satisfy both strength and economic requirements. Minestones containing less than 10 per cent material finer than 75 um are more likely to satisfy both strength and economic constraints.

7.7.4 <u>7 day Strength of the Laboratory Blend Specimens.</u> The average 7 day compressive strength of the laboratory blend-cement mixture was over 100 per cent greater than that of the strongest minestone-cement mixture. The material had the lowest optimum moisture content, and highest dry density, of all the materials studied. The structure of the hardened material is probably similar to that of the coarser grained minestone-cement mixtures but it has a lower voids content and is composed of stronger particles. The laboratory blend-cement mixture achieves twice the suggested average compressive strength with a cement content of only 4 per cent.

7.7.5 Strength Development of Minestone Specimens. It has been reported (38,93) that with the majority of cement stabilised materials a linear relationship exists between the compressive strength and the logarithm of time. Stabilised soils of a silty or clayey nature, however, have been reported (93) to exhibit linear relationships between the logarithm of strength and the logarithm of time at early ages.

A linear regression analysis, assuming both semi-logarithmic and logarithmic relationships was conducted on each set of strength data in Table 7.2. The details of the analysis are given in Appendix A.1. The correlation coefficients for both relationships are given in Table 7.5. The significance of the correlations is difficult to assess because the amount of data available is limited but the values of the coefficients suggest that, for minestone-cement mixtures, the logarithmic relationship gives a slightly better regression line.

The form of the regression line for the logarithmic relationship is:-

$$\log S = \log A + B \log T$$

$$\text{or: } S = AT^B$$

where S = compressive strength (N/mm^2)

T = time (days)

A and B = constants determined from the regression analysis.

The values of A and B for the minestones-cement mixtures are given in Table 7.5. In the case of Parkside minestone mixed with 6 per cent cement there was only two pairs of data and so the values of A and B were determined by assuming a logarithmic relationship. The regression lines for the strength age relationship of each minestone-cement mixture are shown in figure 7.3 to 7.10. The data from Table 7.2 are included in the figures. In the logarithmic relationship the rate at which strength, S, increases with time, T, is dependent upon the values of both A and B. It can be seen from Table 5 that, in all cases, the value of A increased with cement content and that its value was generally higher for the specimens produced from the coarser grained minestones, an exception being the value for the Hem Heath specimens. This suggests that the value of A is a measure of the strength of the aggregate particles, and also the quality of the cementing structure formed during compaction. The low value obtained for Hem Heath specimens may indicate that the material is composed of weaker particles than the similar graded minestones from Snowdown and Wardley. The value of B appears to be independent of cement content but more dependent upon the source of the minestone. It is thought that its value may measure the quality of the hydration process, ie. low

Table 7.5 Results of Linear Regression Analysis

Sample	Cement Content	Constants in: $S = A\ T^B$		Coeff of Correlation ($\times 10^{-2}$)	
	(°/o)	A	B	log	Semi-log
Snowdown-1	5	4.2	0.105	93	93
	10	6.8	0.084	98	98
	15	7.3	0.145	99	99
Snowdown-2	10	4.5	0.306	95	95
Hem Heath	4	1.4	0.403	99	98
	8	2.2	0.383	95	93
	12	2.7	0.405	97	96
Wardley	6	4.2	0.166	99	99
	12	7.3	0.121	81	80
Parkside	6	2.0	0.155	-	-
	12	3.2	0.186	98	98
Blidworth	6	2.6	0.075	92	91
	12	3.6	0.122	72	71
Bagworth	6	1.7	0.200	100	100
	12	2.7	0.114	53	57
Peckfield	6	0.6	0.356	98	96
	12	1.0	0.336	94	93
Lab Blend	4	6.4	0.227	97	95
	8	15.3	0.143	94	91
	12	21.7	0.120	78	75

values indicates a retarded process, and is possibly related to the durability of the mixture. This may become apparent following the examination of durability

The logarithmic relationship may be used to determine the average strength at early ages. The semi-logarithmic relationship has been found (93) to give a better prediction of the long term strength of soil-cement mixtures.

7.7.6 Strength Development of Laboratory Blend Specimens. The logarithmic relationship gave the better regression line for this material. The values of the constants are given in Table 7.5. The regression line and data from Table 7.2 are presented in figure 7.11. The strength development of the specimens appeared to be more dependent upon the value of A than the minestone specimens. The value for specimens containing 12 per cent cement was three times the highest value obtained for the minestone-cement mixtures. This reflects the superior strength of the aggregate and the higher quality of the cementing structure. The value of B was lower than for many of the minestone specimens. This is unlikely to reflect the quality of hydration but results from the greater influence of the value of A.

7.8 Discussion of the Effect of Initial Moisture Content

The effect of the initial moisture content on the compressive strength was examined for four of the minestones - Snowdown-2, Hem Heath, Wardley, Parkside.

7.8.1 Visual Examination. The difficulties experienced when compacting specimens at the optimum moisture content did not occur with the cubes at the higher moisture contents. This suggests that the additional moisture produced an improvement in the workability of the mixtures. It is likely that the combination of higher voids content and improved inter-particle lubrication reduces fracture and degradation of the larger particles during compaction. When stripped from their moulds the specimens appeared to be uniformly compacted throughout the full depth. They appeared to be much more uniform than similar specimens produced at optimum moisture content.

7.8.2 Compressive Strength. To access the effects of moisture content on both density and strength, the compaction and strength data have been superimposed in figure 7.12. The specimens produced from Snowdown and Hem Heath minestones each have different optimum moisture contents for dry density and compressive strength. The highest strengths for both materials were developed by specimens prepared at a moisture content of 1 per cent above optimum. Another feature, common to both materials, is that the specimens prepared at 2 per cent above the optimum produced higher strengths than those prepared at the optimum value. The Wardley and Parkside specimens exhibited the highest strengths when produced at optimum moisture content, with lower strength being recorded as the moisture content was increased above the optimum.

with cement (24) reported improved mixing and greater resistance to immersion in water at moisture contents above the optimum. Similar improvements were also reported in an investigation of cement stabilised London clay (38). Snowdown and Hem Heath minestones have clearly benefited from the increase in moisture content. At the higher moisture contents there was an improvement in the workability of these mixtures so that:-

(a) mixing was improved leading to a more uniform distribution of cement;

(b) less damage was caused to larger particles during mixing and compaction;

(c) the specimens were more uniformly compacted.

These factors gave rise to the increased strength reported for the Snowdown and Hem Heath specimens. It is suggested that the improved strength could also produce improvements in durability.

The benefits of increasing the moisture content are not so apparent from the strengths of Wardley and Parkside specimens. However, the benefits of improved workability may have been masked by the reduction of strength due to the lower dry density of specimens produced at the higher moisture contents. Any benefits will become apparent during the study of durability.

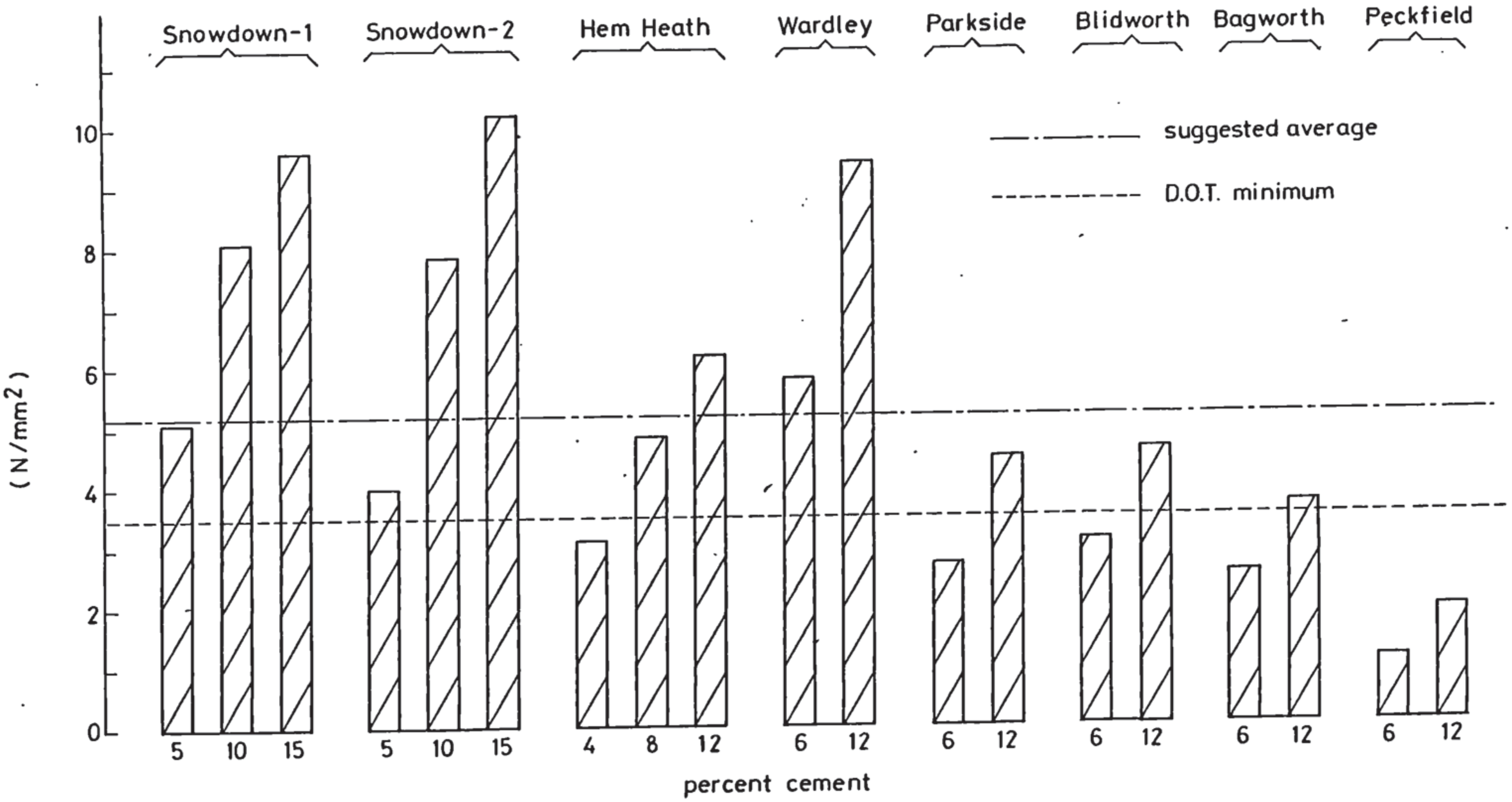


Figure 7.1 Average 7 day compressive strength of cement stabilised minestones

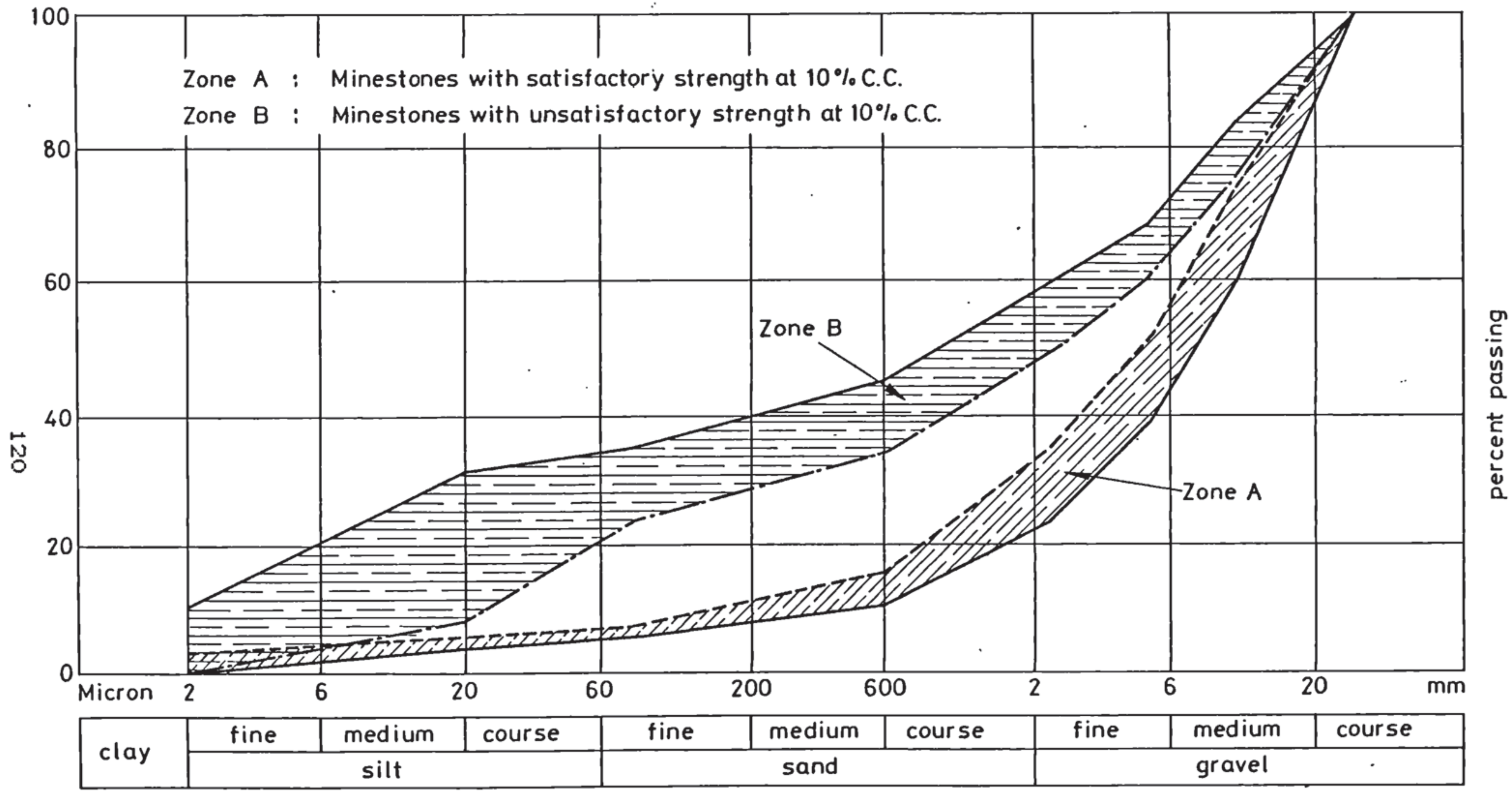


Figure 7.2 The relationship between compressive strength and particle size distribution

STRENGTH DEVELOPMENT

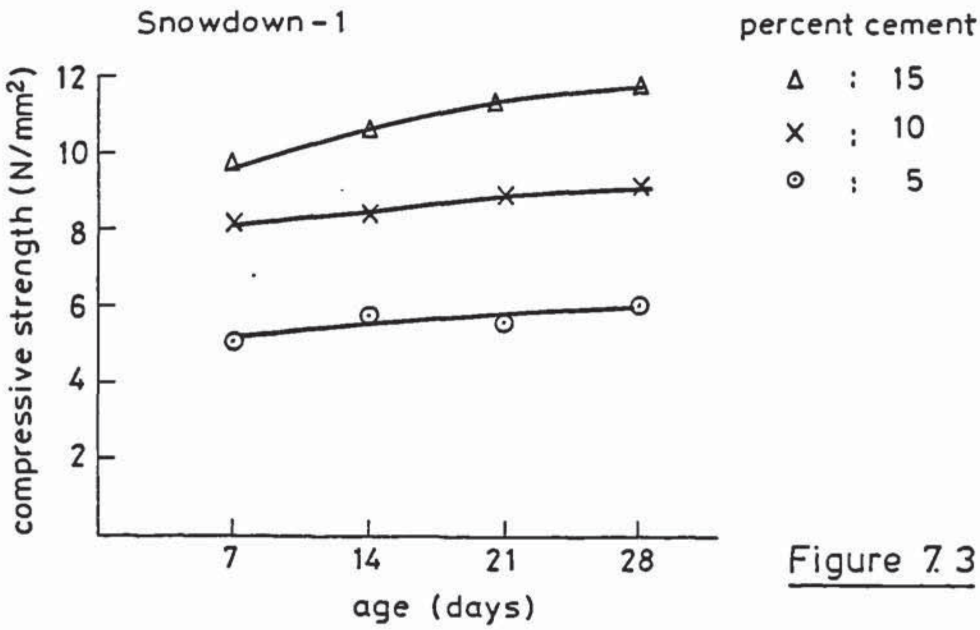


Figure 7.3

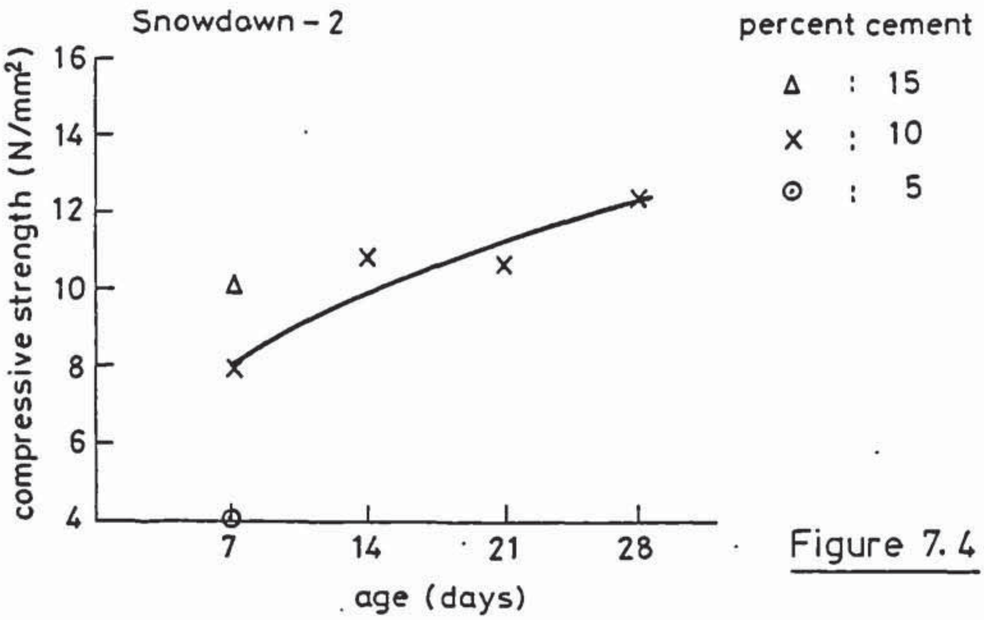


Figure 7.4

Figure 7.5

STRENGTH DEVELOPMENT

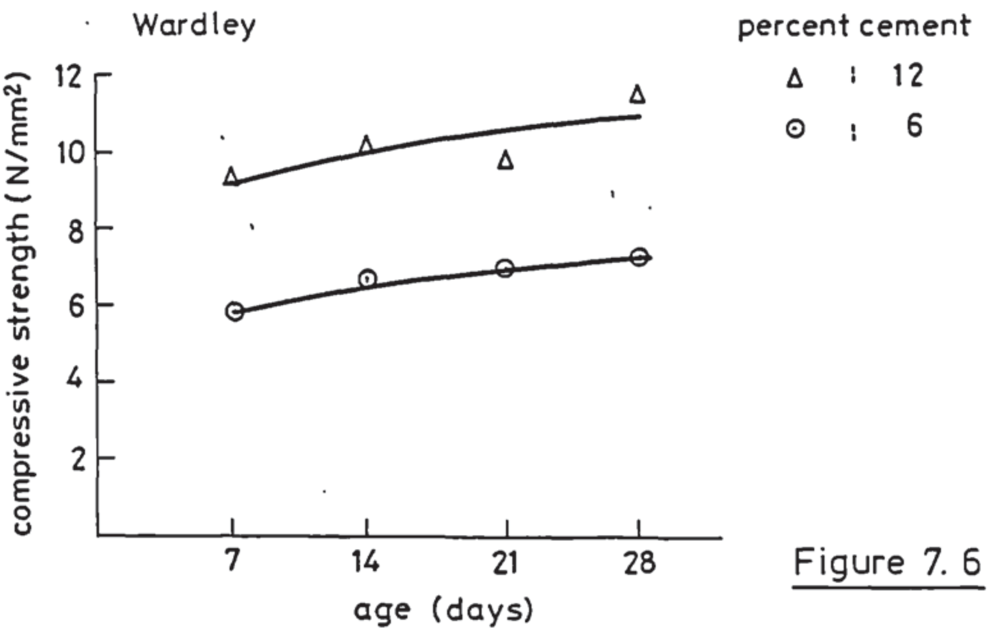


Figure 7.6

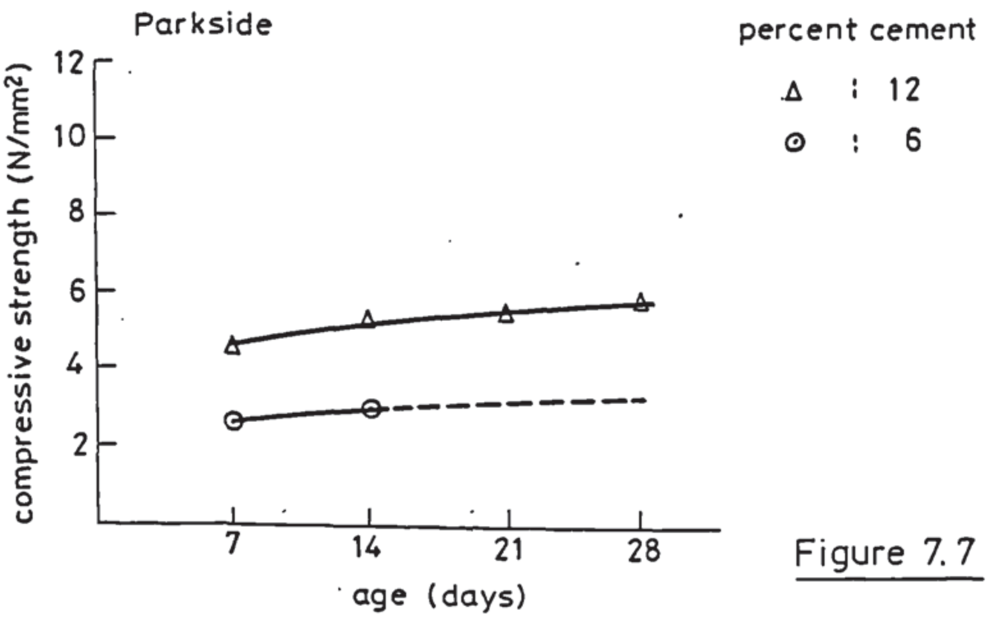


Figure 7.7

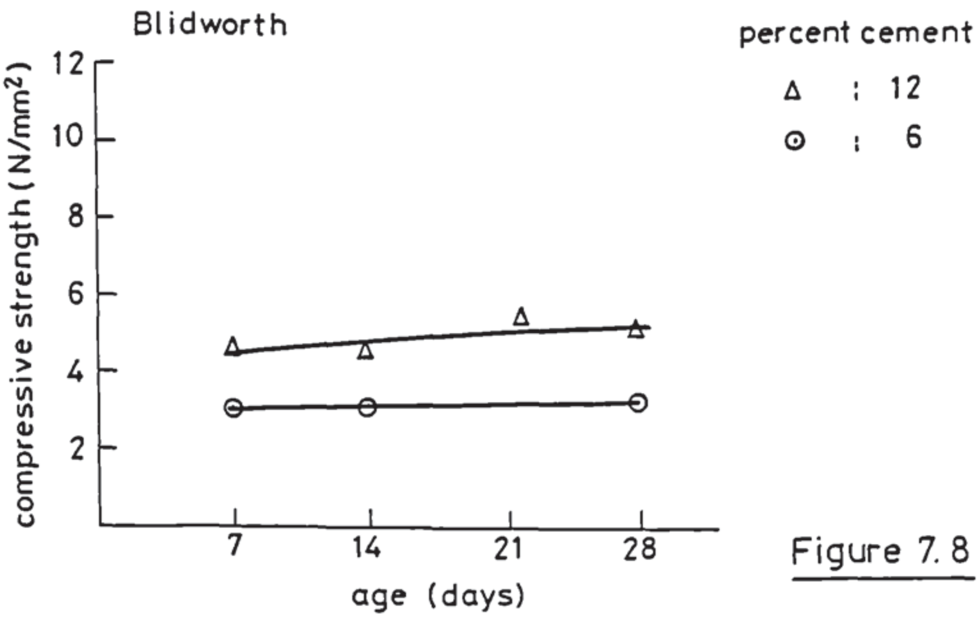


Figure 7.8

STRENGTH DEVELOPMENT

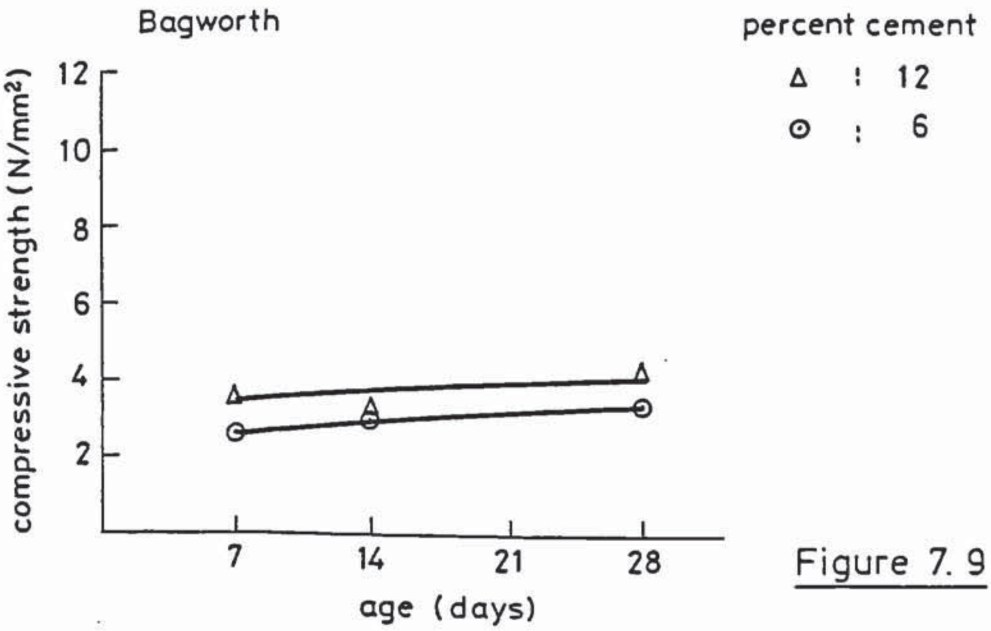


Figure 7.9

Figure 7.10

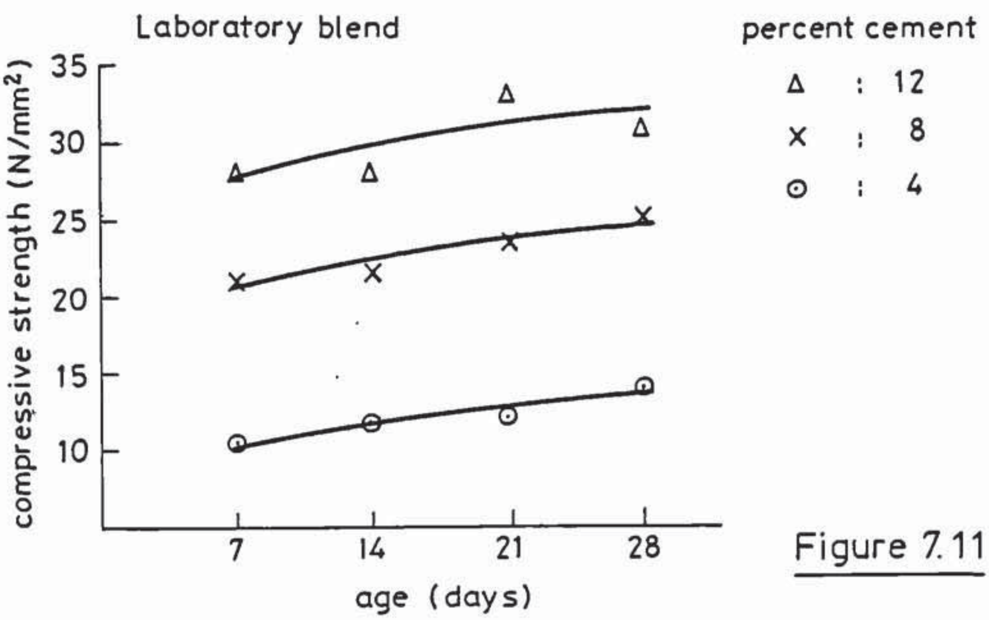


Figure 7.11

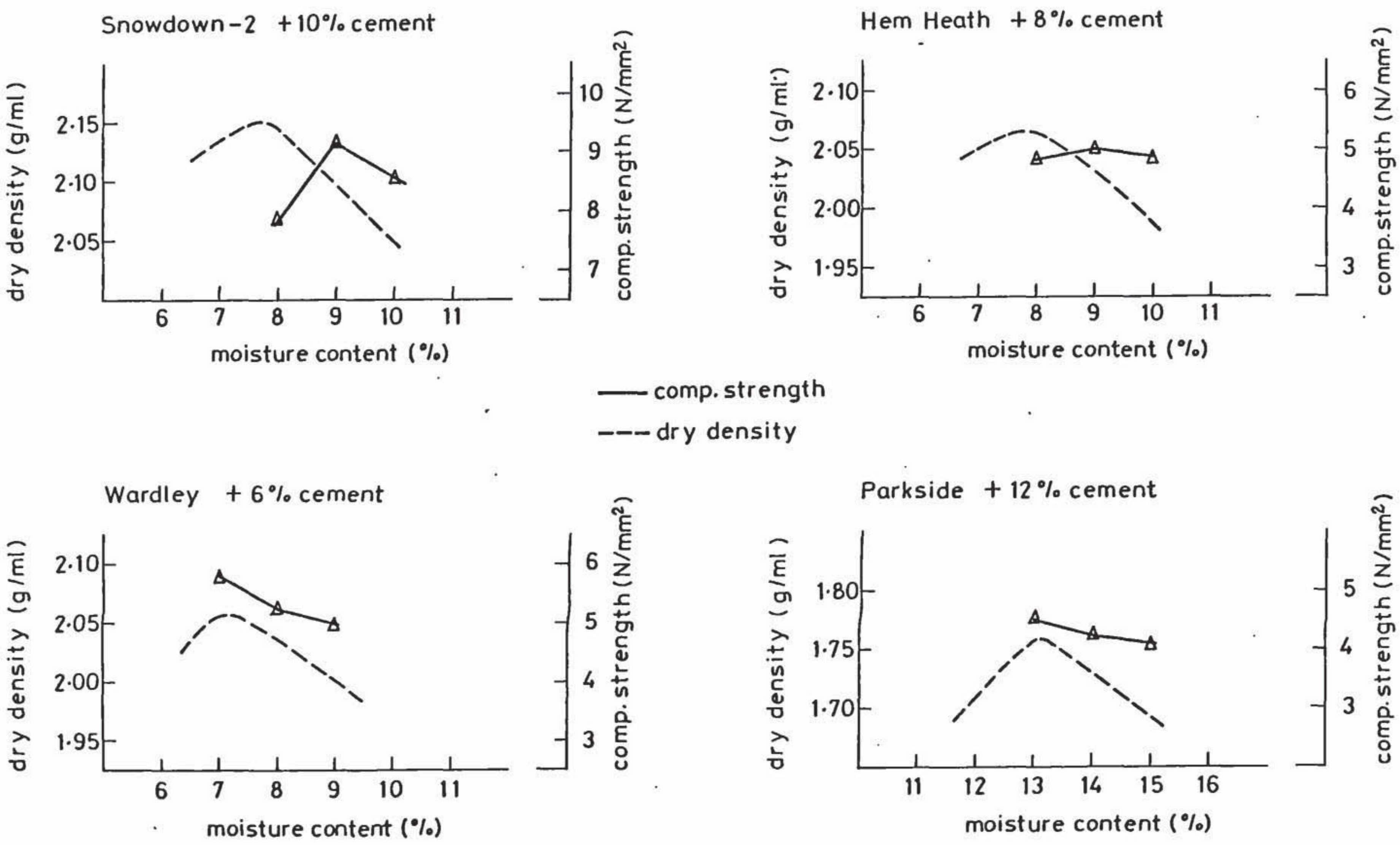


Figure 7.12 Effect of initial moisture content on dry density and compressive strength

CHAPTER 8. EXPERIMENTAL STUDY OF DURABILITY

8.1 Introduction

To assist the formulation of durability criteria applicable to cement bound minestones, a laboratory study was conducted to determine the resistance of specimens to immersion and, separately, to cycles of freezing and thawing. The British Standard Immersion Test (69) is normally considered to reveal the adverse effects that sulphates or sensitive clays may have on the performance of a stabilised soil when exposed to excess moisture. Although it is not currently a mandatory test, it seems very likely that it will be included in a forthcoming revision of the Department of Transport specification (9) and so it was selected for this study. The ASTM freeze-thaw test (57) was selected primarily because it has been correlated with the field performance of soil-cement pavements in USA (72). It was reported (60) to have been used in the development of the earliest British strength criterion (94). The standard procedure (57) was modified to comply with British practice (69) regarding specimen preparation. Both tests permit an assessment of the stability of the material when exposed to moisture changes. Eight minestone samples and a laboratory blend were included in the study. The performance was assessed by measuring the following parameters:-

(a) Dimensional Changes

(b) Mass Changes

(c) Strength Changes

The test programme was undertaken in two phases. The initial programme was concerned with the effect of cement content upon resistance. The second phase was confined to those minestones that produced the best performance in the initial programme and was concerned with the effect of initial moisture content on their performance in the durability tests.

8.2 Initial Considerations

8.2.1 <u>Preparation of Specimens.</u> All the tests were undertaken on 100 mm cubical specimens. For each test a batch of three cubes was produced from the same mix. The methods used to prepare and mould the specimens were identical to those used to produce the compressive strength specimens. A full description of the methods are given in Chapter 7, Section 7.4. The moulded specimens were wrapped in polythene film, placed in polythene bags, and stored in a thermostatically controlled curing room, operating at 20^{o}C, until required. After 7 days curing, the polythene bag and film were removed and the specimen weighed. All specimens that had lost more than 5 grammes during curing were rejected and, if more than one specimen from a batch was rejected, then all three were rejected and a fresh batch prepared.

8.2.2 <u>Assessment of Dimensional Changes.</u> The height and width of the specimens were monitored at regular intervals during the course of the durability tests so that a record could be produced of their dimensional changes. To ensure that these measurements were always made at the same location on each cube, it was necessary to devise a method of accurately relocating the measuring device. Twenty-four hours prior to completion of the curing period six "demec" strain gauge spots were attached to the faces of the cube with an epoxy resin adhesive. A "demec" spot is approximately 3 mm in diameter and has a punched recess at its centre. The spots were arranged on the cube as shown in figure 8.1. The arrangements permitting measurement of:-

(a) The compacted height of the specimen.

(b) The width of the specimen at the centre of each compacted layer.

Two faces of the cube do not have "demec" spots and remain free for locating between the platens of a crushing machine, thus permitting the determination of the unconfined compressive strength at the end of the test. The epoxy resin adhesive proved to be very satisfactory and "demec" spots were only detached from the least resistant specimens when deterioration reached an advanced stage. The dimensions were monitored with a length comparator which had pointed nodes to locate in the punched recesses of two opposing "demec" spots as shown in figure 8.2. The comparator was equipped with a dial gauge graduated to 0.002 mm and proved to be an accurate device for measuring the dimensions of the cubes.

8.3 The Test Methods

8.3.1 <u>The Immersion Test.</u> The test procedure was similar to that of test 12 of BS 1924:1975 (69). Before commencing the test the dimensions of each cube were measured using the length comparator to provide a datum for assessing subsequent performance. The specimens were placed in individual containers which were filled with water to a level approximately 25 mm above the upper surface of the specimen. Individual containers were selected in preference to a single tank because they prevented contamination between specimens and also simplified the control of the water level. The immersed specimens were stored for 7 days in a thermostatically controlled room operating at 20°C. Every 24 hours each specimen was removed from its container, weighed and measured. The weighing and measuring operations were completed in less than 15 seconds and each specimen was immediately replaced in the container so as to avoid excessive drying. At the end of the immersion period the unconfined compressive strength of each specimen was determined using the procedure described in Test 11 of BS 1924:1975 (69).

8.3.2 <u>The freeze-thaw test.</u> The test procedure was similar to that of "Methods of freezing and thawing test of compacted soil-cement mixtures", ASTM designation D 560 (57). The use of 100 mm cubes followed British practice for unconfined compression testing of coarse grained material-cement mixtures (69) and so differed from the ASTM test which uses a cylindrical specimen similar to that used in the British Standard compaction test (2.5 kg rammer method) (64). The cube was selected so as to permit a comparison of the strength of a specimen following freezing and thawing with that of a similar specimen cured at constant moisture content and constant temperature (see Chapter 7).

Before commencing the test, the dimensions of each cube were measured using the length comparator. The specimens were placed in a freezing cabinet operating at -23°C, where they remained for 24 hours. On removal, each specimen was weighed and measured, before being placed on a 13 mm thick porous plate which was half submerged in potable water. This assembly was placed in an environmental cabinet, operating at 21°C and having a relative humidity of 100 per cent, for 24 hours. All the specimens were able to absorb water by capillary action during this thawing period. On removal from the cabinet the weight and dimensional measurements were repeated before return to the freezing cabinet. The test continued for a further eleven freeze-thaw cycles, the cube being weighed and measured at the end of each period of freezing or thawing. Following the final thaw period the unconfined compressive strength of the specimen was determined using the procedure described in Test 11 of BS 1924:1975 (69).

8.4 The Test Programme

8.4.1 Effect of Cement Content. The initial programme was concerned with the effect of cement content upon the resistance of various stabilised mixtures to immersion and to cycles of freezing and thawing. Eight minestone samples and a laboratory blend were studied. The cement contents of the various mixtures were the same as those used in the study of compressive strength. All the specimens were produced at the appropriate optimum moisture content and at a target density that corresponded to the maximum value established in the moisture density relationships. There was an exception, the stabilised specimens produced from the Peckfield minestone, for which the target density was reduced to approximately 93 per cent of the maximum dry density. This was due to the difficulties experienced in producing specimens for assessing compressive strength as discussed in Section 7.6.1.

As specimens of stabilised minestones had not previously been subjected to freeze-thaw testing, it was decided that the initial programme should include an assessment of the repeatability of the test on such materials. Accordingly, the tests on the Snowdown-1 minestone included tests on replicate specimens and these results are commented on in Section 8.6.2.

The mean changes in height, width, and mass, recorded for each batch of three specimens after one day and seven days immersion, are given in Table 8.1. The mean compressive strength of the various mixtures, measured at completion of the immersion test, are given in Table 8.2. This Table also includes the strength of specimens cured at constant moisture content for 7 and 14 days together with the "Resistance Index" (R.I.) (69), which is expressed as the ratio of the compressive strength following immersion to the 14 day compressive strength.

The mean changes in height, width and mass recorded for

Table 8.1 Effect of Cement Content upon Resistance to Immersion:

Dimensions and Mass

Sample	Cement Content	Moisture Content	Immersion Period	Height Change (mm x 10^{-3})		Width Change (mm x 10^{-3})		Mass Change (g)	
	(°/o)	(°/o)	(day)	first day	last day	first day	last day	first day	last day
Snowdown-1	5	8	7	319	820	172	469	ND*	61
- do -	10	8	7	220	624	137	389	ND	62
- do -	15	8	7	195	505	117	318	ND	70
Snowdown-2	10	8	7	115	372	74	251	27	36
Hem Heath	4	8	7	42	63	27	46	21	19
- do -	8	8	7	68	98	49	71	23	30
- do -	12	8	7	93	128	65	91	35	36
Wardley	6	7	7	47	64	30	38	33	38
- do -	12	7	7	78	95	50	68	44	49
Parkside	6	13	7	75	109	55	79	28	28
- do -	12	13	7	73	95	50	66	30	35

* - ND = not determined

(continued...)

Table 8.1 (continued)

Sample	Cement Content (°/o)	Moisture Content (°/o)	Immersion Period (day)	Height Change (mm x 10^{-3})		Width Change (mm x 10^{-3})		Mass Change (g)	
				first day	last day	first day	last day	first day	last day
Blidworth	6	11	7	163	202	117	156	52	50
- do -	12	11	7	155	211	101	148	59	57
Bagworth	6	14	3	1616	failed	952	-	78	-
- do -	12	14	7	877	1910	712	1589	75	102
Peckfield	6	19	3	2299	failed	1171	-	125	-
- do -	12	19	6	914	failed	656	-	118	-
Lab. blend	4	5	7	0	6	0	4	ND*	9
- do -	8	5	7	0	3	0	8	ND	13
- do -	12	5	7	5	16	5	12	ND	24

* - ND = not determined

Table 8.2 Effect of Cement Content upon Resistance to Immersion: Compressive Strength and Resistance Index

Sample	Cement Content ($^o/o$)	Compressive Strength (N/mm^2) 7-day Cure	14-day Cure (a)	7-day Cure + 7-day Imm. (b)	Resistance Index ($^o/o$) (b/a)x100
Snowdown-1	5	5.1	5.7	2.1	37
- do -	10	8.1	8.4	3.9	46
- do -	15	9.7	10.5	6.0	57
Snowdown-2	10	7.9	10.9	5.6	51
Hem Heath	4	3.1	3.8	3.2	84
- do -	8	4.8	5.7	5.1	89
- do -	12	6.2	7.3	6.4	88
Wardley	6	5.8	6.7	4.7	70
- do -	12	9.4	10.2	7.7	75
Parkside	6	2.7	3.1	2.6	84
- do -	12	4.5	5.4	4.4	81
Blidworth	6	3.1	3.1	2.1	68
- do -	12	4.6	4.6	3.4	74
Bagworth	6	2.6	2.9	failed	0
- do -	12	3.6	3.2	1.0	33
Peckfield	6	1.2	1.4	failed	0
- do -	12	1.9	2.1	failed	0
Lab. blend	4	10.3	11.6	10.3	89
- do -	8	21.1	21.6	22.4	103
- do -	12	28.2	28.4	29.3	103

each batch of three specimens, during the first and last freeze-thaw cycles, are given in Table 8.3. The mean compressive strength of the various mixtures, measured at the completion of the freeze-thaw test, are given in Table 8.4. This table also includes the strength of specimens cured at constant moisture content for 7 days together with the "Freeze-Thaw Index" (F.T.I.), expressed as the ratio of the compressive strength following freezing and thawing to the 7 day compressive strength.

8.4.2 Effect of Initial Moisture Content. The study of compressive strength, described in Chapter 7, included an examination of the effect of the initial moisture content upon the strength of specimens produced from Snowdown-2, Hem Heath, Wardley and Parkside minestones. The specimens produced at higher moisture contents appeared to be of a higher quality than those produced at the optimum value. In the case of mixtures produced from Snowdown-2 and Hem Heath minestones, the highest compressive strengths were obtained at a moisture content 1 per cent above the optimum. It was considered that increasing the initial moisture content could, possibly, also be beneficial to the durability of these mixtures. A study was, therefore, made of the effect of the initial moisture content on the resistance of specimens, produced from Snowdown-2, Hem Heath, Wardley and Parkside minestones, to immersion and cycles of freezing and thawing. These specimens were produced at 1 and 2 per cent above the optimum value, with the target densities being determined from the appropriate moisture-density relationship given in Chapter 6. Moisture

Table 8.3 Effect of Cement Content on Resistance to Freezing and Thawing:

Dimensions and Mass

Sample	Cement Content	Moisture Content	Cycles Completed	Height Change ($mm \times 10^{-3}$)				Width Change ($mm \times 10^{-3}$)				Mass Change (g)			
				first		last		first		last		first		last	
	(°/o)	(°/o)		fr'ze	thaw	fr'ze	thaw	fr'ze	thaw	fr'ze	thaw	fr'ze	thaw	fr'ze	thaw
Snowdown-1	5	8	7	- 160	233	failed		- 139	135	-	-	ND*	17	-	-
- do -	5	8	8	- 164	211	failed		- 145	143	-	-	ND	19	-	-
Snowdown-1	10	8	12	- 153	139	2061	1936	- 123	75	982	1021	ND	18	ND	70
- do -	10	8	12	- 160	125	1799	1731	- 133	84	970	972	ND	12	ND	.63
Snowdown-1	15	8	12	- 117	97	1477	1419	- 96	64	673	728	ND	17	ND	66
- do -	15	8	12	- 139	107	1536	1493	- 117	72	749	792	ND	13	ND	70
Snowdown-2	10	8	12	- 139	82	555	685	- 126	59	375	478	- 6	10	27	32
Hem Heath	4	8	12	- 160	82	960	1002	- 175	82	619	725	- 6	17	22	34
- do -	8	8	12	- 246	79	263	415	- 211	62	139	298	- 5	14	19	30
- do -	12	8	12	- 166	71	212	353	- 142	89	105	247	- 2	27	33	40
Wardley	6	7	12	- 187	50	487	550	- 160	33	233	310	- 4	20	23	31
- do -	12	7	12	- 130	85	280	332	- 110	61	133	199	- 3	30	34	44

* - ND = not determined.

(continued...)

Table 8.3 (continued)

Sample	Cement Content	Moisture Content	Cycles Completed	Height Change (mm x 10^{-3})				Width Change (mm x 10^{-3})				Mass Change (g)			
				first		last		first		last		first		last	
	(o/o)	(o/o)		fr'ze	thaw	fr'ze	thaw	fr'ze	thaw	fr'ze	thaw	fr'ze	thaw	fr'ze	thaw
Parkside	6	13	8	- 193	56	failed		- 204	31	-	-	-11	7	-	-
- do -	12	13	12	- 164	32	- 11	147	- 153	28	- 43	111	-10	4	15	22
Blidworth	6	11	6	- 307	215	failed		- 304	103	-	-	-11	33	-	-
- do -	12	11	12	- 220	33	912	853	- 241	87	563	603	- 9	47	55	63
Bagworth	6	14	0	failed				-	-	-	-	-	-	-	-
- do -	12	14	2	- 234	1221	failed		- 209	899	-	-	- 8	98	-	-
Peckfield	6	19	0	failed				-	-	-	-	-	-	-	-
- do -	12	19	2	failed				-	-	-	-	-	-	-	-
Lab. blend	4	5	12	- 69	- 11	- 72	8	- 64	- 9	- 74	- 16	- 10	- 4	- 12	- 5
- do -	8	5	12	- 59	- 4	- 48	16	- 54	- 6	- 63	- 8	- 6	- 4	- 5	0
- do -	12	5	12	- 54	- 2	- 35	17	- 49	- 2	- 44	- 3	- 3	- 1	2	4

Table 8.4 Effect of Cement Content on Resistance to Freezing and Thawing: Compressive Strength and Freeze-Thaw Index

Sample	Cement Content	Compressive Strength (N/mm^2)		Freeze-thaw Index
		7-day cure	7-day cure + 12 F-T cycles	
	(%)	(a)	(b)	(b/a)x100
Snowdown-1	5	5.1	failed	0
Snowdown-1	10	8.1	2.6	32
- do -	10	8.1	3.0	37
Snowdown-1	15	9.7	3.9	40
- do -	15	9.7	4.4	45
Snowdown-2	10	7.9	5.8	73
Hem Heath	4	3.1	1.6	52
- do -	8	4.8	4.5	94
- do -	12	6.2	6.5	105
Wardley	6	5.8	3.8	66
- do -	12	9.4	6.8	72
Parkside	6	2.7	failed	0
- do -	12	4.5	3.5	78
Blidworth	6	3.1	failed	0
- do -	12	4.6	2.1	46
Bagworth	6	2.6	failed	0
- do -	12	3.7	failed	0
Peckfield	6	1.2	failed	0
- do -	12	1.9	failed	0
Lab. blend	4	10.3	13.9	126
- do -	8	21.1	25.1	119
- do -	12	28.2	32.5	115

contents greater than the optimum plus two per cent were not included since two per cent represents the upper limit currently permitted by the Department of Transport (9). The cement contents selected for this part of the study were the same as those used to study the effect of initial moisture content on compressive strength.

The mean changes in height, width, and mass, recorded for each batch of specimens after one day and seven days immersion, are given in Table 8.5. The mean compressive strength of the mixtures, measured at the completion of the immersion test, are given in Table 8.6. This table also includes the strength of specimens cured at constant moisture content for 7 and 14 days, together with the R.I. At this stage of the investigation there was insufficient material to produce the additional specimens required for determining the 14 day strength. The values given in the table were, therefore, estimated as follows:-

$$S_{14} = A \times S_7$$

Where S_{14} = Estimated 14 day strength

S_7 = 7 day strength determined by test

$$A = \frac{\text{14 day strength of specimen at optimum m.c}}{\text{7 day strength of specimen at optimum m.c}}$$

The mean changes in height, width, and mass, recorded for each batch of specimens during the first and last freeze-thaw cycle, are given in Table 8.7. The mean compressive strengths of the mixtures, measured at the completion of the freeze-thaw test, are given in Table 8.8. This table also includes the strength of specimens cured at

Table 8.5 Effect of Initial Moisture Content upon Resistance to Immersion:

Dimensions and Mass

Sample	Cement Content	Moisture Content	Immersion Period	Height Change (mm x 10^{-3})		Width Change (mm x 10^{-3})		Mass Change	
				first day	last day	first day	last day	first day	last day
	($^{o}/_{o}$)	($^{o}/_{o}$)	(day)						
Snowdown-2	10	optimum	7	115	372	74	251	27	36
- do -	10	opt + 1	7	70	180	48	120	19	27
- do -	10	opt + 2	7	60	144	42	102	15	21
Hem Heath	8	optimum	7	68	98	49	71	23	30
- do -	8	opt + 1	7	61	76	48	55	26	29
- do -	8	opt + 2	7	71	85	50	59	23	32
Wardley	6	optimum	7	47	64	30	38	33	38
- do -	6	opt + 1	7	31	39	21	26	23	26
- do -	6	opt + 2	7	33	44	20	31	16	18
Parkside	12	optimum	7	73	95	50	66	30	35
- do -	12	opt + 1	7	50	48	38	32	31	37
- do -	12	opt + 2	7	43	51	37	43	34	39

* - ND = not determined

Table 8.6 Effect of Initial Moisture Content upon Resistance to Immersion:

Compressive Strength and Resistance Index

Sample	Cement Content ($^0/o$)	Moisture Content ($^0/o$)	Compressive Strength (N/mm^2) 7-day Cure	Compressive Strength (N/mm^2) 14-day Cure (a)	Compressive Strength (N/mm^2) 7-day Cure + 7-day Imm. (b)	Resistance Index ($^0/o$) (b/a)x100
Snowdown-2	10	optimum	7.9	10.9 (T)*	5.6	51
- do -	10	opt + 1	9.2	12.7 (E)	7.3	57
- do -	10	opt + 2	8.6	11.9 (E)	7.7	65
Hem Heath	8	optimum	4.8	5.7 (T)	5.1	89
- do -	8	opt + 1	5.0	5.9 (E)	5.5	93
- do -	8	opt + 2	4.9	5.8 (E)	5.2	90
Wardley	6	opitimum	5.8	6.7 (T)	4.7	70
- do -	6	opt + 1	5.2	6.0 (E)	5.0	83
- do -	6	opt + 2	5.0	5.8 (E)	4.8	83
Parkside	12	optimum	4.5	5.4 (T)	4.4	81
- do -	12	opt + 1	4.3	5.2 (E)	3.5	68
- do -	12	opt + 2	4.1	4.9 (E)	3.2	65

* - Method of determination: (T) = Test; (E) = Estimated. (see section 8.4.2)

Table 8.7 Effect of Initial Moisture Content on Resistance to Freezing and Thawing:

Dimensions and Mass

Sample	Cement Content	Moisture Content	Cycles Completed	Height Change (mm x 10^{-3})				Width Change (mm x 10^{-3})				Mass Change (g)			
				first		last		first		last		first		last	
	(°/o)	(°/o)		fr'ze	thaw	fr'ze	thaw	fr'ze	thaw	fr'ze	thaw	fr'ze	thaw	fr'ze	thaw
Snowdown-2	10	optimum	12	- 139	82	555	685	- 126	59	375	478	- 6	10	27	32
- do -	10	opt + 1	12	- 135	42	-20	123	- 122	25	- 51	76	- 7	3	- 5	8
- do -	10	opt + 2	12	- 126	16	-96	39	- 123	6	-113	9	- 8	- 2	- 3	2
Hem Heath	8	optimum	12	- 246	79	263	415	- 211	62	139	298	- 5	14	19	30
- do -	8	opt + 1	12	- 267	65	25	235	- 230	40	- 30	164	- 7	15	12	19
- do -	8	opt + 2	12	- 251	55	- 23	200	- 226	37	- 62	149	- 6	12	8	15
Wardley	6	optimum	12	- 187	50	487	550	- 160	33	233	310	- 4	20	23	31
- do -	6	opt + 1	12	- 197	17	- 33	153	- 176	4	- 77	85	- 7	7	3	14
- do -	6	opt + 2	12	- 152	19	- 81	103	- 158	9	-101	65	- 9	4	- 6	4
Parkside	12	optimum	12	- 164	32	- 11	147	- 153	28	- 43	111	-10	4	15	22
- do -	12	opt + 1	12	- 140	33	-124	81	- 125	27	-120	74	-11	8	11	20
- do -	12	opt + 2	12	- 124	45	- 61	107	- 135	28	- 96	77	-	4	6	15

Table 8.8 Effect of Initial Moisture Content on Resistance to Freezing and Thawing: Compressive Strength and and Freeze-Thaw Index

Sample	Cement Content	Moisture Content	Compressive Strength (N/mm^2)		Freeze-thaw Index
			7-day cure	7-day cure + 12 F-T cycles	
	(°/o)	(°/o)	(a)	(b)	(b/a)x100
Snowdown-2	10	optimum	7.9	5.8	73
- do -	10	opt + 1	9.2	10.1	110
- do -	10	opt + 2	8.6	8.7	101
Hem Heath	8	optimum	4.8	4.5	94
- do -	8	opt + 1	5.0	4.4	88
- do -	8	opt + 2	4.9	4.2	86
Wardley	6	optimum	5.8	3.8	66
- do -	6	opt + 1	5.2	4.3	83
- do -	6	opt + 2	5.0	4.4	88
Parkside	12	optimum	4.5	3.5	78
- do -	12	opt + 1	4.3	3.4	76
- do -	12	opt + 2	4.1	3.4	83

constant moisture content for 7 days, together with the F.T.I.

8.5 Effect of Cement Content on Immersion Resistance

8.5.1 Visual Examination. Visual examination of specimens undergoing the immersion test suggested that the resistance to immersion was largely dependent upon the source of the minestone and to a lesser degree upon the cement content. Many of the specimens endured the full period of immersion, although the degree of resistance varied considerably. The specimens produced from Bagworth and Peckfield minestones, containing 6 per cent cement, completely disintegrated after being immersed for only 3 days, whilst similar specimens containing 12 per cent cement were difficult to handle, without causing damage, after immersion for 5 days. Indeed, after only 1 day under water, these specimens developed a fissure located at the interface of the two compacted layers. With the Peckfield specimens, a number of the "demec" reference spots became detached and, on removal from the water at the completion of the test, the specimens disintegrated completely. Specimens produced from both of the Snowdown minestones were not prone to damage, but the compaction plane again became clearly visible during the course of the test. With the specimens containing 5 per cent cement this was observed after they had been immersed for 2 days and for those containing 10 and 15 per cent cement after 3 days. Specimens produced from Blidworth minestones did not develop a fissure but the surfaces of all the specimens were softened by immersion. The specimens produced from

Hem Heath, Wardley and Parkside minestones appeared to be the most resistant of the minestone-cement mixtures. They were not prone to damage by handling and fissures were not apparent on the faces of the specimens. Similarly, the specimens produced from the laboratory blend showed no signs of distress throughout the test.

8.5.2 Dimensional Changes. From Table 8.1 it can be seen that all the specimens expanded when immersed in water. The expansion of typical specimens from each batch are shown in figure 8.3 to 8.9. Figure 8.3 shows that the specimens produced from Snowdown-1 minestone behaved in a well defined anisotropic manner. At the end of the immersion period the height of the specimens containing 5 per cent cement had increased by 0.8 per cent, whilst the width had only increased by 0.5 per cent. At a cement content of 15 per cent the magnitude of the expansion was reduced marginally but the degree of anisotropy was similar with increases in the height and width of 0.5 and 0.3 per cent respectively. Of the total expansion exhibited by the Snowdown-1 specimens, less than 40 per cent developed during the first day of the immersion period. Indeed figure 8.3 shows that the specimens were still expanding at the end of the test. Specimens produced from Snowdown-2 exhibited slightly less expansion but otherwise their behaviour was similar to that of the specimens produced from Snowdown-1.

It can be seen from figure 8.4 that the Bagworth specimens also behaved in an anisotropic manner but the magnitude of the expansion was greater than that of the Snowdown samples and exceeded 1.5 per cent at the end of the immersion period. An examination of figures 8.3 and 8.4 shows that, after 2 or 3 days immersion the plots of the height and width of both the Snowdown and Bagworth specimens became increasingly parallel. By the end of the immersion period the height and the width are expanding at approximately equal rates. This appears to correlate with the opening of the fissure which was observed to develop on the faces of these specimens between 1 and 3 days after initial immersion (see Section 8.5.1). As the fissure developed it contributed to the increase in the height of the specimen, however, the development of the fissure is relatively short and, eventually, its contribution to the expansion of the specimen diminishes. Further increases in both the height and width are governed by similar phenomena and, therefore, the rates of increase are approximately equal.

The expansion of the specimen produced from Hem Heath, Wardley, Parkside, and Blidworth minestones is shown in figures 8.5, 8.6, 8.7 and 8.8 respectively. The anisotropic behaviour of these specimens is much less marked than that of the Snowdown and Bagworth specimens. This would appear to confirm that the fissures, observed on the faces of the Snowdown and Bagworth specimens, had a significant influence on their behaviour. From Table 8.1 it can be seen that both the height and width of the Hem Heath, Wardley and Parkside specimens increased approximately 0.1 per cent during the immersion period.

This expansion amounts to less than one-fifth of that exhibited by the most stable Snowdown-1 specimens and to only one-twentieth of that exhibited by the Bagworth specimens. The Blidworth specimens expanded approximately 0.2 per cent during the test.

Of the total expansion, exhibited by the Hem Heath, Wardley, Parkside and Blidworth specimens, 70 per cent occurred during the first day of the immersion period and, furthermore, the specimens had stopped expanding before the end of the test. Increasing the cement content of the Hem Heath and Wardley specimens resulted in marginally greater expansion during immersion. On the other hand increasing the cement content of the Parkside and Blidworth specimens from 6 to 12 per cent had no significant effect on the expansion.

From figure 8.9, it is apparent that the Peckfield specimens expanded by between 0.7 and 2.3 per cent after being immersed for only 1 day. Those specimens which endured more than 2 to 3 days under water lost most of their "demec" reference spots as the adhesive could not retain a bond with the rapidly deteriorating specimens.

Specimens produced from the laboratory blend exhibited only negligible expansion during the immersion test. However, the results in Table 8.1 again suggest that the expansion increased marginally when the cement content was raised from 4 to 12 per cent.

8.5.3 Mass Changes. Changes in the mass of a specimen during the course of the immersion test are the results of either changes in the moisture content or loss of material.

The changes in the mass of the specimens after the first and final days of the test are given in Table 8.1. The specimens produced from Snowdown-2, Hem Heath, Wardley, Parkside and Blidworth minestones increased in mass by between 20 and 60 grammes during the first 24 hours. However, by the end of the test, the mass of these specimens had remained unchanged or had only shown a further, slight increase. Since none of these specimens showed signs of serious deterioration these mass changes are attributed to moisture gains which occur within the first 24 hours of the immersion period. The Bagworth and Peckfield specimens absorbed between 75 and 128 grammes of moisture during the first 24 hours. The mass of those Bagworth specimens which endured the full immersion period continued to increase despite losses of solid material due to deterioration. The specimens produced from the laboratory blend absorbed between 9 and 24 grammes of moisture.

The magnitude of the moisture gained by specimens produced from the most resistant minestone and the laboratory blend appeared to increase with increasing cement content.

8.5.4 Volume Change and Degree of Saturation. The volume change and degree of saturation, at the completion of day 1 and day 7 of the immersion period have been computed from the dimensional and mass change data and the values are given in Table 8.9. These calculations show that all the minestone specimens were completely saturated after only 1 day of immersion, whereas the specimens produced from the laboratory blend did not reach saturation even at the end of the immersion period.

8.5.5 Compressive Strength. The results in Table 8.2 show that, with the exception of Hem Heath specimens, the compressive strengths of the minestone specimens, following immersion, were lower than the strength of similar specimens cured at constant moisture content for 7 days. The strength of the Hem Heath specimens appeared to increase marginally during the test. The R.I. of the specimens varied between 33 and 89 per cent. Although the Snowdown specimens did not appear to experience serious distress, they suffered considerable loss of strength. Following immersion, the specimens produced from Snowdown-1, with 5 per cent cement, had a strength equivalent to only 37 per cent of the standard 14 day strength. Increasing the cement content to 15 per cent raised the R.I. to 57 per cent but this was still below the values obtained from many of the other cement-bound minestones. When the failed specimens were examined they were found to break easily along the line of the fissure described in Section 8.5.1. This apparent plane of weakness was not observed on specimens cured at constant moisture content. It was concluded that the plane of weakness, coinciding with the compaction plane, was

Table 8.9 Effect of Cement Content on Resistance to Immersion: Volume and Degree of Saturation

Sample	Cement Content (°/o)	Moisture Content (°/o)	Initial Degree of Saturation (°/o)	Volume Change (ml) first day	Volume Change (ml) last day	Degree of Saturation (°/o) first day	Degree of Saturation (°/o) last day
Snowdown-1	5	8	85	6.6	17.7	-	100.0
- do -	10	8	82	4.9	14.1	-	100.0
- do -	15	8	79	4.3	11.5	-	100.0
Snowdown-2	10	8	88	2.6	8.8	100.0	100.0
Hem Heath	4	8	92	1.0	1.6	100.0	100.0
- do -	8	8	89	1.7	2.4	100.0	100.0
- do -	12	8	86	2.2	3.1	100.0	100.0
Wardley	6	7	85	1.1	1.4	100.0	100.0
- do -	12	7	81	1.8	2.3	100.0	100.0
Parkside	6	13	97	1.9	2.7	100.0	100.0
- do -	12	13	93	1.7	2.3	100.0	100.0

(continued...)

Table 8.9 (continued)

Sample	Cement Content	Moisture Content	Initial Degree of Saturation	Volume Change (ml)		Degree of Saturation (°/o)	
	(°/o)	(°/o)	(°/o)	first day	last day	first day	last day
Blidworth	6	11	95	4.0	5.1	100.0	100.0
- do -	12	11	92	3.6	5.1	100.0	100.0
Bagworth	6	14	97	35.6	failed	100.0	-
- do -	12	14	94	23.2	51.7	100.0	-
Peckfield	6	19	81	54.2	failed	100.0	-
- do -	12	19	79	22.4	failed	100.0	-
Lab.blend	4	5	87	0	0.1	-	93.0
- do -	8	5	84	0	0.2	-	94.0
- do -	12	5	82	0.2	0.4	-	99.0

probably common to all Snowdown specimens but only became a discontinuity when the specimen was immersed in water. The formation of this discontinuity effectively results in a single cubical specimen, having a height to width ratio of 1:1, being divided into two specimens, each having a weight to side ratio of 2:1, as shown in figure 8.25. It has been shown that the strength of a cement stabilised specimen having a height to width ratio of 2:1 is some 20 per cent lower than that of a similar cubical specimen (89). It is, therefore, considered likely that the formation of the discontinuity contributed to the reduced strength of the immersed specimen, with the remainder of the specimen being intact.

The specimens produced from the Hem Heath and Parkside minestones retained more than 80 per cent of the 14 day strength whilst specimens from Wardley minestones retained at least 70 per cent. The resistance of these specimens did not appear to be significantly effected by cement content.

The Blidworth specimens, stabilised with 6 per cent cement, retained 68 per cent of the 14 day strength, whilst, at the 12 per cent cement content, the retained strength increased to 74 per cent. The Bagworth specimens, containing 12 per cent cement, began to show signs of serious distress before the end of the test and this was reflected in the very low final strengths that were obtained. The R.I. of these specimens was only 33 per cent. The Bagworth specimens, containing 6 per cent cement, and all the Peckfield specimens deteriorated before completing the test, so that the R.I. was reported as zero.

The laboratory blend specimens stabilised with 4 per cent cement, retained 89 per cent of the 14 day strength following immersion. The strength of specimens containing 8 and 12 per cent cement appeared to be unaffected by immersion.

8.5.6 <u>Summary.</u> The measurements recorded during measurements on cement-bound minestone specimens show that, within 24 hours of immersion, the specimens expanded rapidly and, furthermore, this expansion was associated with an equally rapid gain in weight due to the absorption of moisture. The magnitude of the initial expansion was largely dependent on the source of the minestone and, to a lesser extent, on the cement content. The results suggest that the initial expansion may be used to predict the subsequent behaviour of the specimens.

Specimens which expanded less than 0.2 per cent at the end of the first day displayed, with the notable exception of specimens produced from Snowdown material, a similar high resistance to immersion. The initial expansion of these specimens represent about 70 per cent of the total recorded at the end of the test and, in addition, the mass remained almost unchanged throughout the remainder of the test. When tested in compression at the end of the test their R.I. was found to exceed 70 per cent. Specimens which expanded more than 0.5 per cent, during the initial

24 hours, continued to expand throughout the period of immersion. Many of these specimens deteriorated completely before the end of the test and the R.I. of the surviving specimens was less than 35 per cent.

Specimens produced from the Snowdown minestones behaved in a very singular manner. Increasing the cement content from 5 to 15 per cent reduced the initial expansion from 0.3 to 0.1 per cent, however, all the specimens continued to expand during the test. A fissure developed on the faces of the specimen and appeared to coincide with a plane of weakness at the location of the compaction plane. The R.I. ranged from 37 to 57 per cent.

The expansion of the laboratory blend was negligible. The moisture gains were small and the strength of specimens containing more than 8 per cent cement was apparently unaffected by immersion. A feature, common to the more resistant minestones and the laboratory blend, was that expansion and moisture gain, increased with cement content.

8.6 Effect of Cement Content on Resistance to Freezing and Thawing

8.6.1 <u>Visual Examination.</u> The freeze-thaw resistance of the minestone specimens appeared to be dependent on the source of the material and to a lesser extent on the cement content. Specimens produced from Snowdown-1 and 2, Hem Heath, Wardley, Parkside and Blidworth minestones endured 12 cycles of freezing and thawing, whereas those produced from Bagworth and Peckfield minestones deteriorated before the completion of the test. When the specimens were removed from the environmental cabinet at the end of the first cycle, it was apparent that they had absorbed water throughout their entire structure. Among the more resistant minestones, the degree of absorption appeared to be higher for specimens containing the least cement.

All the specimens produced from Snowdown-1 developed a fissure, similar to that observed with the immersion specimens, and they had visibly deteriorated before the end of the test. Material was easily dislodged from the surface of the specimen during handling. Specimens produced from Snowdown-2 did not develop fissures, appeared to be more resistant, and were less prone to damage. The Hem Heath, Wardley and Parkside specimens, with the lower cement contents, were prone to damage during handling which resulted in some loss of material. Specimens produced at higher cement contents were not prone to such damage and the loss of material was minimal.

Tests on Blidworth specimens, containing 6 per cent, were terminated after the sixth cycle because they had deteriorated severely. The deterioration of the Blidworth specimens containing 12 per cent cement was less apparent than that of the Snowdown specimens. Although they were not prone to damage during handling, the surfaces of the specimens were progressively softened by the freeze-thaw process. The Bagworth and Peckfield specimens deteriorated rapidly when subjected to the freeze-thaw test. Those with a 6 per cent cement content collapsed on removal from the environmental cabinet at the end of the first cycle. Tests on specimens containing 12 per cent cement were terminated before the end of the test as the severe deterioration resulted in the dimensional and mass measurements becoming unreliable. Deterioration of the Bagworth and Peckfield specimens commenced with the formation of a "shell", which became detached from the main body of the specimen. This was followed by the disintegration of the whole specimen.

The laboratory blend appeared to be very resistant to exposure to freeze-thaw cycles. When the specimens were removed from the environmental cabinet at the end of the first cycle, it appeared that the moisture had only been absorbed into the bottom 20 mm of the specimen. The appearance of the specimen did not change throughout the remaining duration of the test.

8.6.2 Dimensional Changes. The dimensional changes exhibited by typical specimens from the various mixtures are shown in figures 8.10 to 8.16. The mean changes recorded during the first and final cycles are given in Table 8.3.

It can be seen from figure 8.10 that Snowdown-1 specimens, containing between 5 and 15 per cent cement, expanded by more than 1 per cent during the freeze-thaw test. The behaviour of these specimens, like that of those subjected to immersion, is clearly anisotropic and again suggests that the fissure, or compaction plane, had a significant influence on their performance. The specimens contracted by approximately 0.15 per cent during the first freeze period. However, by the end of the first cycle, the height and width of the specimens exceeded the original dimensions by between 0.1 and 0.2 per cent.

The amplitude of the cyclic expansion and contraction diminished rapidly and it is difficult to distinguish between the frozen and thawed dimensions after 6 or 7 cycles. The tests on the specimens containing 5 per cent cement were terminated after the 7th or 8th cycle. At this stage the specimens were expanding during the freeze period and contracting during the thaw period. This behaviour is caused by excessive ice growth during the freeze cycle and indicates serious deterioration (73). Although the specimens containing 10 and 15 per cent cement completed the test it can be seen from Table 8.3 that during the final cycle the frozen heights were also greater than the thawed heights. Tests on replicate specimens produced from Snowdown-1 mixtures gave very similar results and this confirms the repeatability of the freeze-thaw test. The specimens produced from Snowdown-2 minestone appeared to be slightly more resistant. They expanded approximately 0.5 per cent during the test.

The dimensional changes recorded during freeze-thaw tests on the Hem Heath and Wardley specimens are shown in figures 8.11 and 8.12. The specimens with the lowest cement contents, 4 to 6 per cent, displayed a degree of anisotropy which was much less evident at higher cement contents. These specimens, however, displayed a higher resistance than the Snowdown specimens. It can be seen from Table 8.3 that all these specimens contracted by approximately 0.2 per cent during the first freezing period. At the end of the first thaw period they exceeded their original dimensions by less than 0.1 per cent. The Hem Heath specimens containing 4 per cent cement expanded by a further 0.2 per cent at the end of the second cycle and the amplitude of the dimensional changes was reduced from 0.2 to 0.1 per cent. The amplitude remained constant for the remaining cycles but the specimens continued to expand at a rate of approximately 0.1 per cent per cycle. The specimens containing 8 and 12 per cent cement appeared to have a similar level of resistance. The amplitude of the dimensional changes remained constant throughout the test and, on completion, the specimens had expanded less than 0.3 per cent. The behaviour of the Wardley specimens was similar to that of the Hem Heath specimens, although the amplitude of the dimensional changes was slightly smaller.

The resistance of Parkside specimens, shown in figure 8.13, appeared to improve considerably when the cement content was raised from 6 to 12 per cent. It can be seen from Table 8.3 that the specimens containing 6 per cent cement contracted 0.2 per cent during the first freeze period and exceeded their original dimensions by 0.05 per cent at the end of the first cycle. After the second cycle the specimen deteriorated rapidly and, by the fifth cycle, they were expanding during the freeze period and contracting during the thawing period. In addition, the amplitude of these movements was continuing to increase. The specimens had expanded more than 1.0 per cent when the test was terminated on the ninth cycle. The specimens containing 12 per cent cement contracted 0.15 per cent during the first freeze period and exceeded their original dimensions by less than 0.05 per cent at the end of the first cycle. The amplitude of the dimensional changes remained almost constant throughout the test and the final expansion was less than 0.15 per cent. Indeed, during the twelfth freezing period, the height and width of the frozen specimens were, on average, less than the original values.

The changes in the dimensions of the Blidworth specimens are shown in figure 8.14. These specimens contracted by more than 0.2 per cent during the first freeze period. The specimens containing 6 per cent cement exceeded the original dimensions by more than 0.1 per cent at the end of the first cycle. Subsequent deterioration was rapid and the test was terminated after the sixth cycle when the specimen had expanded by approximately 1.0 per cent. During the first cycle, the specimens containing 12 per cent cement exhibited an increase in length of less than 0.1 per cent. The amplitude of the changes in dimension diminished rapidly and, by the sixth cycle it was difficult to distinguish between the frozen and thawed dimensions. Although the specimens endured 12 cycles, it was apparent that they were severely damaged, and the data in Table 8.3 clearly shows that the final frozen dimensions exceeded the final thawed dimensions. An interesting feature of these specimens is that, although they were severely damaged by the freeze-thaw test, the degree of anisotropy is much less marked than that observed with the Snowdown-1 specimens.

The Bagworth and Peckfield specimens displayed little resistance to freeze-thaw cycles. The specimens containing 6 per cent cement did not survive the first cycle. the typical behaviour of such specimens, containing 12 per cent cement, is shown in figure 8.15. Following an initial shrinkage of more than 0.2 per cent, the specimens exhibited considerable expansion by the end of the first cycle - 0.3 per cent for Peckfield and 1.0 per cent for Bagworth. Many of the "demec" reference spots became detached and those remaining were considered to be unreliable because of the severely deteriorated condition of the specimens.

The changes in the dimensions of the laboratory blend specimens are shown in figure 8.16. It can be seen from Table 8.3 that the specimens contracted by less than 0.01 per cent during the freeze period and at the end of the first cycle the dimensions of the specimens were slightly smaller than the original values. The thawed dimensions of the specimens remained approximately constant throughout the test. The increase in cement content, from 4 to 12 per cent, resulted in a reduction in the amplitude of the dimension changes.

8.6.3 <u>Mass Changes.</u> Apart from the Bagworth and Peckfield specimens, which were observed to lose significant amounts of material during the first cycle, the changes in the mass of the specimens, recorded at the end of both the first freeze and thaw periods are attributed entirely to the loss or gain of moisture. It can be seen from Table 8.3 that at the end of the first freeze period, the masses of the minestone specimens had been reduced by between 2 and 11 grammes from their original values. This is equivalent to a reduction in the moisture content of between 0.1 and 0.5 per cent, ie the specimens underwent a degree of drying during the freezing period. It can be seen from Table 8.3 that the degree of drying was higher for those specimens produced from the finer grained Parkside and Blidworth minestones. This is possibly due to the higher moulding moisture content necessary for these materials.

At the end of the first cycle, the masses of the Snowdown-1 specimens had increased by between 10 and 20 grammes, which is equivalent to an increase in the moisture content of approximately 0.5 to 1 per cent. The cement content did not have a significant effect on this increase. The specimens which endured all 12 cycles had increases in mass of approximately 65 grammes, which is equivalent to an increase in the moisture content in excess of 3.0 per cent. These specimens had begun to deteriorate before the end of the test and, therefore, the actual moisture gain were being masked by the loss of material. The moisture content of the Snowdown-2 specimens, which lost only a minimal amount of material during the test, increased by less than 1.5 per cent during the test.

At the end of the first cycle, the Hem Heath and Wardley specimens exhibited increases in mass of between 14 and 30 grammes, indicating that the increase in moisture content had not exceeded 1.5 per cent. At the end of the test, the masses of the specimens suggest that the moisture content had increased by between 1.5 and 2.0 per cent.

During the test, the Parkside specimen appeared to absorb only limited amounts of moisture. At the end of the first cycle, the increases in mass were less than 10 grammes. The specimens containing 12 per cent cement remained intact throughout the test and, on completion, had absorbed less than 25 grammes of moisture, equivalent to an increase in the moisture content of less than 1.5 per cent.

The Blidworth specimens containing 12 per cent cement absorbed considerable amounts of moisture during the test. At the end of the first cycle the mean increase was equivalent to an increase in the moisture content of more than 2.0 per cent. At the end of the test the mass gain was equivalent to an increase in the moisture content of more than 3.0 per cent and was similar to the increase recorded for the Snowdown-1 specimens. The Bagworth specimens, containing 12 per cent cement, had absorbed about 100 grammes of moisture during the first thaw period. The actual moisture gains were, however, again masked by the loss of appreciable quantities of material.

The masses of the laboratory blend specimens decreased by between 3 and 10 grammes during the first freeze period. It can be from Table 8.3 that the magnitude of this reduction was inversely proportional to cement content in the range 4 to 12 per cent. At the end of the first complete cycle all these specimens had masses that were marginally less than the original values. Subsequent changes in the masses of the specimens were minimal.

8.6.4 <u>Volume Change and Degree of Saturation.</u> The volume change and degree of saturation, following the first and final cycles of the freeze-thaw test, were computed from the dimensional and mass change data. These calculated values are given in Table 8.10, together with the degree of saturation of the moulded specimens. The degree of saturation at the end of the freeze period has not been included because the calculation is complicated by ice-growth. The degree of saturation of the minestone specimens increased during the test. The magnitude of the increase appeared to be largely dependent upon the source of the material. The change in the degree of saturation did not appear to be influenced, significantly, by the cement content. The degree of saturation of the laboratory blend specimens remained almost constant throughout the test.

8.6.5 <u>Compressive Strength.</u> The compressive strength of the minestone specimens subjected to the freeze-thaw test were, with the exception of the Hem Heath specimens containing 12 per cent cement, less than the strength of specimens cured at constant moisture content for 7 days. It can be seen from Table 8.4 that the F.T.I. of the

Table 8.10 Effect of Cement Content on Resistance to Freezing and Thawing: Volume and Degree of Saturation

Sample	Cement Content (°/o)	Moisture Content (°/o)	Initial Degree of Saturation (°/o)	Volume Change (ml) first fr'ze	first thaw	last fr'ze	last thaw	Degree of Saturation (°/o) first thaw	last thaw
Snowdown-1	5	8	85	-4.4	5.0	failed		92.0	failed
- do -	10	8	82	-4.3	2.9	37.8	37.2	86.0	95.0
- do -	15	8	79	-3.5	2.5	30.6	31.1	84.0	98.0
Snowdown-2	10	8	82	-3.9	2.0	13.1	16.5	93.0	97.0
Hem Heath	4	8	92	-5.1	2.5	22.1	24.7	100.0	98.0
- do -	8	8	89	-6.7	2.0	5.4	10.1	95.0	99.0
- do -	12	8	86	-4.5	2.5	4.2	8.5	99.0	100.0
Wardley	6	7	85	-5.1	1.2	9.6	11.7	96.0	97.0
- do -	12	7	81	-3.5	2.1	5.5	7.3	96.0	100.0
Parkside	6	13	97	-6.0	1.2	failed		-	-
- do -	12	13	93	-4.7	0.9	-1.0	3.7	94.0	100.0

(continued...)

Table 8.10 (continued)

Sample	Cement Content	Moisture Content	Initial Degree of Saturation	Volume Change (ml)				Degree of Saturation ($^o/o$)	
				first		last		first	last
	($^o/o$)	($^o/o$)	($^o/o$)	fr'ze	thaw	fr'ze	thaw	thaw	thaw
Blidworth	6	11	95	-9.1	4.2	failed		100.0	-
- do -	12	11	92	-7.0	2.1	20.5	20.7	100.0	100.0
Bagworth	6	14	97	failed		failed		-	-
- do -	12	14	94	-6.5	30.5	failed		100.0	-
Peckfield	6	19	81	failed		failed		-	-
- do -	12	19	79	failed		failed		-	-
Lab Blend	4	5	87	-2.0	0.3	-2.2	-0.4	84.0	83.0
- do -	8	5	84	-1.7	-0.2	-1.9	0	81.0	81.0
- do -	12	5	82	-1.5	-0.1	-1.2	0.1	82.0	85.0

minestone specimens which completed the test was between 32 and 105 per cent. The F.T.I. of replicate Snowdown-1 specimens gave very good agreement and appear to confirm the repeatability of the freeze-thaw test.

The F.T.I. of the Snowdown-1 specimens containing 10 per cent cement was only 35 per cent. The F.T.I. for specimens with 15 per cent cement was 43 per cent, which was below the values obtained from the other minestone specimens which endured all 12 cycles. The F.T.I. of the Snowdown-2 specimens containing 10 per cent cement was, at 73 per cent, over twice that of similar Snowdown-1 specimens. An examination of the broken pieces of the Snowdown-1 specimens revealed a plane of weakness similar to that observed on the immersion specimens. This feature was not observed on the Snowdown-2 specimens.

The Hem Heath specimens gave the highest values of F.T.I. At the 8 and 12 per cent cement contents the strength, following the freeze-thaw test, was approximately equal to the 7 day strength. The F.T.I. of the Wardley specimens was approximately 70 per cent. The F.T.I. for both the Hem Heath and Wardley specimens appeared to increase only marginally over the particular range of cement contents studied.

The F.T.I. of the Parkside specimens increased dramatically when the cement content was increased from 6 to 12 per cent. The tests on specimens containing 6 per cent cement were terminated after 8 cycles because of severe deterioration. However, the specimens containing 12 per cent cement endured all 12 cycles and the F.T.I. was 78 per cent which is higher than that of the Wardley specimens.

Although the Blidworth specimens, produced with 6 per cent cement, failed before completion of the test, the specimens with 12 per cent cement had a F.T.I. of 46 per cent. This value is similar to values obtained for Snowdown-1 specimens containing 15 per cent cement. All specimens produced from Bagworth and Peckfield specimens deteriorated before completion of the test.

The compressive strength of all the laboratory blend specimens increased during the freeze-thaw test. The F.T.I. was between 10 and 20 percentage points higher than the values obtained for the Hem Heath specimens.

8.6.6 <u>Summary.</u> The resistance of minestone cement specimens to freezing and thawing appears to be largely dependent on the source of the material and, to a lesser extent, on the cement content. The measurements recorded during freeze-thaw tests show that, despite an initial contraction of between 0.1 and 0.3 per cent during the freezing period, the specimen had expanded by the end of the first cycle. The magnitude of this expansion ranged from less than 0.1 per cent to more than 1 per cent and was associated with an increase in the moisture content of between 0.25 and 5 per cent. No simple correlations were observed between the changes in the dimensions and masses of the minestone specimens during the first cycle and their behaviour during subsequent freeze-thaw cycles.

The least resistant specimens had usually expanded more than 0.1 per cent at the end of the first cycle. These specimens expanded rapidly and their moisture content increased by more than 3 per cent during the subsequent cycles. Many of these specimens did not complete the test and the F.T.I. of those that did was less than 50 per cent. The resistance of the specimens produced from Snowdown-1, Blidworth, Bagworth and Peckfield minestones was not influenced significantly by increasing the cement content.

The specimens produced from Snowdown-2, Hem Heath, Wardley and Parkside minestones displayed a higher degree of resistance. These specimens had expanded less than 0.1 per cent at the end of the first cycle and the moisture content increased by less than 1 per cent during subsequent cycles. The magnitude of both the expansion at the end of the first cycle, and the subsequent increase in moisture content, appeared to be independent of the cement content. However, the magnitude of the expansion at completion of the test was reduced significantly by increasing the cement content. Specimens containing more than 10 per cent cement expanded less than 0.5 per cent and their F.T.I. exceeded 70 per cent.

It is not apparent from this investigation why the resistance of the Snowdown-1 specimen was significantly less than that of the Snowdown-2 specimen. This will be commented on in more detail during the discussion in Chapter 9.

The freeze-thaw test appeared to have only a negligible effect upon the specimens produced from the laboratory blend. The dimensions and mass of the specimens remained almost unchanged during the test and the strength of all the specimens increased. There was no significant difference in the resistance of specimens containing between 4 and 12 per cent cement.

8.7 Effect of Initial Moisture Content on Immersion Resistance

8.7.1 Visual Examination. The effect of initial moisture content on the immersion resistance of Hem Heath, Wardley and Parkside specimens was not apparent from a visual examination. However, the Snowdown-2 specimens, produced at 1 and 2 per cent above optimum moisture content, did not develop the fissure which was observed on similar specimens produced at the optimum value. All specimens in this phase of the investigation remained intact throughout the immersion period.

8.7.2 Dimensional Changes. It can be seen from Table 8.5 that, with the exception of Hem Heath specimens, expansion during immersion was reduced when the moisture content increased. Typical results obtained from the various mixtures are shown in figures 8.17, 8.18, 8.19 and 8.20. In figure 8.17, the anisotropic behaviour of the

Snowdown-2 specimens is seen to diminish as the moisture content increased. This correlates with the absence of the fissure that was apparent with specimens produced at the optimum moisture content. The magnitude of the expansion at the end of the first day was reduced from about 0.1 per cent, for specimens produced at optimum, to 0.05 per cent, when the initial moisture content was increased by 2 per cent. Similarly, the expansion at the end of the test was reduced from more than 0.25 per cent to approximately 0.1 per cent. Furthermore, it can be seen from figure 8.17 that specimens produced at 1 per cent above optimum were still expanding at the end of the test, whereas the specimens at 2 per cent above appeared to cease to expand after 5 days under water. The expansion of the Hem Heath specimens, shown in figure 8.18, was unaffected by increasing the initial moisture content. The expansion of the Wardley and Parkside specimens is shown in figures 8.19 and 8.20. It is apparent from these plots, and from the data, that an increase in moisture content to 1 per cent above the optimum value reduced expansion during the test to less than 0.05 per cent. This compares with expansion of between 0.07 and 0.1 per cent for specimens produced at the optimum.

8.7.3 Mass Changes. It is clear from the results in Table 8.5 that the masses of all the specimens increased during the immersion test. Since all the specimens remained intact, these changes must be attributed to moisture gain. For the Snowdown-2 and Wardley specimens an increase of 2 per cent in the initial moisture content resulted in a reduction of some 50 per cent in the amount of moisture absorbed during the test. However, an increase in the initial moisture content of the Hem Heath and Parkside specimens produced no effect on the amount of moisture that they absorbed during the immersion test.

8.7.4 <u>Volume Change and Degree of Saturation.</u> The volume change and degree of saturation are summarised in Table 8.11. The Snowdown-2 specimens produced at optimum moisture content were completely saturated at the end of the first day under water. However, the degree of saturation of Snowdown specimens produced above the optimum was only 96 per cent at the end of the first day, although, by the completion of the test, this had risen to 100 per cent. All the specimens produced from Hem Heath, Wardley and Parkside minestones were completely saturated at the end of the first day.

8.7.5 <u>Compressive Strength.</u> The results in Table 8.6 show that, with the exception of the Hem Heath specimens, the compressive strength following immersion were lower than the strengths of similar specimens cured at constant moisture content for 7 days. The strength of the Hem Heath specimens appeared to increase marginally during the test. The relationship between initial moisture content and the R.I. appeared to be dependent upon the source of the minestone.

The Snowdown-2 specimens, produced at the higher initial moisture contents, retained higher strengths than

Table 8.11 Effect of Initial Moisture Content on Resistance to Immersion: Volume and Degree of Saturation

Sample	Cement Content (°/o)	Moisture Content (°/o)	Initial Degree of Saturation (°/o)	Volume Change (ml) first day	Volume Change (ml) last day	Degree of Saturation (°/o) first day	Degree of Saturation (°/o) last day
Snowdown-2	10	optimum	82	2.6	8.8	100.0	100.0
- do -	10	opt + 1	88	1.7	4.2	96.0	100.0
- do -	10	opt + 2	91	1.4	3.5	96.0	100.0
Hem Heath	8	optimum	89	1.7	2.4	100.0	100.0
- do -	8	opt + 1	92	1.6	1.9	100.0	100.0
- do -	8	opt + 2	93	1.7	2.0	100.0	100.0
Wardley	6	optimum	85	1.1	1.4	100.0	100.0
- do -	6	opt + 1	92	0.7	0.9	100.0	100.0
- do -	6	opt + 2	94	0.7	1.0	100.0	100.0
Parkside	12	optimum	93	1.7	2.3	100.0	100.0
- do -	12	opt + 1	93	1.3	1.1	100.0	100.0
- do -	12	opt + 2	92	1.2	1.4	100.0	100.0

all the minestone specimens. However, the R.I. of the specimens produced 2 per cent above the optimum moisture content was only 65 per cent. Although this represents an improvement on the R.I. of similar specimens produced at the optimum value, it is still less than the values obtained from other minestones. An examination of the broken specimens revealed the presence of the plane of weakness that had also been observed in the specimens produced at optimum moisture content. This was a little surprising in view of the absence of any visible evidence of this flaw prior to the compressive strength test. It was concluded that this plane of weakness had again contributed to the reduction in strength.

The R.I. of the Hem Heath specimens was approximately 90 per cent and did not appear to be influenced by an increase in the initial moisture content. The R.I. of the Wardley specimen produced at both 1 and 2 per cent above the optimum value was 83 per cent, a 13 percentage point improvement on the value obtained with specimens produced at the optimum. The Parkside specimens produced at the optimum moisture content had Resistance Indices greater than 80 per cent, and when the moisture content was increased the values obtained were less than 70 per cent.

8.7.6 Summary. The effect of initial moisture content on the immersion resistance of minestone specimens appeared to be largely dependent upon the source of the material. Although an increase in the initial moisture content may reduce the magnitude of the changes in dimension and mass, this does not necessarily result in reduced strength loss. The resistance of the Snowdown-2 specimens was improved considerably by increasing the initial moisture content, however, both the final expansion, and the strength loss, still exceed those of other minestone specimens. Increasing the initial moisture content of the Wardley specimens improved their stability, as expressed by the changes in dimension and mass, and also significantly increased the R.I. The Hem Heath specimens appeared to be the most resistant and their behaviour, during the test, was apparently unaffected by increases in the initial moisture content. The expansion of Parkside specimens was reduced only marginally when the initial moisture content was increased. This apparent improvement in resistance was not reflected by the R.I. which was reduced significantly.

8.8 Effect of Initial Moisture Content on the Resistance to Freezing and Thawing

8.8.1 Visual Examination. It was evident from the appearance of the Snowdown and Wardley specimens that their resistance to freezing and thawing had been improved considerably by increasing the initial moisture content. On removal from the thaw cabinet, at the end of the first cycle, the specimens produced at the higher moisture contents had clearly absorbed much less moisture than similar specimens produced at the optimum value. The absorbed moisture had only risen to height of some 20 mm above the base of the specimen. The appearance of these specimens did not change during subsequent cycles and their quality appeared to be totally unaffected by cyclic freezing and thawing. There was no evidence of any improvement in the resistance of the Hem Heath and Parkside specimens produced above the optimum moisture content. On removal from the cabinet at the end of the first cycle, all the specimens had absorbed moisture to their full height. The specimens were not prone to damage and did not lose any material during the test.

8.8.2 <u>Dimensional Changes.</u> Typical plots of the changes in dimension recorded during freeze-thaw tests, on the four minestone-cement mixtures, are shown in figures 8.21 to 8.24. The changes recorded during the first and last cycles are given in Table 8.7.

The dimensional stability of specimens produced from Snowdown-2, Hem Heath and Wardley minestones was improved significantly when the moisture content was increased above the optimum value and this is shown clearly in figures 8.21 to 8.23. The anisotropic behaviour which was evident at optimum moisture content, was absent at the higher moisture contents. It can be seen from Table 8.7 that the expansion at the end of the first cycle was reduced. In the case of Snowdown-2 and Wardley minestones this expansion was negligible when the moisture content was increased 2 per cent. The expansion during subsequent cycles was also reduced as the moisture content increased. Total expansion of the Snowdown-2 specimens, which exceeded 0.5 per cent at optimum moisture content, was negligible at the highest moisture content. The total expansion of Wardley specimens was reduced by 80 per cent to less than 0.1 per cent, whilst that of Hem Heath specimens was reduced by 50 per cent to less than 0.2 per cent. The amplitude of the dimensional changes, which remained constant throughout the test, was approximately 0.15 per cent.

The effect of initial moisture content on the dimensional stability of Parkside specimens is shown in figure 8.24. With these specimens only a marginal reduction was recorded in the expansion when the moisture content was raised to 2 per cent above the optimum.

8.8.3 Mass Changes. The changes in the masses of the specimens, are given in Table 8.7 and these are attributed entirely to moisture loss or gain. The amount of moisture absorbed during the first thaw period, by the Snowdown-2 and Wardley specimens, was progressively reduced as the initial moisture content increased. At the end of the first cycle the mass of each of the specimens, produced at 2 per cent above optimum, was approximately equal to the original value. Similarly, the moisture that they absorbed during the subsequent cycles was less than that absorbed by the specmens produced at optimum. Although the moisture content varied with the cyclic freezing and thawing, the specimens produced at the highest moisture contents did not exhibit a progressive increase in moisture content during the course of the test.

Increases in the initial moisture content of the Hem Heath and Parkside specimens had only a minimal effect of the amount of moisture absorbed during the first thaw period. However, the amount of moisture absorbed during subsequent cycles was reduced.

The moisture content of specimens produced at 2 per cent above optimum had increased by between 0.1 and 0.9 per cent at the end of the test.

8.8.4 Volume Changes and Degree of Saturation. The volume change and degree of saturation, following the first and last freeze-thaw cycles, are given in Table 8.12. The improved stability of the Snowdown-2 and Wardley specimens, suggested by the dimension and mass data, is reflected in the calculated values of the degree of saturation. The degree of saturation, which was observed to increase during tests on specimens produced at optimum moisture content, remained almost unchanged when the initial moisture content was raised 2 per cent. The increase in the degree of saturation during tests on Hem Heath and Parkside specimens, produced at 2 per cent above optimum moisture content, was marginally less than for similar specimens, produced at the optimum value.

8.8.5 Compressive Strength. The results of the compressive strength tests are summarised in Table 8.8. These show that the strength retained by the Snowdown-2 and Wardley specimens, produced above the optimum moisture content, was greater than the strength retained by similar specimens produced at the optimum value. However, the strength retained by the Hem Heath and Parkside specimens was reduced as the initial moisture content was increased.

Table 8.12 Effect of Initial Moisture Content on Resistance to Freezing and Thawing: Volume and Degree of Saturation

Sample	Cement Content	Moisture Content	Initial Degree of Saturation	Volume Change (ml)				Degree of Saturation (°/o)	
				first		last		first	last
	(°/o)	(°/o)	(°/o)	fr'ze	thaw	fr'ze	thaw	thaw	thaw
Snowdown-2	10	optimum	82	-3.9	2.0	13.1	16.5	93.0	97.0
- do -	10	opt + 1	88	-3.8	0.9	- 1.2	2.7	89.0	91.0
- do -	10	opt + 2	91	-3.7	0.3	- 3.2	0.6	90.0	92.0
Hem Heath	8	optimum	89	-6.7	2.0	5.4	10.1	95.0	99.0
- do -	8	opt + 1	92	-7.3	1.5	- 0.4	5.6	99.0	99.0
- do -	8	opt + 2	93	-7.0	1.5	- 1.5	5.0	98.0	98.0
Wardley	6	optimum	85	-5.1	1.2	9.6	11.7	96.0	97.0
- do -	6	opt + 1	92	-5.5	0.2	- 1.9	3.2	96.0	98.0
- do -	6	opt + 2	94	-4.7	0.4	- 2.8	2.3	96.0	98.0
Parkside	12	optimum	93	-4.7	0.9	-1.0	3.7	94.0	100.0
- do -	12	opt + 1	93	-3.9	0.9	-3.8	2.3	94.0	100.0
- do -	12	opt 2	92	-3.9	1.0	-2.5	2.6	93.0	96.0

The relationship between initial moisture content and F.T.I. again appeared to be dependent upon the source of the minestone. The value for the Snowdown-2 specimens increased substantially when the moisture content was raised above optimum. It increased by more than 30 percentage points to a maximum value of 110 per cent, at 1 per cent above optimum, and then decreased slightly to 101 per cent, at 2 per cent above optimum. These were the only specimens, in this phase of the investigation, to develop additional strength during the freeze-thaw test. An examination of the broken specimens did not reveal the plane of weakness observed on the immersion specimens. The F.T.I. for the Wardley specimens also increased as the moisture content was raised. It increased from 66 per cent to 83 per cent, when the moisture content was raised 1 per cent, and increased further, to 88 per cent, when the moisture content was raised by 2 per cent. For the Parkside specimens the index appeared to be unaffected by increased moisture content, with all three mixes producing values of approximately 80 per cent. An increase in the moisture content of the Hem Heath specimens appeared to have a slightly detrimental effect with the index being reduced by 8 percentage points, to 86 per cent, when the moisture content was raised by 2 per cent.

8.8.6 Summary. The stability of specimens undergoing freeze-thaw testing was improved when the initial moisture content was raised above the optimum. This was clearly demonstrated by the reductions in the dimensional and mass changes recorded throughout the tests. This improvement, and its effect on the compressive strength retained at the end of the test, was dependent upon the source of the minestone. The stability of the Snowdown-2 and Wardley specimens, produced at 2 per cent above optimum, was much improved when compared with that of similar specimens produced at the optimum value. This improvement was reflected by increases in the retained strength and F.T.I.

Snowdown-2 specimens, which were the least resistant samples when produced at optimum moisture content, showed the greatest resistance when the initial moisture content was increased. The stability of the Hem Heath specimens, undergoing cyclic freezing and thawing, was also improved by increasing the initial moisture content, but to a lesser degree. However, this improvement was not reflected by the retained strength, or F.T.I. which were reduced progressively as the moisture content was raised. The behaviour of the Parkside specimens was only marginally improved by increasing the initial moisture content, with the effect on retained strength and F.T.I. being negligible.

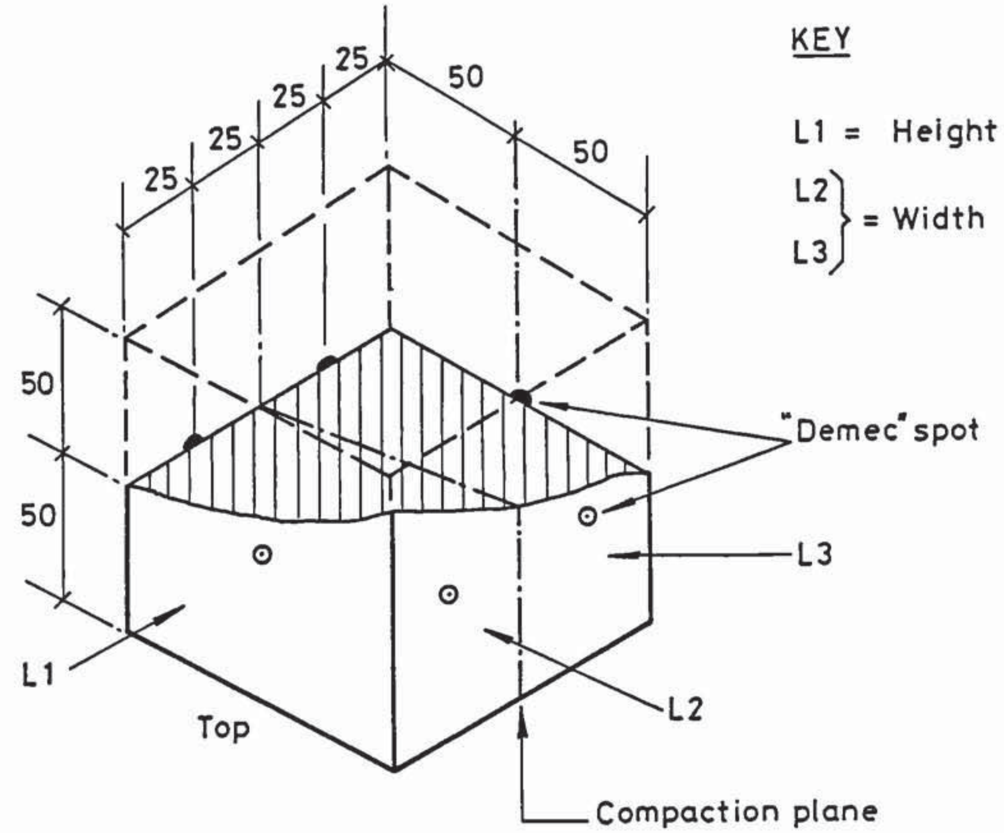


Figure 8.1 Location of Demec Spots

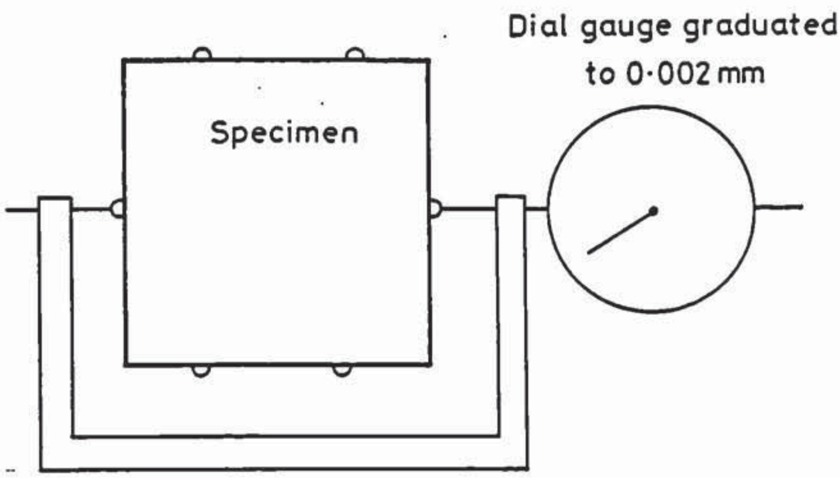
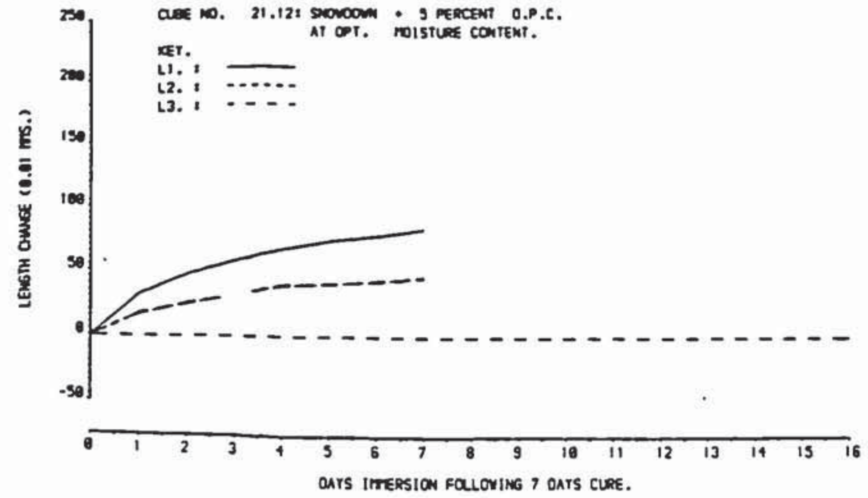
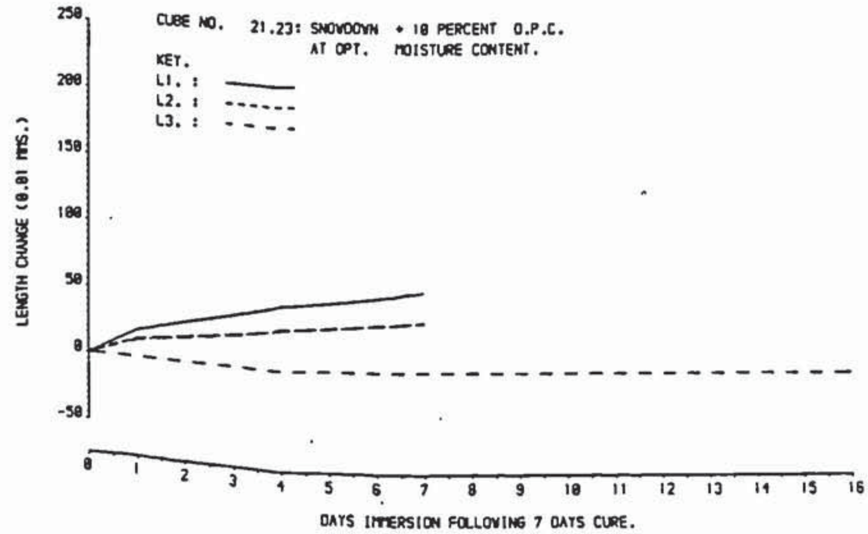


Figure 8.2 Diagrammatic sketch of Length Comparator





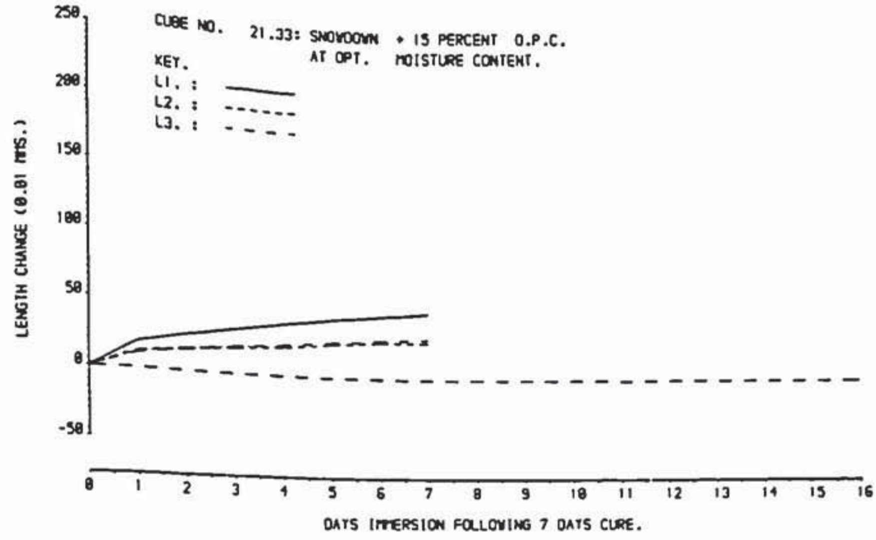
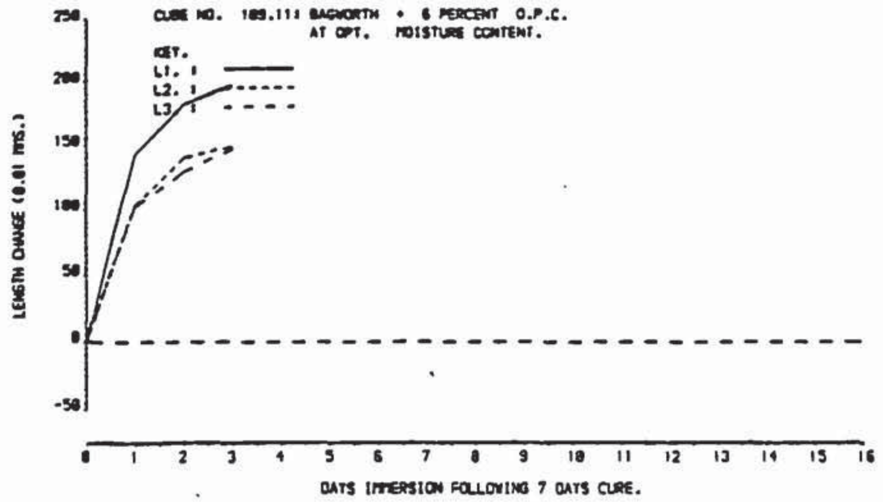


Fig 8.3 Effect of Cement Content upon Resistance to Immersion: Snowdown-1



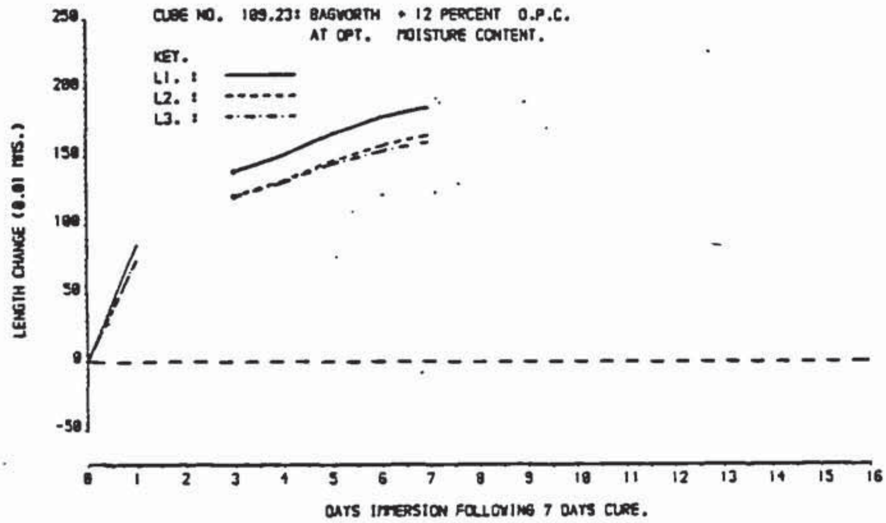
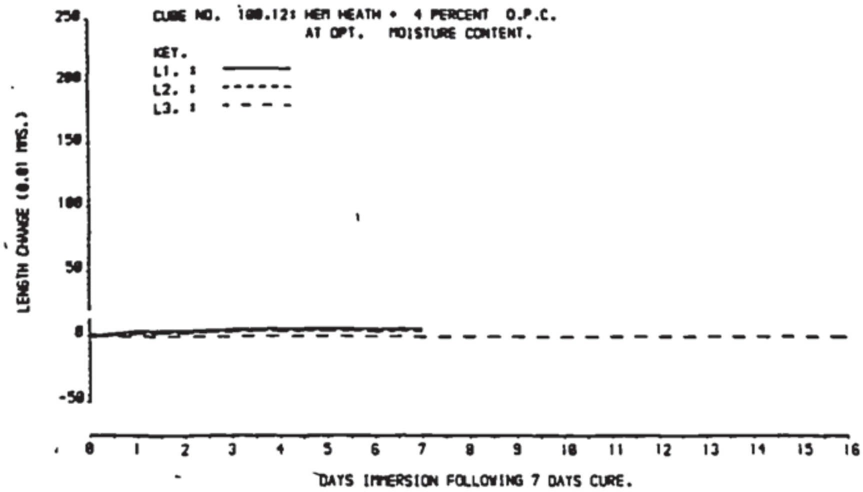
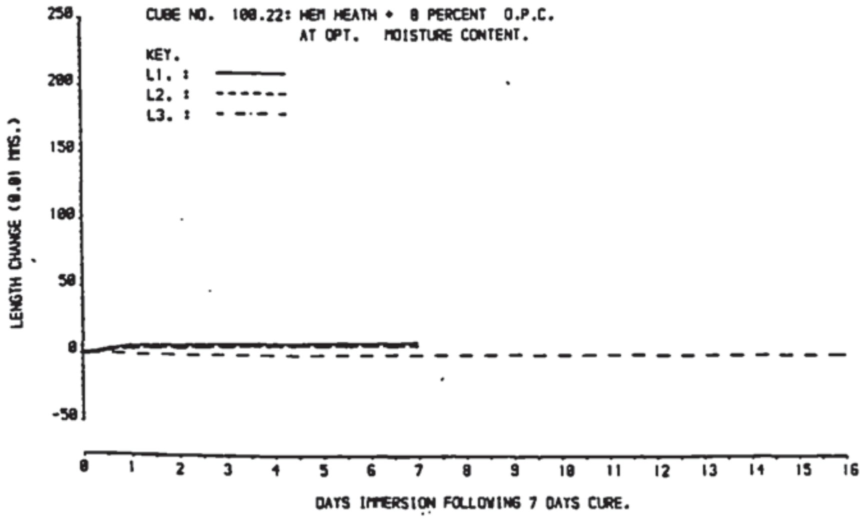


Fig 8.4 Effect of Cement Content upon Resistance to Immersion: Bagworth





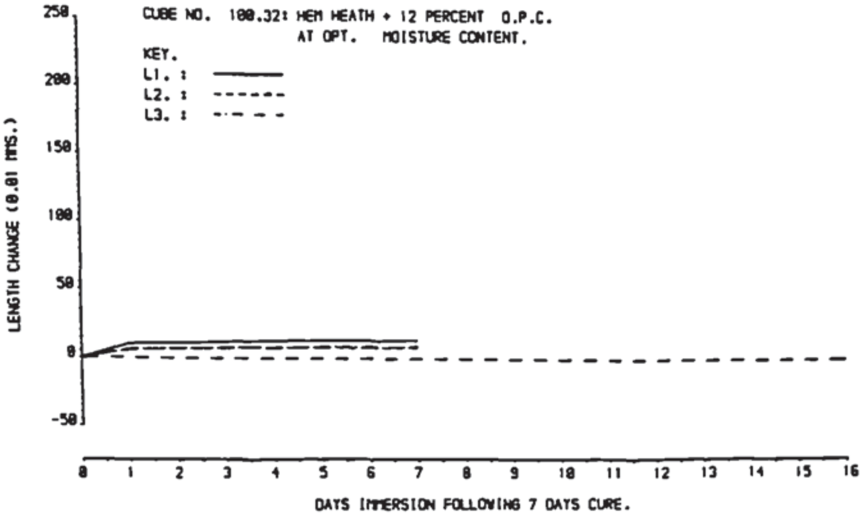
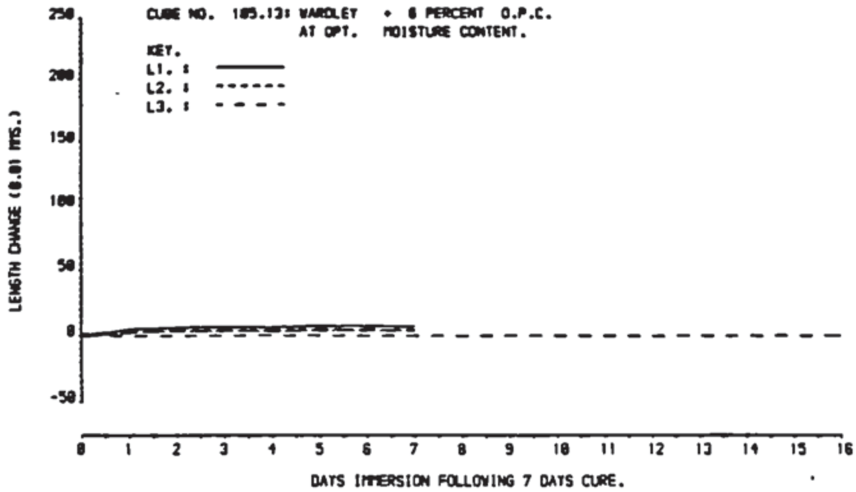


Fig 8.5 Effect of Cement Content upon Resistance to Immersion: Hem Heath



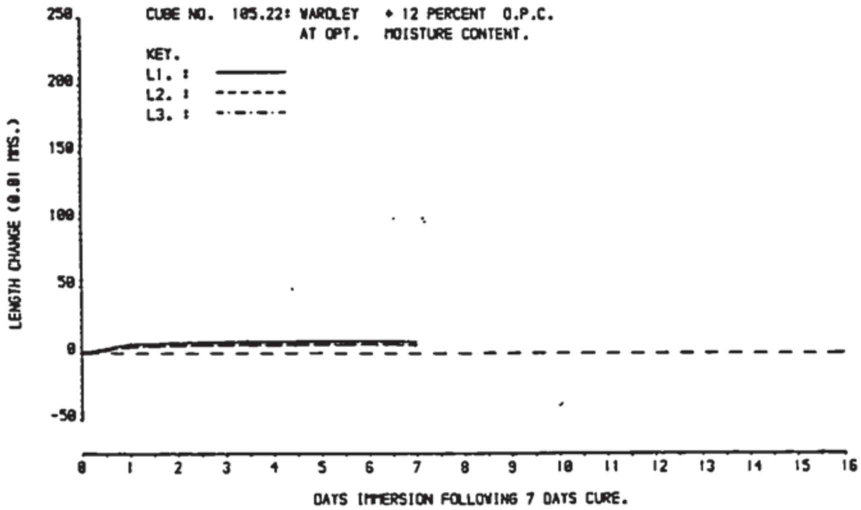
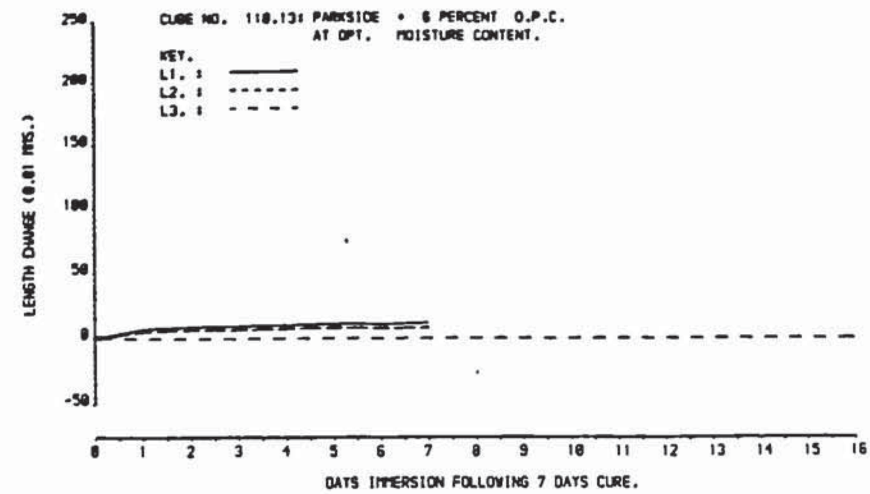


Fig 8.6 Effect of Cement Content upon Resistance to Immersion: Wardley



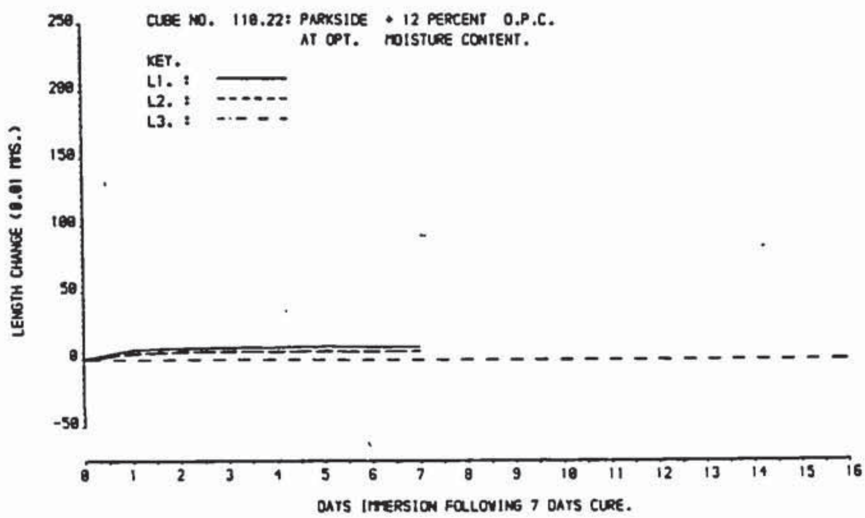
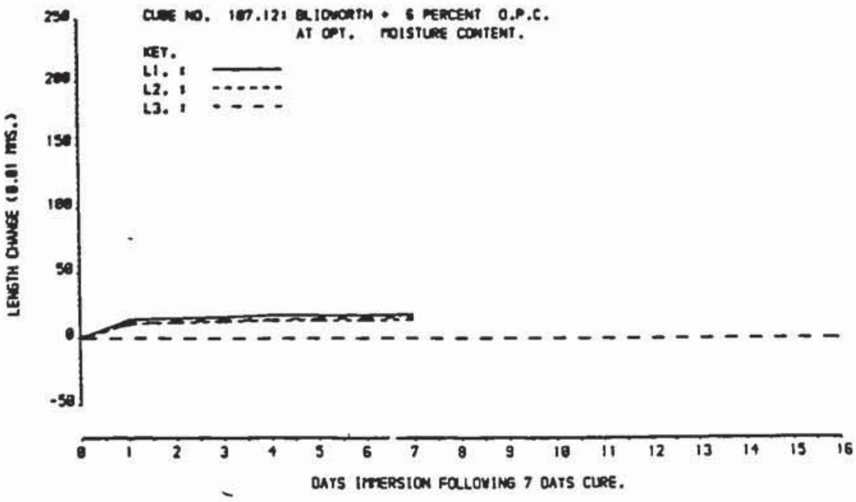


Fig 8.7 Effect of Cement Content upon Resistance to Immersion:
Parkside



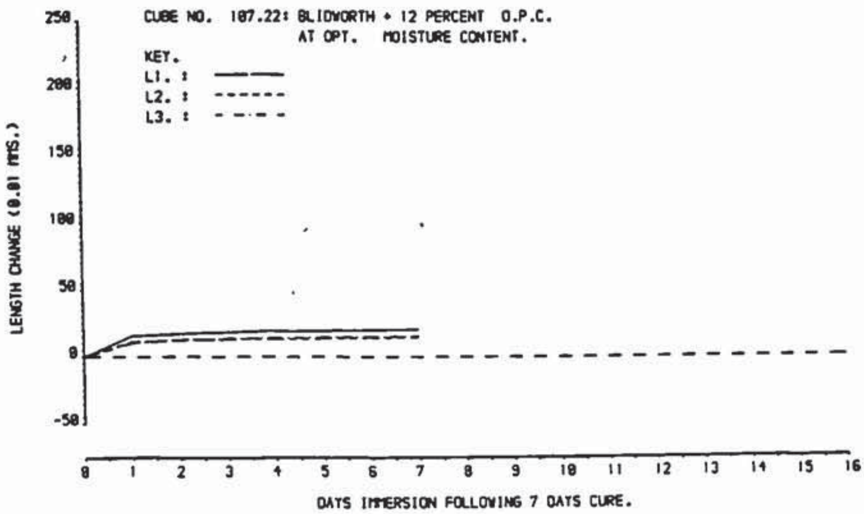
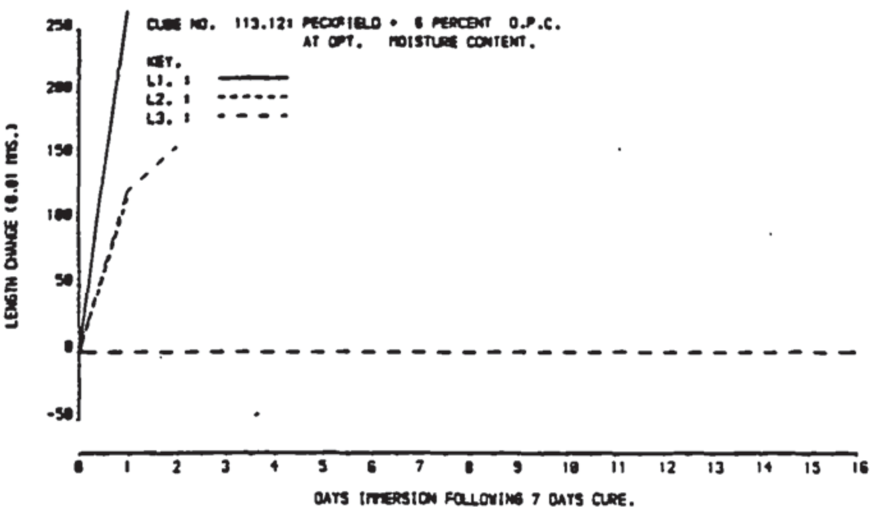


Fig 8.8 Effect of Cement Content upon Resistance to Immersion: Blidworth



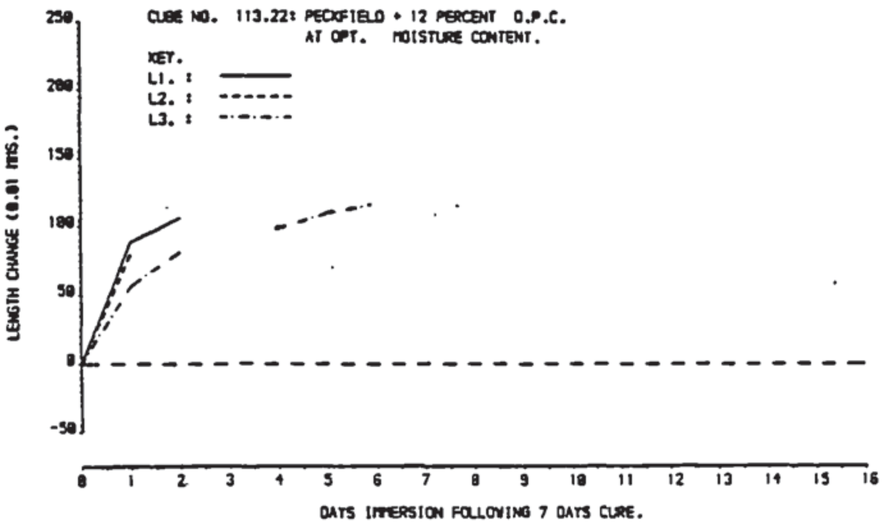


Fig 8.9 Effect of Cement Content upon Resistance to Immersion: Peckfield

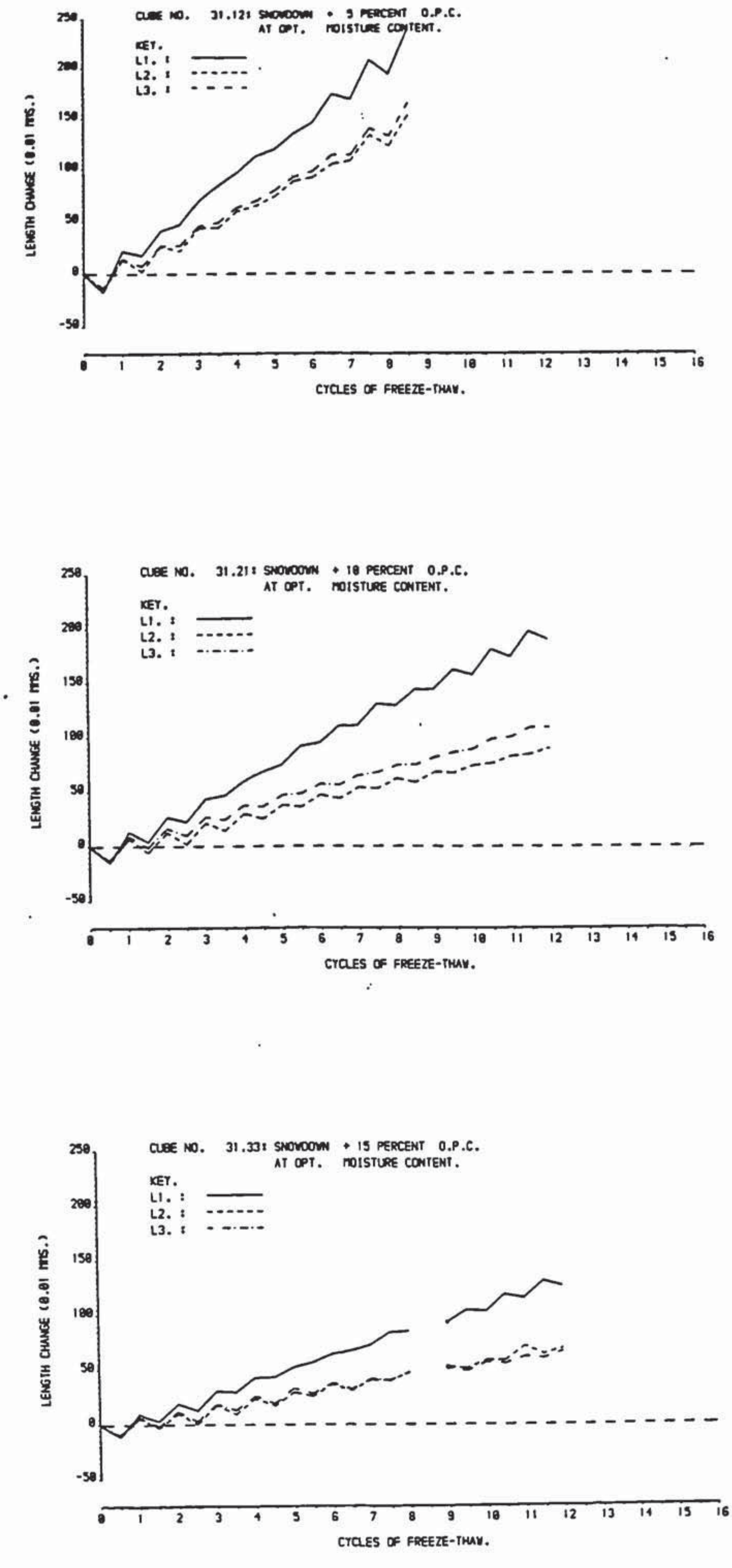


Fig 8.10 Effect of Cement Content upon Resistance to Freezing and Thawing: Snowdown-1

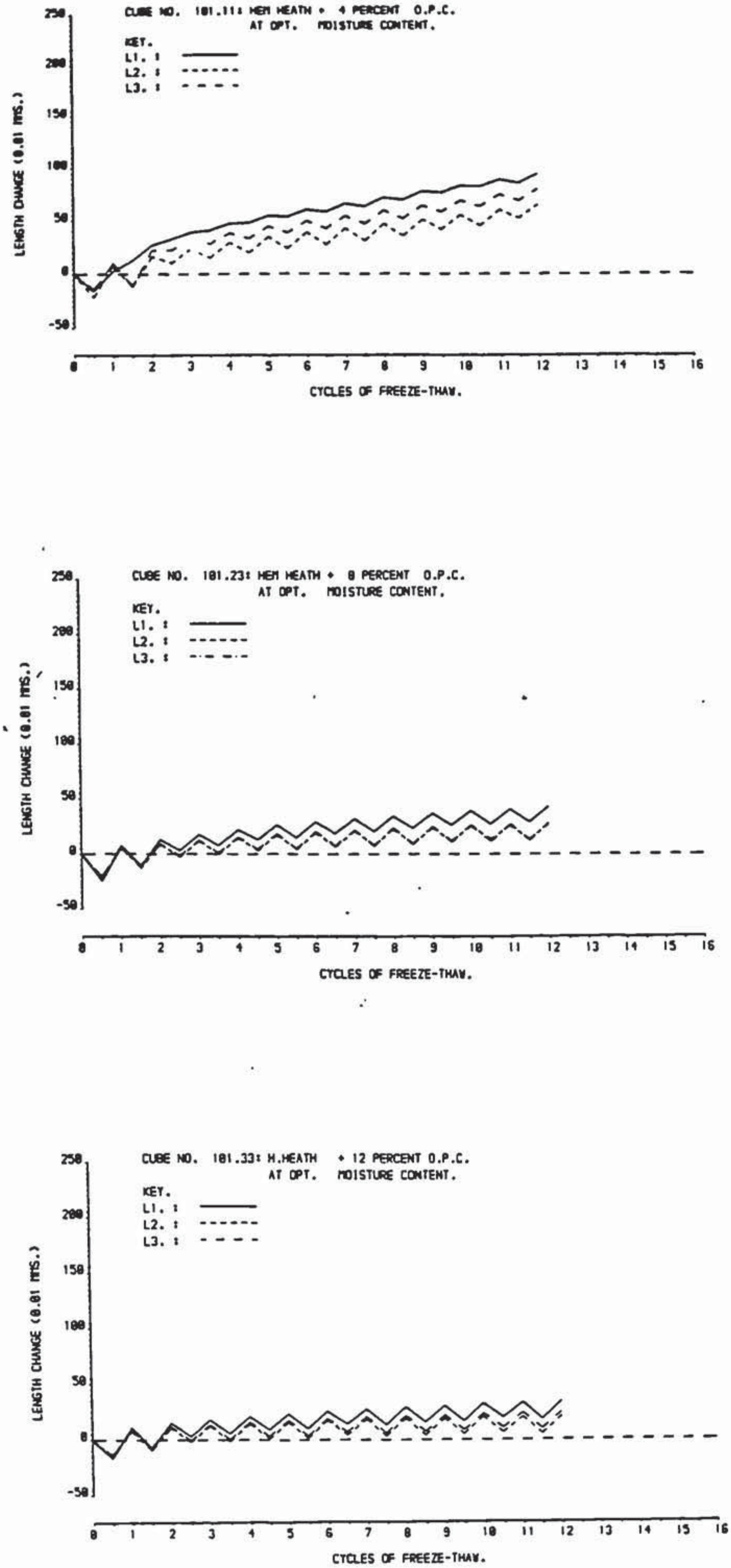
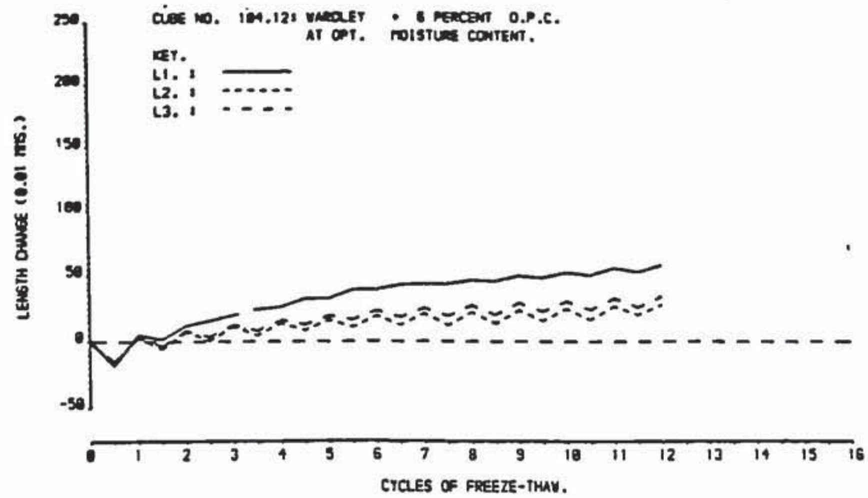


Fig 8.11 Effect of Cement Content upon Resistance to Freezing and Thawing: Hem Heath



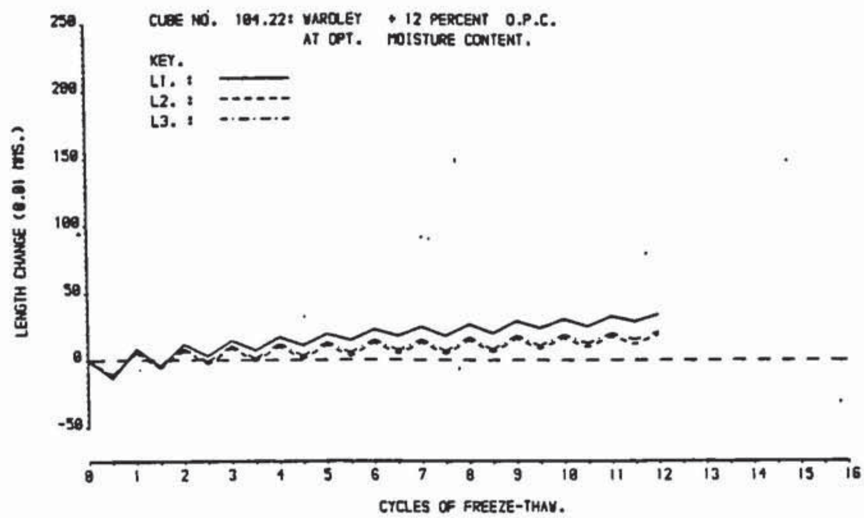
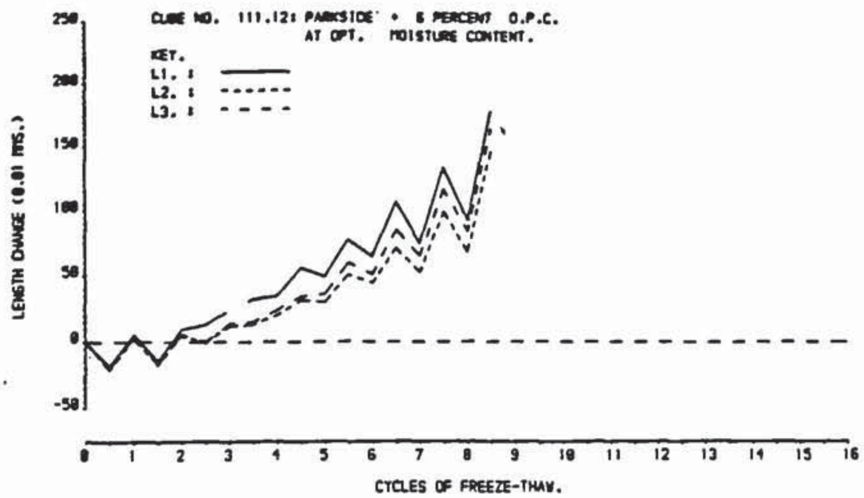


Fig 8.12 Effect of Cement Content upon Resistance to Freezing and Thawing: Wardley



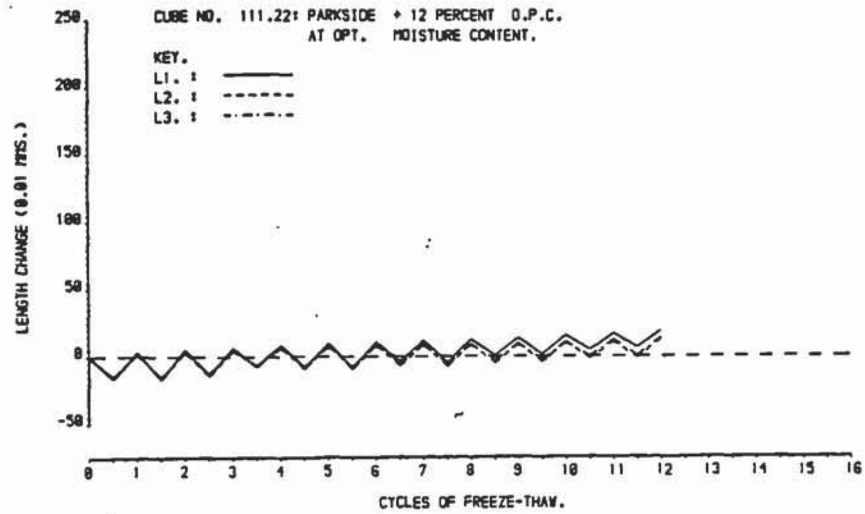
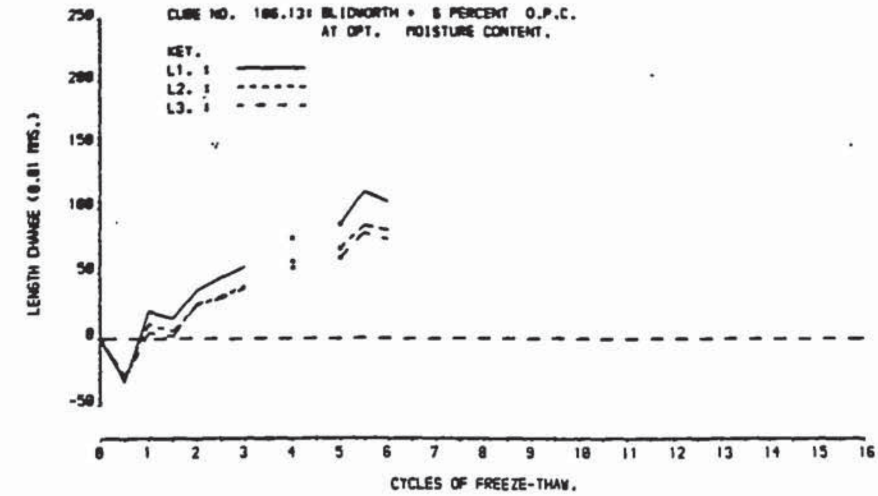


Fig 8.13 Effect of Cement Content upon Resistance to Freezing and Thawing: Parkside



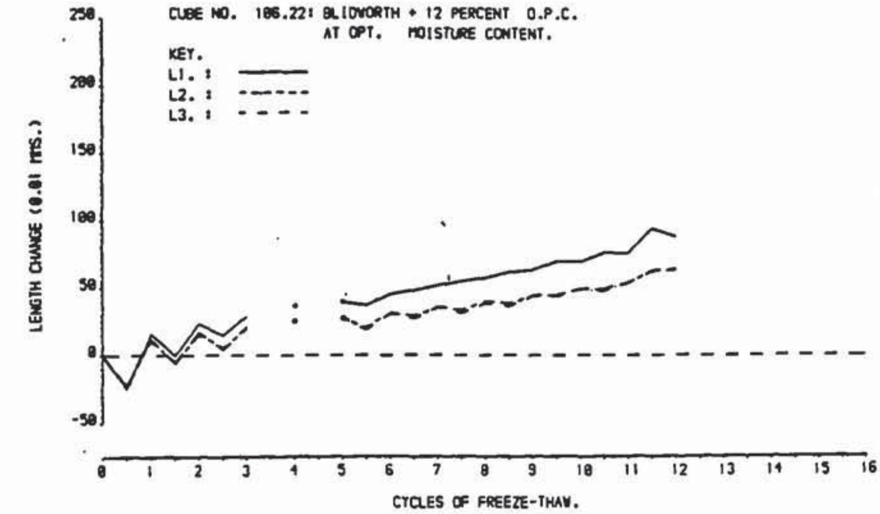
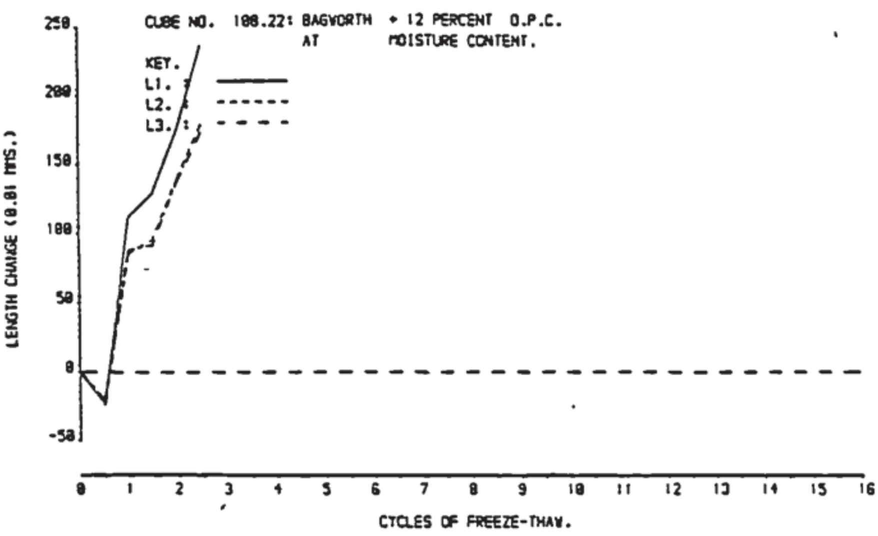


Fig 8.14 Effect of Cement Content upon Resistance to Freezing and Thawing: Blidworth



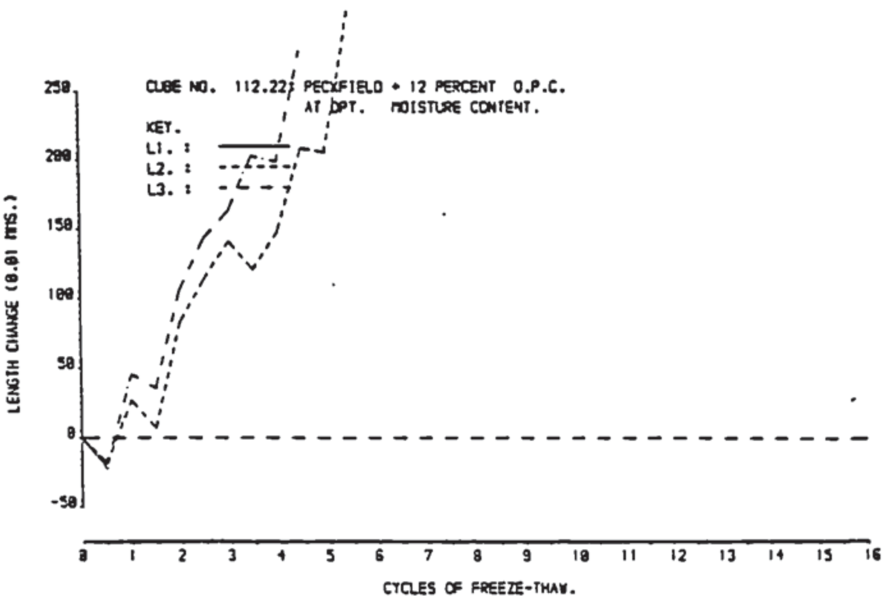
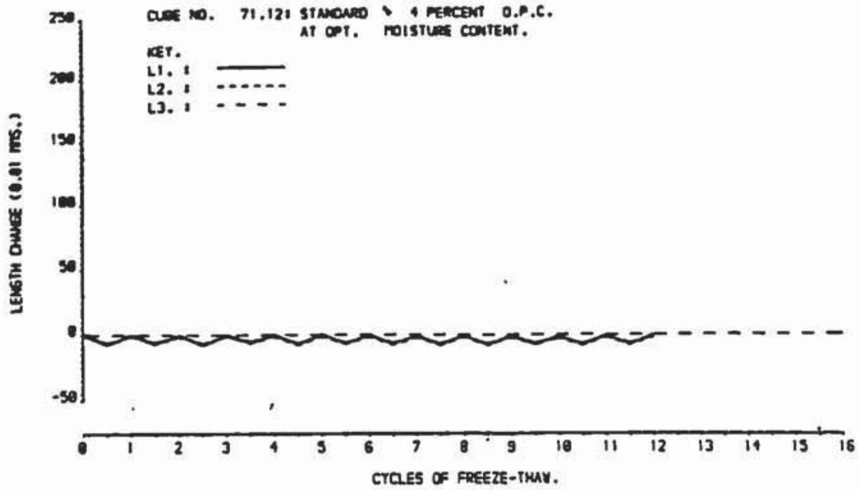
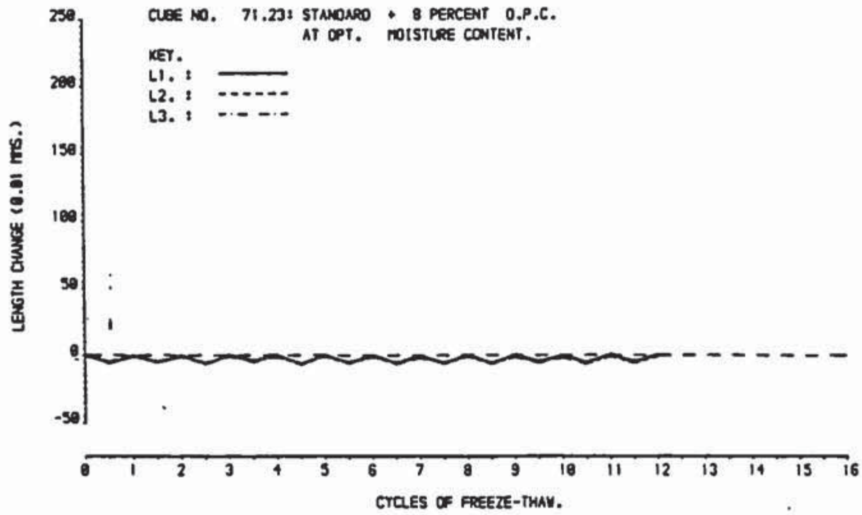


Fig 8.15 Effect of Cement Content upon Resistance to Freezing and Thawing: Bagworth/Peckfield





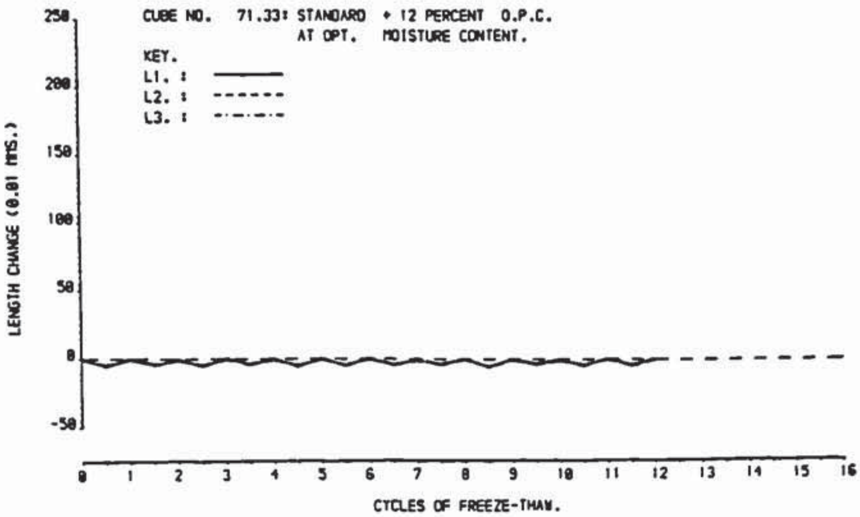
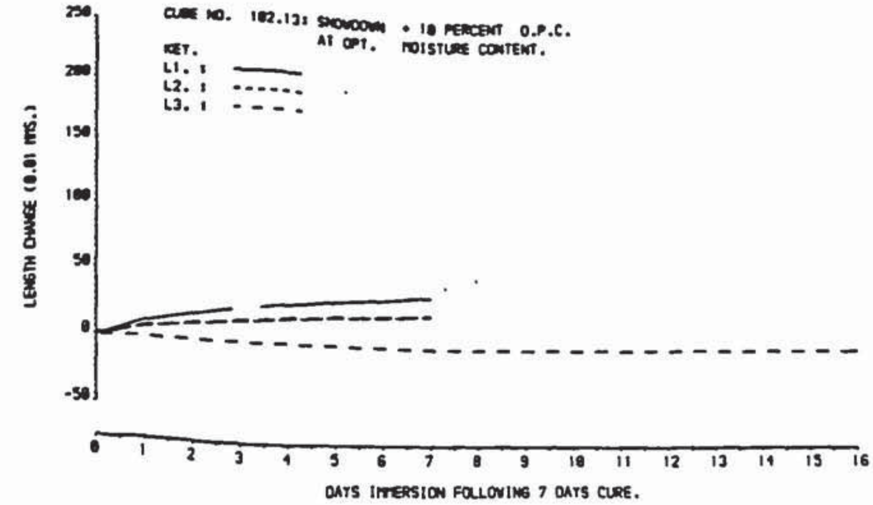
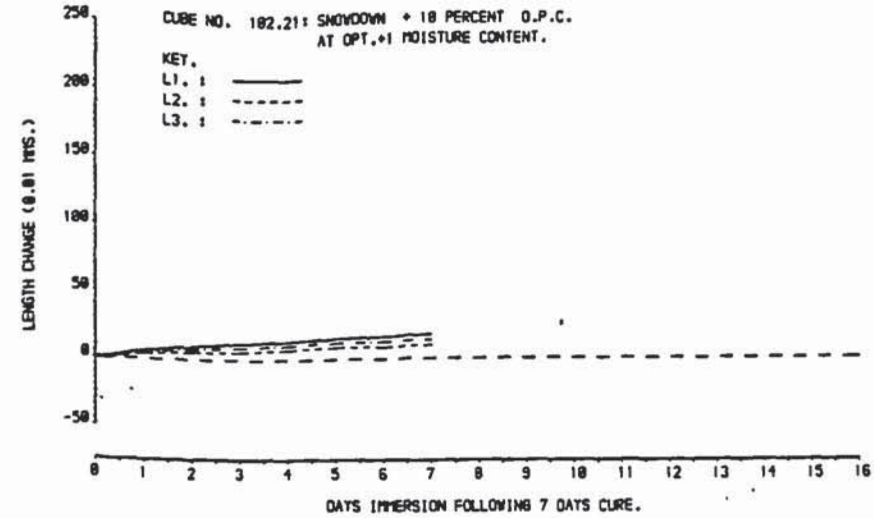


Fig 8.16 Effect of Cement Content upon Resistance to Freezing and Thawing: Laboratory Blend





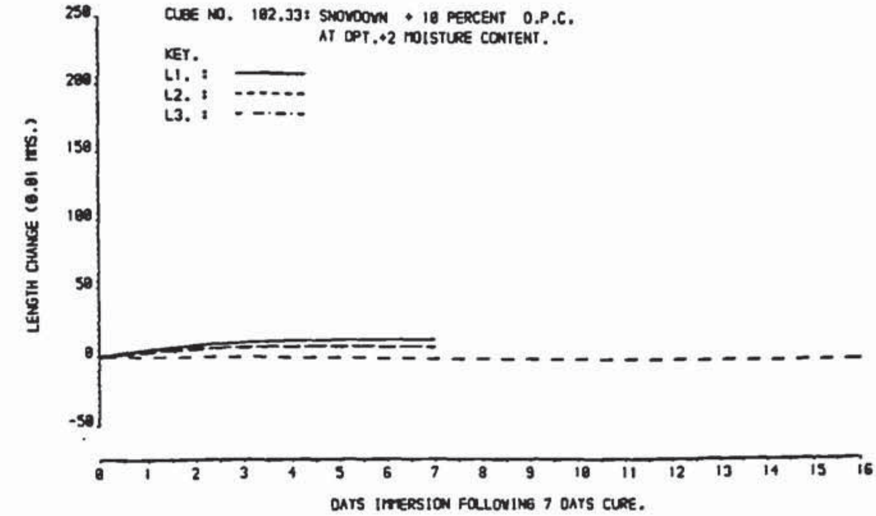
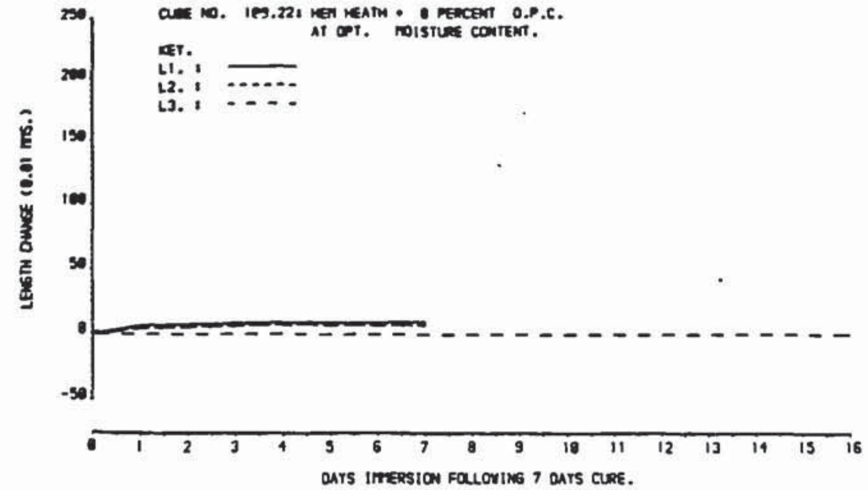
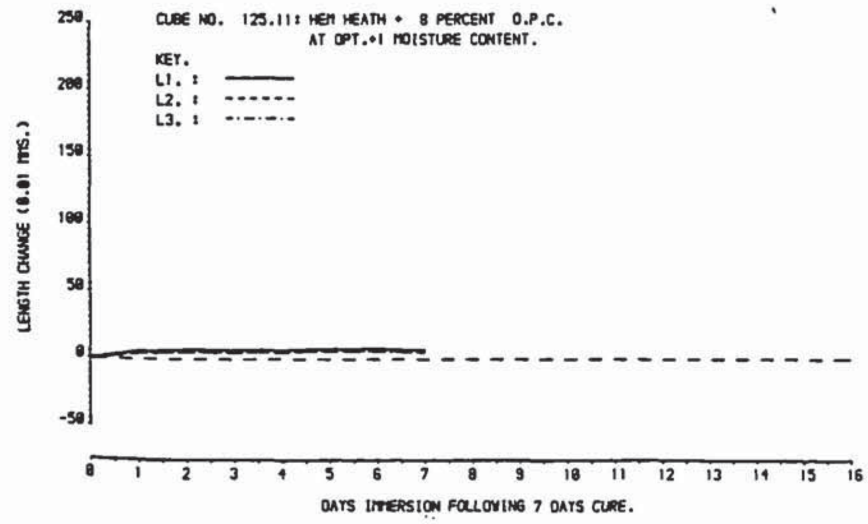


Fig 8.17 Effect of Moisture Content upon Resistance to Immersion: Snowdown-2





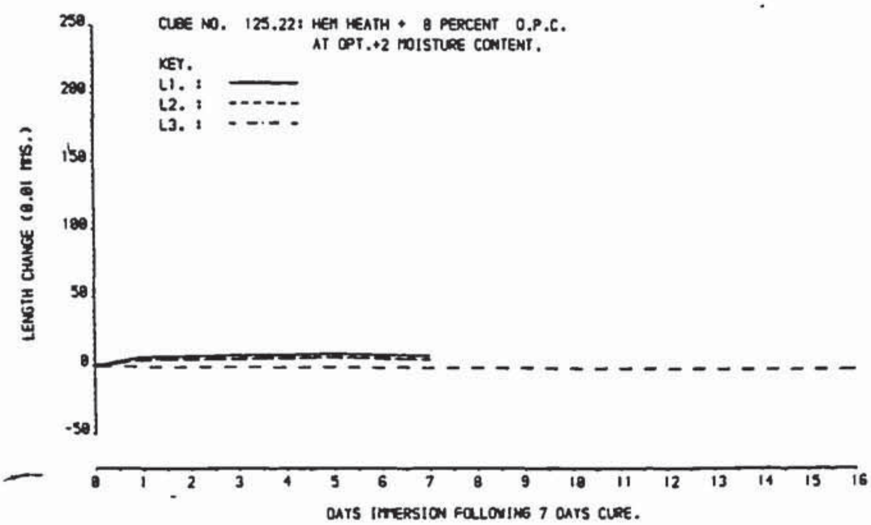
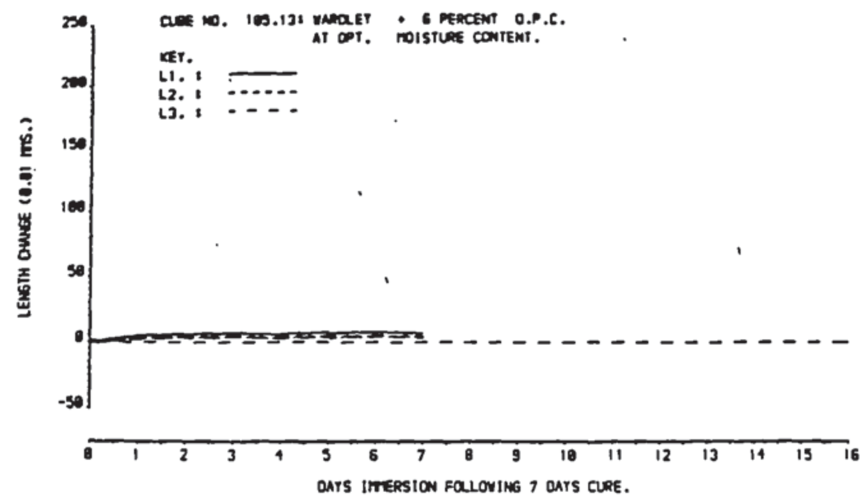
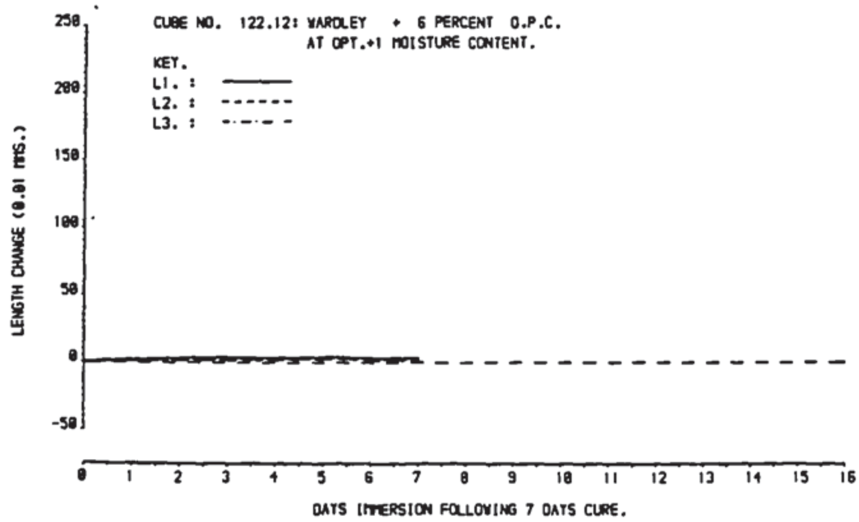


Fig 8.18 Effect of Moisture Content upon Resistance to Immersion: Hem Heath





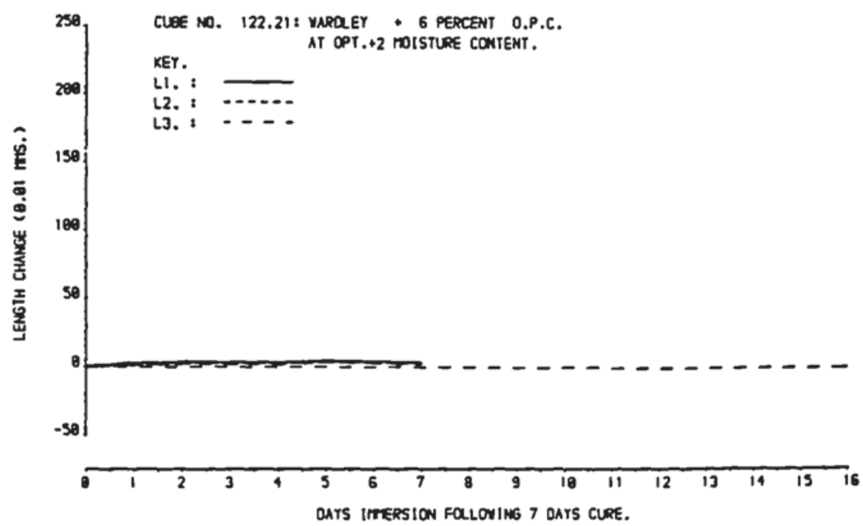
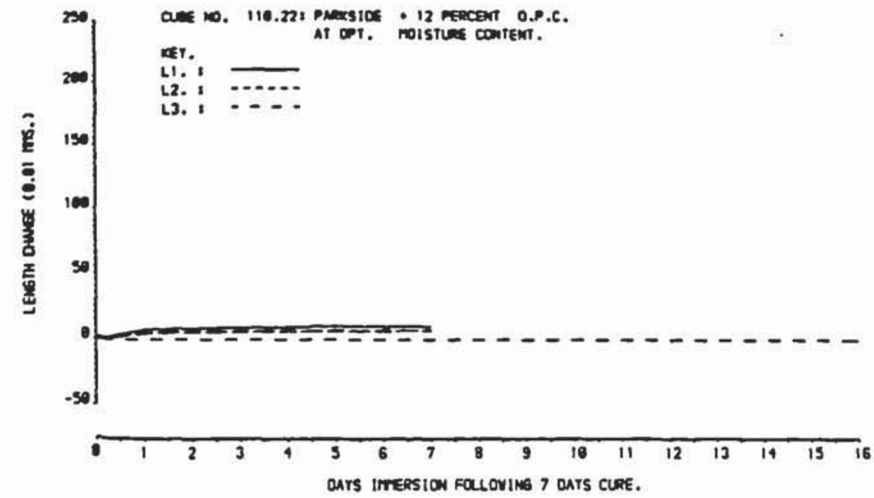
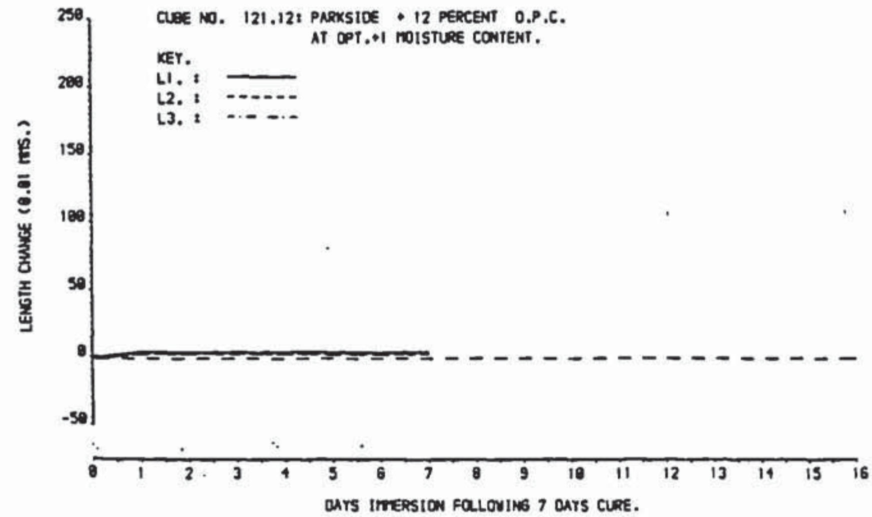


Fig 8.19 Effect of Moisture Content upon Resistance to Immersion: Wardley





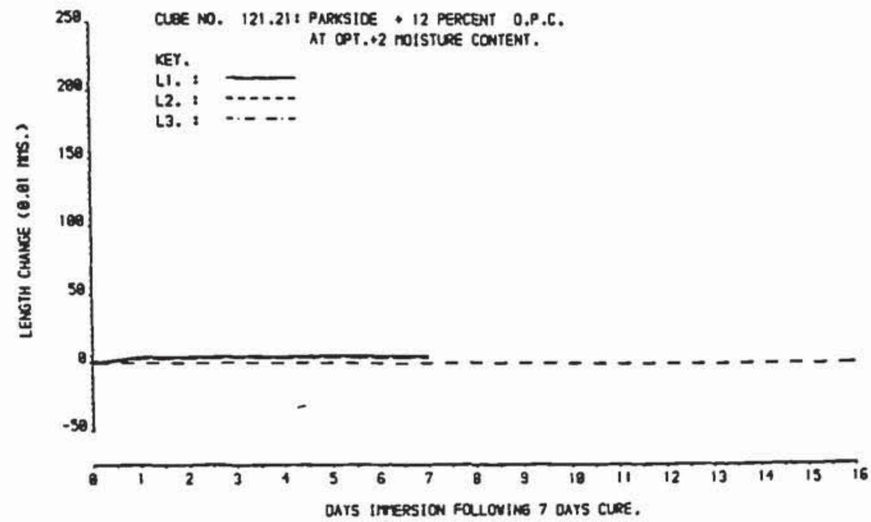
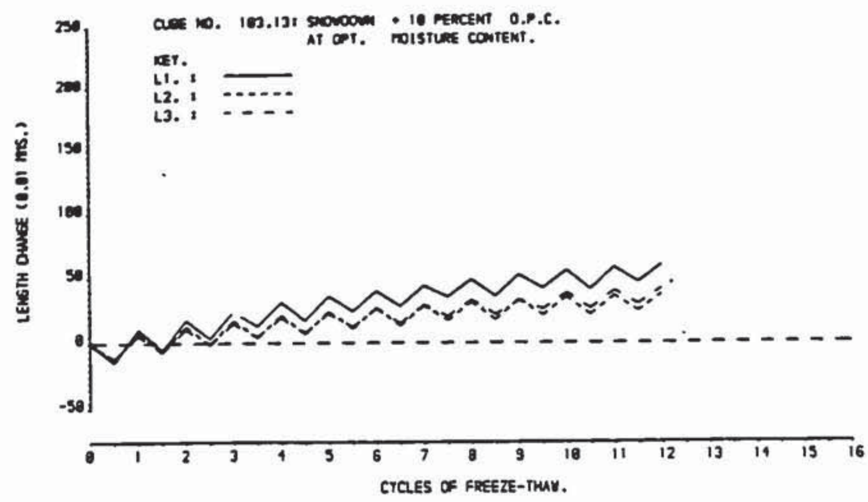
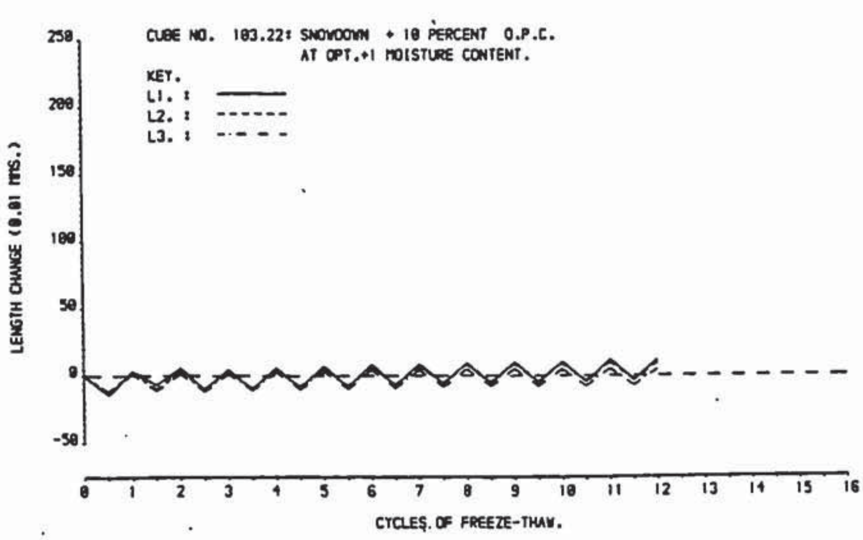


Fig 8.20 Effect of Moisture Content upon Resistance to Immersion: Parkside





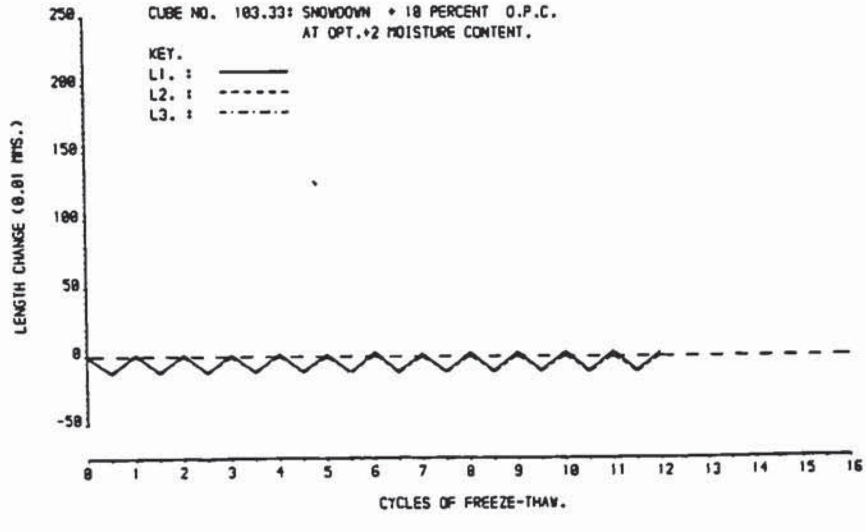
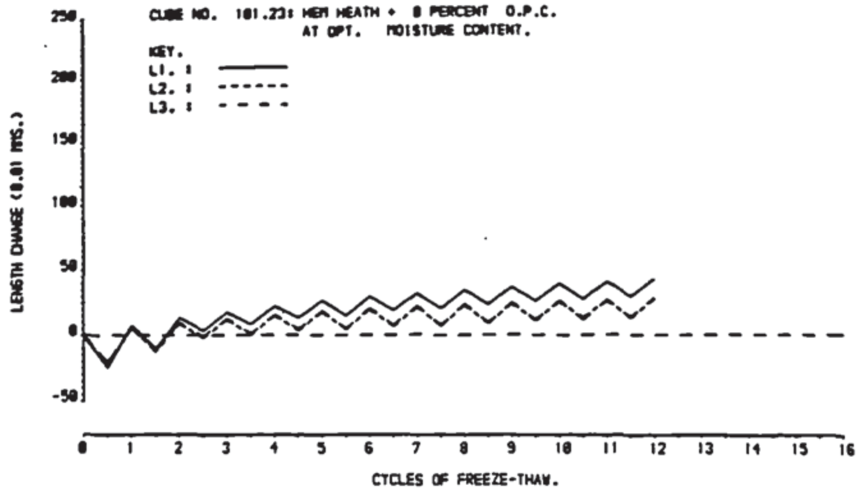
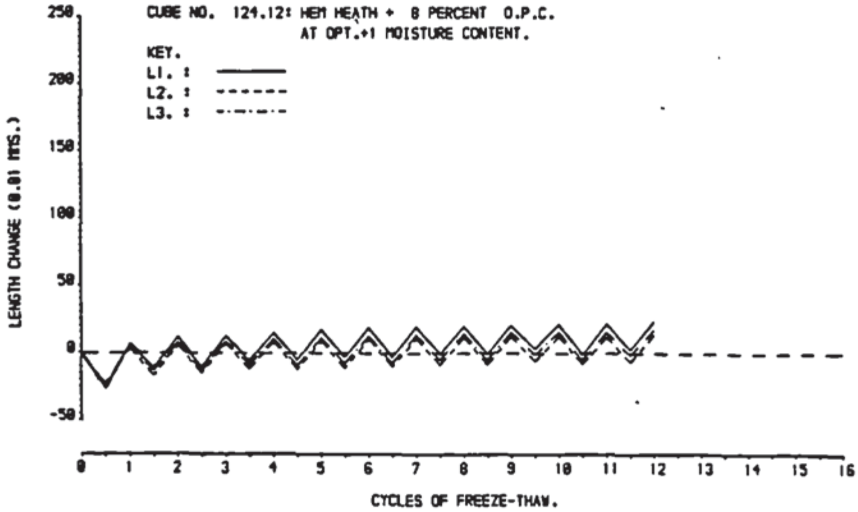


Fig 8.21 Effect of Moisture Content upon Resistance to Freezing and Thawing: Snowdown-2





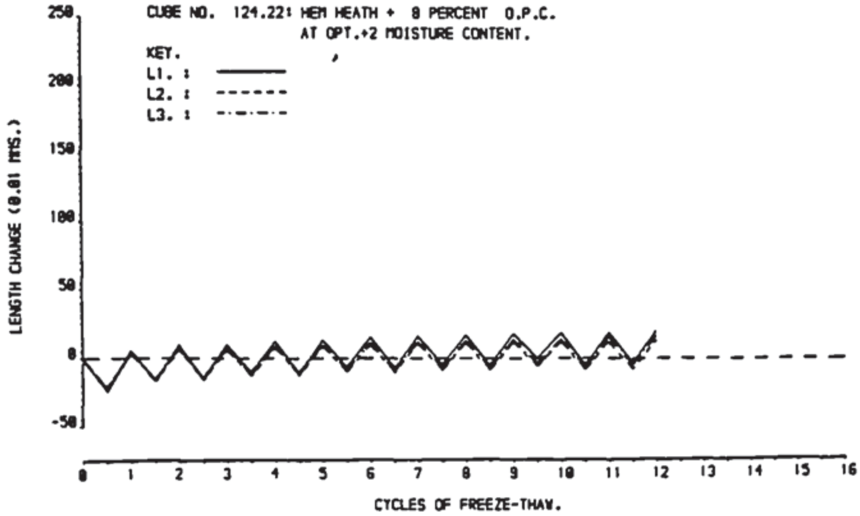
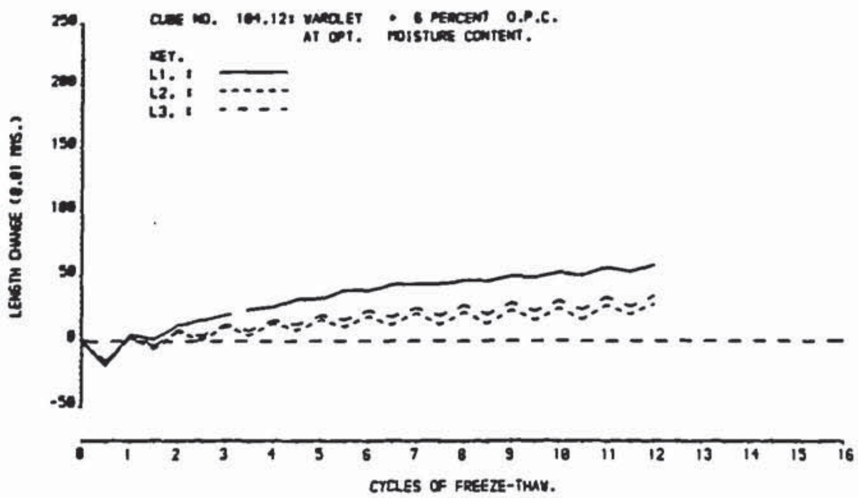
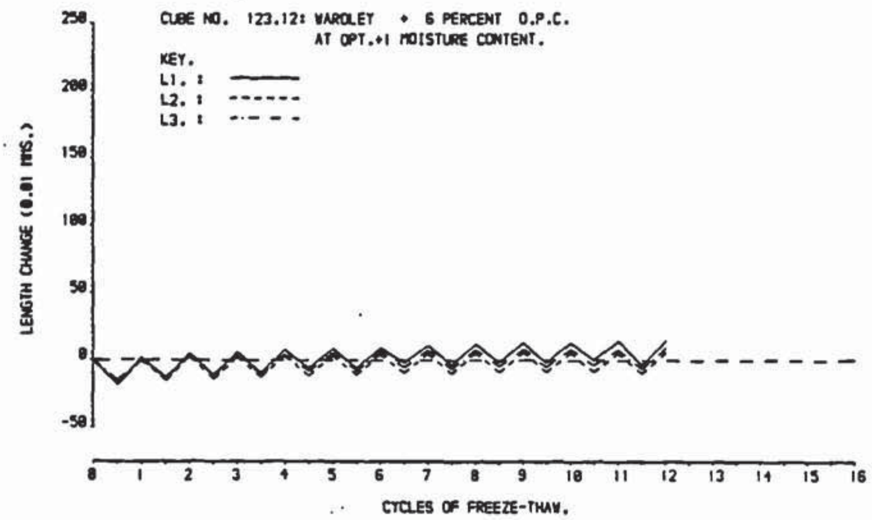


Fig 8.22 Effect of Moisture Content upon Resistance to Freezing and Thawing: Hem Heath





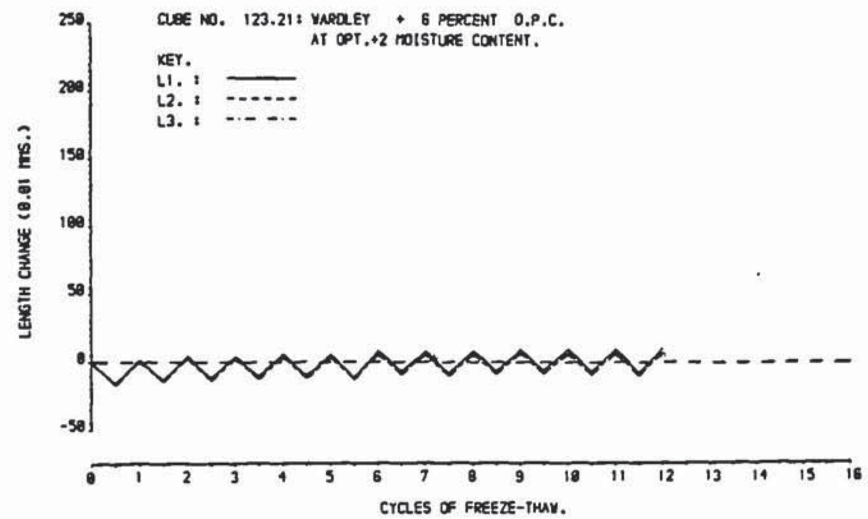
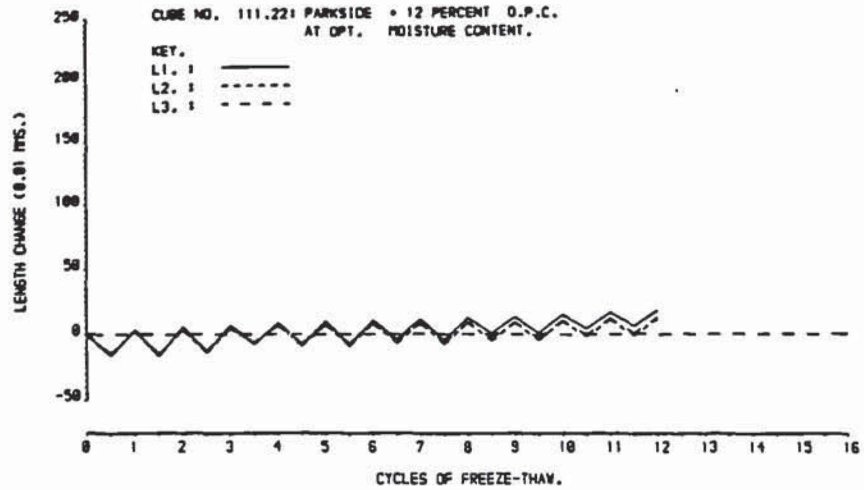
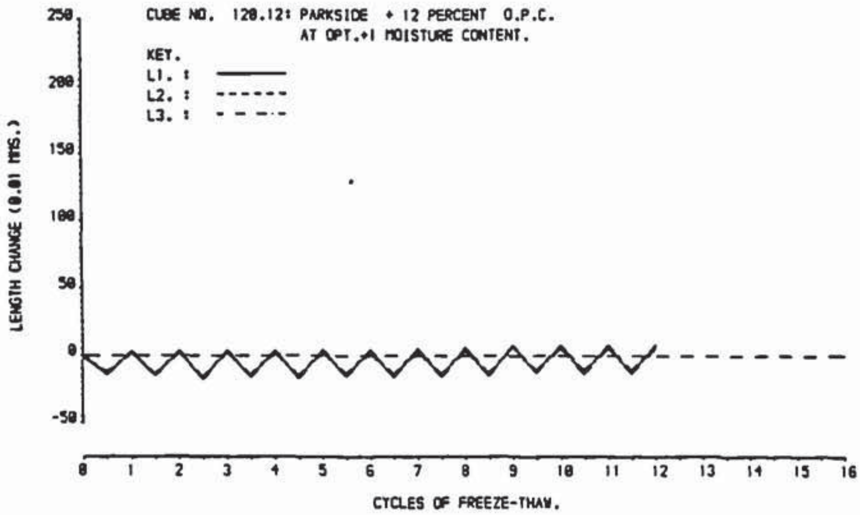


Fig 8.23 Effect of Moisture Content upon Resistance to Freezing and Thawing: Wardley





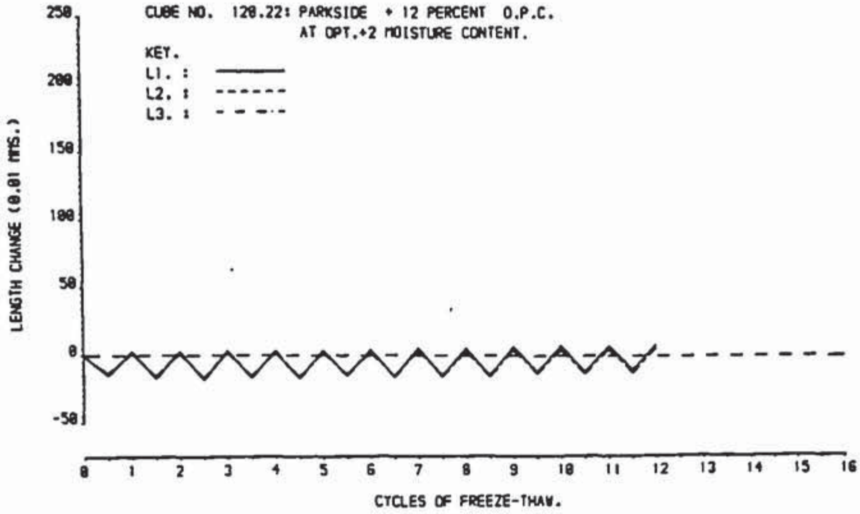
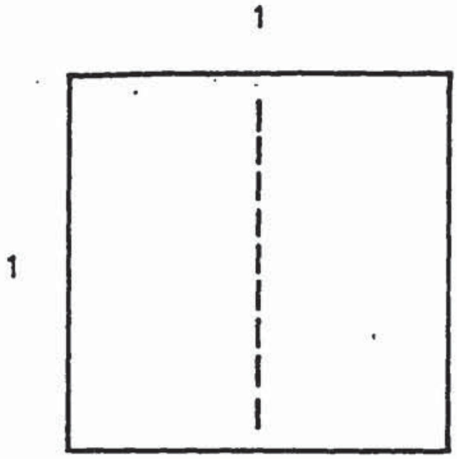


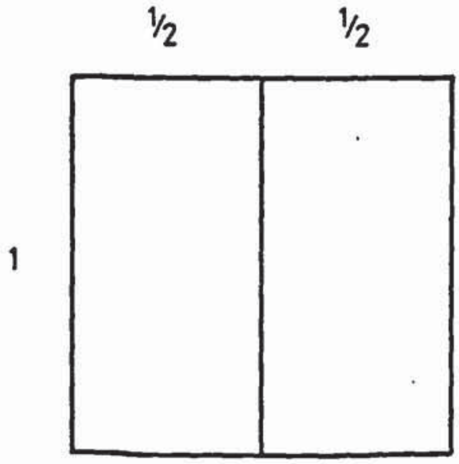
Fig 8.24 Effect of Moisture Content upon Resistance to Freezing and Thawing: Parkside



Snowdown Specimen cured
at constant moisture content
for 7 days

Single Cubical Specimen

Height : Width = 1



Snowdown Specimen cured
at constant moisture content
for 7 days followed by immersion

Discontinuity divides
specimen into 2

Height : Width = 2

Figure 8.25 Effect of Apparent Plane of Weakness on Effective Shape of Specimen

CHAPTER 9 DISCUSSION OF DURABILITY TESTS

9.1 Introduction

The results of the immersion and freeze-thaw tests, reported in Chapter 8, are discussed individually. Initially the changes in volume and moisture content are examined, and an attempt made to define the causes of these changes by considering certain physical and chemical characteristics of the raw material. As the discussion develops consideration is given to the effect on compressive strength. The resistance to the immersion and freeze-thaw tests is assessed in terms of the following parameters:

(a) Strength following test or absolute quality,

(b) Strength loss during test or change in quality,

(c) Volume change during test.

The results are compared with various published criterion and the discussion concludes by comparing the immersion and freeze-thaw resistance of the various mixtures.

9.2 Immersion Tests

9.2.1 <u>Specimen Stability.</u> All the minestone specimens were observed to expand during the first day of the immersion test. Furthermore this expansion was associated with moisture gain which completely saturated the specimens. The magnitude of this initial expansion was largely dependent upon the source of the material and, to a lesser degree, the cement content. It was also observed that the expansion of certain minestone specimens was influenced by initial moisture content. Subsequent behaviour in the test was also influenced by the source of the minestone. Expansion of the more resistant specimens ceased before the end of the test, with the initial expansion accounting for more than 70 per cent of the total recorded. These particular specimens gained very little additional moisture during the test. The less resistant specimens continued to expand in excess of twice the initial expansion, and were still expanding at the end of the test. This expansion was accompanied by a progressive moisture gain during the test. The expansion of the specimens produced from a laboratory blend of gravel sand and brickearth was negligible. Moisture gain was marginally less than that exhibited by the more resistant minestone specimens, and the specimens were not completely saturated at the end of the test.

The expansion of soil-cement specimens during immersion has usually been attributed to the presence of sulphates or expansive clay minerals (84). In the presence of excess moisture, sulphates can react with the constituents in cement to form the compound ettringite which, because it occupies a greater volume than any of its reactants (84), leads to expansion and, possibly, disruption of cemented materials. Minestones contain clay minerals and in the presence of lime, liberated during hydration of cement, these may also react with sulphates to produce ettringite (80). All the minestones contain sulphates which will influence the resistance of cement stabilised specimens to immersion and contribute to any expansion observed.

The role of expansive clay minerals in the expansion of specimens of cement bound minestone is unlikely to be significant. An investigation of the clay fraction of the minestones (Chapter 6) showed that the predominant minerals were kaolinite, illite, and chlorite. These minerals are not expansive and will not, therefore, swell significantly upon contact with water. Although expansive, mixed layered minerals were observed in a number of the minestones they only constitute a minor proportion of the mineralogy and, therefore, their influence on the behaviour of the clay fraction is considered to be negligible.

There is one additional factor which may significantly influence the expansion of minestone-cement specimens. Minestones contain a high proportion of mudstone which is susceptible to breakdown when immersed in water (4). In an investigation (4) of the breakdown of British coal measure rocks, the breakdown of immersed samples of desiccated mudstone was attributed to "air breakage", often referred to as slaking (95). The mechanism of air breakage was described as follows:-

> "...during dry periods evaporation from the surfaces of rock fragments promotes high suctions...which on rapid immersion in water becomes pressurised by capillary pressures developed in the outer pores. Failure of the minestone skeleton along the weakest plane ensues and an increased surface area is exposed to a further sequence of events."

Another mechanism, proposed by Nakano (96), attributes the breakdown during immersion to the adsorbtion of water onto the faces of clay particles. In the vicinity of the negatively charged faces of the clay particles the forces acting to orientate the water molecules are very strong. The molecules become organised such that the water is in a solid rather than liquid state, and is referred to as adsorbed water (95). In the presence of excess moisture additional water may be adsorbed and, therefore, the effective solid volume associated with each particle increases. This leads to inter-particle swelling which is reflected in an increase in the volume of the soil structure. It has been acknowledged that following initial breakdown of excavated coal measure rocks by air breakage, inter-particle swelling becomes progressively more important with time (4). Inter-particle swelling is not to be confused with the swelling of expansive clay minerals. It is thought that cement stabilised specimens which are produced at or above optimum moisture content are unlikely to be very susceptible to air breakage, though the consumption of water by the hydration process may create local "dry zone" within the specimen such that air breakage may occur. It is considered likely that inter-particle swelling, resulting from the adsorbtion of water, will contribute to the expansion of cement bound minestone specimens.

The major factors influencing the expansion of the immersed minestone specimens would, therefore, appear to be sulphate content, which will govern the extent of ettringite formation, and moisture gain, which may generate inter-particle swelling due to adsorbtion. It was also evident from the results of the tests that the grading of the minestone may influence the expansion of the cemented specimens, with the finer minestones, generally, exhibiting more expansion than the coarser minestones.

The increases in volume and mass recorded during tests on specimens, produced at the appropriate optimum moisture contents and containing 12 per cent cement (10 per cent in the case of Snowdown), are given in Table 9.1. The Table includes details relating to composition of the material finer than 75 um and together with the total and water soluble sulphate contents. Similar information, but for the specimens produced at initial moisture contents of between optimum and optimum plus 2 per cent, is given in Table 9.2.

The water soluble sulphate contents of the minestones are very similar with an average value of 0.14 per cent, however, their total sulphate contents cover a wider range. In figure 9.1 the increase in specimen volume at the end of the first day, is shown plotted against the total sulphate content. The expansion of those specimens produced from the coarser grained minestones - Snowdown-2, Hem Heath and Wardley - appear to increase with sulphate content in an approximately linear fashion. The sulphate content of Snowdown-1 was not determined but, by extrapolation, the volume increase of 0.49 per cent implies that the sulphate content was approximately 1.0 per cent.

Table 9.1 Summary of Volume and Mass Data Recorded During Immersion Tests on Specimens Produced at Optimum Moisture Content

Sample	Cement Content	Moisture Content	Fine Fractions (o/o)		Sulphate Content (o/o SO_3)		Volume Increase (o/o)		Mass Change (g)	
	(o/o)	(o/o)	<75 um	<2 um	Total	Water Soluble	First Day	Last Day	First Day	Last Day
Snowdown-1	10	8	6	1.5	-	-	0.49	1.41	-	62
Snowdown-2	10	8	6	2	.45	.13	0.26	0.88	27	36
Hem Heath	12	8	6	2	.31	.15	0.22	0.31	35	36
Wardley	12	7	7	3	.21	.14	0.18	0.23	44	49
Parkside	12	13	25	6	.21	.19	0.19	0.23	30	35
Blidworth	12	11	23	10	.20	.10	0.36	0.51	59	57
Bagworth	12	14	31	0	.32	.11	2.32	5.17	75	102
Peckfield	12	19	35	0	.37	.14	2.24	failed	118	-
Lab Blend	12	5	8	1	-	-	0.02	0.04	-	24

Table 9.2 Summary of Volume and Mass Data Recorded During Immersion Tests on Specimens Produced at Various Moisture Contents

Sample	Cement Content	Moisture Content	Fine Fractions (o/o)		Sulphate Content (o/o SO_3)		Volume Increase (o/o)		Mass Change (g)	
	(o/o)	(o/o)	<75 um	<2 um	Total	Water Soluble	First Day	Last Day	First Day	Last Day
Snowdown-2	10	Optimum					0.26	0.88	27	36
- do -	10	Opt + 1	6	2	.45	.13	0.17	0.42	19	27
- do -	10	Opt + 2					0.14	0.35	15	21
Hem Heath	8	Optimum					0.17	0.24	23	30
- do -	8	Opt + 1	6	2	.31	.15	0.16	0.19	26	29
- do -	8	Opt + 2					0.17	0.20	23	32
Wardley	6	Optimum					0.11	0.14	33	38
- do -	6	Opt + 1	7	3	.21	.14	0.07	0.09	23	26
- do -	6	Opt + 2					0.07	0.10	16	18
Parkside	12	Optimum					0.19	0.23	30	35
- do -	12	Opt + 1	25	6	.21	.19	0.13	0.11	31	37
- do -	12	Opt + 2					0.12	0.14	34	39

Total sulphate contents exceeding 1.0 per cent have been measured in samples obtained from the Snowdown stockpile (97). The expansion of Parkside specimens was almost identical to that of the Wardley specimens which had the same total sulphate content. However, the expansion of the Blidworth specimens, which had a similar sulphate content, was twice that of the Wardley specimens. Sherwood observed (80) that the degree of sulphate attack was proportional to the clay content. It can be seen from Table 9.1 that, whilst Parkside and Blidworth minestones contain a similar proportion of material finer than 75 um, the clay sized fraction of the Blidworth sample is almost twice that of the Parkside sample. The greater expansion of the Blidworth specimens is, therefore, possibly due to the higher clay content. The sulphate contents of the Bagworth and Peckfield minestones were similar to that of Hem Heath yet expansion of the specimens during the first 24 hours of immersion was ten times that of the Hem Heath specimens. The Bagworth and Peckfield minestones contained the highest proportion of fine material. Both contained more than 30 per cent finer than 75 um, however, apparently, neither material contained individual particles finer than 2 um. It was evident from the sedimentation analysis that the fine material was probably highly aggregated. The aggregation, or flocs, will contain clay particles and, therefore, the clay content of these materials is possibly higher than that of the other minestones. Whilst this would increase their susceptibility to sulphate-clay reactions it is considered unlikely that sulphates are responsible for the highly expansive behaviour of the Bagworth and Peckfield specimens.

In figure 9.2 the increase in specimen volume at the end of the test is shown plotted against sulphate content. The terminal expansion of the specimens produced from Hem Heath, Wardley, Parkside and Blidworth specimens were only marginally greater than those recorded at the end of the first day. However, in the case of the Snowdown-2 specimens, the final expansion was some three times that recorded at the end of the first day. The Snowdown-1 specimens also behaved in a similar manner as can be seen from Table 9.1. The nature of the expansion of Snowdown specimens is significantly different to that of other minestones which have only slightly lower sulphate contents. This would appear to suggest that, in the case of Snowdown specimens, sulphates are not the major cause of expansion although as mentioned earlier, sulphates could have influenced the Snowdown-1 specimens which, overall, exhibited much larger expansions than the Snowdown-2 specimens. In figure 9.3 the increase in volume at the end of the test is shown plotted against the moisture (mass) gain. There does not appear to be any simple relationship between the expansion and the moisture gain, but there are certain features in the data which are considered of interest. The Bagworth specimens had severely deteriorated by the end of the test and, therefore, the true moisture gain was masked by a significant loss of material. The amount of moisture taken up by these specimens was probably at least twice that gained by the other specimens and is reflected in a very high volume increase. The Peckfield specimens had taken up the highest amount of moisture at the end of the first day and, although they had similar volume increases to the Bagworth specimens, they totally deteriorated before the completion of the test. The data for specimens produced from the Hem Heath, Parkside and Blidworth minestones, and the laboratory blend, do appear to follow an approximately linear relationship between expansion and moisture gain. The expansion of the specimens produced from the two Snowdown samples also appears to be related to moisture gain, but not the same relationship as for the Hem Heath, Parkside and Blidworth specimens. From figure 9.3 it is clear that the amount of moisture taken up by the Snowdown-2 specimens was similar to that taken up by the Hem Heath and Parkside specimens, but the resulting expansion was considerably greater. Similarly the Snowdown-1 specimens expanded considerably more than the Blidworth specimens.

The highly expansive behaviour of the Bagworth and Peckfield specimens is attributed to inter-particle swelling. These specimens were very hygroscopic which may suggest that the potential for constituents in the material, to adsorb water is very high. The composition of the fine fraction of both these materials suggests that the clay particles were aggregated and so formed flocs. It is considered possible that, when the specimens were immersed, water was adsorbed onto the faces of the clay particles causing the flocs to swell. The aggregated flocs constitute a significant proportion of the whole sample and, therefore, the swelling flocs have a considerable influence on the volume of the specimen.

Before discussing the role of inter-particle swelling in the expansion of specimens produced from the other minestones, it is convenient to consider the effect of the initial moisture content. Figure 9.4 shows the effect of initial moisture content on the behaviour of four cement bound minestones subjected to the immersion test. It can be seen that the amount of moisture gained by the Snowdown-2 specimens was progressively reduced as the initial moisture content was raised. Furthermore, at 1 per cent above optimum the increase in volume, was more than halved. The amount of moisture gained by the Wardley specimens was also progressively reduced, however, the volume increase was reduced only marginally, although it remained the least susceptible to swelling of all the minestones. Increasing the moisture content of Hem Heath and Parkside specimens appeared to have no effect on the moisture gains, although the volume increases were reduced slightly.

The reduction in the amount of moisture gained by the Snowdown-2 and Wardley specimens is attributed to a reduction in the amount of water adsorbed onto the clay particles. Increasing the initial moisture content provided additional water for adsorption prior to compaction of the specimen. The potential for further adsorption during the immersion test was reduced and consequently the component of expansion resulting from inter-particle swelling was also reduced. The expansion of Snowdown-2 specimens appears to be particularly sensitive to initial moisture content. It is concluded that specimens produced at or below optimum moisture content are likely to be very susceptible to inter-particle swelling. It is believed that this sensitivity may explain the difference between the behaviour of Snowdown-1 and Snowdown-2 specimens. Clearly, only a small difference in the moisture regime of two samples would be sufficient to produce wide differences in their resistance to immersion. The expansion of the Hem Heath, Wardley and Parkside specimens was less sensitive to initial moisture content and it is, therefore, concluded that, within the moisture range of optimum to optimum plus 2 per cent, the component of expansion due to inter-particle swelling is probably negligible.

The fissure which occurred at the compaction plane of some of the immersed specimens, may also be attributed to the susceptibility of certain cement bound minestones to inter-particle swelling. In order to ensure adequate bond between the compaction layers, the upper surface of the lower layer was scarified. The action of scarifying the minestone fractures the larger, weak particles exposing new surfaces which are thought to be particularly susceptible to adsorbtion. Thus, when the specimens are immersed, the expansion forces generated at the compaction plane are greater than elsewhere in the specimen, resulting in the formation of a fissure along the compaction plane. The fact that Snowdown-2 specimens produced above optimum moisture content did not develop a fissure can be attributed to increased workability which eased the task of scarifying and, therefore, resulted in fewer fractured particles.

In figure 9.5 the total expansion of the specimen produced at optimum plus 2 per cent is shown plotted against the total sulphate content, also shown is the behaviour of corresponding specimens produced at optimum moisture content (the dotted line in the figure). It would appear that, at optimum plus 2 per cent, the inter-particle swelling is significantly reduced and the expansion is governed mainly by sulphate content.

9.2.2 Compressive Strength. The disruptive effect of immersion on the compressive strength of cemented minestone, produced at optimum moisture content, is illustrated in figure 9.6. The figure compares the strength at the end of the immersion test with the 7 day strength, immediately before immersion. With the exception of the Hem Heath specimens, compressive strength was reduced during the immersion period. The figure also shows that for any particular mixture, the magnitude of the strength loss was approximately constant over the range of cement content used.

The resistance to immersion is normally expressed in terms of the Resistance Index (R.I.), defined (69) as the ratio of the compressive strength following immersion to the strength of a similar specimen cured at constant moisture content for 14 days. It is usually expressed as a percentage and it has been suggested (70) that for satisfactory performance the R.I. should exceed 80 per cent. The R.I.'s of the various cemented minestones are shown plotted against the 7 day compressive strength in figure 9.7. The figure illustrates, again, that the resistance to immersion is largely dependent upon the source of the minestone. In general the R.I. increased with 7 day compressive strength, however, the only specimens to exceed the 80 per cent criterion were those produced from the Hem Heath and Parkside minestones. The Department of Transport specifies a minimum 7 day strength of 3.5 N/mm^2 (9). It can be seen from figure 9.7 that merely satisfying this requirement did not guarantee adequate resistance to immersion. Indeed it would appear that the resistance to immersion is not necessarily governed by absolute compressive strength.

The compressive strength of concrete specimens cured at constant moisture content and tested in a dry condition may be as much as 25 per cent higher than that of similar specimens which have been soaked prior to testing (98). The higher strength has been attributed (99) to:

(a) higher density of the dry cement paste,
(b) initial tensile stresses in the paste due to shrinkage being locally restrained by the (coarse) aggregate,
(c) possible development of hydrostatic pressures in the saturated paste.

The cement content of concrete is higher than that used in minestone-cement mixtures and, in addition, the restraint provided by the weaker minestone aggregate would generate lower tensile stresses in the paste. It would, therefore, seem reasonable to assume that a reduction in the strength of immersed minestone specimens of some 5-10 per cent may be attributed to these phenomena. Greater strength losses are almost certainly due to the additional disruption caused by expansion of the specimen and, therefore, the resistance index is largely governed by volume increase. The relationship between resistance index and volume increase is shown in figure 9.8. It can be seen that the R.I.'s of those specimens which underwent volume increases greater than 0.3 per cent were less than the recommended minimum value of 80 per cent (70). However, the volume increase of those specimens which did exceed this value was less than 0.3 per cent. A volume increase of 0.3 per cent is equivalent to a linear expansion of 0.1 per cent. This appears to agree with Packard and Chapman (73) who suggested that expansion during the ASTM freeze-thaw test (57) should not exceed 0.1 per cent. The specimens produced from Wardley minestone increased in volume by approximately 0.2 per cent, ie. less than 0.1 per cent linear expansion, however, the corresponding Resistance Indices were marginally less than 80 per cent.

The effect of the initial moisture content on the strength retained following the immersion test can be seen from figure 9.9. The strength of the Snowdown-2 specimens increased by approximately 40 per cent when the initial moisture content was raised above optimum. At optimum plus 2 per cent their retained strengths were 50 to 100 per cent greater than those of any of the other minestone specimens. Increasing the initial moisture content of the Hem Heath and Wardley specimens had only a negligible effect on the retained strength, whilst the strength of the Parkside specimens appeared to be reduced marginally. By comparing figures 9.10 and 9.4, it is clear that the effect of initial moisture content on retained strength mirrors its effect on the volume increase.

The effect of initial moisture content on the R.I. is shown in figure 9.10. Despite the comparatively high strength retained by Snowdown-2 specimens, produced above optimum, the R.I. increased only marginally. This, of course, is due to the fact that the 14 day strength of Snowdown-2 specimens is also increased when the initial moisture content is raised above optimum. At optimum plus 2 per cent the R.I. was 65 per cent, 15 percentage points below the recommended minimum. The R.I. of Wardley specimens increased by 13 percentage points, and exceeded the recommended minimum when the initial moisture content was raised above optimum. The effect of initial moisture content on the R.I. of Hem Heath specimens was negligible and appears to correlate with the effect on moisture gain and volume increase shown in figure 9.4. The R.I. of Parkside specimens was reduced by more than 10 percentage points when the initial moisture content was raised. The effect of moisture content on the R.I. of Parkside specimens does not appear to correlate with the insignificant effects on moisture gain and volume increase. It is suspected that the relatively high R.I. of specimens produced at optimum may be unrepresentative and is due on anomalous result.

The strength retained following the immersion test is, presumably, a measure of the absolute quality of the material following 7 days immersion, whereas the R.I. is a measure of the change in quality during the test. It may be argued that, providing the causes of disruption are not continuing at the end of the test, it is the absolute quality rather than the change in quality that should be used to assess the suitability of a material for inclusion in pavement construction. The dimensional measurements, presented in Chapter 8, suggested that all the specimens produced at optimum plus 2 per cent had stopped expanding before the end of the test which suggests that disruption had ceased. It would, therefore, appear that, in terms of absolute quality, Snowdown-2 specimens, produced at optimum plus 2 per cent, were superior to any of the other minestone specimens.

It is concluded that the R.I. should not be used alone to determine the immersion resistance of cement bound minestone specimens, indeed of all such cemented materials. Dimensional and, possibly, moisture content measurements, made during the immersion test, will help to assess the degree and nature of the disruption.

9.3 Freeze-Thaw Tests

9.3.1 Specimen Stability. The freeze-thaw behaviour of cement bound minestone specimens, produced at the optimum moisture content, appeared to be largely dependent upon the minestone and, to a lesser degree, the cement content. The specimens contracted when frozen for the first time, however, on completion of the first complete cycle all had expanded above their original dimensions. This expansion was accompanied by a moisture gain. The behaviour during subsequent cycles varied considerably. Of those specimens completing all 12 cycles, the more resistant ones continued to contract when frozen and to expand when thawed. During the course of the test the amplitude of the cyclic changes in dimension diminished slightly and the thawed dimension was observed to increase marginally with each cycle. This net expansion was accompanied by a moisture gain of up to 2 per cent.

The least resistant specimens underwent considerable expansion during the test and absorbed more than 3 per cent additional moisture. The amplitude of their cyclic changes in dimension diminished rapidly and and by the end of the test many of these specimens were expanding when frozen and contracting when thawed. None of the specimens produced from Bagworth and Peckfield minestones completed the test. These specimens had expanded by more than 0.3 per cent at the end of the first cycle and it was evident that they had absorbed a considerable amount of moisture. By the third cycle they were expanding when frozen and contracting when thawed.

The behaviour during the first cycle, and the amount of moisture absorbed during the test, appeared to be almost independent of the cement content. However, expansion during the course of the test was reduced significantly when the cement content was raised. Higher cement contents appeared to be of particular benefit to the Hem Heath and Parkside specimens.

Freeze-thaw tests on four cement bound minestones showed that the stability of both the dimensions, and the moisture content, improved significantly when the moisture content was raised by 2 per cent above the optimum. All the specimens produced from a laboratory blend of gravel, sand and brickearth, proved to be very resistant to cycles of freezing and thawing. Apart from the cyclic changes, their dimensions, and moisture contents, remained almost constant throughout the test.

The coefficient of thermal expansion for soil-cement mixtures is approximately 10 umm/mm/^{o}C (73). During each cycle of the freeze-thaw test the temperature of the specimen is reduced from 21^{o}C to -23^{o}C and then, 24 hours later, allowed to return to 21^{o}C. The cyclic change in volume due to thermal effects is, therefore, approximately 0.13 per cent. Volume changes exceeding, or contrary to, those which can be explained by thermal effects, have been attributed to one or more of the following (73):

(a) re-distribution of moisture,

(b) excessive ice growth,

(c) absorption of additional moisture.

During the freezing of a soil-cement specimen unfrozen water, in the smaller pores of the fine fractions, migrates towards the ice forming in larger pores (73). This re-distribution of moisture, which is similar to that reported to occur in concrete (100), essentially dries the fine fraction causing shrinkage (44). For non-deteriorating specimens, undergoing the freeze-thaw test, the degree of shrinkage on freezing increases with clay content (73). With the onset of deterioration the tensile strength of the material is reduced and its ability to resist ice growth diminishes. As deterioration progresses, shrinkage during freezing is increasingly counteracted by excessive ice growth and at severe deterioration the frozen volume of the specimen exceeds the thawed volume. Packard and Chapman (73) considered the absorption of additional moisture to be only a secondary factor in the volume change behaviour of freeze-thaw specimens. However, they acknowledged that the absorption of water would make a specimen more susceptible to frost damage.

After observing the freeze-thaw behaviour of specimens produced from a variety of soil types, Packard and Chapman (73) concluded that deterioration was due to a combination of the following mechanisms:

(a) differential volume changes,

(b) hydraulic pressure,

(c) excessive ice growth.

Neville (44) reports that differential shrinkage induces stresses in concrete which, if developed rapidly enough, may result in surface cracking. Soil-cement exhibits greater freezing shrinkage than concrete and, furthermore, the various constituents of the soil are likely to have a wide variety of volume change characteristics. It is argued, therefore, that the differential volume change mechanism has a greater effect on soil-cement than on concrete (73). The freezing of water increases its volume by approximately 9 per cent (44). As ice forms in the larger pores of a specimen it places the excess water under a hydraulic pressure. The magnitude of this pressure is dependent upon the resistance to flow, ie. the permeability immediately surrounding the pore in which the ice is forming. Packard and Chapman (73) believed that initial weakening of soil-cement specimens was due to mechanisms such as differential volume changes and hydraulic pressures. During this stage the volume change pattern of the specimen is constant. If a sufficient degree of weakening occurs the forces produced by the formation of ice in the pores will cause dilation (44). This expansive component counteracts the thermal and drying shrinkage and hence the frozen dimensions of the specimen increases. As the test proceeds the influence of the ice growth mechanism becomes increasingly significant until, at severe deterioration, the length change pattern, and also deterioration, are governed almost entirely by the freezing and thawing of water contained in the specimens.

It was stated, previously, that Packard and Chapman (73) considered the absorption of moisture during the freeze-thaw test to be of only secondary importance and was not a primary cause of deterioration. However, the immersion tests, discussed in Section 9.2.1, showed that the minestone specimens underwent expansion which was associated with the absorption of additional moisture. This expansion was disruptive and, almost without exception, resulted in a reduction of the compressive strength during the course of the test. The expansion was attributed to:

(a) inter-particle swelling, due to the adsorbtion of water molecules onto the clay particles,

(b) ettringite, a highly expansive mineral formed when, in the presence of excess moisture, sulphates in the minestones react with constituents in cement.

Consequently, it is believed that the absorption of moisture is a significant factor influencing the volume change behaviour, and hence any deterioration of cement-bound minestone specimens undergoing freezing and thawing. It almost certainly contributes to the initial weakening described by Packard and Chapman (73).

The changes in the volume and mass of specimens recorded during the first and last cycles of the freeze-thaw test are given in Table 9.3. These data relate only to those specimens produced at optimum moisture content. The Table includes the change in frozen volume, defined as the difference between the frozen volumes on the first and twelfth cycles. Other information in the Table relates to cement content, initial moisture content, and composition of the fraction of raw material finer than 75 um. Similar data, but for specimens produced at initial

Table 9.3 Summary of Volume and Mass Data recorded during Freeze-Thaw Tests on Specimens Produced at Optimum Moisture Content

Sample	Cement Content	Moisture Content	Fine Fraction (o/o)		Volume Change (o/o)				Increase in Frozen Volume	Mass Change (g)			
	(o/o)	(o/o)	<75 um	<2 um	F	T	F	T	(o/o)	F	T	F	T
Snowdown-1	5				-.44	.50	-	-	-	ND	-	-	-
- do -	10	8	6	1.5	-.43	.29	3.78	3.72	4.21	ND	15	ND	67
- do -	15				-.35	.25	3.06	3.11	3.41	ND	15	ND	68
Snowdown-2	10	8	6	2	-.39	.20	1.31	1.65	1.70	- 6	10	27	32
Hem Heath	4				-.51	.25	2.21	2.47	2.72	- 6	17	22	34
- do -	8	8	6	2	-.67	.20	.54	1.01	1.21	- 5	14	19	30
- do -	12				-.45	.25	.42	.85	0.87	- 2	27	33	40
Wardley	6	7	7	3	-.51	.12	.96	1.17	1.47	- 4	20	23	31
- do -	12				-.35	.21	.55	.73	0.90	- 3	30	34	44
Parkside	6	13	25	6	-.60	.12	-	-	-	-11	7	-	-
- do -	12				-.47	.09	-.1	.37	0.37	-10	4	15	22
Blidworth	6	11	23	10	-.91	.42	-	-	-	-11	33	-	-
- do -	12				-.70	.21	2.05	2.07	2.75	- 9	47	55	63

ND = Not Determined

(continued...)

Table 9.3 (continued)

Sample	Cement Content	Moisture Content	Fine Fraction (°/o)		Volume Change (°/o)				Increase in Frozen Volume	Mass Change (g)			
	(°/o)	(°/o)	<75 um	<2 um	F	T	F	T	(°/o)	F	T	F	T
Bagworth	6	14	31	0	-	-	-	-	-	ND	-	-	-
- do -	12				-.65	3.05	-	-	-	- 8	98	-	-
Peckfield	6	19	35	0	-	-	-	-	-	-	-	-	-
- do -	12				-	-	-	-	-	-	-	-	-
Lab Blend	4				-.20	-.03	-.22	0	0	-10	- 4	-12	- 5
- do -	8	5	8	1	-.17	-.02	-.19	0	0	- 6	- 4	- 5	0
- do -	12				-.15	-.01	-.12	0	0	- 3	- 1	2	4

ND = Not Determined

Table 9.4 Summary of Volume and Mass Data recorded during Freeze-Thaw Tests on Specimens Produced at Various Moisture Contents

Sample	Cement Content	Moisture Content	Fine Fraction (°/o)		Volume Change (°/o)				Increase in Frozen Volume	Mass Change (g)			
	(°/o)	(°/o)	<75 um	<2 um	F	T	F	T	(°/o)	F	T	F	T
Snowdown-2		Optimum			-.39	.20	1.31	1.65	1.70	- 6	10	27	32
- do -	10	Opt + 1	6	2	-.38	.09	-.12	.27	.26	- 7	3	- 5	8
- do -		Opt + 2			-.37	.03	-.32	.06	.05	- 8	- 2	- 3	2
Hem Heath		Optimum			-.67	.20	.54	1.01	1.21	- 5	14	19	30
- do -	8	Opt + 1	6	2	-.73	.15	-.04	.56	.69	- 7	15	12	19
- do -		Opt + 2			-.70	.15	.15	.50	.55	- 6	12	8	15
Wardley		Optimum			-.51	.12	.96	1.17	1.47	- 4	20	23	31
- do -	6	Opt + 1	7	3	-.55	.02	-.19	.32	.36	- 7	7	3	14
- do -		Opt + 2			-.47	.04	-.28	.23	.19	- 9	4	- 6	4
Parkside		Optimum			-.47	.09	-.10	.37	.37	-10	4	15	22
- do -	12	Opt + 1	25	6	-.39	.09	-.38	.23	.01	-11	8	11	20
- do -		Opt + 2			-.39	.10	-.25	.26	.14	ND	4	6	15

ND = Not Determined

moisture contents of between the optimum and the optimum plus 2 per cent are given in Table 9.4.

At the end of the first freeze period the contraction of the minestone specimens ranged from 0.35 to 0.91 per cent, and these are between 2.5 and 7 times greater than can solely be explained by thermal effects. The contraction of the specimens produced from the laboratory blend was 0.2 per cent and is almost entirely due to thermal effects. In figure 9.11 the initial contraction of the various mixtures tested has been plotted against (a) silt content, and, (b) clay content. Figure 9.11(a) suggests that there is no simple correlation between the initial contraction and the silt content. However, figure 9.11(b) clearly shows that, for the majority of the mixtures, the initial contraction increased with clay (<2 um) content. This apparent correlation is consistent with the generally accepted correlation between freezing contraction and the size of the fine fraction (44,73). Furthermore, it suggests that the clay content, rather than the silt content, governs the initial freezing contraction of these particular mixtures. This seems reasonable since, with the migration of moisture from the fine fraction to the ice forming in the larger pores, water will be removed from the adsorbed layer on the surface of the clay particles. The removal of adsorbed water reduces the volume of the clay particles which will be reflected in the volume of the specimen.

It would appear from figure 9.11(b) that the initial contraction of the Bagworth specimens was not governed by clay content. However, the fine fraction of both the Bagworth and Peckfield minestones was observed to be highly aggragated. It is suspected that the aggregations, or flocs, contain clay particles and, therefore, the true clay content probably exceeds that of the other samples. During the programme of immersion tests the Bagworth and Peckfield specimens were considered to be particularly susceptible to inter-particle swelling caused by the adsorbtion of water onto the surfaces of clay particles present in the flocs. It is thought that the Bagworth and Peckfield specimens are similarly susceptible to the removal of adsorbed water and, therefore, subject to considerable freezing shrinkage.

It has been suggested that the resistance of a soil-cement specimen to freezing and thawing may be gauged by the change in frozen dimensions (73). Furthermore, Packard and Chapman (73) found that the following criterion correlated with the established weight loss criterion (72) used to assess the performance of specimens subjected to the ASTMS freeze-thaw test (57).

> "Twelfth cycle frozen length not exceeding first cycle frozen length by 0.005 in."

This is equivalent to an increase in the frozen dimensions of approximately 0.1 per cent or, in terms of volume, 0.3 per cent.

Figure 9.12 shows the effect of cement content on the increase in the frozen volume of specimens produced at optimum moisture content. During the course of the test the frozen volume of the majority of minestone specimens increased by considerably more than 0.3 per cent. Increasing the cement content improved dimensional stability but, with the exception of Parkside specimens, the increases in frozen volume were still more than twice the recommended maximum. Of the specimens completing the test, those produced from Snowdown-1 appeared to be the least resistant in terms of their volume increase. At 15 per cent cement content the frozen volume increased by more than 3 per cent. Specimens produced from Snowdown-2, however, appeared to be more resistant with the frozen volume increasing by less than 2 per cent with only 10 per cent cement. Increasing the cement content of Hem Heath specimens from 4 to 8 per cent reduced the increase in frozen volume by over 50 per cent. However, increasing the cement content to 12 per cent did not achieve any significant improvement. The resistance of Wardley specimens appeared to improve marginally when the cement content was raised from 6 to 12 per cent. The increase in frozen volume was reduced by half a percentage point to less than 1 per cent.

The resistance of the Parkside specimens increased dramatically when the cement content was raised. All the specimens containing 6 per cent cement deteriorated before completing the test. However, at 12 per cent cement content the frozen volume increased by less than 0.4 per cent. Increasing the cement content had a similar effect on Blidworth specimens, although those with 12 per cent cement still exhibited an increase in frozen volume of approximately 2 per cent. This does, however, suggest that they were more resistant than any of the specimens produced with Snowdown-1 minestone sample. All the specimens produced from the laboratory blend satisfied the acceptance criterion relating to frozen volume.

The effect of the initial moisture content on the increase in frozen volume of specimens produced from four mixtures is shown in figure 9.13. The dimensional stability of many of the specimens was improved significantly when the initial moisture was raised above optimum. The Snowdown-2 and Wardley specimens showed the greatest improvement, with the increases in frozen volume being reduced by more than 1 percentage point to less than 0.2 per cent. The Snowdown-2 specimens, produced at the optimum plus 2 per cent, displayed the highest freeze-thaw resistance of all the minestone specimens tested. The increase in the frozen volume of Parkside specimens was reduced from 0.37 per cent, at optimum, to less than 0.2 per cent, at optimum plus 2 per cent. The Snowdown-2, Wardley, and Parkside specimens, produced at optimum plus 2 per cent, satisfied the frozen volume criterion (73). Although the increase in the frozen volume of Hem Heath specimens was halved, when the initial moisture content was raised by 2 per cent, it was, at 0.55 per cent, almost twice the recommended criterion.

Before attempting to define the mechanisms which govern the freeze-thaw behaviour of cement bound minestone, it is convenient to consider the effect of cement content, and initial moisture content, upon:

a) the initial contraction during the first freeze period,

b) the moisture gained during the test.

The effects of cement content upon initial contraction, and moisture gain, are shown in figures 9.14 and 9.15 respectively. Similarly, the effect of initial moisture content are shown in figures 9.16 and 9.17.

With the exception of one result, that for the Hem Heath specimens containing 8 per cent cement, it is clear from figure 9.14 that increasing the cement content reduced the initial contraction by between 0.05 and 0.2 percentage points. These reductions, which represent only a small part of the contraction attributed to drying shrinkage, increased with the proportion of fine material in the raw minestone. It is concluded that, as the strength of the specimens increased with cement content, so too did their ability to resist shrinkage strains, generated by the loss of adsorbed water from the clay particles. Figure 9.16 shows that the initial contraction, during the first freeze period, appears to be independent of initial moisture content within the range optimum to optimum plus 2 per cent. This suggests that the improved resistance of specimens produced above optimum, as shown in figure 9.13, can not be attributed to phenomena associated with initial contraction.

The amount of moisture absorbed by the minestone specimens produced at optimum moisture content appeared to be governed largely by the source of the material. The least resistant specimens, Snowdown-1, Blidworth and Bagworth, were characterised by high moisture gains exceeding 60 grammes. Peckfield specimens, which deteriorated rapidly, also absorbed a considerable amount of moisture. The result for Snowdown-1 is particularly interesting since the amount of moisture gained was approximately twice that gained by Snowdown-2, and other similarly graded minestones. The mass of the specimens produced from the laboratory blend remained almost constant throughout the test. Raising the initial moisture content appears to have had a particularly significant effect on the amount of moisture gained by specimens produced from certain minestone-cement mixtures as can be seen from figure 9.17. The Snowdown and Wardley specimens, produced at the optimum moisture content, absorbed at least 30 grammes of additional moisture. When the initial moisture content was raised 2 per cent the mass of similar specimens remained within 4 grammes of the original weight throughout the test. Increasing the initial moisture content of the Hem Heath specimens had a negligible effect on the amount of moisture absorbed during the first thaw period, however, subsequent gains were reduced by more than 80 per cent. The amount of moisture absorbed by the Parkside specimens did not appear to be influenced by initial moisture content.

It is apparent that, for these specimens, the degree of resistance to cyclic freezing and thawing is very dependent on the amount of moisture absorbed during the thaw periods.

By absorbing moisture the specimens became susceptible to the same phenomena which are thought to be responsible for the expansion of similar specimens subjected to the immersion test, particularly inter-particle swelling. However, the extra water would also influence the ice formation during freezing, which could further contribute to deterioration. It is considered, however, that the freeze-thaw resistance of the majority of the minestone specimens was governed by the degree of susceptibility to these expansive phenomena, rather than to differential volume change or to the hydraulic pressure mechanisms.

The results of the immersion tests on Bagworth and Peckfield specimens suggested that they were particularly susceptible to inter-particle swelling. When subjected to the freeze-thaw test similar specimens absorbed a great deal of moisture during the first thaw period. This is largely attributed to a demand from the clay particles to satisfy their adsorbtion requirements. The resulting inter-particle swelling is reflected in a net increase in volume at the end of the first thaw period. The increased moisture content also raises the degree of saturation and therefore, during the subsequent freeze period the hydraulic pressures generated by ice growth is also increased. As the test proceeds the specimens deteriorate rapidly due to:

(a) swelling pressure, generated by the adsorption of water,

(b) increasing hydraulic pressure and, eventually, heave due to increasingly excessive ice growth.

The specimens had completely deteriorated before completing the test.

The immersion resistance of the Blidworth specimens was similar to that of the Snowdown-2 specimens. The expansion of Blidworth specimens was thought to be influenced mainly by the moderately high clay content of the raw minestone. Although the freeze-thaw resistance of the Blidworth specimens was similar to that of the Snowdown-1 specimens, the Blidworth specimens had absorbed almost three times as much moisture at the end of the first thaw period. This higher absorption is thought to be associated with the mechanical properties of the raw minestone, which resulted in particularly high capillary suctions in the Blidworth specimens. The dramatic increase in moisture content results in higher hydraulic pressures during the freezing periods. The behaviour of the specimens during subsequent cycles is governed almost entirely by this excessive ice growth. The amplitude of the cyclic changes in volume diminished rapidly and specimens completing the test were severely deteriorated.

Immersion tests on Snowdown-2 specimens suggested that their susceptibility to inter-particle swelling was particularly sensitive to initial moisture content. Raising the initial moisture content 2 per cent halved the amount of moisture absorbed and, apparently, eliminated the component of expansion due to inter-particle swelling. Wardley specimens were also sensitive to initial moisture content. The amount of water absorbed during the immersion test was halved when the initial moisture content was raised 2 per cent. However, the effect on the accompanying expansion was minimal. The terminal expansion of both Snowdown-2 and Wardley specimens produced at optimum plus 2 per cent appeared to be largely dependent upon the sulphate content.

The freeze-thaw resistance of Snowdown-2 and Wardley specimens was also found to be sensitive to initial moisture content. At optimum, the specimens had absorbed moisture at the end of the first thaw period and had expanded above their original volumes. The expansion is attributed mainly to inter-particle swelling. Both the amount of moisture absorbed and the volume of the specimens increased with each cycle. With the exception of specimens produced from Snowdown-1 containing 5 per cent cement, all the specimens completed the test. However, it was apparent that the specimens were deteriorating progressively. During the course of the test the amplitude of the cyclic changes in volume was progressively reduced. This is interpreted as an indication that excessive ice growth was contributing to the deterioration.

The Snowdown-2 specimens produced at optimum plus 2 per cent absorbed very little moisture during the freeze-thaw test and underwent only very limited expansion. It would appear that the adsorption requirement of the clay particles was satisfied at the mixing stage and, therefore, the specimens did not absorb additional moisture during the thaw periods. In the absence of excess moisture, not only is inter-particle swelling eliminated but the formation of entringite is inhibited. The amplitude of the cyclic changes in volume remained constant throughout these tests. Clearly, these volume changes were governed entirely by the cyclic thermal effects and cyclic redistribution of moisture in the specimens. Since there was no evidence to suggest the onset of deterioration, the specimen must also have satisfactorily resisted the hydraulic pressure generated by ice growth. The Snowdown-2 specimens produced at optimum plus 2 per cent were, apparently, completely resistant to the freeze-thaw test.

Increasing the initial moisture content of the Wardley specimens by 2 per cent almost completely eliminated expansion during the test. The amount of moisture absorbed was marginally higher than by the Snowdown-2 specimens. The significant reduction in expansion is again attributed to the elimination of any inter-particle swelling and an inhibition of the reactions which produce entringite. The cyclic changes in volume remained constant throughout the test and there was no evidence of serious deterioration. The Wardley specimens produced at optimum plus 2 per cent also appear to be resistant to the freeze-thaw test.

The expansion of Hem Heath specimens during the immersion test appeared to be related to sulphate content and, therefore, due to the formation of entringite. The magnitude of the expansion was independent of the initial moisture content which suggests that inter-particle swelling was negligible. In the freeze-thaw test, both absorption and expansion, at the end of the first thaw period were independent of initial moisture content. However, subsequent absorption and expansion were reduced as the moisture content was raised. The expansion is attributed mainly to phenomena associated with excess moisture. The amplitude of the cyclic changes in volume was greater than that of any of the more resistant minestone specimens. It is, therefore, suspected that the Hem Heath specimens were particularly susceptible to differential volume changes. The amplitude of the cyclic changes decreased steadily during the test indicating excessive ice growth, and, perhaps, signalling the onset of deterioration.

The behaviour of the Parkside specimens during the immersion test was similar to that of the Hem Heath specimens. Expansion was independent of the initial moisture content and appeared to be related to sulphate content. In the freeze-thaw test the specimens containing 12 per cent cement, and produced at the optimum moisture content, appeared to be the most resistant, in terms of moisture and volume stability, of all the cement bound minestone specimens. Expansion during the test was reduced very slightly when the initial moisture content was raised but, otherwise, the behaviour appeared to be almost independent of the initial moisture content. The amplitude of the cyclic changes remained almost constant throughout the test. It would appear that Parkside specimens, containing 12 per cent cement are capable of resisting the various mechanical and chemical mechanisms operating during the course of the freeze-thaw test.

9.3.2 <u>Compressive Strength.</u> It has been suggested that during the 12 cycles of the freeze-thaw test there is an opportunity for a strength gain equivalent to about 10 days additional curing (73). The effect of freeze-thaw cycling can be assessed from figure 9.18, where the terminal compressive strength is plotted against the corresponding 7 day value of similar specimens prior to testing. This graph includes only data for specimens produced at the optimum moisture content. It shows that the disruptive effects of freezing and thawing were sufficient to counteract any benefits from the additional curing and, in most cases, the freeze-thaw cycling produced a reduction in strength. Only the Hem Heath specimens, containing 8 and 12 per cent cement, maintained their 7 day strength at the end of the test. The strength of the specimens produced from the laboratory blend increased by between 10 and 26 per cent during the freeze-thaw test and confirms that there is an opportunity for curing to continue.

A convenient method of expressing the freeze-thaw resistance, in terms of strength change, is the Freeze-Thaw Index (F.T.I.). This is defined as the ratio, expressed as a percentage of the compressive strength following the freeze-thaw test to the strength of similar specimens cured at constant moisture content for 7 days. In figure 9.19 the F.T.I.'s of the various minestone cement mixtures are shown plotted against the 7 day compressive strength. Although the F.T.I. increased with compressive strength as the cement content was raised, there does not appear to be any simple correlation which could be applied, universally, to such specimens in general.

The current strength requirement of the Department of Transport specification (9) which requires a minimum 7 day value of 3.5 N/mm^2, was derived from an earlier criterion (94) which was believed (60) to correlate with the established weight loss criteria applied to the American freeze-thaw tests (56,57). Furthermore, the following criteria relating to F.T.I. have been correlated (73) with weight loss criteria:

(i) F.T.I. $<$ 90°/o : non-resistant

(ii) 90°/o $<$ F.T.I. $<$ 145°/o : may or may not be resistant

(iii) F.T.I. $>$ 145°/o : resistant

Both criteria have been superimposed over the data in figure 9.19. It can be seen that, whilst many of the cement bound minestone specimens exceed the recommended 7 day strength, the majority have clearly been severely disrupted by the freeze-thaw test. Although the specimens produced from Hem Heath minestone, containing 8 and 12 per cent cement, appear to satisfy both criteria, their F.T.I.'s, 94 and 105 per cent respectively, do not prove conclusively that they have adequate resistance. It is concluded that the freeze-thaw resistance of cement bound minestone cannot be assessed by the 7 day compressive strength.

In figure 9.20 the F.T.I. is plotted against the increase in frozen volume during the test. The acceptance criteria for both F.T.I. and frozen volume, recommended by Packard and Chapman (73), have been superimposed on the data. It is apparent that those specimens which exhibited the higher volume increases also had the greatest loss in strength during the test. None of the minestone-cement specimens, produced at optimum moisture content, satisfied both acceptance criteria. Although Hem Heath specimens exceed the recommended minimum value for F.T.I. their frozen volume had increased by at least twice the recommended 0.3 per cent. On the other hand, the frozen volume of the Parkside specimens, containing 12 per cent cement had increased by only 0.37 per cent yet the F.T.I. was, at 78 per cent, 12 percentage points below the recommended minimum.

Tests on four cement bound minestones suggested that the stability of both the dimensions and moisture content of the specimens was improved when the initial moisture content was raised. The degree of improvement varied according to the source of the minestone. The Snowdown-2 and Wardley specimens appeared to benefit most, whilst Hem Heath and Parkside specimens exhibited only marginal improvements. The effect of the improved dimensional stability on F.T.I. is shown in figure 9.21. The F.T.I. of Snowdown-2 and Wardley specimens were increased significantly as the initial moisture content was raised. The strength of Snowdown-2 specimens produced at optimum + 1 per cent increased by 10 per cent during the freeze-thaw test. This result confirms that there is an opportunity for cement bound minestone to gain strength during the freeze-thaw test. The fact that the majority lost strength indicates that the nature of the mechanisms operating on cemented minestone during the freeze-thaw test is such as to more than counter this potential strength gain. The effect on the F.T.I. of raising the initial moisture content of Hem Heath and Parkside specimens was almost insignificant. The results for Hem Heath suggest, if anything, a reduction in the F.T.I. as the initial moisture content was raised.

The Snowdown-2 specimens, produced above optimum moisture content, satisfy both criteria pertaining to F.T.I. and frozen volume. The Wardley and Parkside specimens produced at the higher moisture content satisfy the criterion for frozen volume but have F.T.I.'s less than 90 per cent. The Hem Heath specimens, produced above optimum moisture content, failed to satisfy either of these criteria.

The F.T.I. criteria imply that the index may be used alone to identify the most resistant specimens, having an F.T.I. of at least 145 per cent, and the non-resistant, having an F.T.I. less than 90 per cent. When used to determine the resistance of cemented minestone the F.T.I. may be used to identify non-resistant specimens, however, it does not appear to correlate with the dimensional stability of the specimens as gauged by the change in frozen volume. It is suggested, therefore, that the F.T.I.

does not provide a sensitive measure of the freeze-thaw resistance of cement bound minestone specimens. The data presented in figure 9.21 also suggest that minestone specimens exhibiting an acceptable degree of dimensional stability have an F.T.I. in excess of approximately 80 per cent. This appears to agree with the F.T.I. criteria which was often applied (70) to specimens subjected to the British Freeze-Thaw Test, deleted from BS 1924 in 1967 (68).

From the foregoing discussion it could appear that the strength of cement bound minestone may be reduced by up to 20 per cent during the freeze-thaw test without any evidence of serious deterioration. Although this strength loss may be acceptable, the material must retain adequate strength for the purpose envisaged, ie. sub-base and, possibly, road-base construction. Thompson and Dempsey (76) acknowledged that a degree of strength loss during the freeze-thaw test was acceptable providing the strength retained was adequate for the intended end use. They proposed that a "minimum tolerable strength" should be established and that the strength retained at the end of the freeze-thaw test should exceed this value. Both the test method and the criteria established were tailored to the prevailing climatic conditions in various regions of the U.S.A (76). Although a minimum tolerable strength has not been established for pavement materials used in the U.K. it might possibly be argued that the strength retained by materials displaying adequate resistance to freezing and thawing should also exceed the minimum 7 day strength requirement of 3.5 N/mm2 (9).

In figure 9.22 the primary measure of resistance, considered to be the change in frozen volume, is shown plotted against strength following the test, the F.T.I. of each mixture is also shown. Only the most resistant minestone specimens, satisfying the volume change criterion and/or having an F.T.I. exceeding 80 per cent, are represented in the figure. The Snowdown-2 and Wardley specimens not only satisfied the volume change and F.T.I. criteria but their retained strength exceeded the 7 day requirement. The Parkside specimens exhibited a very high resistance to freezing and thawing, however, their 7 day strengths were lower than those of the Snowdown and Wardley specimens. During the test the strength was reduced to the minimum 7 day requirement. The strength retained by the Hem Heath specimens exceeded the minimum 7 day requirement, however, the change in frozen volume exceeded the recommended maximum. It is suspected, as was discussed in Section 9.3.1, that the Hem Heath specimens had begun to deteriorate. The retained strength does not provide a sensitive measure of the resistance of cemented minestones to freezing and thawing. However, the strength retained by minestone specimens which exhibit satisfactory resistance should also exceed the minimum 7 day strength requirement of 3.5 N/mm^2 (9).

9.4 Comparison of Immersion and Freeze-thaw Resistance

The various measures of resistance to the immersion and freeze-thaw tests, strength following test, R.I./F.T.I., and volume change, are compared in figures 9.23, 9.24 and 9.25 respectively.

9.4.1 <u>Strength Following Test.</u> When comparing the strength following test the difference between the curing regimes imposed upon the specimens by the immersion and freeze-thaw test must be taken into account. Notwithstanding the effects of immersion on curing, the immersion specimens are cured at constant temperature for 14 days. At the end of the freeze-thaw tests, the specimens are considered (71) to have cured for a period equivalent to approximately 17 days at constant temperature (20^{o}/oC). Replicate specimens displaying a very high level of resistance to both tests would, therefore, be expected to retain marginally higher strengths following the freeze-thaw test that following the immersion test. The strength retained at the end of each test are compared in figure 9.23, and it can be seen that, for the majority of cement bound minestones, the strength retained following the freeze-thaw test is equal to, or less than the strength retained following the immersion test. This would appear to suggest that the freeze-thaw test is a more severe test of the durability of most cement bound minestones. The Snowdown-2 specimens appear to be an exception, with the higher strengths being retained by the specimens subjected to the freeze-thaw test. It would appear that the resistance of these specimens to freezing and thawing is comparable, if not greater than, their resistance to immersion. The strength retained by the highly resistant specimens produced from the laboratory blend was highest at the end of the freeze-thaw test. This appears to be consistent with the curing regimes. Indeed the strength retained by the laboratory blend following the freeze-thaw test are some 11 to 35 per cent greater than those following the immersion test. For the Snowdown-2 specimens these increases are 4 to 13 per cent so that its relative performance, whilst better than the other cement bound minestones, is not as good as that achieved by the laboratory blend.

In figure 9.23, the Department of Transport's minimum 7 day strength requirement of 3.5 N/mm^2 (9) has been superimposed over the test data. At the end of both tests, the strength retained by Snowdown-2, Hem Heath, Wardley and laboratory blend specimens exceeded the 7 day minimum. Although the strength retained by the Snowdown-1 and Parkside specimens at the end of the immersion test exceeded the 7 day minimum, the strength retained following the freeze-thaw test was below the minimum value. The strength retained by the Blidworth, Bagworth and Peckfield specimens, at the end of both tests, was less than the 7 day minimum.

9.4.2 R.I. and F.T.I. A comparison of the R.I. and F.T.I. not only has to account for the different curing regimes but is also complicated by the differences in the methods of determining their respective values. R.I. is based on the 14 day strength of specimens cured, at constant moisture content, whereas the F.T.I. is based on specimens cured for only 7 days. Replicate specimens displaying a very high degree of resistance to both tests would, therefore, be expected to have a higher value for F.T.I. than for R.I. The values are compared in figure 9.24 which shows that the ratio between R.I. and F.T.I. varies with the individual minestone. The individual R.I's. obtained from Snowdown-1, Blidworth, and Bagworth minestones, are significantly higher than the corresponding F.T.I's. The R.I's. of specimens produced from Hem Heath and Wardley minestones, are similar to their F.T.I's. The results for Parkside specimens are somewhat erratic, but generally the F.T.I. is marginally higher than the appropriate R.I. The F.T.I. of the more resistant Snowdown-2 specimens is significantly higher than their R.I. The comparison between R.I. and F.T.I. again suggests that the freeze-thaw test is the more severe test for most cement bound minestones, the exception again being Snowdown-2. The F.T.I. of the laboratory blend was, in all cases, higher than their R.I. as was expected of such highly resistant specimens.

In figure 9.24, the recommended minimum values for the indices, 80 per cent for the R.I. (70) and 90 per cent for the F.T.I. (73), have been superimposed over the test data. It is clear that the only minestone specimens to come anywhere near satisfying both criteria were produced from Hem Heath and Wardley minestones. The R.I. and F.T.I. for these specimens were approximately equal to recommended minimum values. Whilst the Snowdown-2 specimens easily satisfied the F.T.I. criteria, their R.I's were below the recommended minimum value. Specimens produced from the other minestones failed to satisfy both criteria. The specimens produced from the laboratory blend easily satisfied both criteria.

9.4.3 Volume Change. The method of assessing the durability of soil-cement mixtures by measuring volume changes has been employed since research conducted in the U.S.A. in the 1930's (23). Packard (71) refined the method and later, Packard and Chapman (73) recommended a criterion which could be used to assess the performance in the ASTM Freeze-Thaw Test (57). In Section 9.2.2 it was suggested that this criteria may also be valid for specimens subjected to the immersion test. The volume changes recorded during the immersion and freeze-thaw tests are compared in figure 9.25. For the majority of cement bound minestones the volume change recorded during the freeze-thaw test is significantly greater than that recorded during the immersion test. This would appear to support the strength related data in figures 9.23 and 9.24 and confirms that the freeze-thaw test is the more severe test for many cement bound minestones. The exception is again seen to be the results for the more resistant Snowdown-2 specimens which underwent greater volume change during the immersion test. It is apparent that these specimens were more susceptible to immersion than to cyclic freezing and thawing.

The recommended maximum change in the frozen volume of a specimen undergoing the freeze-thaw test, and which is also thought to apply to volume increase during the immersion test, is 0.3 per cent. In figure 9.25, this criterion has been superimposed over the test data. In addition to the specimens produced from the laboratory blend, only the most resistant specimens produced from Wardley and Parkside minestones satisfy the criterion in both tests. The Snowdown-2 specimens satisfy the criterion in the freeze-thaw test, but expanded more than the recommended maximum in the immersion test.

In general these results are compatible with the relationships shown in figures 9.23 and 9.24, with those specimens which had low F.T.I's. and low compressive strengths generally exhibiting the largest volume changes. A similiar trend can be seen with the immersion test results. A close examination of the data reveals that the results for the Hem Heath specimens do not appear to fit this pattern, particularly with regard to the performance in the freeze-thaw test. The expansion of these specimens following immersion was below the 0.3 per cent criterion and the R.I. values were greater than 80 per cent. However, expansion during freeze-thaw testing exceeded the 0.3 per cent value but the F.T.I's. were close to the 90 per cent value. Thus, whilst the specimen did undergo expansion during the test, the strength results indicate that this was not sufficient to cause severe disruption of the specimen.

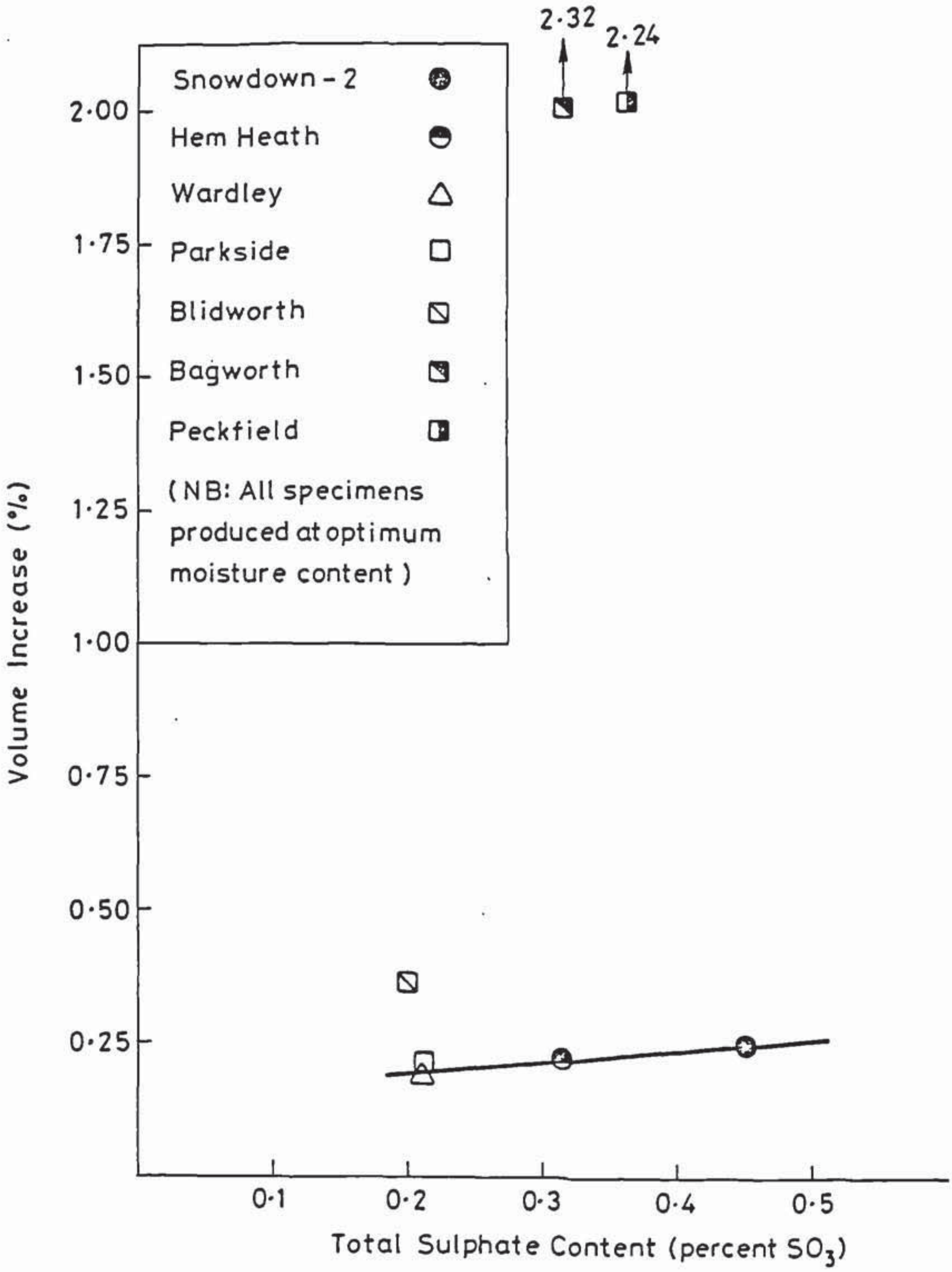


Figure 9.1 · Volume Increase after first day of Immersion Test plotted against Total Sulphate Content

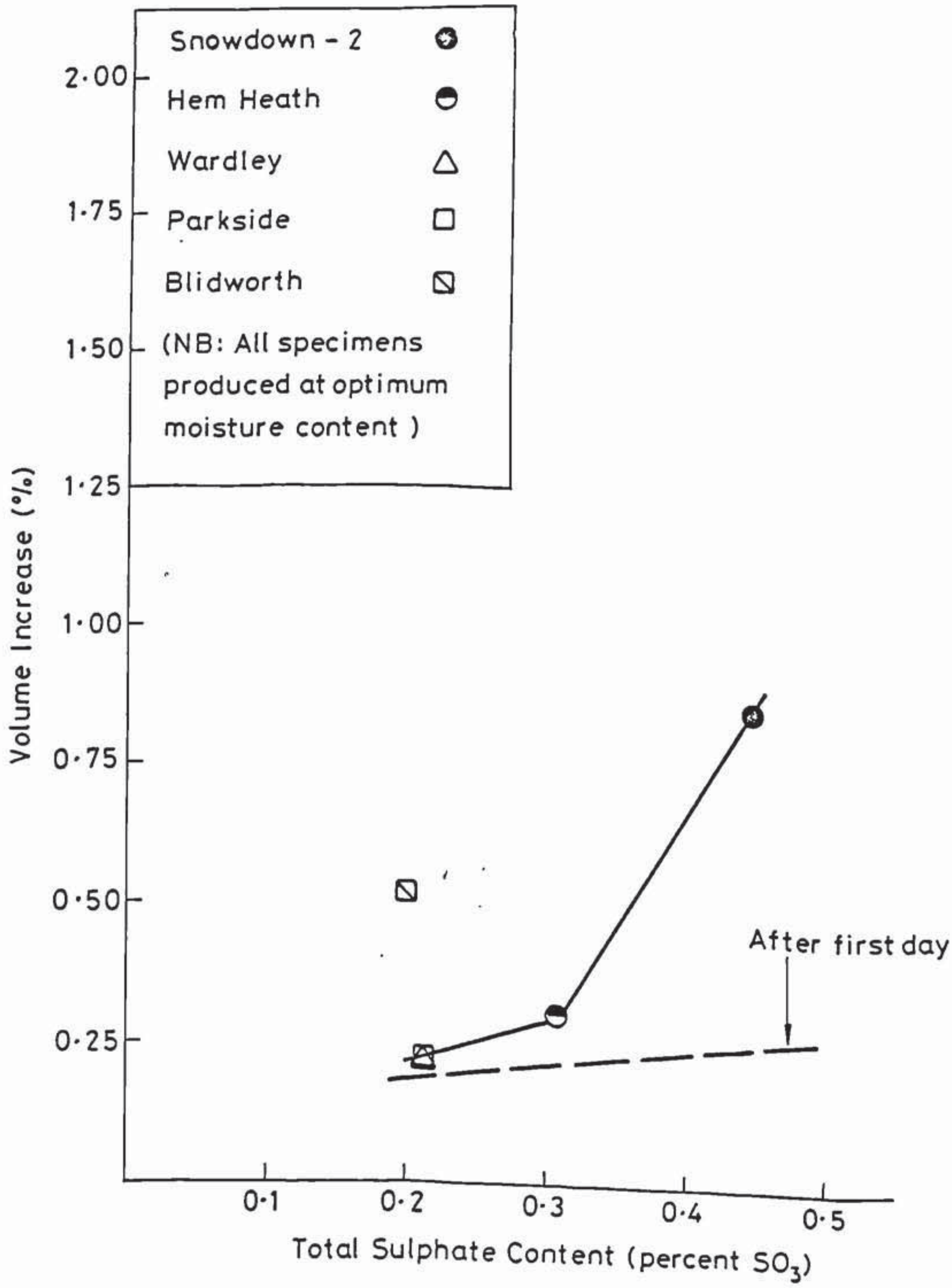


Figure 9.2 Volume Increase at end of Immersion Test plotted against Total Sulphate Content

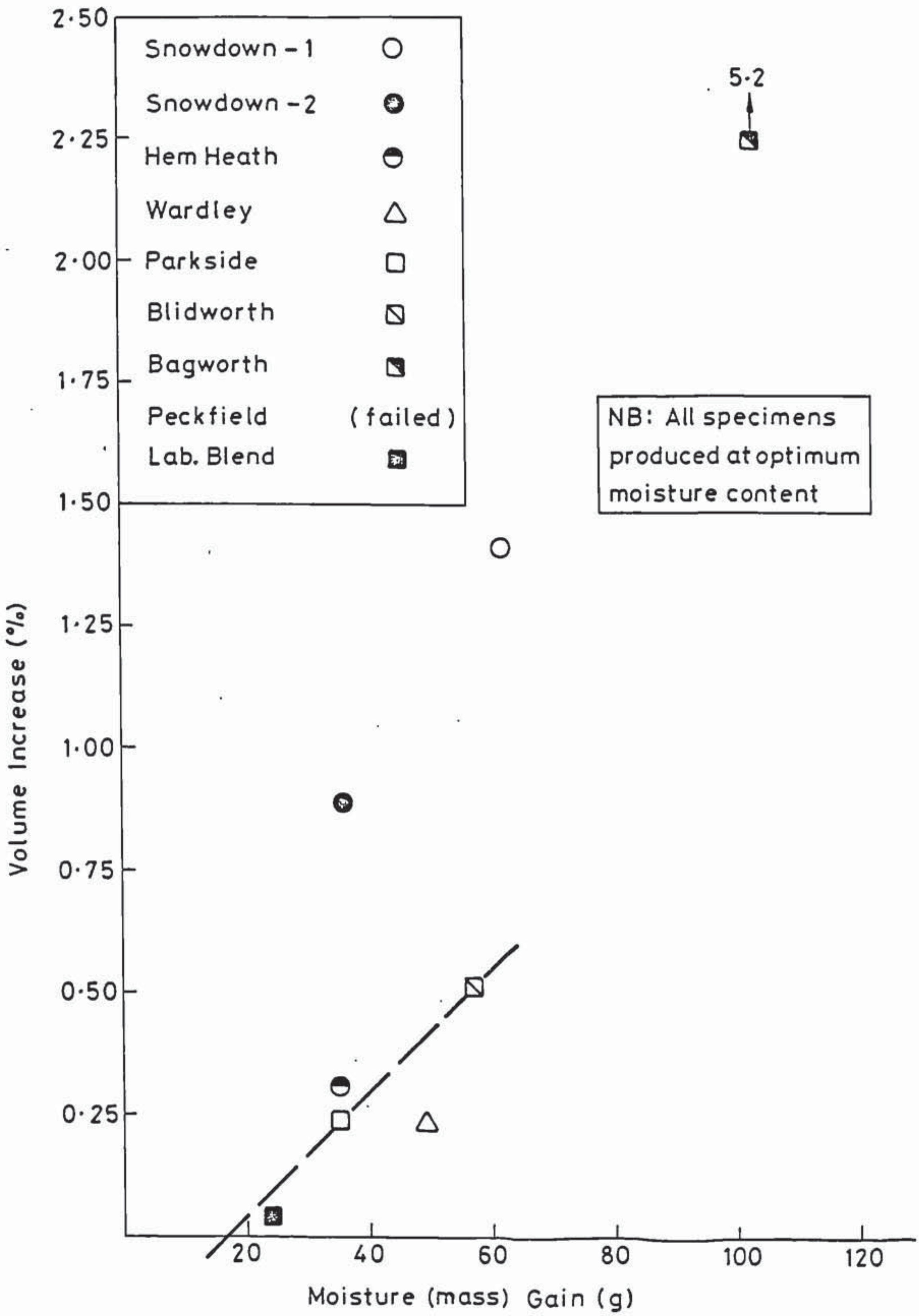


Figure 9.3 Volume Increase at the end of the Immersion Test plotted against Moisture Gain

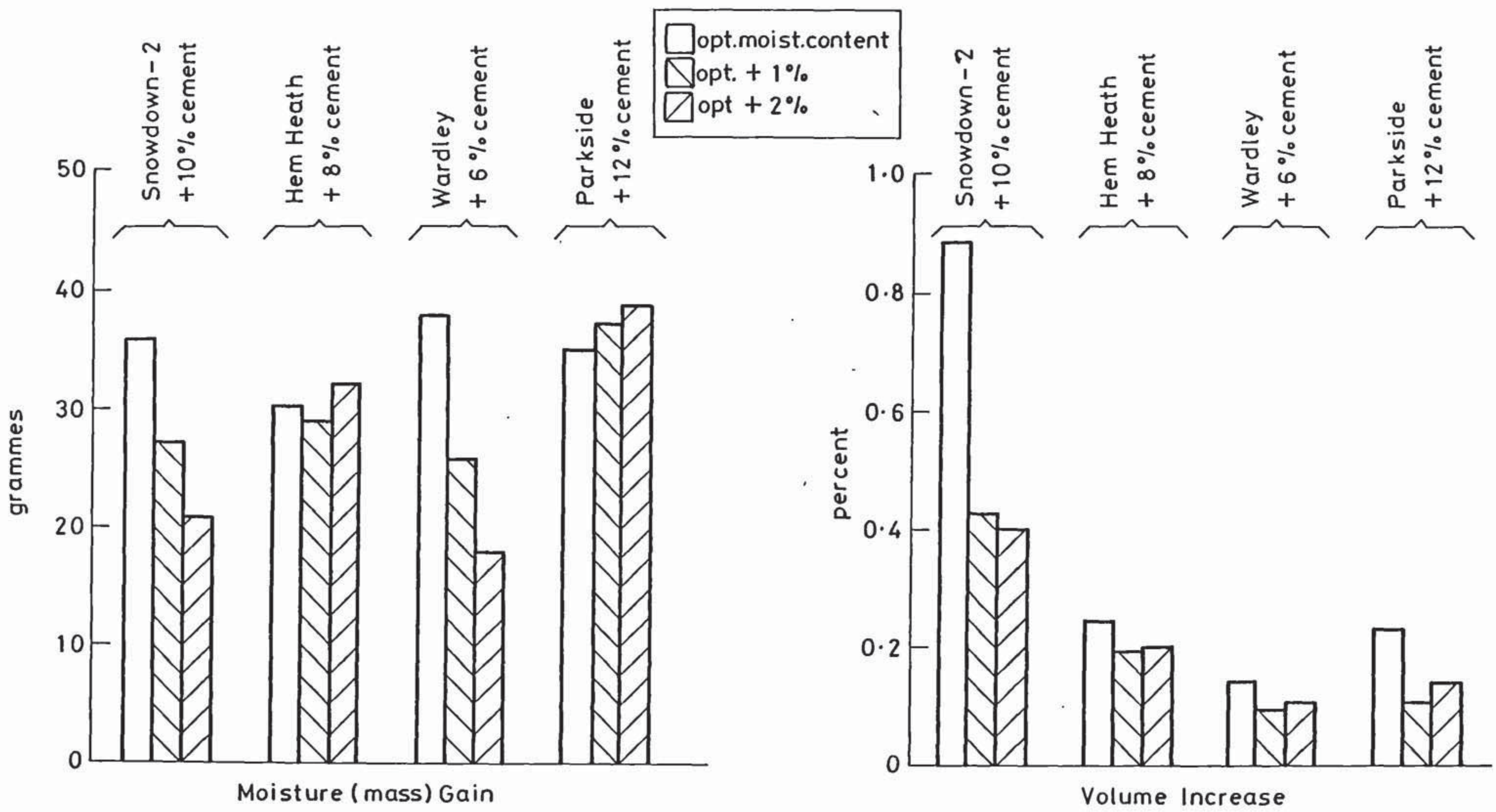


Figure 9.4 Effect of Initial Moisture Content on the behaviour of specimens subjected to Immersion

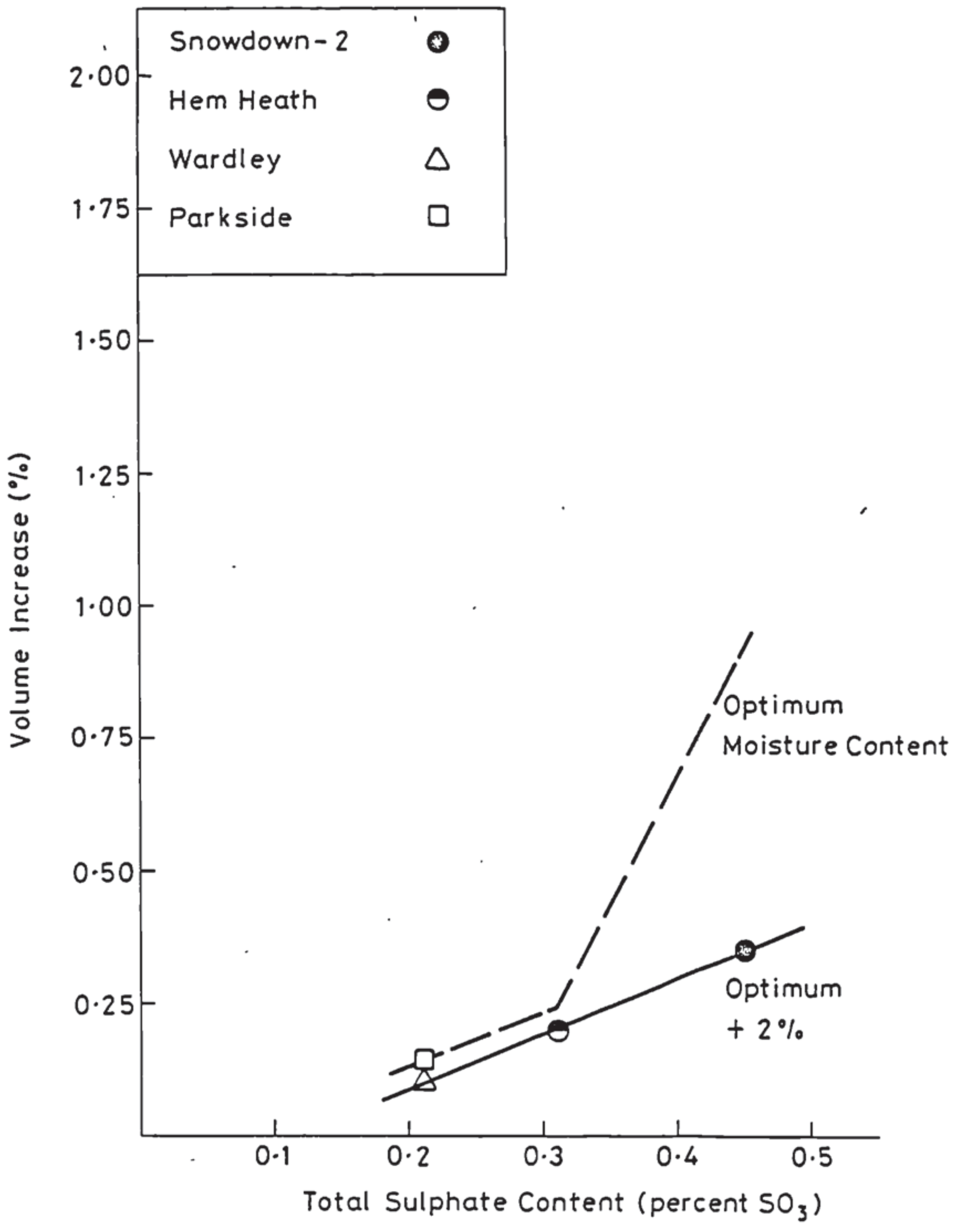


Figure 9.5 Effect of Initial Moisture Content on the relationship between Total Volume Increase and Total Sulphate Content

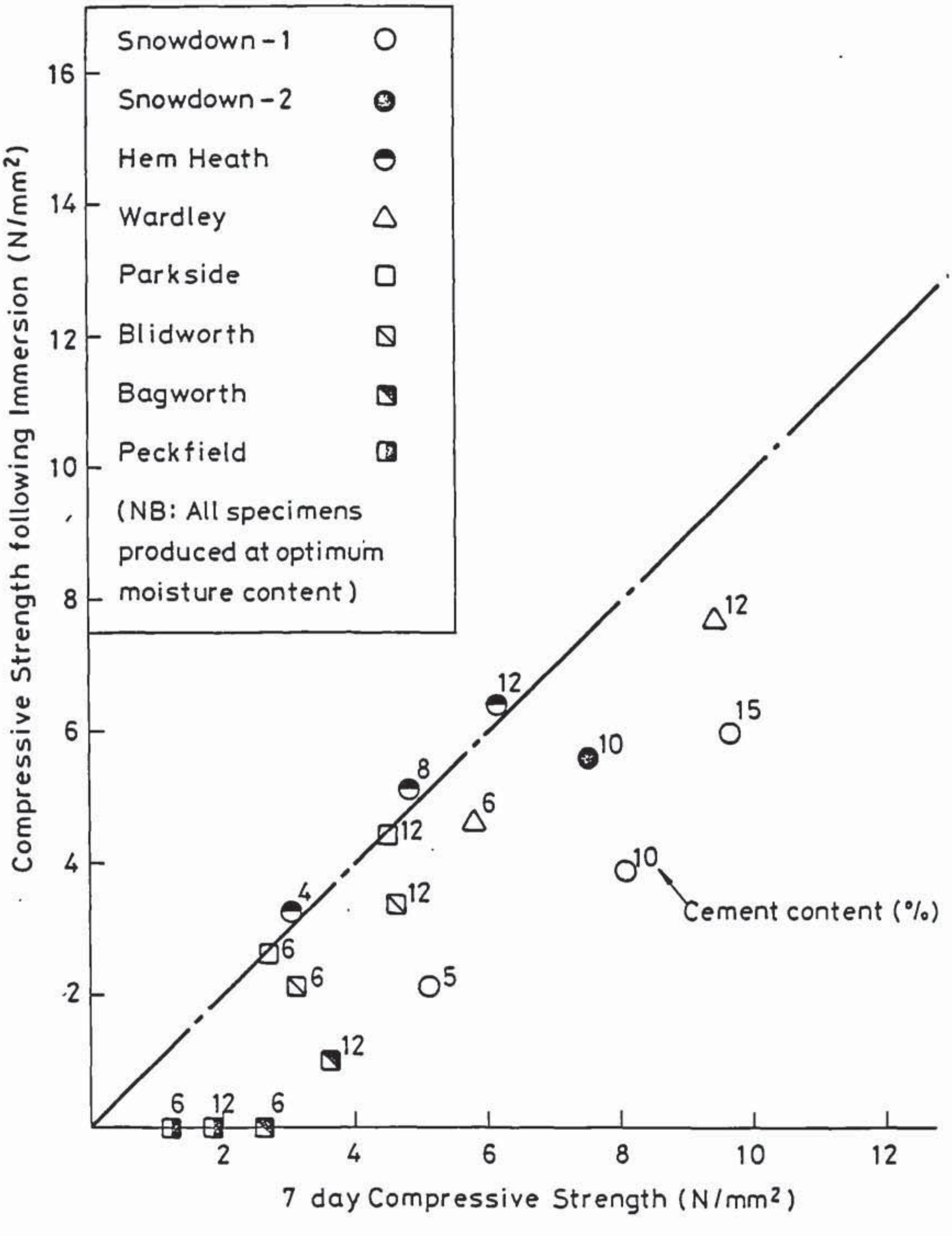


Figure 9.6 Effect of Immersion on Compressive Strength

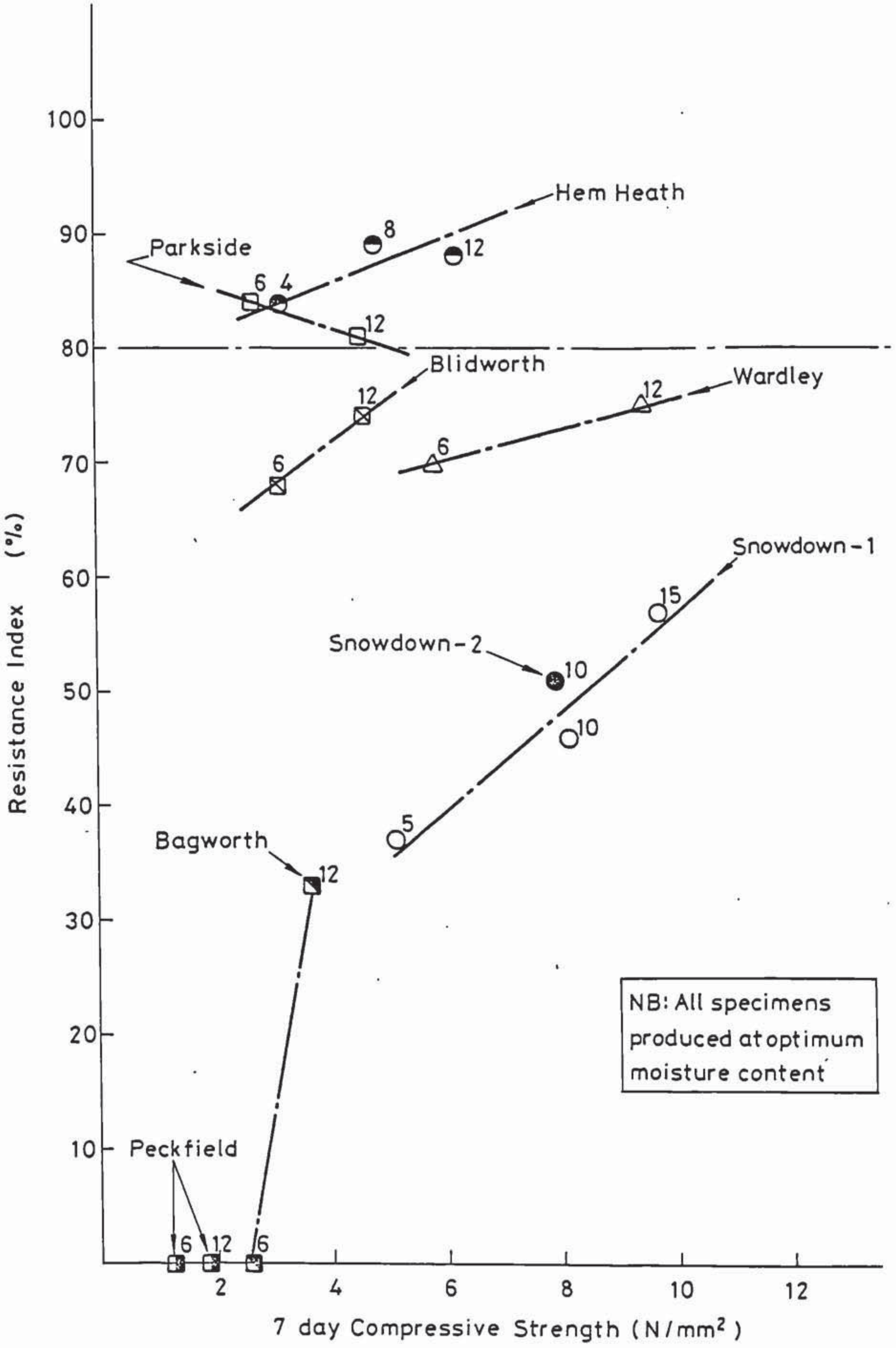


Figure 9.7 The Resistance Index of Cement Bound Minestone plotted against Compressive Strength

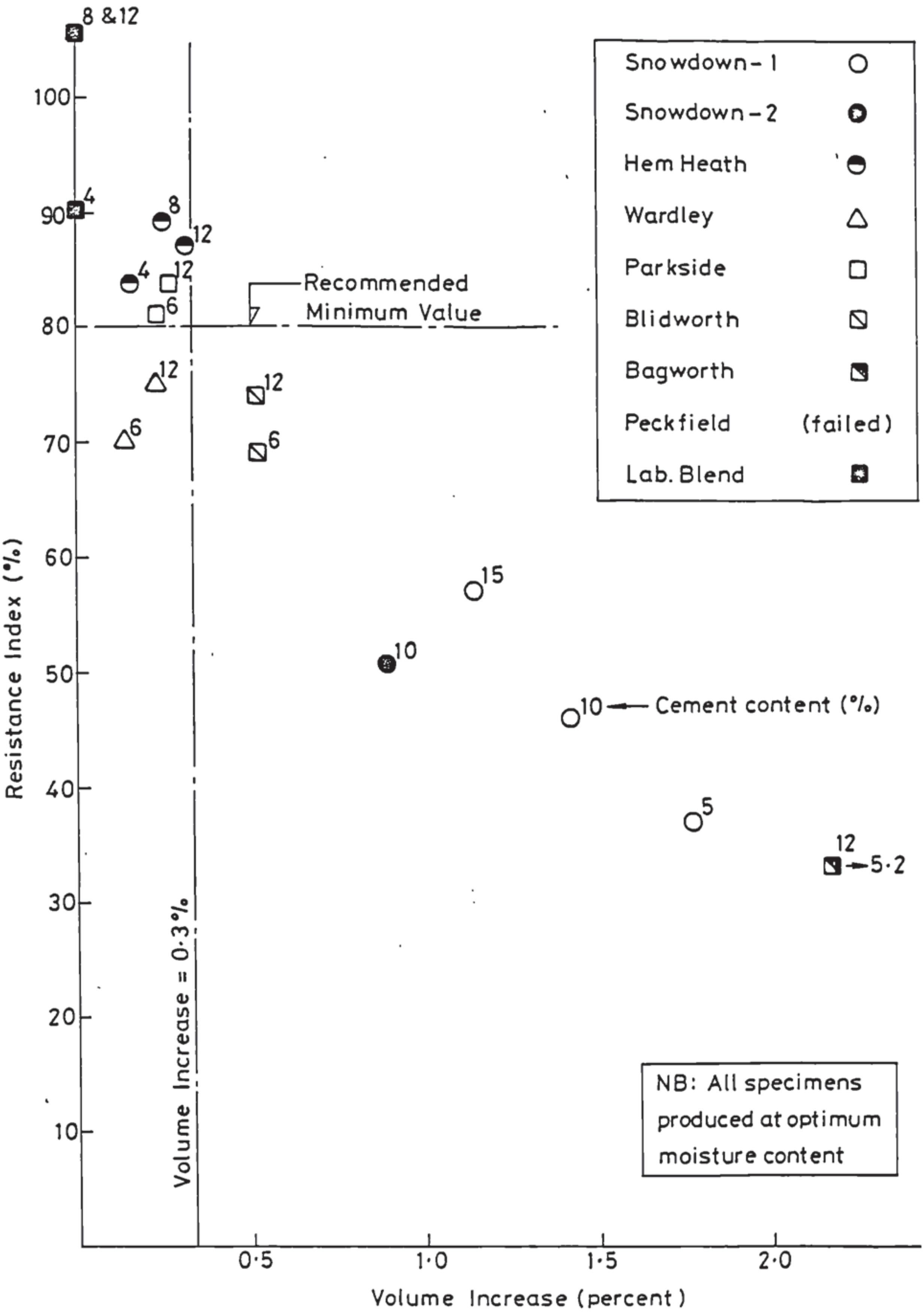


Figure 9.8 Resistance Index plotted against Volume Increase

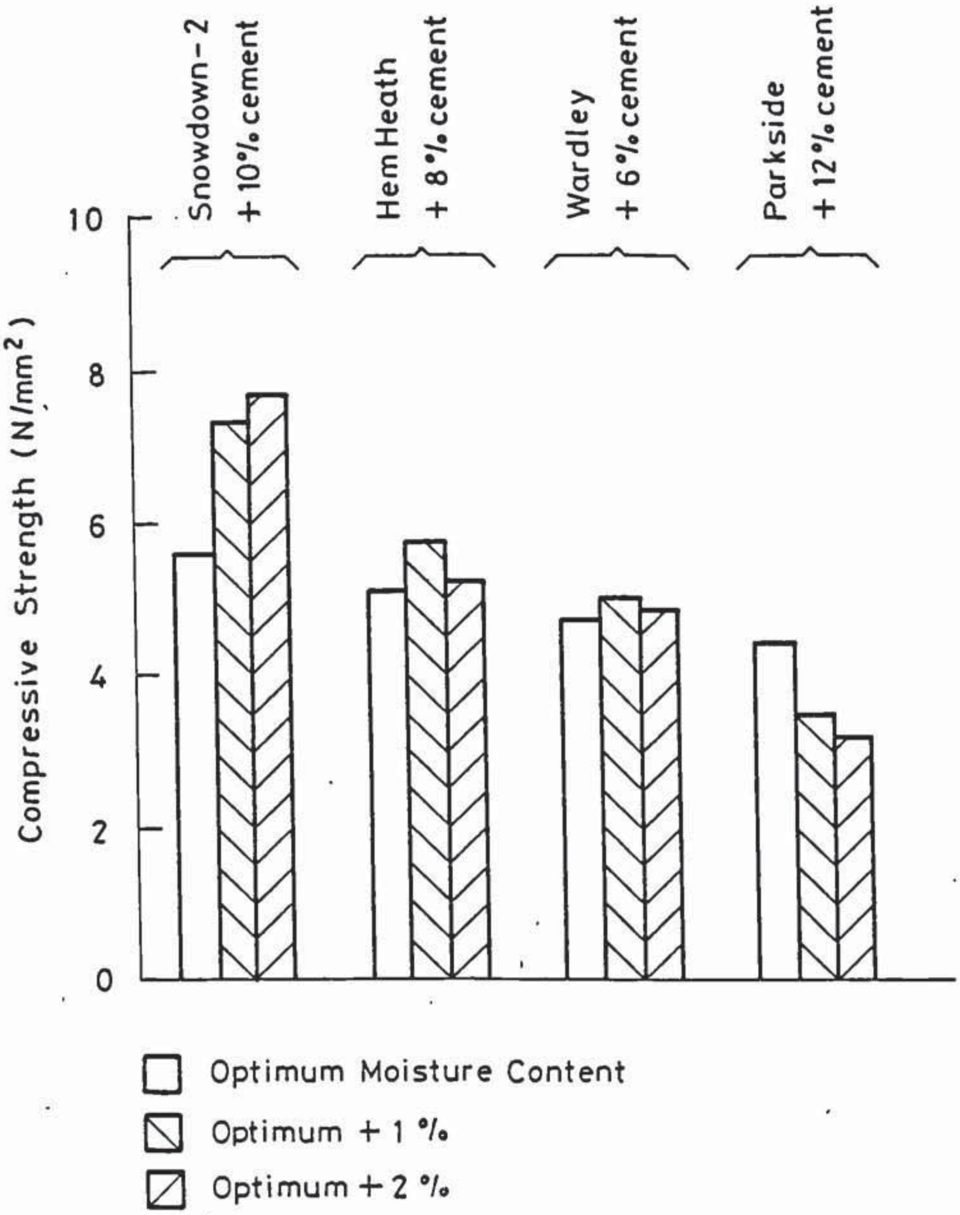


Figure 9.9 Effect of Initial Moisture Content on Compressive Strength following the Immersion Test

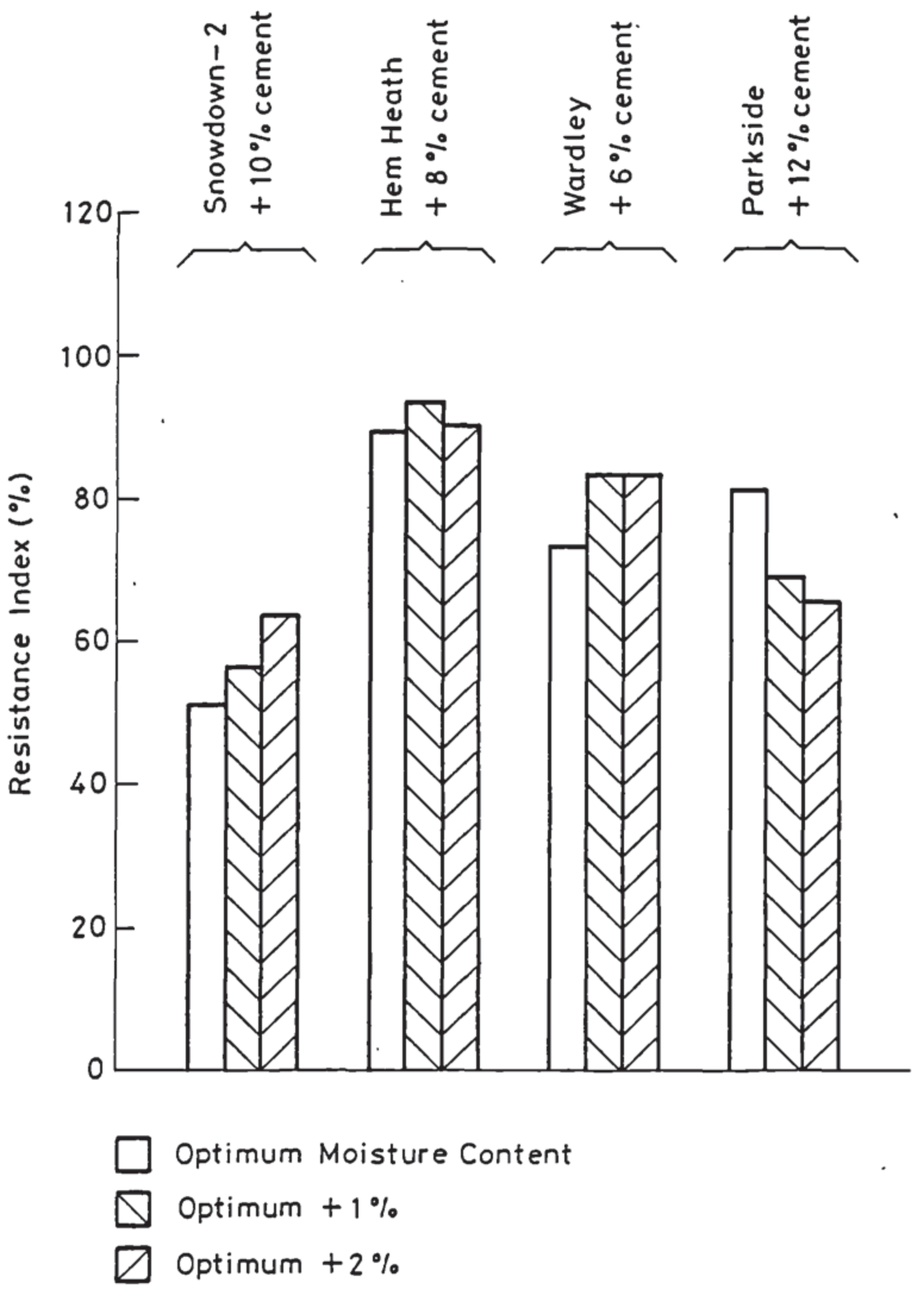


Figure 9.10 Effect of Initial Moisture Content on Resistance Index

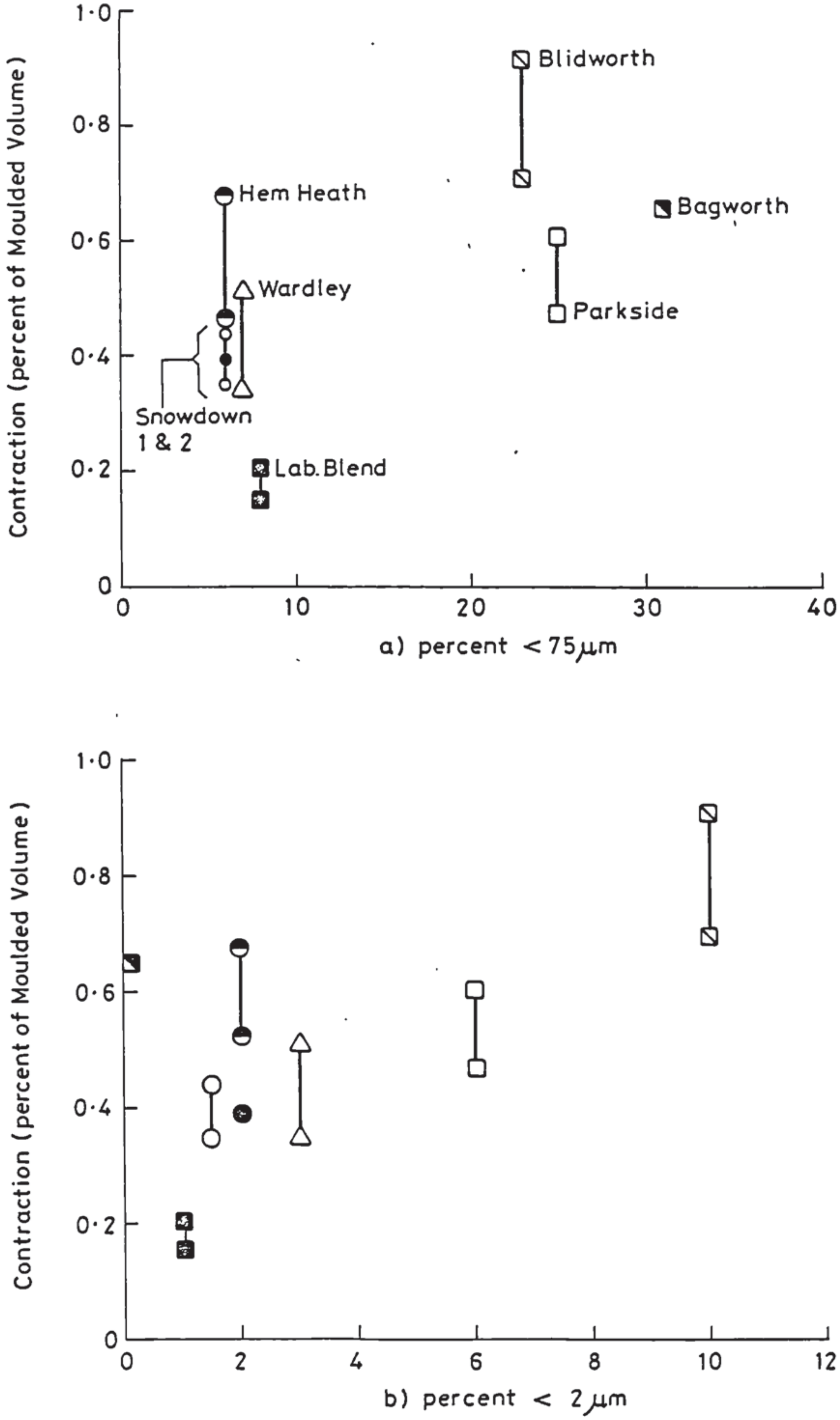


Figure 9.11 Contraction during first Freeze Period plotted against a) Silt Content; and b) Clay Content of Raw Material

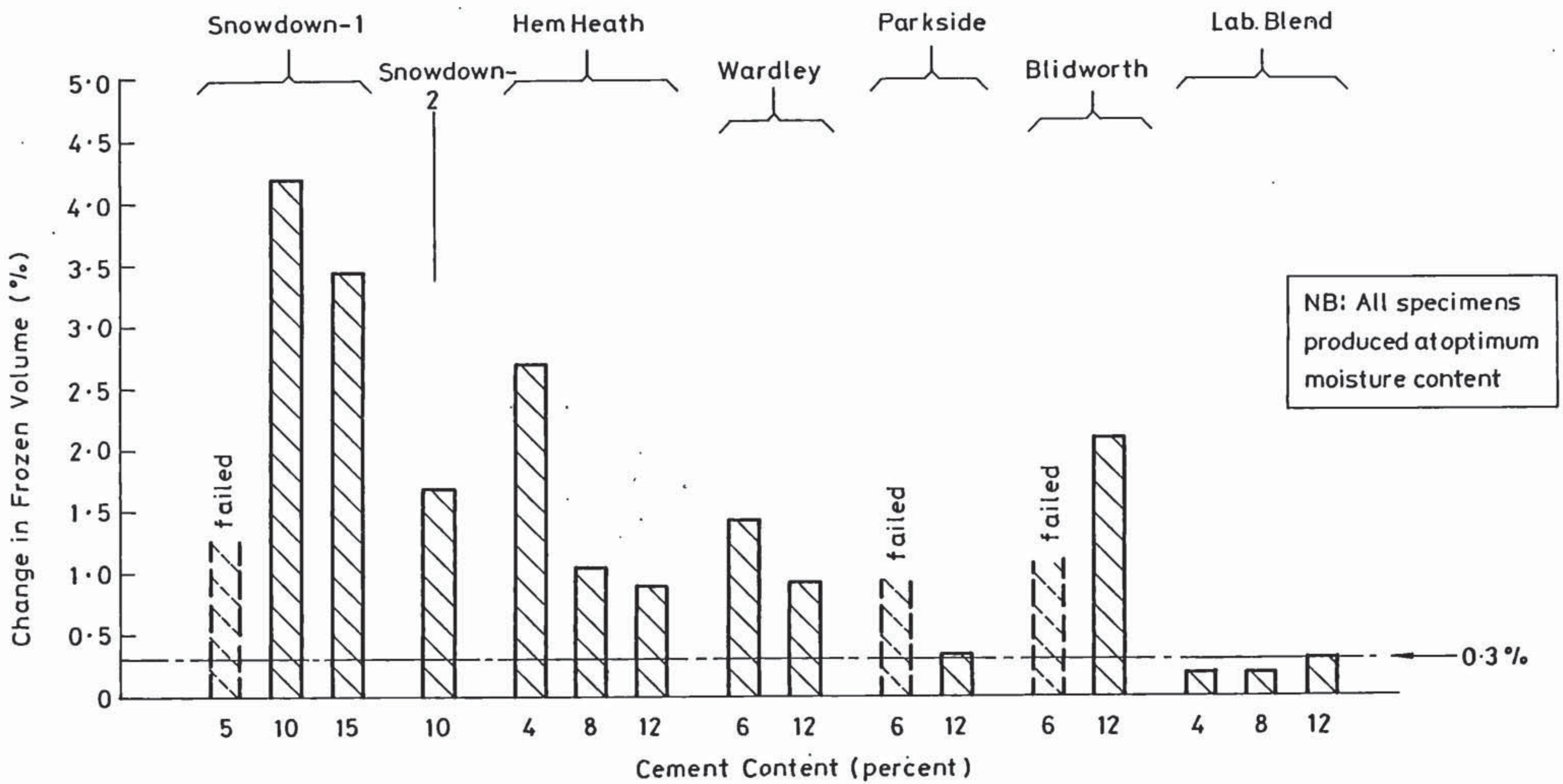


Figure 9.12 Effect of Cement Content on the change in the Frozen Volume during Freeze-Thaw Test

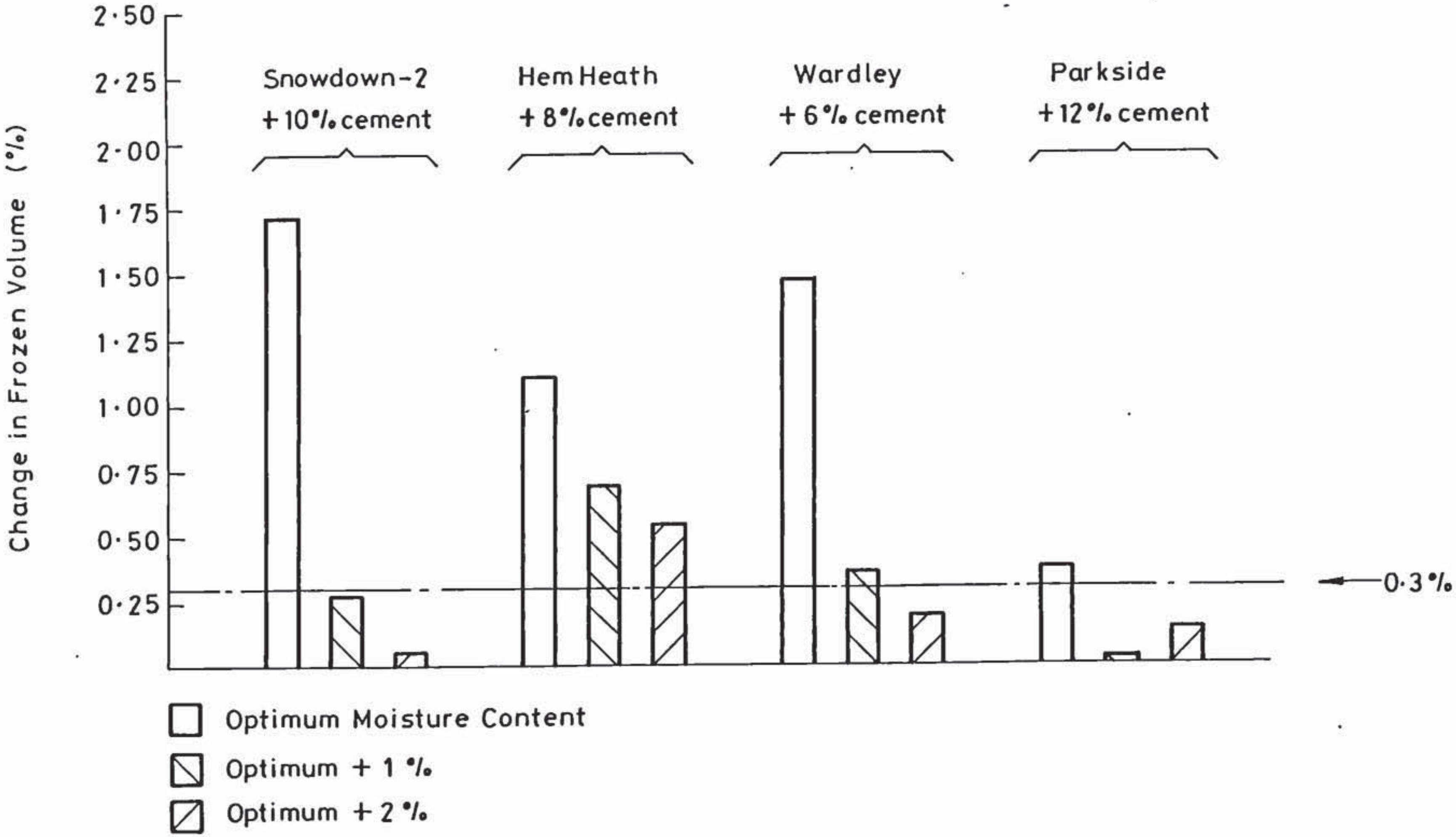


Figure 9.13 Effect of Initial Moisture Content on the Change in the Frozen Volume during the Freeze-Thaw Test

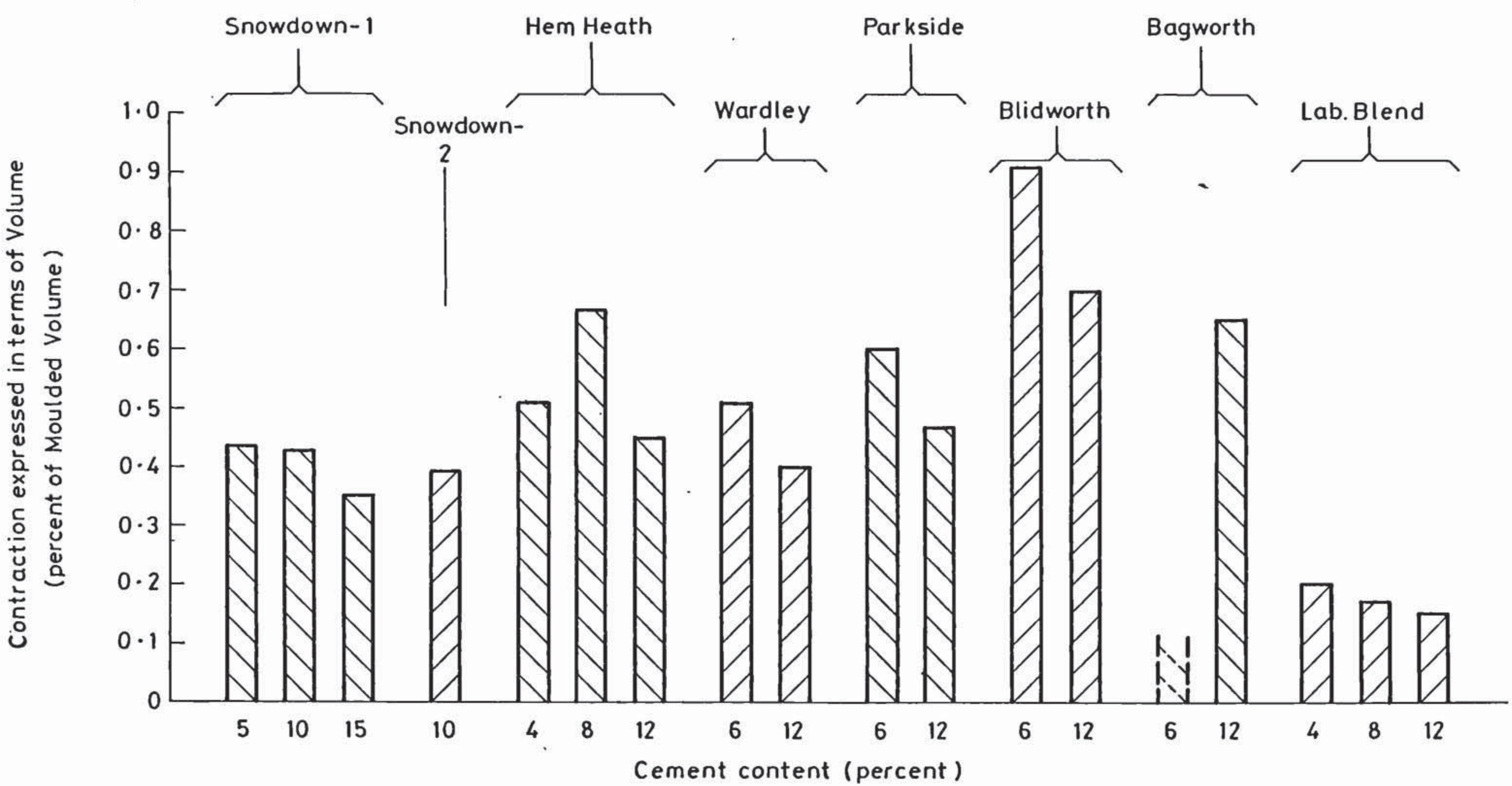


Figure 9.14 Effect of Cement Content on Contraction during the first Freezing Period

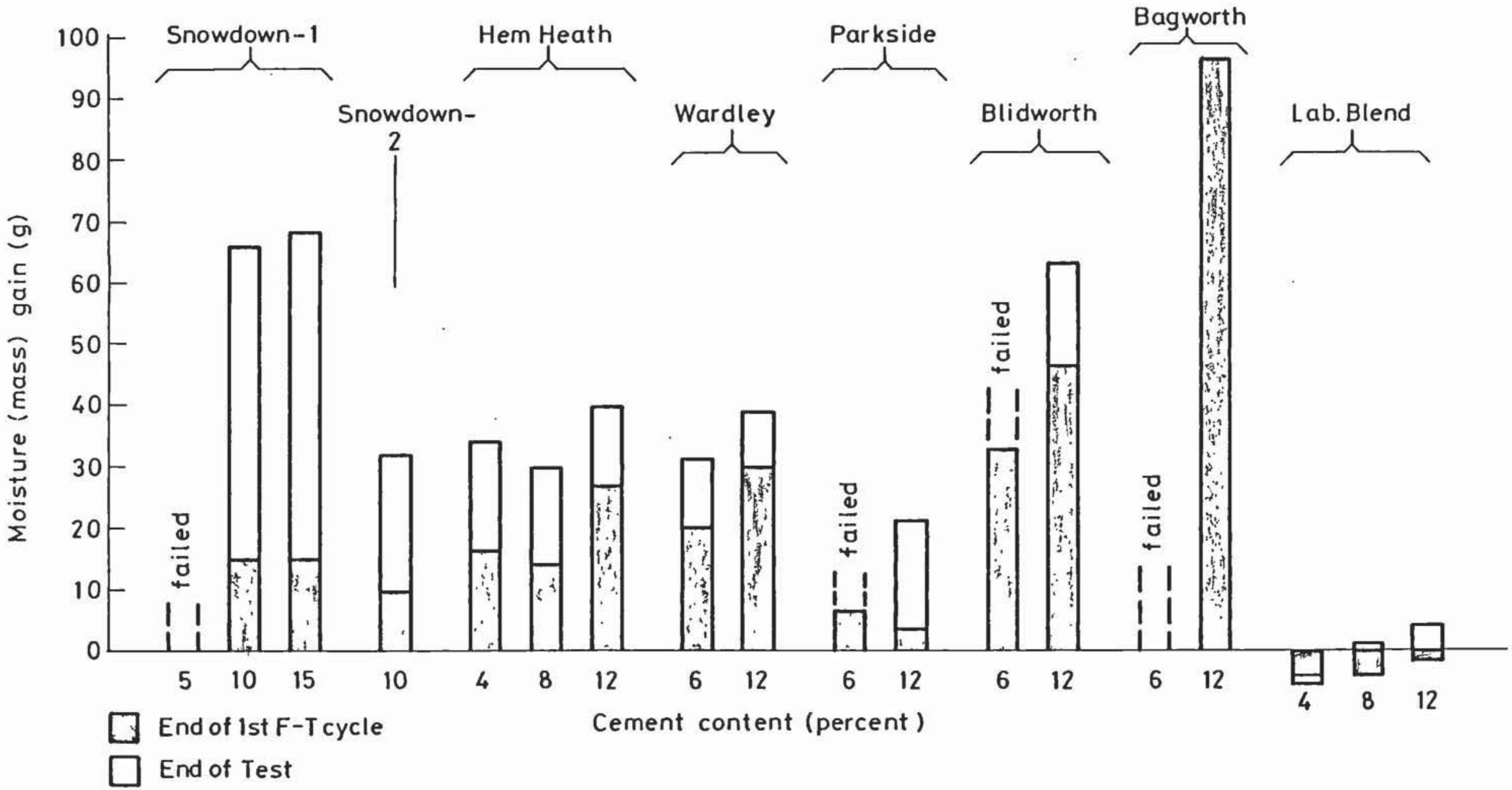


Figure 9.15 Effect of Cement Content on Moisture Gain during the Freeze-Thaw Test

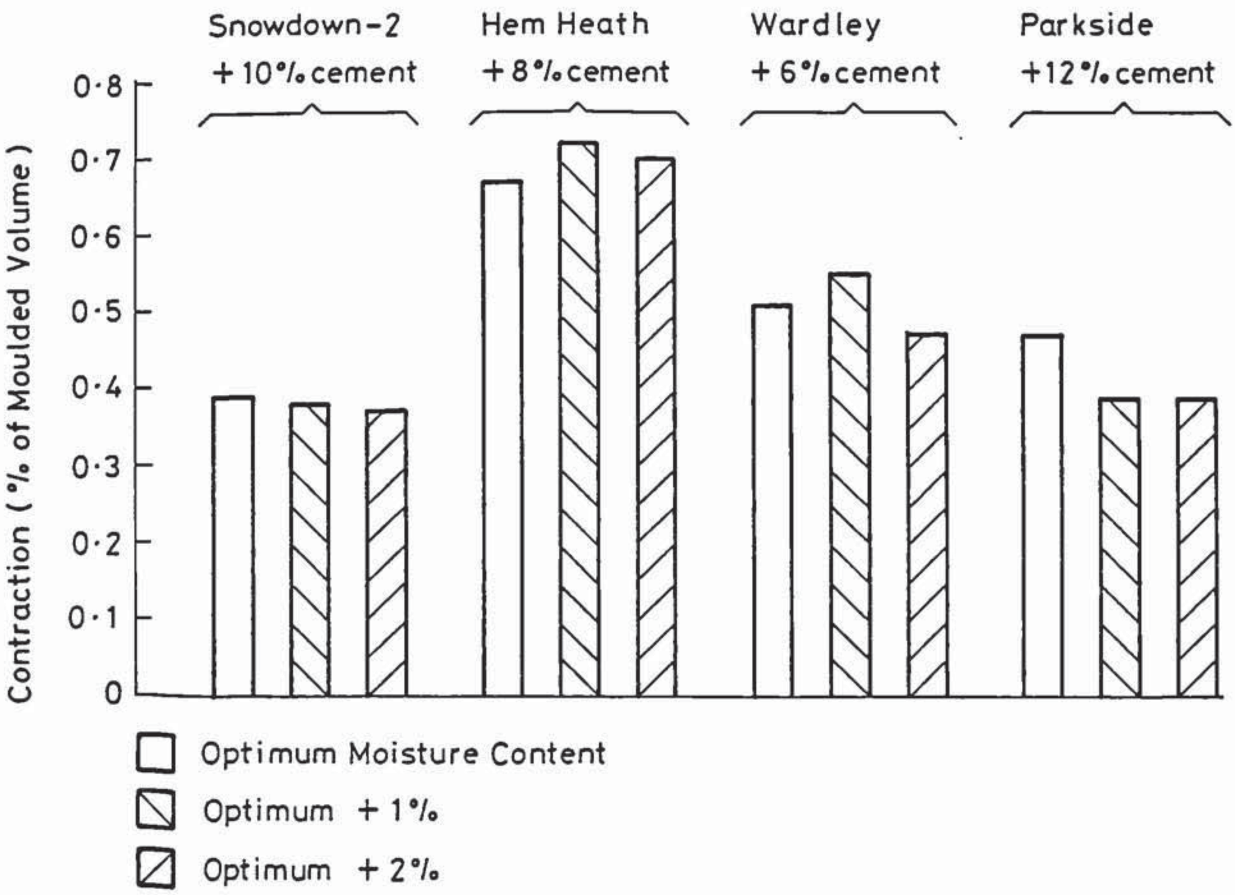


Figure 9.16 Effect of Initial Moisture Content on Contraction during first Freezing Period

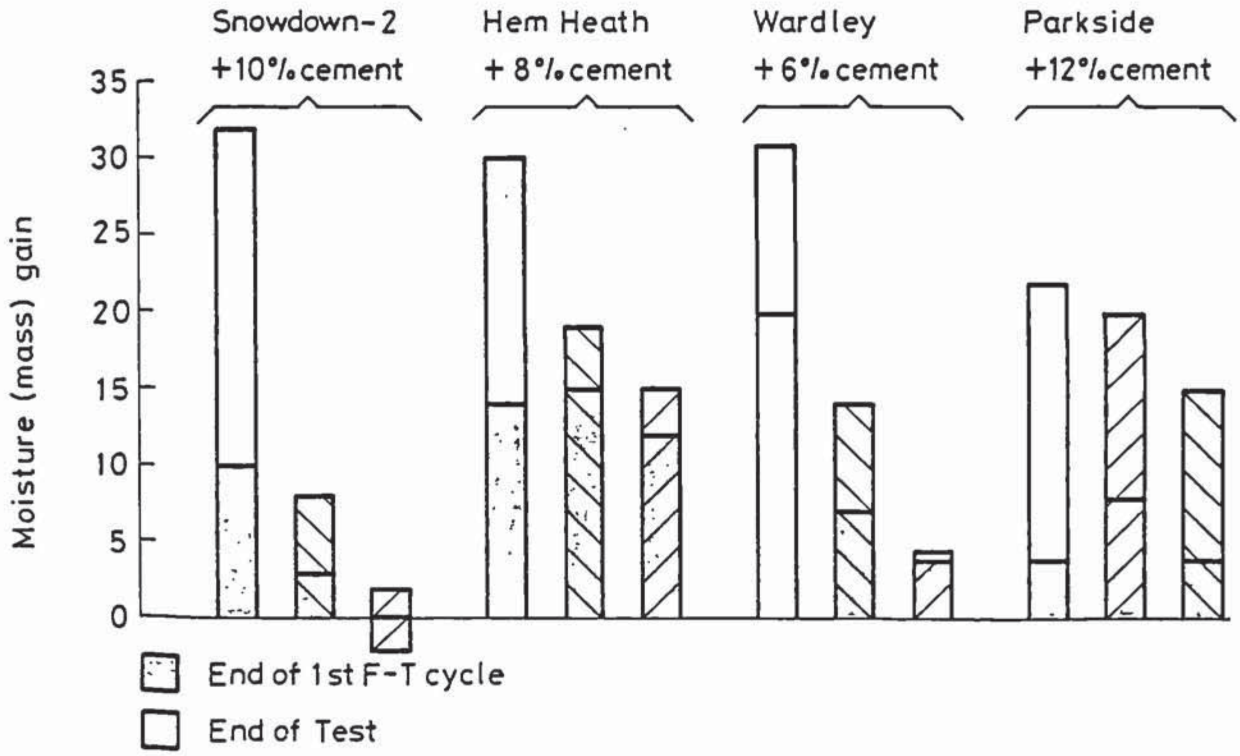


Figure 9.17 Effect of Initial Moisture Content on Moisture Gain during Freeze-Thaw Test

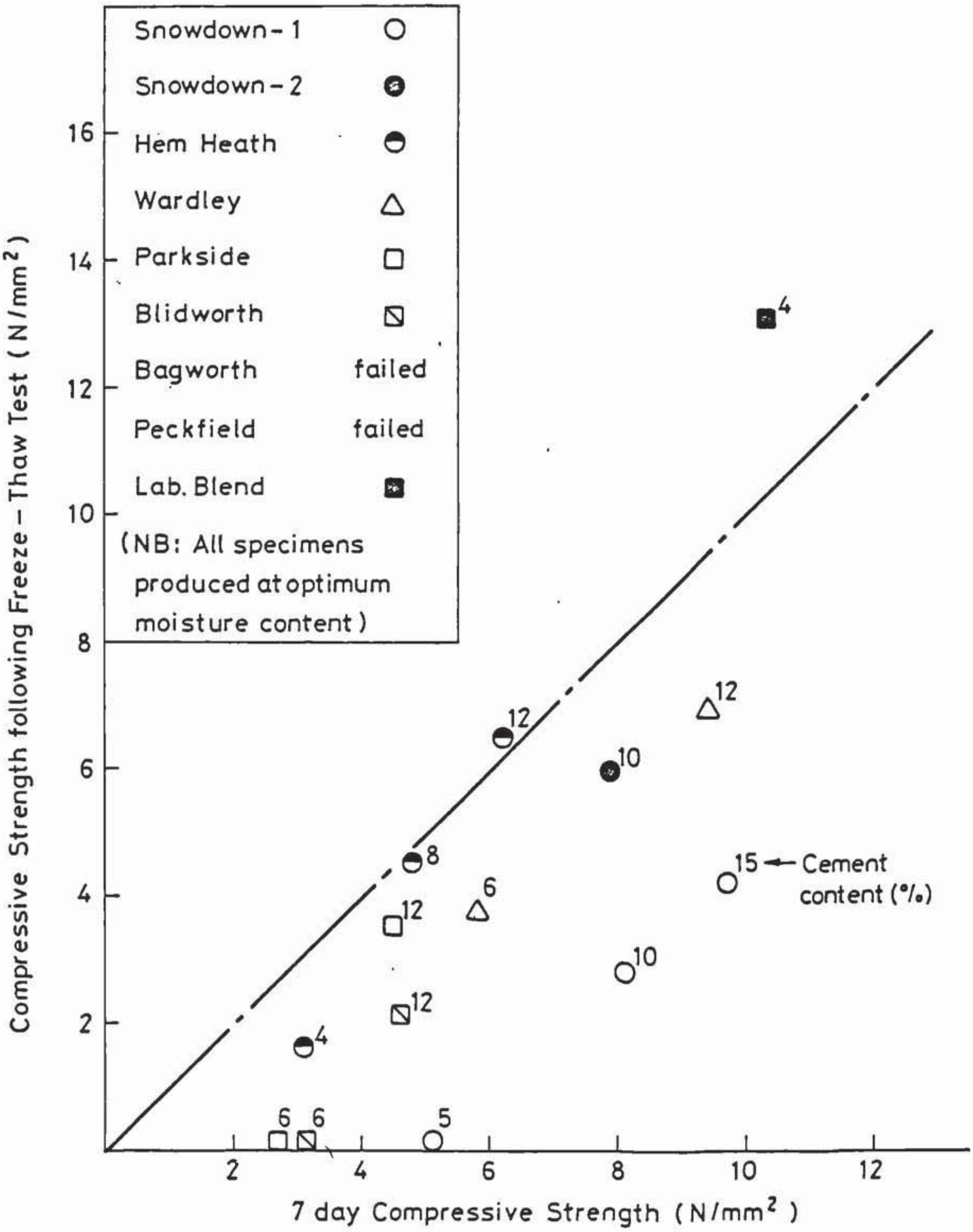


Figure 9.18 The effect of Freezing and Thawing on Compressive Strength

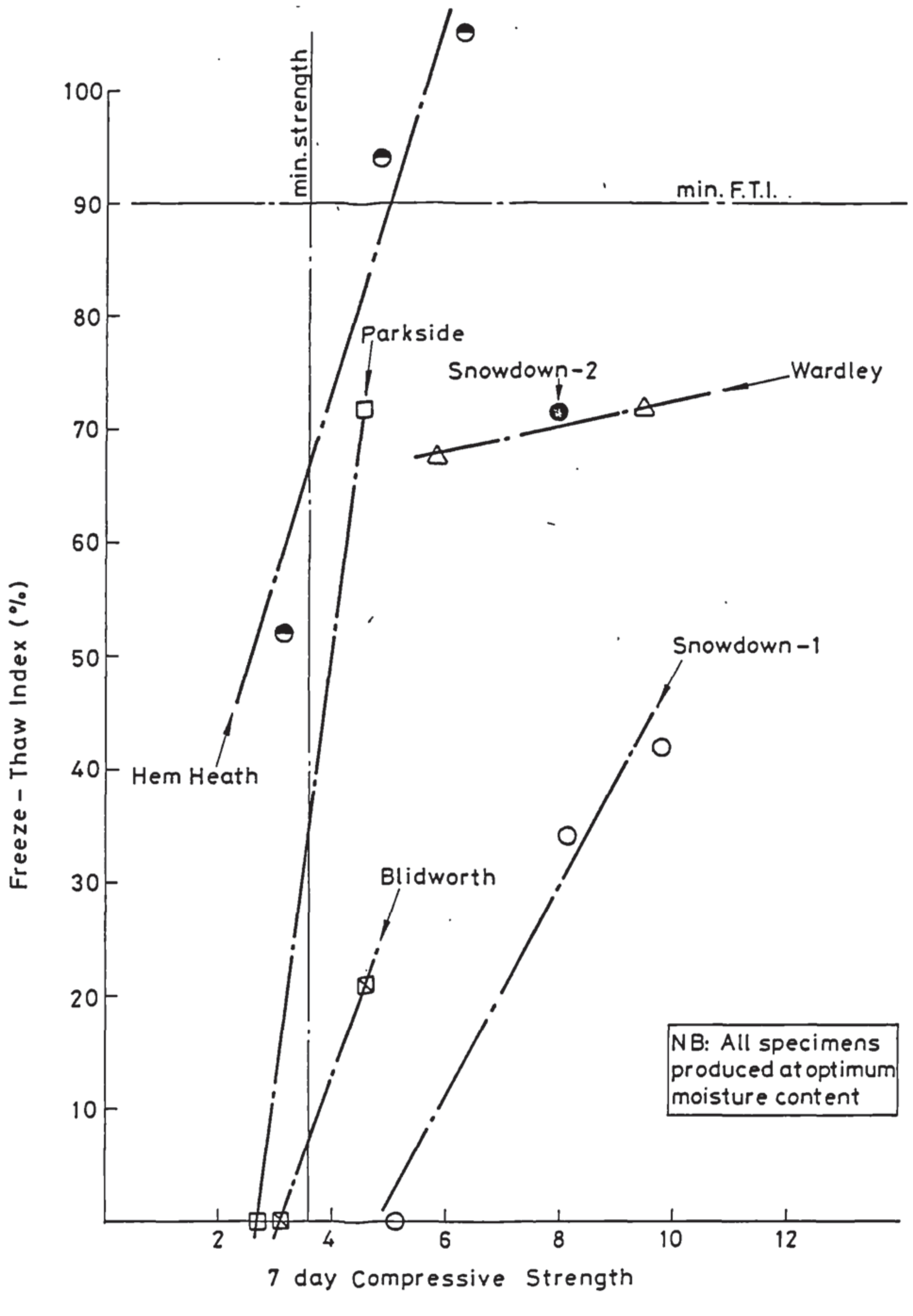


Figure 9.19 Freeze-Thaw Index plotted against 7 day Compressive Strength

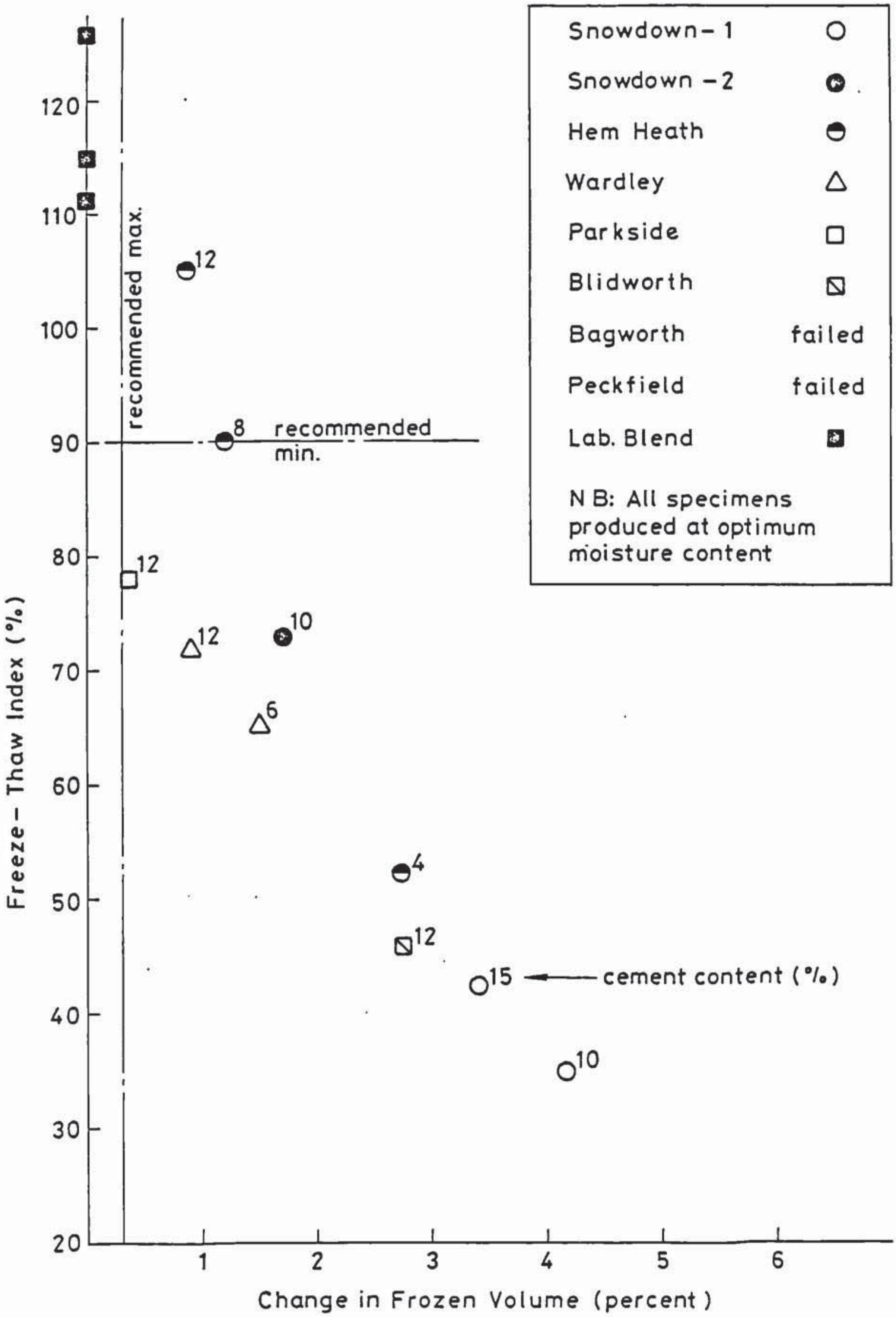


Figure 9.20 Freeze-Thaw Index plotted against Change in Frozen Volume

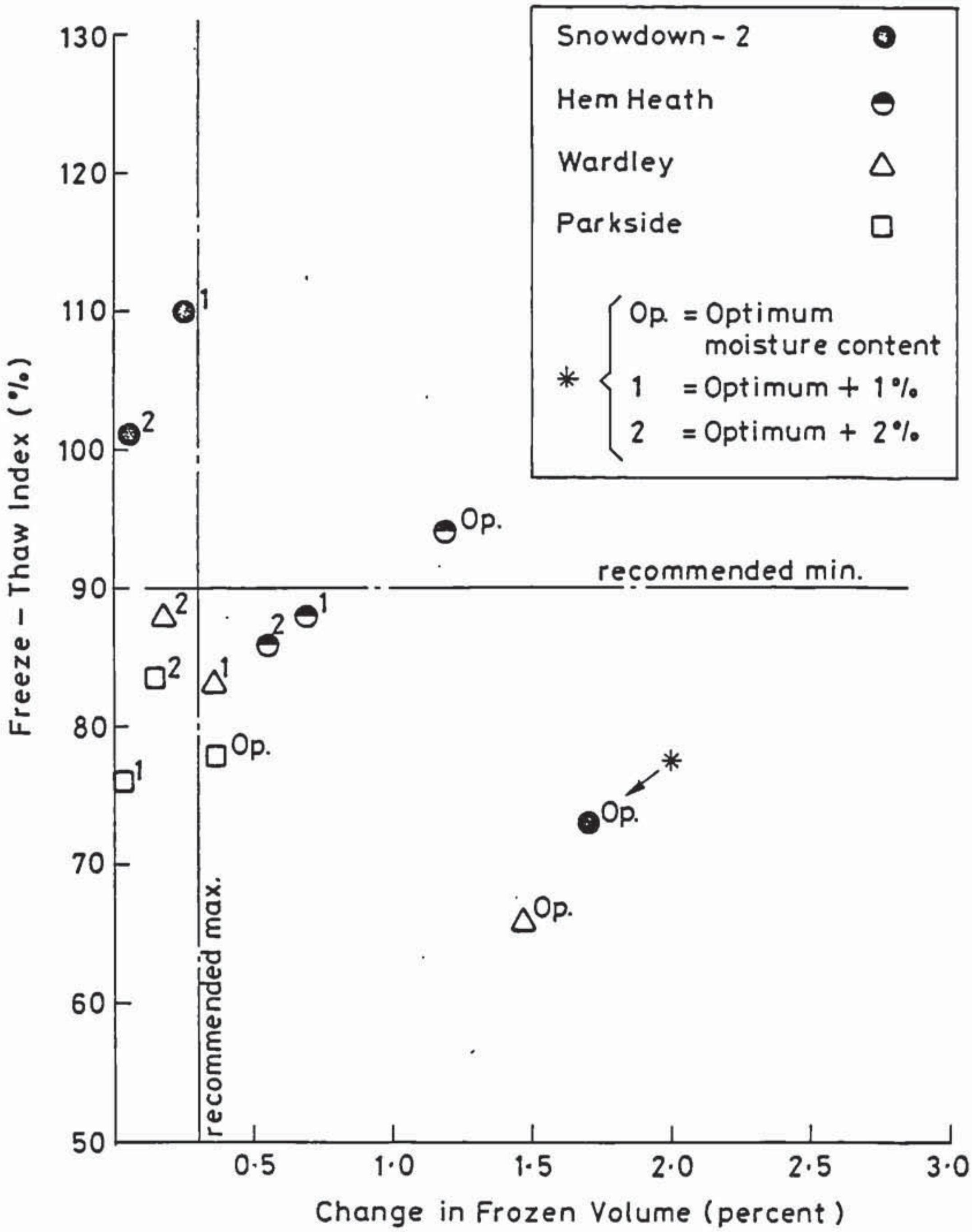


Figure 9.21 Effect of Initial Moisture Content on Relationship between Freeze-Thaw Index and change in Frozen Volume

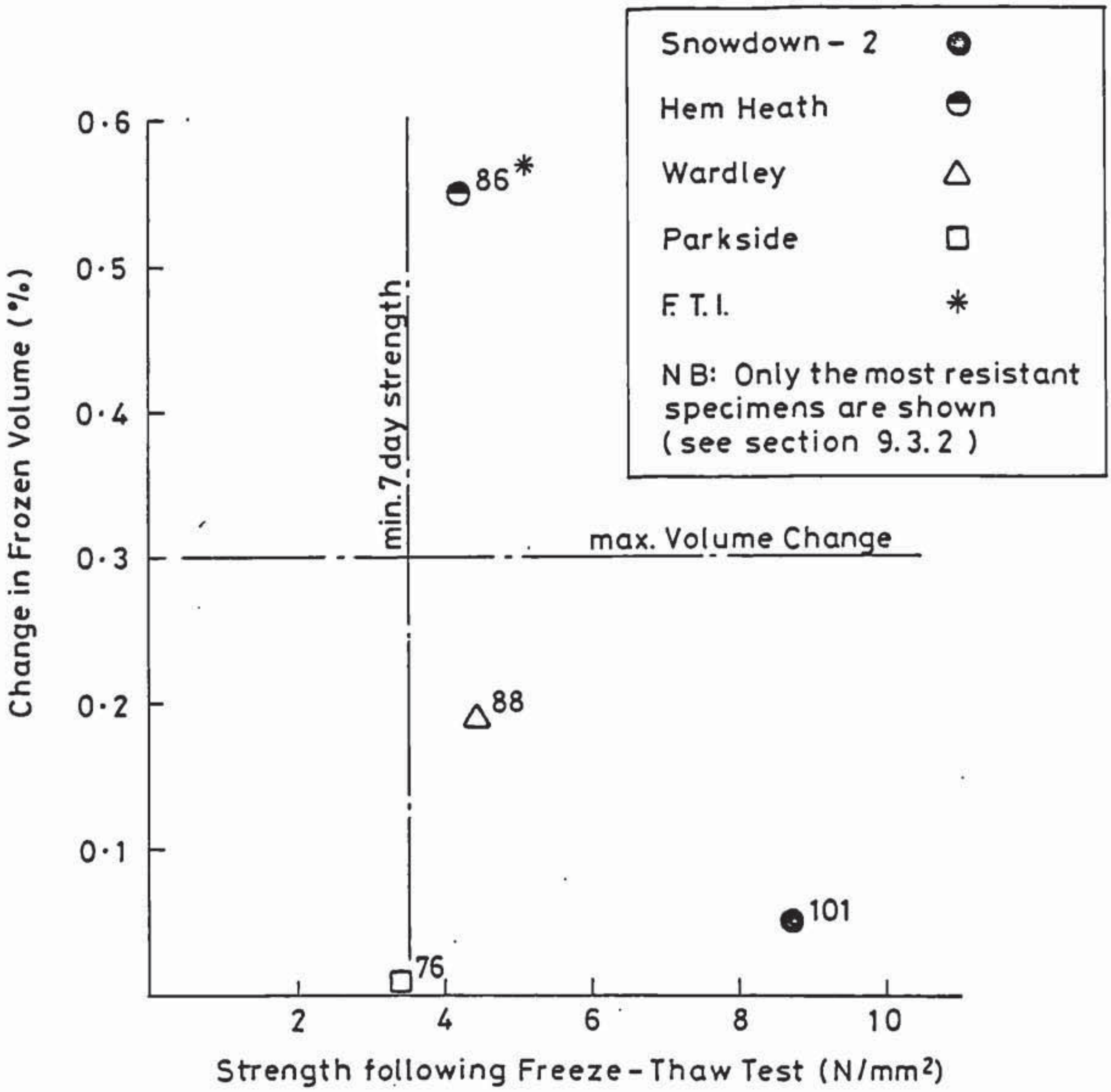


Figure 9.22 Change in Frozen Volume plotted against Strength following Test

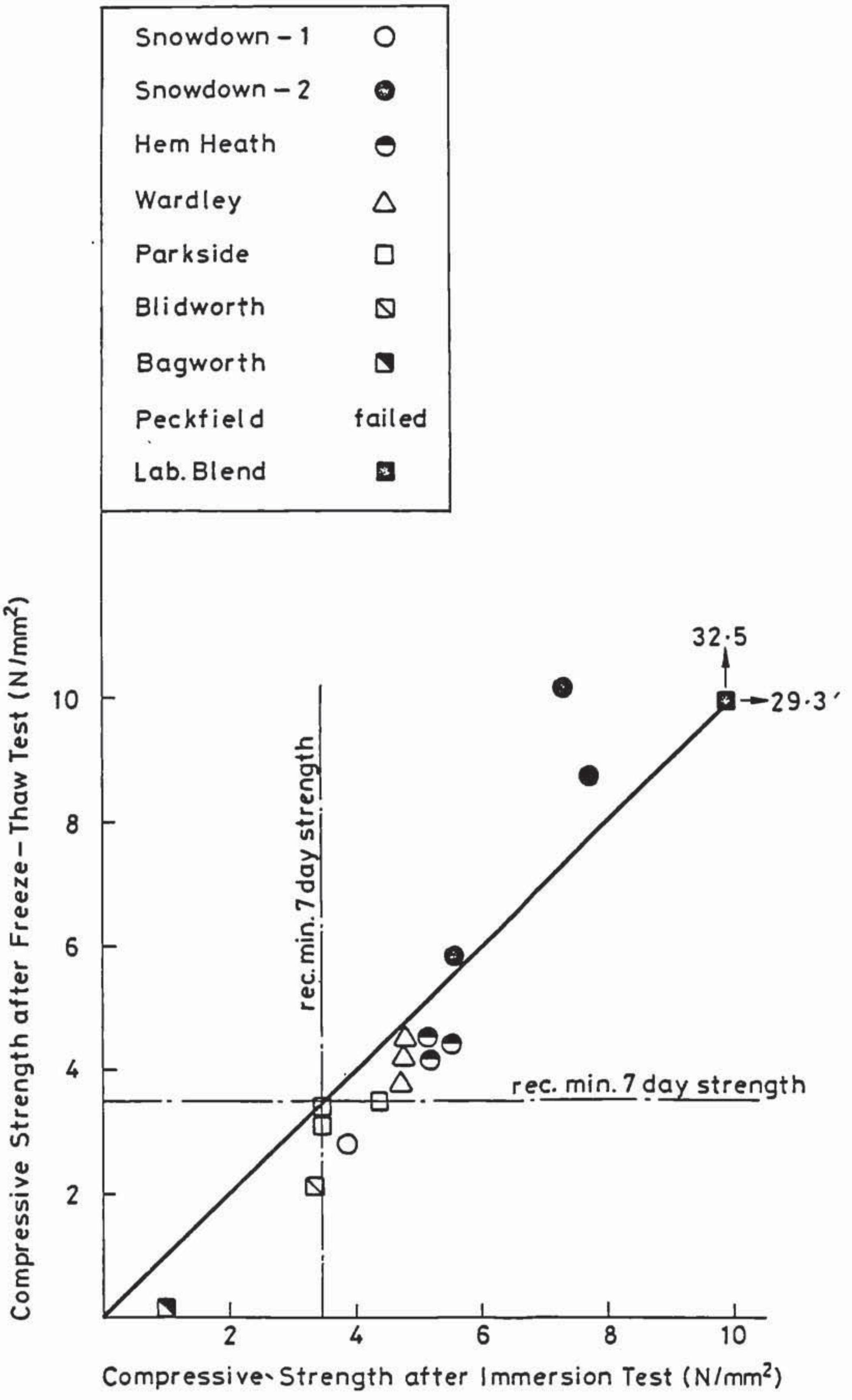


Figure 9.23 Comparison of Compressive Strength following Immersion & Freeze-Thaw Test

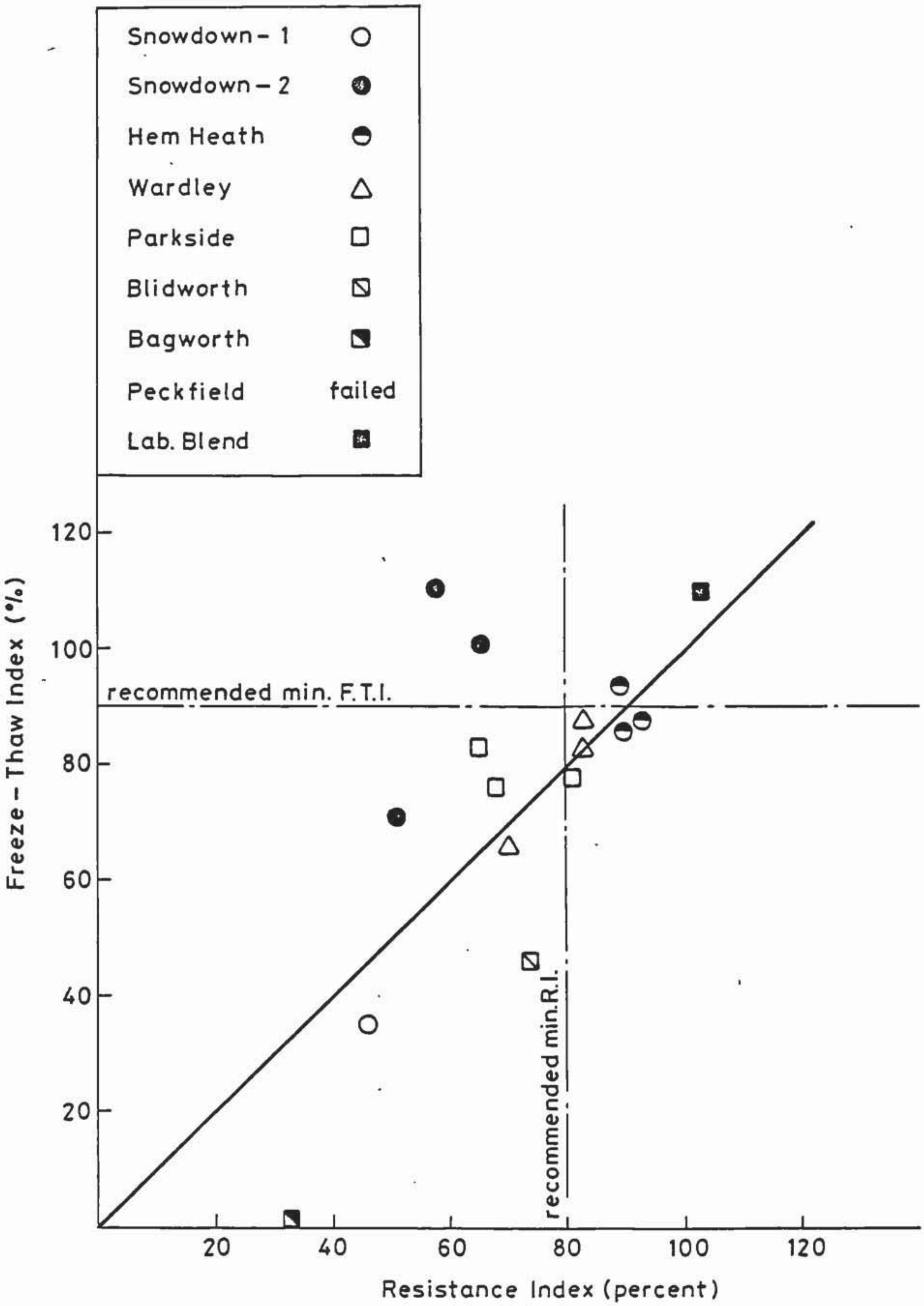


Figure 9.24 Comparison of Resistance Index (R.I.) and Freeze-Thaw Index (F.T.I.)

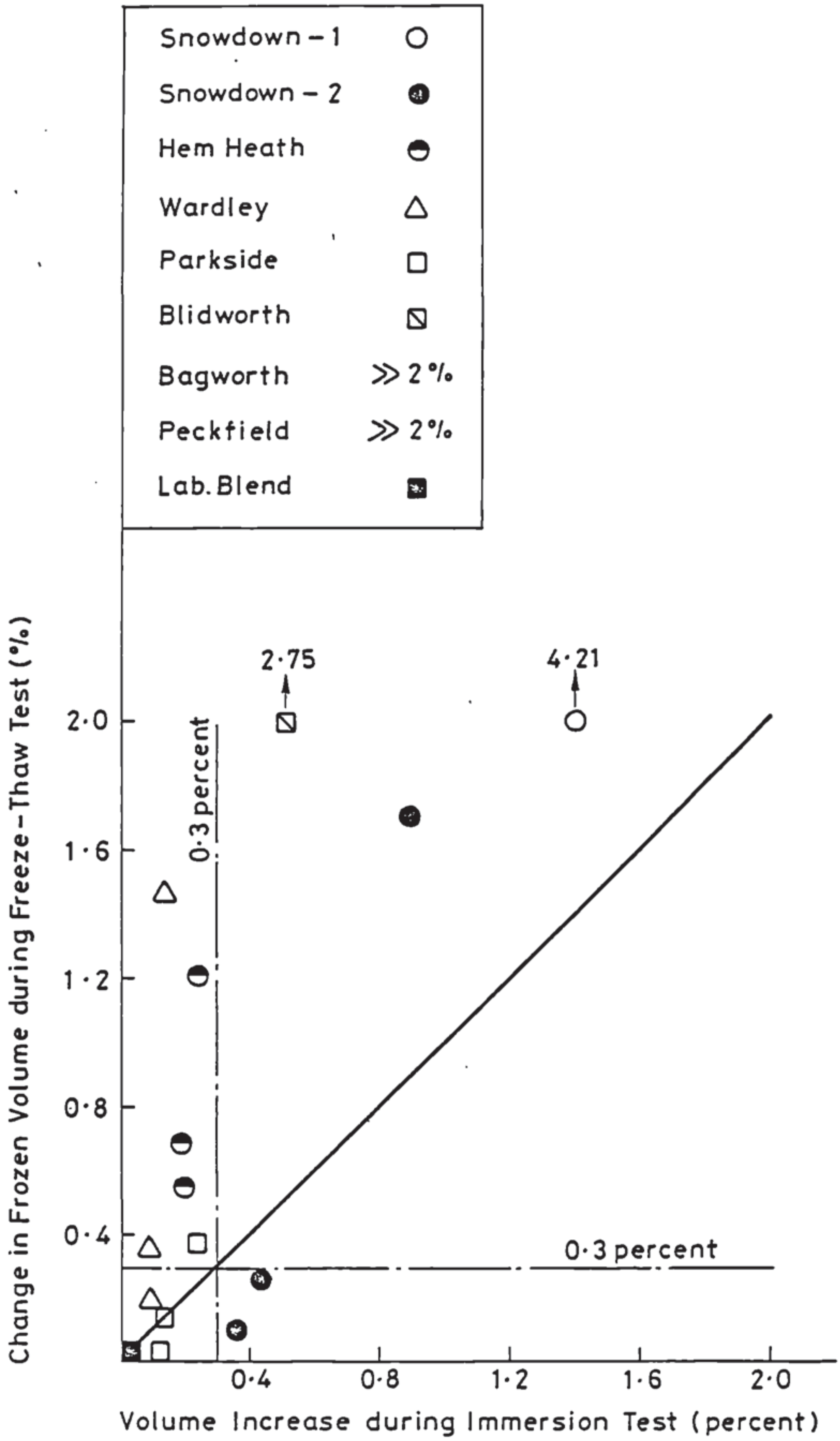


Figure 9.25 Comparison of Volume Changes during Immersion and Freeze-Thaw Tests

CHAPTER 10 MEASURING AND ASSESSING THE DURABILITY OF CEMENT BOUND MINESTONE

10.1 Introduction

The investigation of compressive strength, discussed in Chapter 7, showed that certain cement bound minestones, containing economically viable amounts of cement, satisfied the Department of Transport, 7 day strength criterion (9). Kettle and Williams, using the T.R.R.L. Frost Heave Test (13) to judge the frost susceptibility, reported (15) that cement bound minestones which contained sufficient cement to satisfy the strength criterion would normally also satisfy the heave criterion (13), and so be classified as non-frost susceptible. It would appear, therefore, that certain cement bound minestones satisfy the major criteria governing the acceptability of cement bound materials for sub-base and road-base construction. However, cement bound minestone is yet to be accepted as a viable alternative to expensive good quality aggregate for the construction of public roads. It is suggested that the current method of assessing cement bound materials is a major hurdle to a wider acceptance of cement bound minestones. These methods (9) are, possibly, not appropriate to low grade materials and do not provide sufficient information, particularly with regard to durability, to allow such materials to be specified with confidence. Although BS 1924:1975 (69) describes a test to assess the immersion resistance of cement bound soils it offers no guidance on the interpretation of the results. Furthermore, it has been argued (15) that the current frost heave criterion is not an appropriate measure of the frost susceptibility of cement bound materials. If cement bound low grade materials, including minestone, are to receive serious consideration as alternative pavement materials, it is essential that the methods for assessing their quality should include durability tests to provide an adequate record of their response to changes in moisture content and temperature. In addition, the criteria for judging performance must be established by correlating behaviour of the laboratory specimens with field performance. Before proposing methods and criteria appropriate to cement bound minestone, it is necessary to review the various measures of performance that have been used in this experimental study.

10.2 Dimensional Stability

Accurate measurements of the volume of cement bound minestone specimens during the durability test provide a sensitive measure of the degree of resistance to deterioration. Interpretation of the record of volume changes depends upon the characteristics of the particular test environment.

10.2.1 Immersion Test. The volume of all the minestone specimens tested increased during the test. The volume of the more resistant specimens increased during the first 1 to 4 days immersion and then remained constant for the remaining duration of the immersion period. This was interpreted as indicating that the effects of immersion, including any deleterious effects, had ceased. The volume of the less resistant specimens increased steadily throughout the test and was still increasing at the end of the immersion period. This almost certainly indicated serious and continuing deterioration.

10.2.2 Freeze-Thaw Test. The cyclic nature of this test produces cyclic changes in volume. Durable specimens display a consistent cyclic pattern for the duration of the test with, possibly, a slight overall increase in the frozen volume. Deviation from a previously consistent pattern, such as a reduction in the amplitude of the cyclic changes, indicates the onset of deterioration. The deterioration of the least resistant specimens is governed mainly by ice growth and therefore the frozen volume of such specimens may exceed the thawed volume.

10.3 Mass Changes

The record of mass changes compliments the volume change data. However, it was found that material was lost from the majority of specimens, including even the most resistant and, therefore, mass data do not provide as sensitive a measure of resistance as volume change. The durability of cement bound minestone specimens appeared to be governed, to a large extent, by the absorption of water and mass measurements provide a means of estimating the amount of moisture gained.

10.4 7 Day Compressive Strength

This is, currently, the primary method of assessing the quality of cement bound materials. Tests on cement bound minestones showed that satisfying the D.O.T. minimum 7 day strength criterion (9) did not necessarily guarantee adequate resistance to either immersion or freezing and thawing. Whilst the 7 day compressive strength will continue to provide a most satisfactory method for the on site quality control of cement bound minestone it does not provide a sensitive measure of durability.

10.5 Resistance Indices

Resistance indices provide a measure of the effects of a durability test on the compressive strength of a specimen.

10.5.1 Resistance Index. BS 1924:1975 (69) defines the Resistance Index (R.I.) as the ratio of the compressive strength following immersion to the strength of a similar specimens cured at constant moisture content for 14 days. The immersion test (69) is likely to be included in a forthcoming revision of the Department of Transport Specification for Road and Bridge Works (9). It is anticipated that the test specimens will be required to have an R.I. of not less than 80 per cent (101), although there does not appear to be any substantive evidence to support this criterion. The results of immersion tests on cement bound minestone suggest that the R.I. does not:

a) indicate the extent of any deterioration,

b) demonstrate if such deterioration has ceased or is continuing.

The most resistant specimens, produced from the Snowdown-2 minestone, had an R.I. of only 65 per cent yet the strength retained at the end of the test was higher than that of other minestone specimens which had R.I. values exceeding 80 per cent. Furthermore, the volume change data for the Snowdown-2 specimens suggested that the effects of immersion had ceased before the end of the test. It is concluded that the R.I. should not be used alone to judge the immersion resistance of cement bound minestone specimens.

10.5.2 Freeze-Thaw Index. The Freeze-Thaw Index (F.T.I.) is defined as the ratio of compressive strength at the end of the test to that of a similar specimen cured at constant moisture content for 7 days. Packard and Chapman (73) suggested that the F.T.I. does not provide a sensitive measure of the resistance of materials having an F.T.I. of between 90 and 145 per cent. The F.T.I. of the cement bound minestones ranged from 0 to 110 per cent. It is concluded, therefore, that the F.T.I. should not be used alone to judge the resistance of cement bound minestones. The results suggested that the F.T.I. of minestone specimens displaying an acceptable degree of dimensional stability exceeded 80 per cent.

10.6 Retained Strength

The strength retained by a specimen subjected to immersion or freeze-thaw test provides a measure of the absolute quality following 7 days immersion or 12 cycles of freezing and thawing. Like other strength related criteria, it does not indicate the degree of resistance or the extent of any deterioration and should not be used alone to judge the durability of cement bound minestone. The results of tests on minestone specimens showed that the strength retained by the more resistant specimens exceeded 3.5 N/mm^2, ie the minimum 7 day strength required by the Department of Transport (9). It could be argued that 3.5 N/mm^2 represents the lowest strength required to ensure the structural integrity of the pavement and, therefore, providing the effects of immersion or freezing and thawing, have ceased before the end of the test, specimens which retain a higher strength may be considered to possess adequate durability.

10.7 Measuring the Durability of Cement Bound Minestone

It is recommended that any laboratory studies of cement bound minestone should include the B.S.I. Immersion Test (69) and a modified A.S.T.M. Freeze-Thaw Test (57). The method of preparing the specimens and the test procedures have been described in Section 8.2 and 8.3 respectively. The procedures include regular monitoring of both the dimensions and mass of the specimen, as well as the usual strength determinations. These tests are considered to be appropriate to cement bound minestone. The immersion test provides a measure of:

(a) The effect of sulphates which are present in all minestone,

(b) The susceptibility of the mudstone component of a minestone sample to inter-particle swelling associated with the adsorption of moisture.

The freeze-thaw test provides a measure of the susceptibility to cycles of freezing and thawing which are believed (23) to simulate the forces generated by moisture content. Such a study should examine the effects of both cement content and initial moisture content on performance on the durability test.

10.8 Criteria for Assessing the Durability of Cement Bound Minestone

The results of the experimental studies suggest that the judgement of the performance in the durability tests should be based upon both volume change and strength. The following criteria are considered to be appropriate to cement bound minestone.

(a) <u>Immersion Test</u>

Volume Change:	On completion of the test the volume of the specimen should be stable and should not exceed the original volume by more than 0.3 per cent.
Strength Retained:	The strength retained by specimens satisfying the volume change criterion should exceed 3.5 N/mm^2, ie the minimum 7 day strength required by the D.O.T. (9).

(b) <u>Freeze-Thaw Test</u>

Volume Change:	The 12th cycle frozen volume should not exceed the 1st cycle frozen volume by more than 0.3 per cent.

Freeze-Thaw Index: F.T.I. > 80 per cent.

Strength Retained: The strength retained by specimens satisfying the above criteria should exceed 3.5 N/mm^2.

These criteria are provided for guidance and must, eventually be correlated with field behaviour. Ideally, all such criteria that are established by experimental testing are only valid for the particular material studied. Their extension to other materials is always liable to require some correction or modifications to render them meaningful for the other/new materials. Indeed, the apparent discrepancy in the results of freeze-thaw tests on the Hem Heath specimens (Section 9.4.3) may be an indication of the need to develop a specific criterion for individual materials. The Hem Heath specimens would be rejected on the basis of volume change but both the F.T.I. and the retained strength indicate satisfactory performance in the test. The volume expansion could be a result of surface disruption, which is unlikely to damage the core of the specimen, so that the retained strength values would be significant, whereas volume expansion due to disruption within the specimen would be likely to lead to a severe loss of strength. Certainly, when interpreting such a result, all the information should be considered before taking a decision on the performance of an individual material.

CHAPTER 11 CONCLUSIONS

11.1 Introduction

The quality and durability of a number of cement bound minestones have been studied in the laboratory. The programme of tests examined the compressive strength, the resistance to immersion in water, and the resistance to cycles of freezing and thawing. The results of the tests suggest that the current methods for assessing cement bound materials, for consideration in the construction of public roads, are not appropriate to cement bound minestone. Alternative methods of measuring and assessing the durability of cement bound minestone have, consequently, been proposed. Although these methods were established by tests on cement bound minestones, it is believed they should also apply to other cement bound materials.

11.2 General Conclusions from the Study of Compressive Strength

1. The compressive strength of cement bound minestone appeared to be governed largely by the grading of the raw material.

2. Minestones containing less than 10 per cent material finer than 75 um satisfied the Department of Transport's 7 day strength requirement (9) at cement contents considered to be economically viable. The comparatively high strength of these materials was attributed to:

(a) their granular nature which promotes the conventional structure of strong particles coated with cement and bonded together at the points of contact,

(b) the relatively high dry density and hence low voids content of the compacted material.

3. Minestones containing more than 20 per cent material finer than 75 um failed to satisfy the current 7 day strength requirement when mixed with economically viable amounts of cement. The low strength was attributed to:

(a) their cohesive nature which resulted in a material comprising weak particles in a poorly stabilised fine matrix,

(b) the low dry density and hence high voids content of the compacted material.

4. The compressive strength of specimens produced from a laboratory blend of gravel, sand, and brickearth was over 100 per cent greater than that of the strongest cement bound minestone specimens. The high strength was attributed to very high dry density and a correspondingly low voids content. Indeed it may be that this reference material was closer to dry lean concrete than to cement stabilised soils produced with the minestone.

5. The compressive strength of certain minestones was increased when the initial moisture content was raised above optimum.

11.3 General Conclusions from the Experimental Study of Durability

11.3.1 General. The resistance of cement bound minestone specimens to both immersion and freezing and thawing, appeared to be governed largely by the source of the raw material and, to a lesser degree, by the cement content. The ability to satisfy the minimum 7 day strength requirement (9) of the Department of Transport did not necessarily guarantee satisfactory performance in the durability tests. The well graded, coarser grained minestones were, generally, more resistant than the poorer graded finer grained minestones. Specimens produced from minestones containing more than 30 per cent material finer than 75 um failed to complete either test. The dimensional stability of the minestone specimens was generally improved when the initial moisture content was raised above the optimum. The degree of this improvement depended on the source of the minestone and ranged from marginal to significant, with the resistance of Snowdown-2 specimens being improved considerably when the initial moisture content was raised 2 per cent above optimum. The laboratory blend displayed a very high degree of resistance to both tests.

11.3.2 Immersion Test.

1. All the minestone specimens expanded when immersed in

water. This expansion was associated with a moisture gain.

2. The degree of expansion during the test appeared to be governed mainly by phenomena associated with the breakdown of the mudstone fragments present in the minestone. This breakdown has been attributed to inter-particle swelling caused by the adsorption of water onto clay particles. The expansion was also influenced, although to a lesser degree, by the presence of sulphates.

3. Expansion during the test was reduced, albeit by varying degrees, when the initial moisture content was raised above the optimum value. It was concluded that raising the moisture content provided additional water for adsorption prior to compaction. The potential, therefore, for further adsorbtion and hence inter-particle swelling, was reduced. All the specimens produced at two per cent above the optimum had stopped expanding before the end of the test.

4. The compressive strength of cement bound minestone was, almost without exception, reduced during the immersion test.

5. The laboratory blend displayed a very high degree of resistance to immersion. The expansion during the test was negligible and the compressive strength appeared to be unaffected.

11.3.3 Freeze-Thaw Test.

1. The initial freezing contraction of the minestone specimens was greater than can solely be explained by thermal effects. The additional contraction was attributed to the removal of adsorbed water from the surfaces of the clay particles as moisture migrated towards ice forming in the larger pores.

2. At the end of the first cycle, all the minestone specimens had expanded above their original volumes. This expansion was accompanied by a moisture gain.

3. The behaviour of specimens during subsequent cycles varied considerably. The more resistant specimens contracted when frozen and expanded when thawed. During the test the amplitude of these cyclic changes diminished slightly, whilst the frozen dimension increased marginally. The less resistant specimens underwent considerable expansion during the test. The amplitude of the cyclic changes diminished rapidly and, by the end of the test, the specimens were expanding when frozen and contracting when thawed.

4. The resistance of cement bound minestone specimens to freezing and thawing is almost certainly influenced by their susceptibility to the phenomena responsible for the expansion when subjected to the immersion test ie inter-particle swelling and the presence of sulphates.

5. The stability of both the volume and mass of cement

bound minestone specimens improved as the initial moisture content was raised above the optimum value. This improvement is attributed to a reduction in the disruptive effects of inter-particle swelling. All the specimens produced at optimum plus 2 per cent displayed a high degree of stability during the freeze-thaw test.

6. The compressive strength of the minestone specimens was, with the notable exception of the most resistant Snowdown-2 specimens, reduced during the freeze-thaw test.

7. The laboratory blend displayed a very high degree of resistance to freezing and thawing. The cyclic changes in volume were almost entirely due to thermal effects. The effects on the development of compressive strength was negligible.

11.4 Conclusions Regarding Durability Criteria

1. The current method of assessing cement bound materials is not appropriate to low quality materials such as minestone. It places great emphasis on the 7 day compressive strength, but does not adequately assess the probable durability of such materials.

2. The Department of Transport Specification for Road and Bridge Works (9) should be revised to include a criterion for judging performance in the immersion test. Furthermore, consideration should be given to the introduction of a test to measure the resistance to

freezing and thawing.

3. Criteria have been proposed for assessing the resistance of cement bound minestone specimens to immersion and freezing and thawing. The results of the experimental study suggest that dual criteria, based on volume change and compressive strength, are to be preferred to the single strength loss criterion currently applied to the immersion test.

4. The criteria proposed for cement bound minestones are, probably, also valid for other cement bound low quality materials.

CHAPTER 12 RECOMMENDATIONS FOR FURTHER WORK

12.1 Introduction

This research has been confined to an experimental study conducted on laboratory specimens. Whilst such laboratory studies provide valuable information concerning the quality and durability of cement bound minestone, they do not replace the experience to be obtained from full scale field experiments. It is recommended that future research into cement bound minestone should, where possible, relate the information and experience obtained from tests on laboratory specimens to the performance of the cement bound minestone used, particularly, by the National Coal Board for the construction of haul roads, hardstandings, etc. The correlation of laboratory results with field performance will help to validate, or otherwise, the durability criteria proposed in chapter 10. The proposals for further work include, therefore, both laboratory scale and field scale studies.

12.2 Laboratory Scale Studies

(1) The experimental study of durability should be extended to other cement bound minestones being considered for use in pavement construction. It would also be interesting to test a range of cement bound materials to study their responses using the criteria developed for cement bound minestone. This could include other industrial by-products such as quarry waste, china clay sand and slate together with such natural materials as

chalk and silty sands.

(2) A programme of laboratory study should be implemented to examine, further, the phenomena responsible for the expansion and/or disruption of cement bound minestone. In such a study it is recommended that emphasis should be placed on changes occurring at the cement-minestone interface, and so full use should be made of such techniques as thin-sectioning, SEM and pore fluid expulsion.

Both programmes would be used to substantiate the durability criteria derived in this study and, where appropriate, modify them to produce a good correlation between laboratory and field performance.

12.3 Field Scale Studies

(1) When a cement bound minestone is used for pavement construction samples of the material used should be subjected to a programme of tests, conducted on laboratory specimens, which include the immersion and freeze-thaw procedures described in chapter 6.

(2) Whenever possible, newly constructed pavements incorporating cement bound minestone should be the subject of a programme of core sampling. The cores are to be obtained at various ages commencing as soon as is practically possible after compaction and continuing until, at least, the end of the first winter season following construction. Tests on these cores should

include immersion and freeze-thaw procedures similar to those used for laboratory specimens. Furthermore the cores should analysed for the presence of expansive or disruptive minerals using the techniques indicated in section 12.2(2).

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APPENDIX A1 REGRESSION ANALYSIS OF STRENGTH DEVELOPMENT DATA

A1.1 Semi-logarithmic Regression Analysis

A1.1.1 Regression line

$$S = A + B.\log T$$

where: S = compressive strength - N/mm^2

T = time - days

$$A = \overline{S} - B.\overline{\log T}$$

$$B = \frac{Z_{\log T.S}}{(Z_{\log T})^2}$$

$$(Z_{\log T})^2 = \Sigma \log T_i^2 - n.(\overline{\log T})^2$$

$$(Z_S)^2 = \Sigma\ S_i^2 - n.(\overline{S})^2$$

$$(Z_{\log T.S}) = \Sigma \log T_i .\ S_i - n.(\overline{\log T}.\overline{S})$$

$$\overline{S} = \frac{\Sigma S_i}{n}$$

$$\overline{\log T} = \frac{\Sigma \log T_i}{n}$$

n = 4

i = 7,14,21&28 days

A1.1.2 Coefficient of correlation

$$r = \frac{Z_{\log T.S}}{(Z_{\log T}).(Z_S)}$$

A1.2 Logarithmic Regression Analysis

A1.2.1 Regression line

$$S = A.T^B$$

or $$\log S = \log A + B.\log T$$

where:

S = compressive strength – N/mm^2

T = time – days

$$\log A = \overline{\log S} - B.\overline{\log T}$$

$$B = \frac{Z_{\log T.\log S}}{(Z_{\log T})^2}$$

$$(Z_{\log T})^2 = \Sigma \log T_i{}^2 - n.(\overline{\log T})^2$$

$$(Z_{\log S})^2 = \Sigma \log S_i{}^2 - n.(\overline{\log S})^2$$

$$(Z_{\log T.\log S}) = \Sigma \log T_i . \log S_i - n.(\overline{\log T}.\overline{\log S})$$

$$\overline{\log S} = \frac{\Sigma \log S_i}{n}$$

$$\overline{\log T} = \frac{\Sigma \log T_i}{n}$$

$n = 4$

$i = 7,14,21\&28$ days

A1.2.2 Coefficient of correlation

$$r = \frac{Z_{\log T.\log S}}{(Z_{\log T}).(Z_{\log S})}$$

A1.3 Results

The results of the regression analysis are given in Table A1.

Table A1 Results of Regression Analysis

Material	Cement Content	Semi-log Analysis $S = A + B.\log T$			Log Analysis $S = A.T^B$		
	(°/o)	A	B	C	A	B	C
Snowdown-1	5	4.0	1.33	0.93	4.2	0.105	0.93
- do -	10	6.4	1.64	0.98	6.8	0.084	0.98
- do -	15	6.6	3.57	0.99	7.3	0.145	0.99
Snowdown-2	10	2.2	6.93	0.95	4.5	0.306	0.95
Hem Heath	4	0.0	3.78	0.98	1.4	0.403	0.99
- do -	8	0.0	5.50	0.93	2.2	0.383	0.95
- do -	12	-0.6	7.59	0.96	2.7	0.405	0.97
Wardley	6	3.7	2.50	0.99	4.2	0.166	0.99
- do -	12	6.8	2.90	0.80	7.3	0.121	0.81
Parkside	6	-	-	-	2.0	0.155	-
- do -	12	2.7	2.21	0.98	3.2	0.186	0.98
Blidworth	6	2.6	0.57	0.91	2.6	0.075	0.92
- do -	12	3.4	1.41	0.71	3.6	0.122	0.72
Bagworth	6	1.4	1.36	1.00	1.7	0.200	1.00
- do -	12	2.5	1.05	0.57	2.7	0.114	0.53
Peckfield	6	0.1	1.23	0.96	0.6	0.356	0.98
- do -	12	0.3	1.86	0.93	1.0	0.336	0.94
Lab. Blend	4	5.4	5.60	0.95	6.4	0.227	0.97
- do -	8	14.9	6.72	0.91	15.5	0.143	0.94
- do -	12	21.5	7.47	0.75	21.7	0.120	0.78

APPENDIX A2 CLAY MINERAL ANALYSIS.

The mineralogical analysis of the clay fraction of the various samples was determined by the X-ray Diffraction technique. The work was carried out by the Department of Geological Sciences under the supervision of Dr. J. Morton, who also assisted with the interpretation of the diffractograms. A full description of X-ray diffractometry is given by Zussman. (1)

The source of radiation used was Cobalt Kα, selected because it pushes up the diffraction angle, and reduces background noise, in the area of interest. The samples were mounted by the suction method. In this method the samples are sucked through a ceramic disk and are, therefore, orientated - ie they are flat.

Each sample was scanned three times as follows:-

Scan No.	Treatment.
1	untreated
2	glycolated
3	heated (550^{o}C)

The X-ray diffractograms obtained are shown in figures A.1 to A.7

A very approximate estimate of the proportion of each mineral present in the clay fraction was determined by measuring the area under each of the peaks in the diffractogram obtained from the untreated sample.

(1.) Zussman, J. Physical Methods in Determinative Mineralogy. 2nd. edition. Academic Press, London. 1977. pp. 391-473

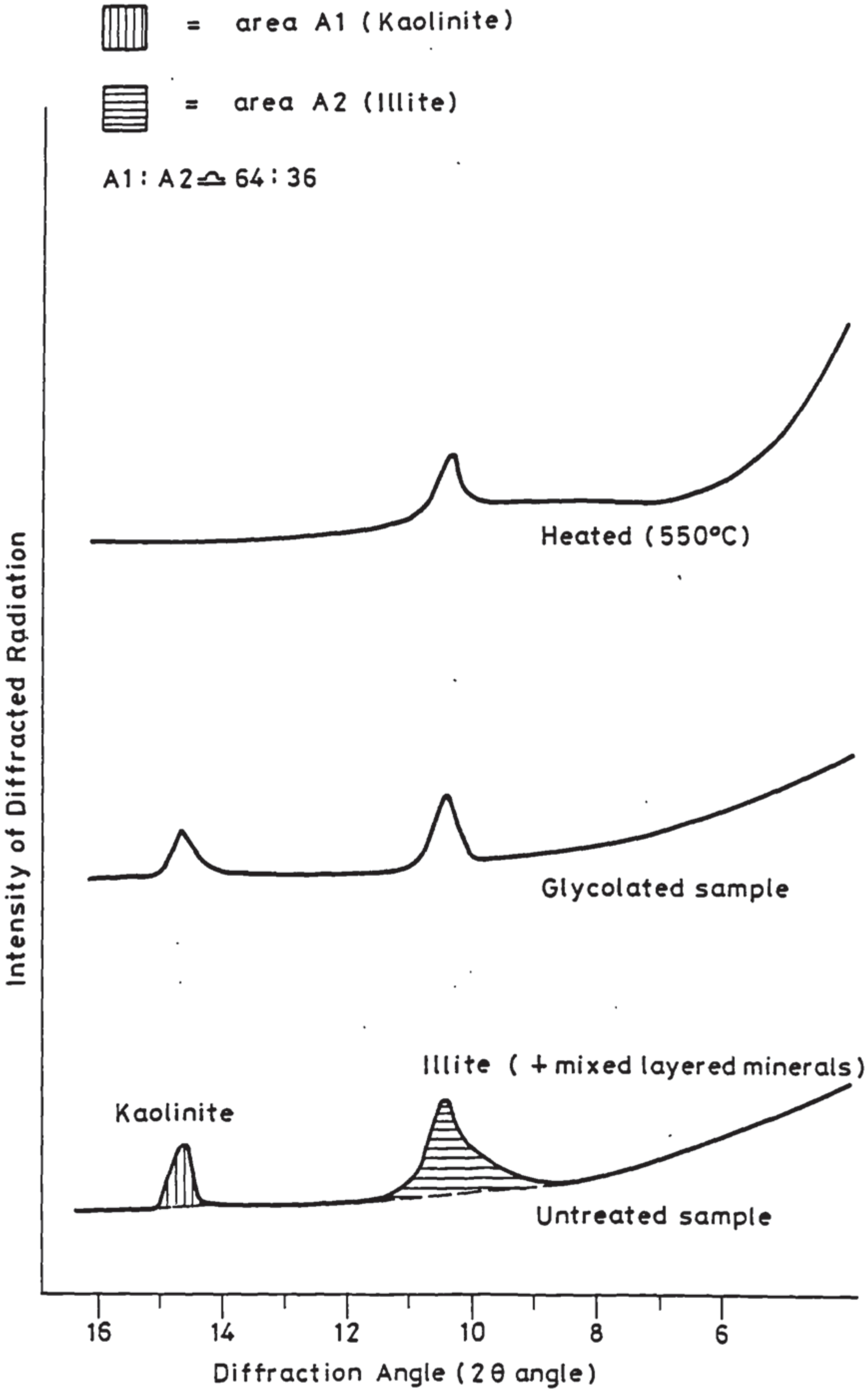

Figure A.1 X-ray Diffractograms of Snowdown-1 Clay Fraction

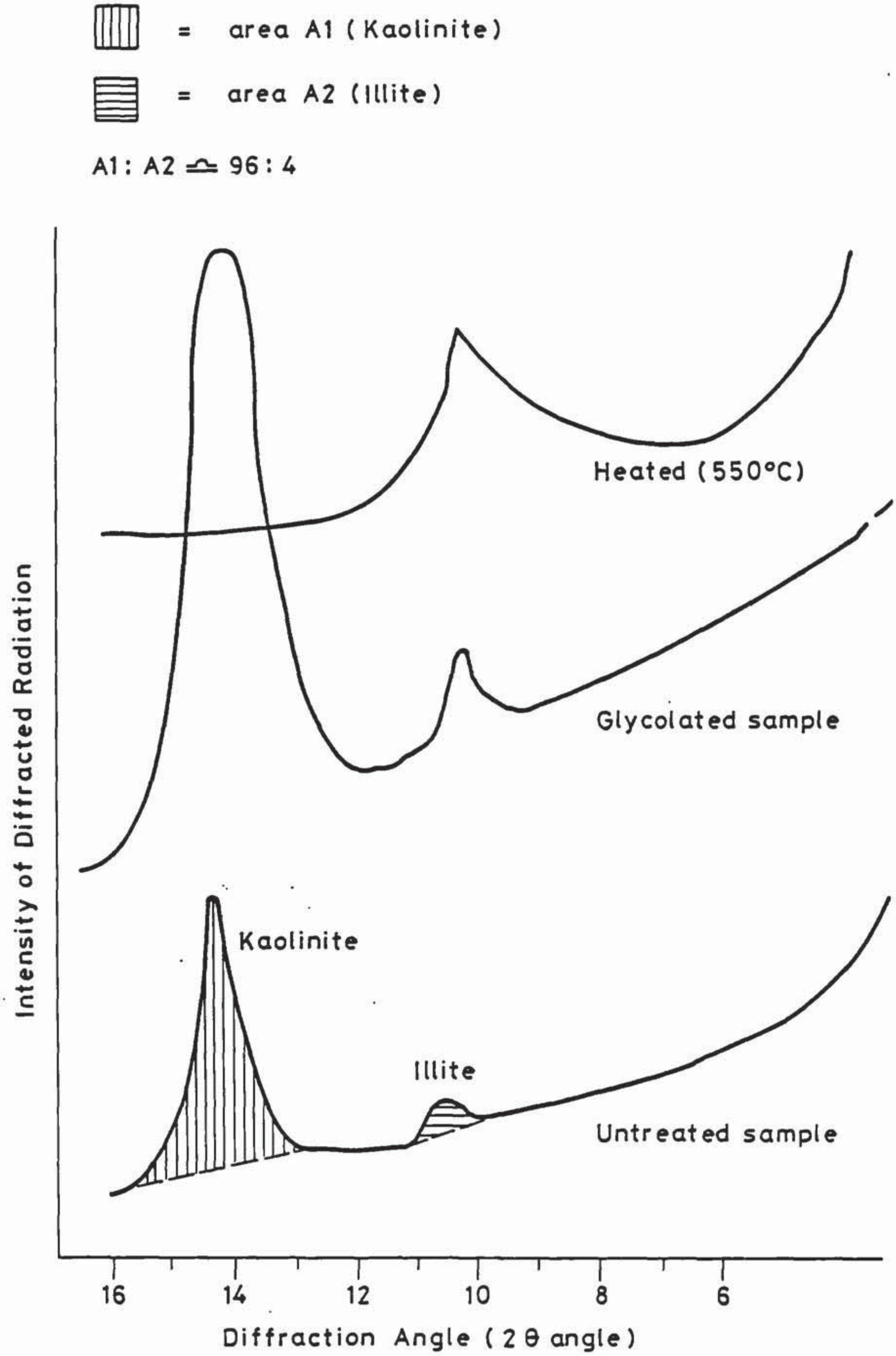

Figure A.2. X-ray Diffractograms of Hem Heath Clay Fraction

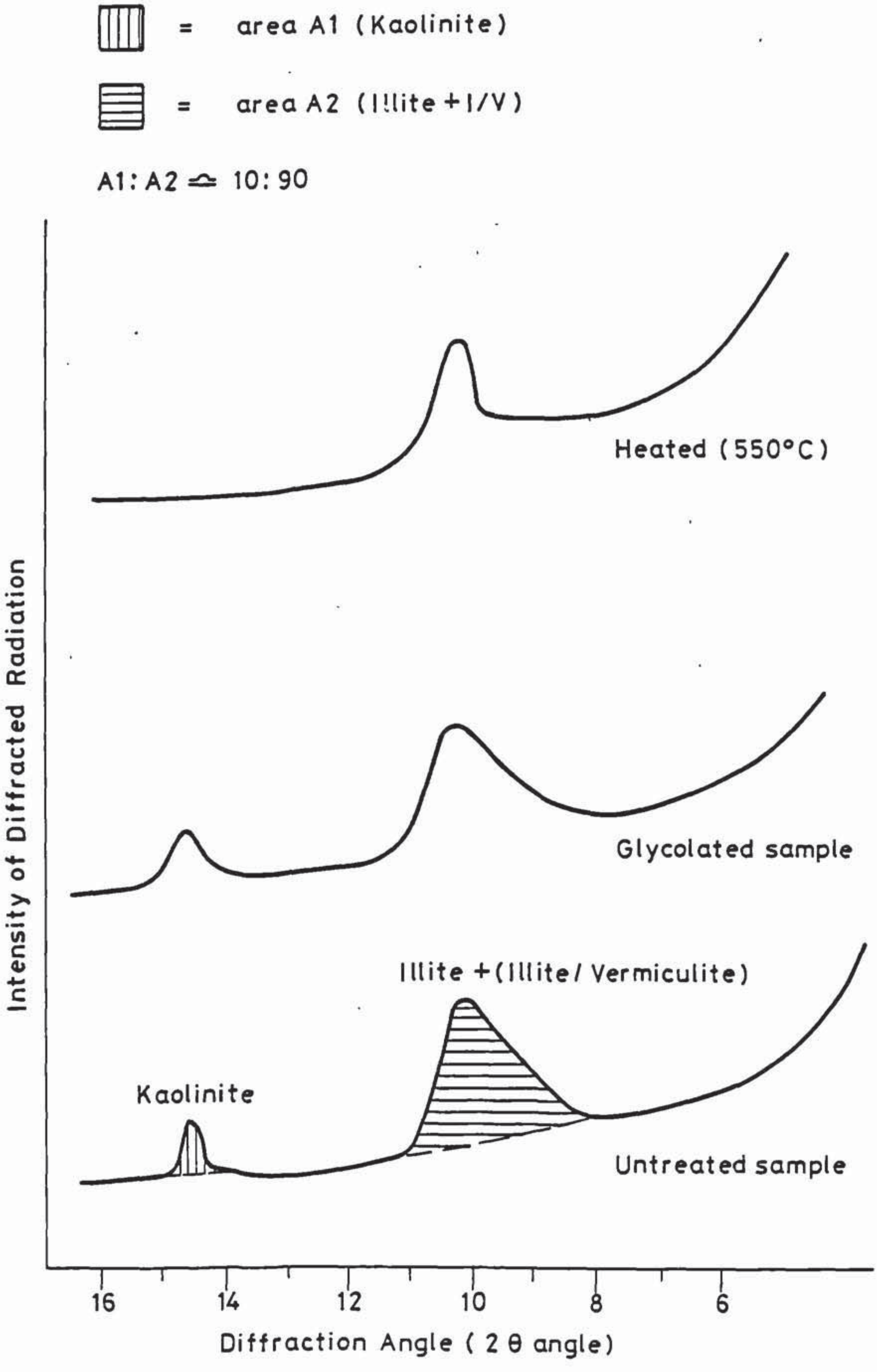

Figure A.3 X-ray Diffractograms of Parkside Clay Fraction

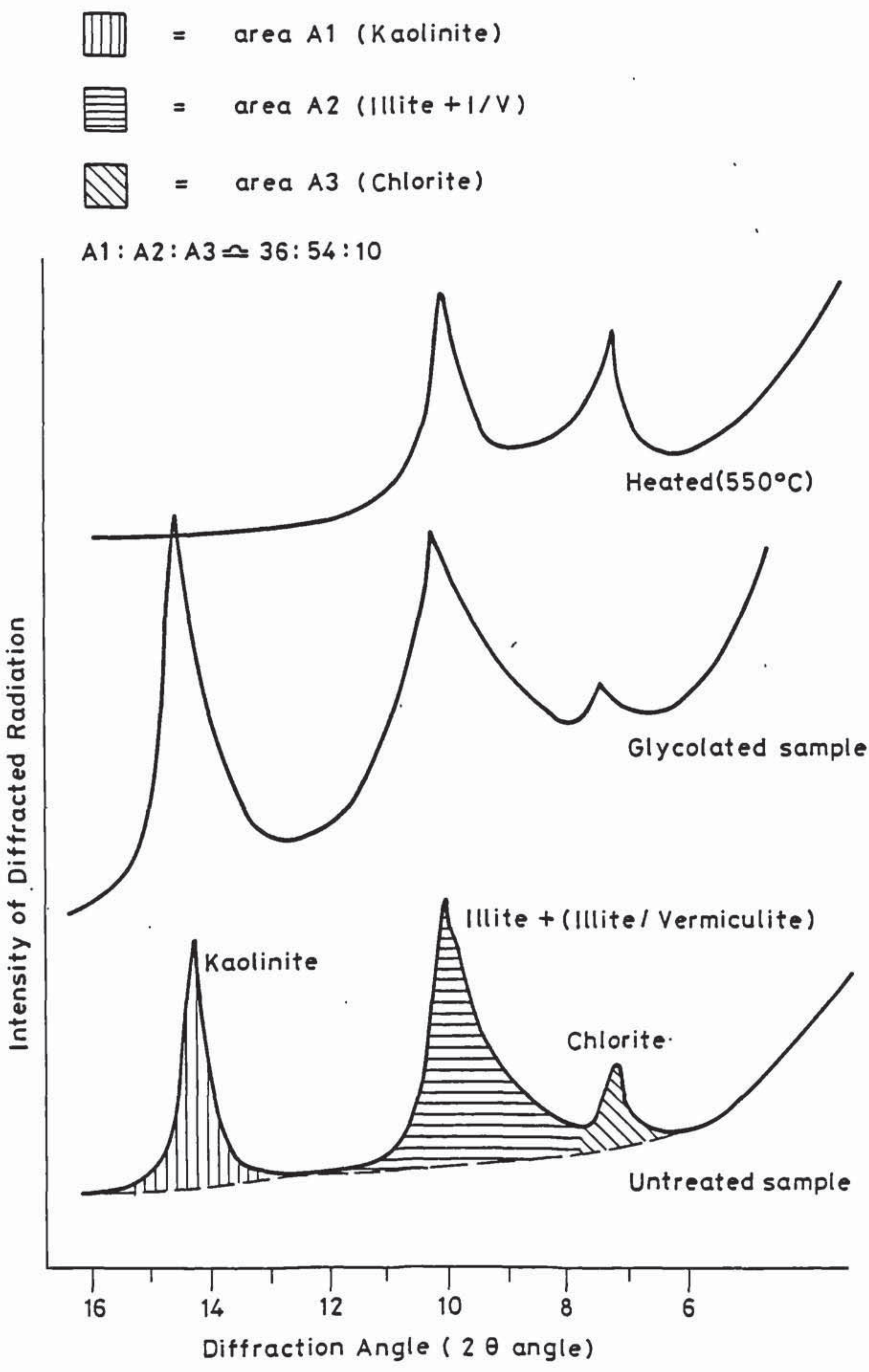

Figure A.4 X-ray Diffractograms of Blidworth Clay Fraction

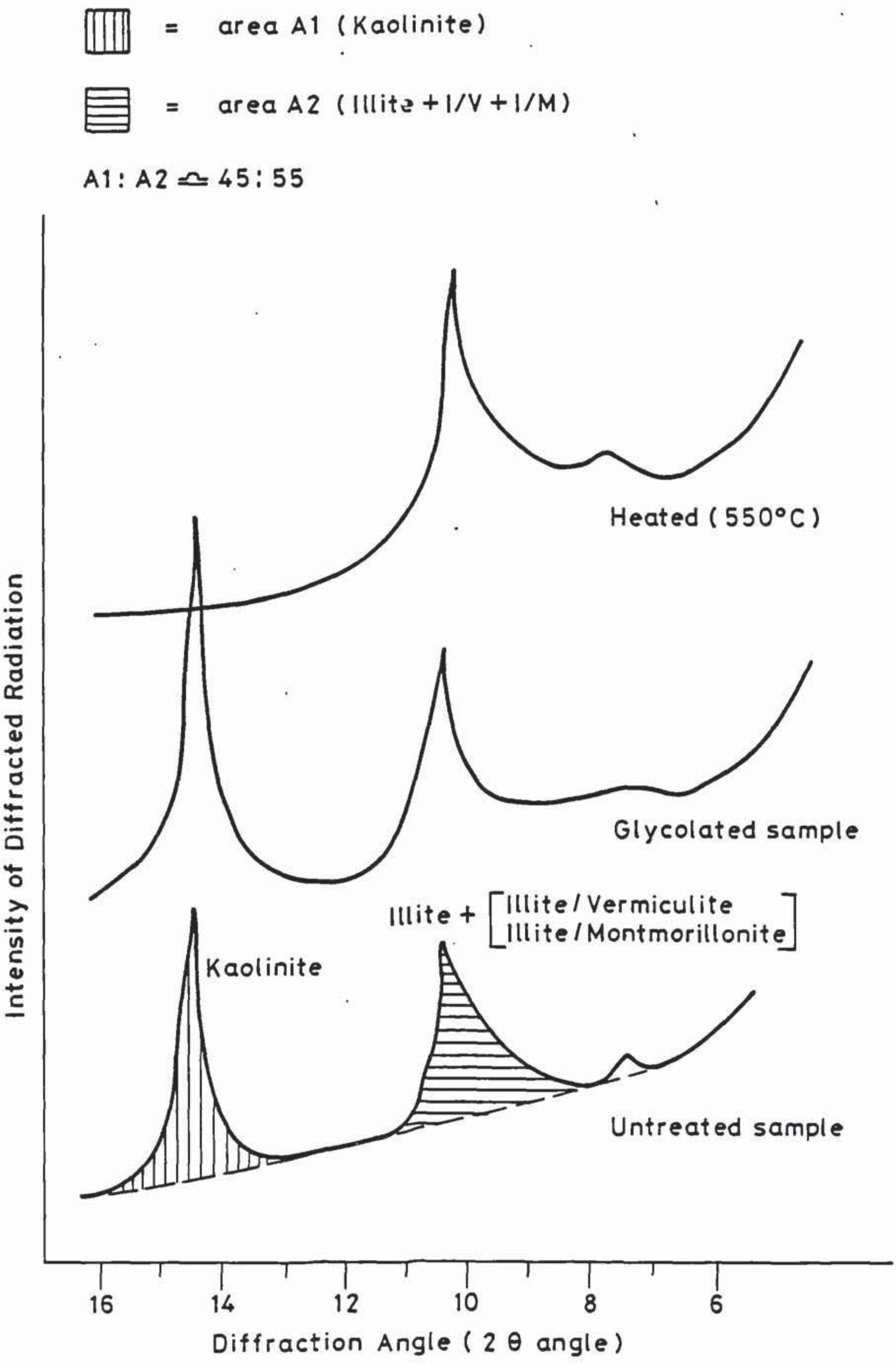

Figure A.5 X-ray Diffractograms of Bagworth Clay Fraction

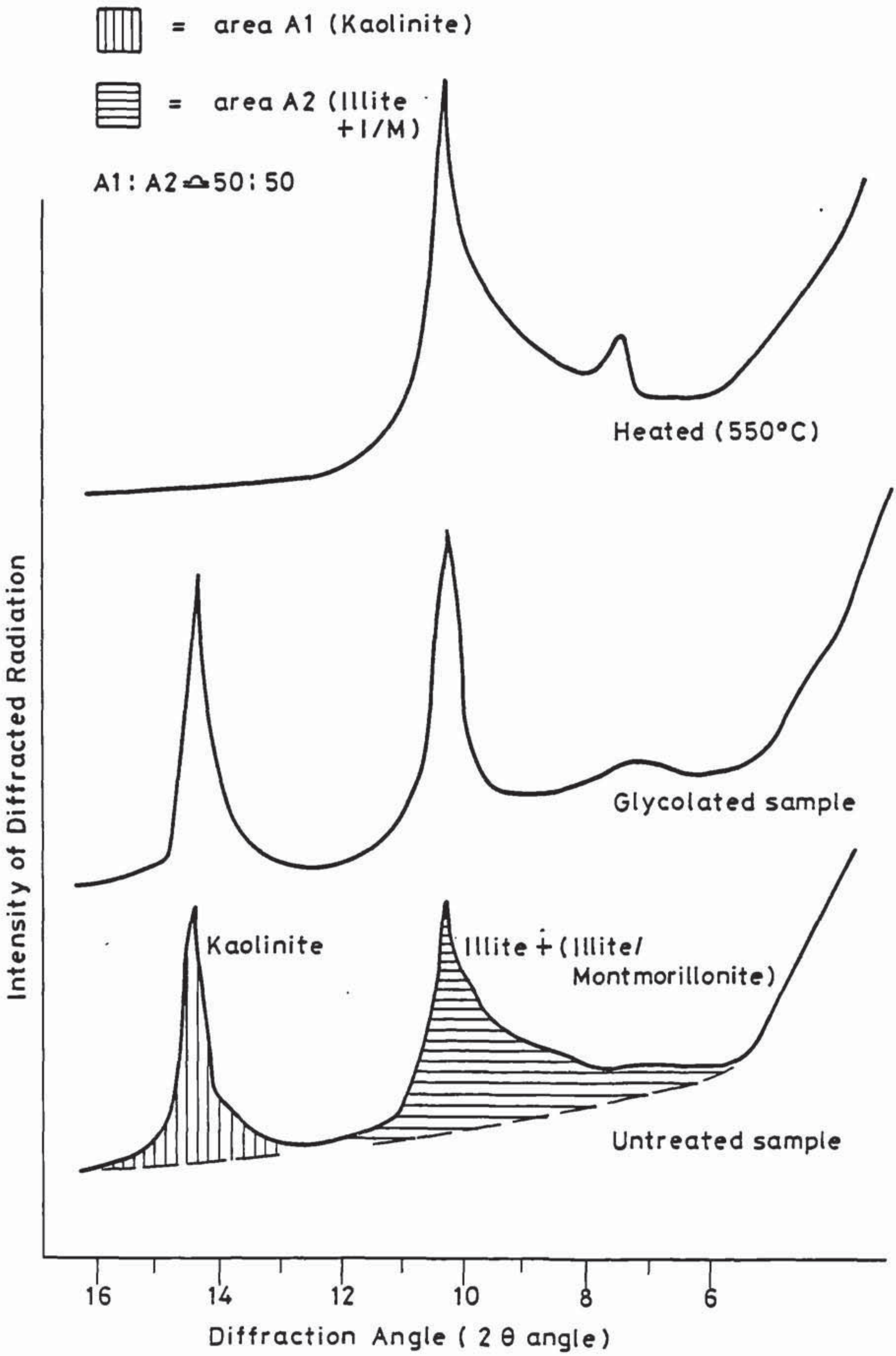

Figure A.6 X-ray Diffractograms of Peckfield Clay Fraction

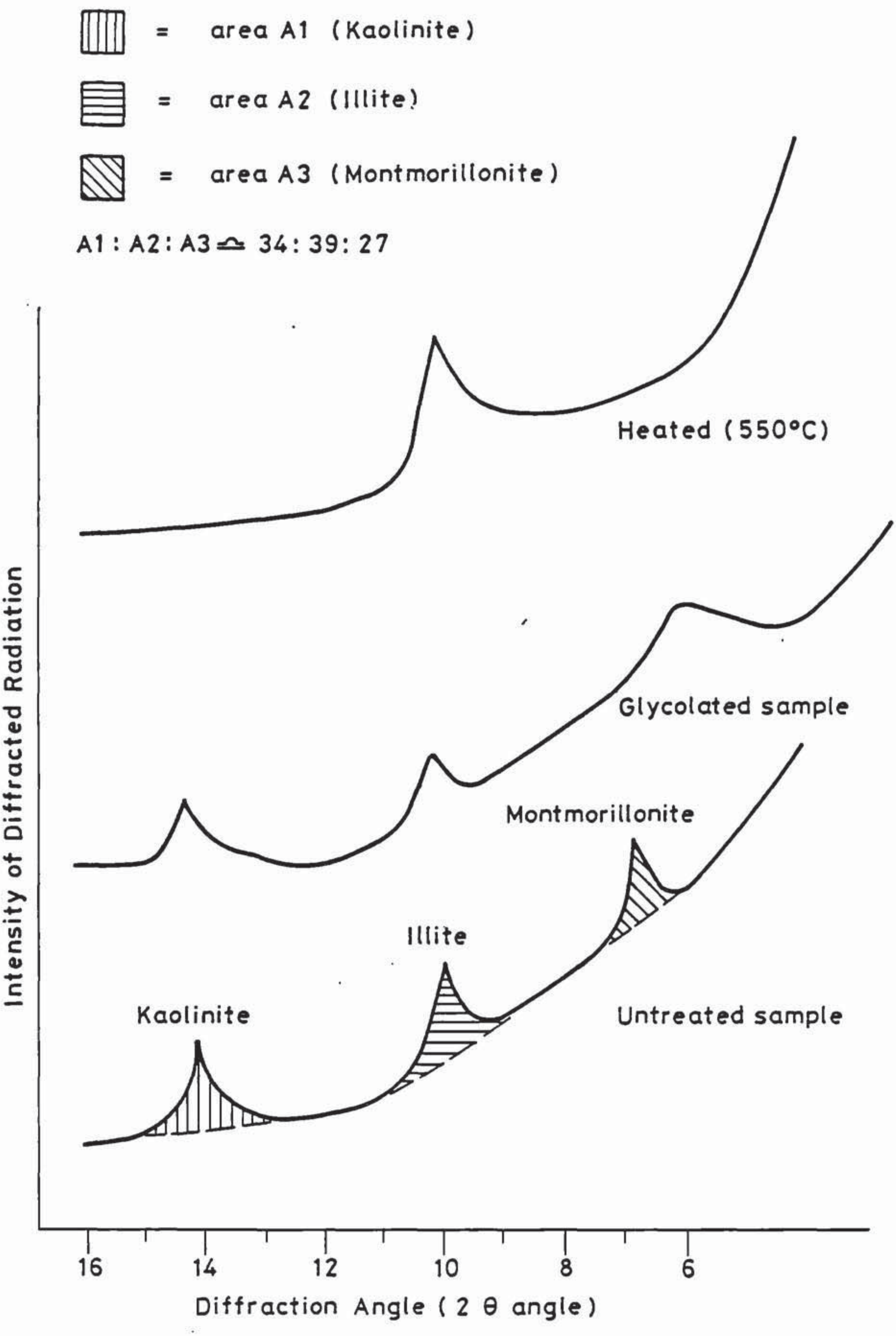

Figure A.7 X-ray Diffractograms of Lab.-blend Clay Fraction

APPENDIX A3 METHOD FOR DETERMINING THE CATION EXCHANGE CAPACITY OF CLAYS

1. Soil Samples

   Air dried at 20°C and graded to suit requirements.

2. Saturation

   Take approximately 2.0 g of soil and weigh accurately (1 mg) and place in 250 ml conical flask; add approximately 100 ml of 0.2 N Barium Nitrate solution. Agitate for 2 hours.

3. Cleaning

   Filter through double* millipore filter (0.45 um); wash with double distilled water in 10 ml amounts until filtrate shows no precipitate sulphuric acid.

   * double, as a control filter, once per set

4. Leaching

   Remove filter with sample (ensure ALL soil is transferred) to 250 ml beaker, wash filter with 100 ml precisely of 0.1 N nitric acid. Agitate by hand or magnetic stirrer for 10 minutes and allow to stand. (Filter to remain submerged).

   Repeat with clean filter as control.

5. Atomic Absorption Sample

Pipette a clean 50 ml sample of the leach, centrifuge if necessary and store in a clean 50 ml plastic bottle.

Repeat with clean filter as control.

6. Ba Determination

Barium concentration in leach to be determined by Atomic Absorption Spectrometry.

Calcs.

Ba conc in leach (mg/ml) = Ba taken up by sample

$$= w$$

$$\frac{w}{\text{wt of sample}} \times 100 = \text{ug of Ba for 100 g sample of soil}$$

$$\frac{w \times 100}{\text{wt of sample}} \times \frac{1}{1000} \times \frac{2}{137.36}$$

$$= \text{m equiv/100 g soil}$$