THE CHARACTERISATION OF THE PLASTIC PROPERTIES OF MASONRY MORTARS

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ABSTRACT

This programme of work was initiated as a result of recent major changes within Europe that have impacted upon the type of masonry mortars that are in use and the appropriate test procedures.

A new suite of test method standards, the EN 1015 series, has been issued by CEN over the last few years and supports two new mortar standards, BS EN 998-1 and BS EN 998-2, which are for plastering and rendering and masonry mortars respectively.

In addition, there has been a large increase in the use of factory made mortar. Factory production, particularly as a dry mortar, leads itself to the production of more complex mortar formulations, and some mortars now contain as many as eight separate admixtures. These changes have lead to a large increase in the number of mortars that are highly air entrained, whilst the use of retarders and other admixtures in these mortars means that they frequently have pronounced thixotropic and other time dependant properties. All of these changes have in turn led to greater complexity in the fields of test methods and procedures.

This thesis describes an investigation to define the properties of plastic mortar from first principles, then to compare these properties with the results produced by existing test methods to confirm if the latter were adequate. It was found that there were some properties for which several test methods existed, but some for which there were none. A test programme was then developed to investigate in detail and then to compare

some relevant test methods, including new European ones and the older British ones that they replaced.

Finally, two new, procedures were developed, one based on rheometry, one on the measurement of the early heat of hydration. Recommendations are made that these test procedures be further developed with a view to their adoption as standard tests.

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NOTATION AND ABREVIATIONS

ACI	American Concrete Institute
AEA	Air entraining agent
AFNOR	Association Francaise de Normalisation
ASTM	American Society for Testing and Materials
BRE	Building Research Establishment
BS	British Standard
CAA	Cement admixtures association
C&CA	Cement and Concrete association
C_2S	Di calcium silicate
C_3A	Tri calcium aluminate
C_3AH_6	Tri calcium alumino hydrate
C_4AF	Tetra calcium alumino ferrite
C_3S	Tri calcium silicate
$C_3S_2H_6$	Calcium silicate hydrate
CEN	European Committee For Standardization (Comite Europeen De Normalisation)
Cp	Centipoise
CR	CEN report (in BSI)
d	Shear
D	Sieve size in BS EN 13139 through which 100% of the sand must pass
d.g.a	Differential gravimetric analysis
DIN	Deutsches Institut Fur Normung
d.t.a	Differential thermal analysis

e.m.f	Electromotive force
EN	European Standard
F	Stress
F_o	Yield value
ISO	International Standards Organisation
nr	No retarder
OPC	Ordinary Portland cemant
PD	Published (bsi) document
PFA	Pulverised fuel ash
r1	Radius of the inner cylinder in the Haake Viscometer
r2	Radius of the outer cylinder in the Haake Viscometer
ret	Retarder
RILEM	Reunion Internationale des Laboratoires et Experts des Materiaux, Systemes de Constructions et Ouvrages
RPM	Revolutions per minute
SD	Standard deviation
U	Plastic viscosity
μm	Micron

1. INTRODUCTION

The work covered in this project evolved within RMC Materials in its earliest stages, as increasingly sophisticated mortars were developed and manufactured at a network of producing plants and three laboratories, one concentrating wholly on research and special projects. As Kingston University became a leader in the field of masonry collaboration was both logical and fruitful.

Much of the work was carried out in RMC's laboratories, which were particularly well equipped for research into mortar and the related test methods, and provided an excellent adjunct to the Kingston facilities. For many years work at RMC had investigated the plastic and hardened properties of mortar and from 1988 until the present date had been closely involved in the evolution of the new European mortar test methods, which culminated in the BS EN 1015 series of standards, published from 1999 until 2004. Even now, following publication, work continues on these standards with the first revisions and minor corrections already in hand.

Although RMC produced mortars for plastering, rendering and floor screeding, this research programme was restricted to masonry mortars and these were only of the general purpose type as defined in BS EN 998 as "masonry mortar without special characteristics".

This clearly excluded specialist mortars, some of which are becoming increasingly used. Lightweight masonry mortars are often required where thermal properties are important, which is now commonly the case in both the UK and continental Europe following on from the important environmental conferences that culminated in the Rio Accord and Kyoto Protocol, with their emphasis on reducing global warming and energy consumption. Thin joint mortars also find increasing application and their use benefits masonry structurally as well as thermally. These materials are currently being studied at Kingston but were not addressed in detail in this project although one specialist test method, that for correction time, was appraised.

The research programme also excluded very specialist materials like repair mortars, grouts and related void filling materials.

1.1 DEFINITIONS, BACKGROUND AND CONTEXT

In the context of this work, mortar is defined as a mixture of binder, aggregate and, optionally, admixture, used for the construction of masonry or for screeding. Fillers and/or additives may also be included in the material. Although today in most of the developed world the binder is usually Portland cement, or at least is based on Portland cement, and the aggregate invariably sand, this is not inevitably the case and certainly was not always the case. Historically a wide variety of aggregates and binders, including various soils, organic materials and fibrous matter have been used, as discussed more fully in the literature survey which is chapter 2 of this thesis. Binders in ancient history could be forms of ashphalt, gypsum or hydraulic lime. Various pozzolanas were used, together with a plethora of naturally occurring materials that acted as admixtures. Although not strictly within the scope of this project some of these are addressed in areas of the work where relevant, as although often dismissed as outdated and of historical interest only, they sometimes assist in a greater understanding of fundamentals that are still valid today.

In a wider context, the word mortar is often used to describe the finer phase in a concrete, primarily the cement and sand. Whilst not necessarily incorrect, this can be confusing because although perhaps outwardly similar, the two may have very different properties and requirements. This does not mean that work carried out on the latter is not of relevance to masonry mortars, merely that it must be carefully considered. Indeed, in some cases, the work may actually be directly applicable to masonry but in many cases will only be of limited application as the mortar properties will be inappropriate. Additionally and importantly the substrate, application and actual service environment are likely to differ greatly.

Concrete mortar in practice is often less homogenously distributed and less consistent throughout a member than is masonry mortar. This is because throughout the depth of a pour, particularly in tall sections, variations may occur in placing, consistence and compaction. Segregation and bleeding may also affect the homogeneity of the material throughout the depth of an element. These factors are not relevant in the case of most masonry mortars, although interface suction may have more effect throughout the whole of a mortar element, particularly as the suction of the masonry unit tends to increase and joint width to decrease, (the two possibilities not being linked).

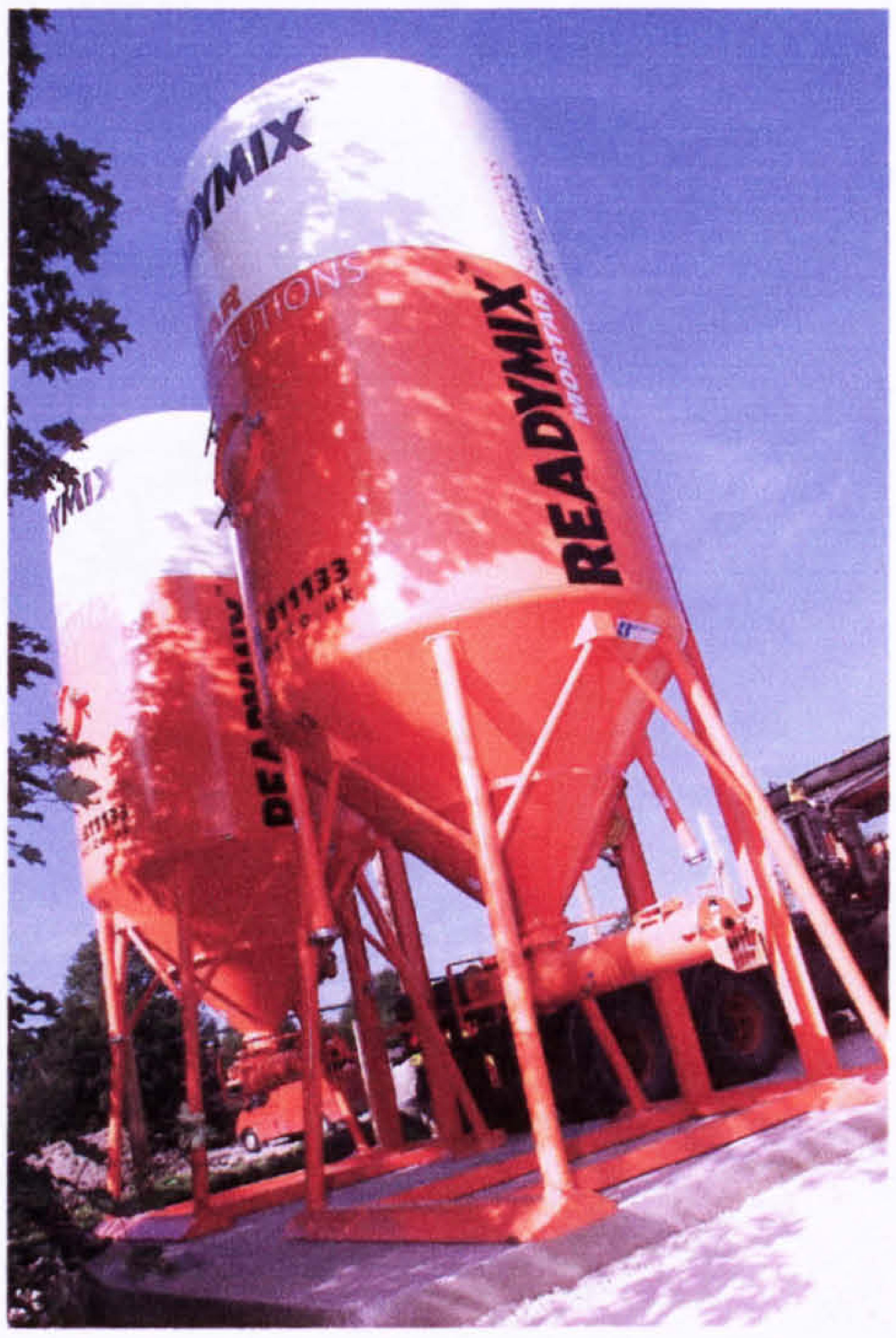
A wide range of cement types is covered by this project. Much work on mortars is restricted to Portland cements, often to ordinary Portland cement alone, although a great variety of materials are used as mortar binders. The range of available materials for which current requirements exist has now been standardised within much of Europe by the publication of the new European cement standard EN197 and it is this range of materials that are considered in the context of this work. Thus mortar binders will be taken to include cements based on Portland cement clinker that can be both unblended but also as a blended composite with pozzolanic materials like pulverised fuel ash, ground granulated blastfurnace slag and finely divided limestone.

Further cement types have recently become more widely used in some specialist applications and in particular, those based on various forms of calcium sulfate find application in floor screeding mortars, which flow into place with little requirement for the input of placing energy and are often referred to as "flow applied". Although these screeding mortars are not generally covered within this work, the applicability of existing tests and the accuracy and applicability of the stated requirements will also be considered for some of the cement and binder types used therein.

The range of mortar types in general usage in much of Europe has also increased widely within the last decade with a much greater amount now being made away from the building site in purpose made factories. These factories range in size from small batching plants making mortars and perhaps concretes in the wet state, to multi million pound factories making specialist dry mortars. All of these forms of mortar are generally referred to as factory made or ready to use mortars.

The wet ready to use mortars are basically composed of cement, sand, water and perhaps lime and other admixtures or additions, with the addition of a chemical cement set retarder. They are produced to a consistence that is appropriate for use without the need for further addition of water or mixing and are delivered to the building site in transit mixing vehicles, (truck mixers,) or in specially designed purpose made vehicles. Dry ready to use mortars are either produced in bags or, more usually for the larger building sites, for delivery and/or use in silos. These invariably contain an integral mixer and provision for water and electrical power to be connected, as shown in figure 1.1 below.

Figure 1.1 A typical dry silo mortar container



Both developments have resulted in a large increase in the amount of factory made mortars with known cement contents, and a concomitant reduction in the amount of site made materials where the achievement of the correct cement content and mix proportions may sometimes be in doubt.

These developments have brought with them the need for new test methods, particularly in the case of the retarded materials where effective and convenient ways of measuring their early life properties were lacking at the time that they began to be placed on the market. Appropriate lime types will also be considered as binders, as blends with Portland or other cements and as the sole binder in the mortar. Here too changes have taken place within the last decade, with less lime being used in mortars overall in Europe and North America, although the perception remains in parts of the industry that the addition of lime to cement based mortars is beneficial, particularly with respect to the plastic properties.

In the context of this project, sands will cover predominately natural fine aggregates, the term now adopted for sands, although for clarity and to differentiate this material from the fine aggregate fraction of concrete, the term sand will continue to be used in this work, although artificial aggregates are not expressly excluded. The definition taken from the new standard (BS EN 13139, 2003), now adopted throughout the CEN member states, does not include or address the term sand but instead states "*fine aggregate is the designation given to the smaller* <u>*aggregate*</u> *sizes with D less than or equal to 4mm*", (authors underlining).

BS EN 13139 requires that 100% of the material is required to pass a sieve size of 2D, ie 8mm. This appears to permit material that would be too coarse for use in most mortars but the UK published guidance document (PD 6682-3:2003), gives a requirement that for masonry mortars 100% must pass the 4mm sieve, a slightly more practical grading for most masonry mortars than that given in the superseded British Standard (BS 1199 and 1200:1996).

In this work, the definition of a mortar sand is not, however, necessarily constrained by this 4mm upper bound, as for some applications, particularly in repair and conservation and for some stonework, a larger maximum may well be acceptable. Instead it is taken to be "a material that is mainly finer than 5 millimetres", a definition based on a now obsolete British Standard, but one that is nevertheless helpful.

As well as grading, the particle shape is also taken into consideration, as this interacts with grading, which when usually expressed tends to assume a reasonable degree of sphericity.

The consideration of mortar properties and requirements is based on international criteria, rather than being restricted to the UK or even to Europe. The test methods considered are similarly based although the majority will be European.

All of the requirements of mortar are grouped into two categories, those for the hardened properties and those for the plastic properties. This is a robust classification, serving equally for all mortar types including specialist mortars like those used for floor screeding, grouting and other less widely used applications, but is only useful as a very general overview.

Although at first sight the hardened properties appear to be of far greater interest than the plastic properties, the focus of this work is very much on the latter for several reasons. Firstly, it is an area that has attracted only a minority of recent research interest, the majority being directed towards the hardened properties, which has meant that much of the work in this field, and many of the test methods that are still in use are rather dated and sometimes lacking in accuracy and scientific rigour. (As an example, one current American test for plastic properties was published in 1920 when work on modern rheological concepts was unknown, Emley, 1920).

Secondly, the much greater use of factory made mortars with accurately known cement/binder contents and thus in general more reliable hardened properties seems to have directed the recent focus of attention more at the plastic properties. Lastly, there has been a great upsurge in the amount of bricklaying and blocklaying that is carried out by sub contract bricklaying companies, which now execute the vast majority of works on large and medium sized contracts, with the bricklayer directly employed by the main contractor being a real rarity. Many of these sub contractors are substantial companies employing several hundreds of bricklayers.

This change has also been accompanied by a pronounced reduction in the supervision of work by Clerks of the Works and/or related supervisory personnel. Much masonry, therefore, is now effectively carried out on a basis that relates solely to the number of units laid or the area of masonry produced, with the focus of the sub contractors increasingly on productivity, with little regard for longer term hardened properties. This has meant that the subcontracted masons look very closely at the plastic properties, as by optimising these from their point of view, to assist the laying process, they can maximise their incomes.

1.2 THE OBJECTIVES OF THE WORK AND A GENERAL OUTLINE

This project was undertaken whilst working with a specialist mortar producing company and having close involvement in the development of new European standards for products and test methods. As a result of that work it had become more and more apparent that the tests used for the determination of many of the properties of mortars were inadequate. They produce numbers and values, and these are frequently used for comparison, but it is often unclear what basic properties are actually being measured. The situation obtained particularly in respect of the plastic as opposed to the hardened properties.

This led to the test development and programme of work that initiated this project, culminating in a much improved test for the setting characteristics that is not only much more accurate and reproducible than the existing standard tests, but is also easier and more practical and measures a definable and precise parameter. In addition, the work led to a recommendation for the use of a more sophisticated test procedure, based on rheometry, for use in the very early life plastic state of the mortar.

It was also clear that the tests used for the determination of many of the earlier life plastic properties were inadequate but because they were usually being used in a specialist environment by very experienced personnel, their experience and knowledgeable application of the tests enabled some of the inadequacies to be masked.

Nevertheless, it was obvious that the tests in use did not fully reflect some of the key properties of the material. The majority of tests are said to measure a single property but actually measure two or more interacting properties, with it not being possible to separate and quantify the contribution of each. Moreover, these key properties and their influences do not generally appear to be fully understood. As factory made mortars were developed, they were modified much more with admixtures. Amongst other applications, these were used to obtain high air contents, to prolong setting times, (perhaps for several days) and on occasions, for application to high suction surfaces, to resist the sometimes deleterious effects of substrate suction in the plastic state.

At the same time as these trends were occurring another influence became increasingly important. As bricklayers became more scarce and in many cases formed sub contracting companies, the demands for the plastic properties to be more and more "operative friendly" increased. The final hardened properties of the mortars gradually assumed a lesser role, the benefits of clean, so called "well-graded" sands became virtually forgotten, and initial ease of use became more and more a consideration. As testing of site made mortars became minimal, these were often adulterated with domestic detergents and were rarely made using washed sands. Dirtier materials containing silt and clay were favoured because they were easier to use, with apparently superior plastic properties (as assessed by the operatives), although these often subjective assessments were not examined to determine exactly what judgemental criteria were used.

As factory made mortars were required to achieve equal ease of use they were increasingly formulated with higher air contents and additional sophisticated admixtures became more commonplace. Even these materials sometimes contained sands that were not washed.

These influences produced mortars with increasingly complex and interacting rheological properties. The sands sometimes reacted with the admixtures in a manner that was unpredictable in terms of the plastic properties, yet quantitative test methods for studying these matters were not in use. Even the basic mortar properties themselves appeared to be rarely appreciated, with the influence of the thixotropic nature of most of the systems and the instability of some of the higher air contents often being ignored

This work was intended to quantify these individual properties and requirements in much more detail than had usually been the case, and to start with a rigorous consideration, based on first principles, of exactly what the plastic properties actually comprise. For instance, it is common to refer to the workability when consistence would be more appropriate. In addition, one-point tests, that is tests that measure only one parameter without fixing or measuring other relevant parameters simultaneously, are often used for the plastic properties that are not really appropriate for non-Newtonian fluid systems like mortars. There is also often a lack of clarity concerning exactly what some of the existing one-point tests are measuring and it was hoped to clarify this.

It is commonplace to refer to the use of rheological measurements or rheological parameters whereas rather than use this rigorous scientific approach the commentators merely used inadequate one point tests like slump to BS EN 12350-2, 2000 or dropping ball value, BS 4551-1, 1998. Although many of these tests are said to measure a unique property it is suggested that they are often measuring the same general parameter and this too was to be investigated in the context of a science based rheological background, rather than empirical tests based on ease of use and robustness of equipment. Rheology, often thought of as the science of fluids, but more accurately the science of the deformation and flow of matter, Domone, 1999, has been used to develop sophisticated production and control procedures in many other industries, for example in foodstuffs, lubricants and health and beauty products, but has never really been used within the field of masonry mortars, even though the issues arising with mortars are very relevant to rheological considerations.

It was hoped that the programme would lead to a position enabling either the validation of one or more of the existing test methods, perhaps in modified and improved form, or the proposal of alternatives better able to fulfil the function of quantifying the properties and requirements in an objective and reproducible way.

2. REVIEW OF LITERATURE

This chapter reviews previous work in the fields to be studied. It is divided into sections, each covering a key aspect, as discussed below.

The requirements and properties of mortar are considered initially in the terms in which they were originally defined by users and technologists, and an attempt is then made to classify or group these into logical categories that integrate with contemporary terminology. This is not easy but an objective approach is sought, although many of the definitions are subjective in varying degrees and some are difficult to interpret.

The next section covers the tests and test requirements. This is hoped to serve both as a comprehensive treatment of the majority of the procedures that are said to have proved successful, and also a critical review of some of the new European test methods that were in the process of introduction at the time that much of this work was being carried out.

The final section covers some aspects of rheology and viscometry but only those that are of direct relevance to this investigation and necessary for their use as tools in the work, rather than in the degree of depth appropriate to a specialist study of those subjects.

2.1 TRADITIONAL REQUIREMENTS FOR THE PLASTIC MORTAR PROPERTIES

The earliest of all references of relevance to mortars, found in the writings of the great Roman writer Vitruvius, stated that mortar was required to "bind ...the stones together", Vitruvius, c 27 BC. Palladio, 1570, identified rapid hardening and durability as needs whilst Ronelet, 1812, identified and tested for compressive strength, tensile strength and adhesion, as was recommended later by the Royal Institution of British Architects, 1911. Many references cover the perhaps rather obvious designed hardened properties of strength, often in bond and compression, sometimes in tension. The majority of mortar specifications rely on references of this form, generally simple and non-demanding although there are exceptions. Thus in respect of hardened properties, Perander and Raman, 1985, stated that for rendering mortars a low modulus of elasticity is desirable, failure rate increased with the modulus of elasticity of the mortar, with major fluctuations in temperature and moisture not leading to defects in low modulus mortars.

Alternatively, the properties may be specified obliquely by closely prescribing the mix proportions and material properties, as in the procedure used by Baronio et al, 2000, who used type of binder, type and grading of aggregate and binder:aggregate ratio to characterise mortars used for historical matching. Plummer, 1962, reported on work carried out at the University of Wisconsin and the University of California, and suggested that, although the data were not conclusive, there appeared to be an optimum ratio of lime to cement for the attainment of maximum tensile bond to clay brick of between 1 and $^1/_4$ and recommended that for what he termed highly plastic limes and for moderate brick suctions the ideal ratio was 1 to $^1/_2$, but this suggestion was based only on experimental work and no accompanying theoretical basis was given.

Plummer also reported that the use of more plastic limes increased the water retentivity substantially, as did the use of air entrainment, but again this conclusion was based on a very limited amount of data.

Finally, Plummer stated that the strength of mortars depended to a large degree on the binder content and the water cement ratio and that *since in most masonry structures, compressive strength of mortar is of secondary importance to bond strength, workability and water retentivity these properties are usually given primary consideration in specifying mortars*. This is an interesting comment and it is believed that Plummer was certainly correct in principle with this hypothesis but perhaps lacked accuracy in his detail, as it is rare to find the plastic properties of mortar that he believed to be important actually being specified in practice.

Some workers in the field addressed compressive strength, flexural strength and elasticity, as for example Bromlet, 2000. Other recent references have included much more comprehensive methodologies for the characterisation and testing of the hardened properties of the mortar, thus Middendorf et al, 2000, listed a great number of hardened properties, *colour, density, porosity, pore size distribution, capillary water absorption under atmospheric pressure and under vacuum, drying behaviour, water vapour permeability, freeze thaw resistance, the coefficients of thermal and moisture expansion, compressive strength, bending strength, adhesive strength, dynamic modulus of elasticity and resistance to abrasion.* This is a comprehensive list of hardened properties but of course successful attainment of these is partially dependent on the plastic properties, as the interaction of the wet mortar with the bricks and blocks will profoundly affect some of the hardened properties listed.

Specific references to plastic properties are less common and usually much less comprehensive, thus Teutonico et al, 1994, stated in the preamble to the Smeaton research project that the plastic properties that were of concern were workability, moisture content, rate of hardening (sic) and shrinkage. It is often not appreciated that the initial and early life interaction with the units, particularly if they have high suction rates, has a major influence on the final hardened properties, as outlined above. This has now been largely accepted in Germany, where a doctoral thesis has addressed this issue, Reichers, 2000, and where a DIN standard specifies tests on mortars that have been subjected to the suction of a unit prior to testing, DIN 18555-9, 1999. Harrison, 1983, in a BRE published note, embodied this principle in his test for mortar durability, casting specimens against a real background, to achieve a realistic pore structure in his test mortars.

One of the earlier scientifically based workers to be specifically concerned with the plastic mortar properties was Emley, 1920, who identified the importance of water retentivity and devised equipment to measure this, as discussed further in section 2.2.2. He realised that a mortar could spread easily, and apparently possess good plasticity, but be deficient in its reaction to high suction backgrounds. Emley was quite advanced in his thinking, stating that a mortar with enhanced water retentivity would spread further than a comparable mortar with reduced water retentivity. Emley also identified a further plastic property, which he called the sand carrying capacity. Sand carrying capacity is not a term used in current scientific terminology and is dependant on several properties, rather than being a fundamental property. Emley proposed nearly one hundred years ago, that the property was related to the plastic properties of the binder and the sand grading. Other factors not identified by Emley or even present at that time, such as air content and interaction of some complex admixtures such as cellulose ethers, if present in the system, also have a profound influence.

More recently, Backman, 1953, stated that the plastic properties were affected by the grain size distribution and the water retention of the lime where present, going on to suggest that admixtures could be used to improve them. Perander and Raman also stated that surface cracking, adhesion and application were influenced by the workability and while discussing workable mortars did not state how these should be characterised. Piepenburg, 1970, stated that workability was more important than strength.

It was recently stated that *Plasticity, water retention, soundness and sand carrying capacity are perhaps the most important properties of mortar,* Thompson, 2000. Thompson also quotes Emley, 1920, as stating that "spreadability" was a key property, and one that was tested for in the Emley plastimeter still in use today in the USA, ASTM C 110, 2003.

The majority of references to the wet mortar properties, (more correctly the plastic properties), use imprecise and sometimes confusing terminology, often using the word workability to encompass all of the working properties without further analysis, sometimes using workability as an alternative expression for consistence. This is a fundamental concern because the word and property "workability" can have meanings that are different but often confusingly so, as they are nevertheless reasonably similar in some ways. Thus, in pure scientific interpretation, the term may be taken to relate closely to the amount of work required to move, deform, penetrate or similarly act upon the material. However, in concrete technology, which is sometimes used to cover mortar, it is often used purely and simply as the value obtained from a standard slump test, (for example using BS EN 12350-2, 2000), which although perhaps simple is hardly correct. Confusingly, however, other tests for concrete plastic property values are also said to measure workability, for example, the degree of compactability test, BS EN 12350-4, 2000, which replaces the old British test for compaction factor in the UK, BS 1881-103:1993 and the vebe value, BS EN 12350-3, 2000, although perhaps the comparison with mortars is not strictly valid here as each of these concrete tests tends to be used with a particular concrete type, vebe for stiffer mixes, compaction factor for more flowable generally wetter ones.

The publication of EN 206 has now changed the imprecise term "workability" in the context of concrete to the more satisfactory term "consistence", which has been used in the field of mortars for several decades, although not always with precision, as in C&CA TDH 8425,1980, which states that *the minimum amount of water should be used and the mixture beaten with a rod to give a putty like consistency.* In contrast, the ACI, 1994, defines mortar consistence as *that property that determines the ease and homogeneity with which it can be mixed, placed, consolidated and finished.*

Other terms are also used in a loose and unclear context, as for example, Ashurst, 1997, who stated whilst discussing some forms of hydraulic limes, *these limes are harsher working,* which is clearly rather subjective. Early analysis of the plastic properties and terms is far less common than for the hardened ones, although in a report issued by the BRE as far back as 1927, Cowper, 1927, stated that *plasticity...is a difficult property to define. Such simple statements as "the property which makes it easy to spread" and "the yielding property of a wet mixture to change of form" convey the general form but are lacking in scientific precision or quantitative meaning.* Later work sometimes used less analytical terminology, as for example Davidson, J., 1961, which stated that *The plasticity or workability of the mortar defines its ability to flow or spread into all the area of the brick face, the bumps and hollows, the cracks and crevices....* In later work he expanded on this definition, to say that, *workability is the property that enables a mortar to spread, when trowelled, into all cracks and crevices of the units. Workability is a combination of several properties including plasticity, consistency and cohesion.*

Although largely determined by aggregate grading and material proportions, the ultimate workability depends on water content, Davidson, 1976. It is believed that the property defined in the last sentence is consistence, although the earlier part of the definition is not actually inaccurate. Davidson went on to say that *It is the result of the roller bearing effect of the aggregate particles lubricated by the cementitious slurry,* which may be partially correct but which omits a great deal. In still later work, Davidson 1974, his definition became more complex and all embracing, as he stated that *In reality, it is a combination of several properties, including plasticity, consistency and cohesion.*

A recent definition, ASTM, 1993, describes workability as...*that property of freshly mixed concrete or mortar which determines the ease with which it can be mixed, placed, consolidated and finished.... This definition appears to have been taken from the identically worded one proposed by the ACI,1990.* Other workers also adopted the ACI definition, Rankine, 1998, and in this example went on to add that *Perhaps the closest quantifiable relative to workability is consistence, which describes the mobility or ease of flow, as measured by the "static" slump test or the more "dynamic" flow test.*

The ACI definition possesses some succinctness, but does not help to quantify or identify any individual contributing influences. In contrast to the looseness of expression used in some relatively recent references to concrete, as for example as cited above, the earlier work of Tattersall and Banfill 1983, had expressed the issue quite accurately, although their far seeing work was not widely adopted in the UK. Thus they suggested that there could be considered to be three groups of terms in the field, qualitative, quantitative empirical and quantitative fundamental, as outlined below with acknowledgement to those two authors.

Qualitative properties should only be used as general descriptive terms and not in an attempt to quantify, quantitative empirical should be used in a simple quantitative way and finally quantitative fundamental, are capable of rigorous quantitative analysis. These are shown in table 2.1 below, together with some of the properties that may be associated with them.

Table 2.1 after Tattersall and Banfill, the terminology of plastic properties

Qualitative	**Quantitative empirical**	**Quantitative fundamental**
Workability	Slump	Viscosity
Flowability	Compacting factor	Mobility
Compactability	Vebe time	Yield value
Stability	Flow	Consistency
Finishability	N/a	N/a
Pumpability	N/a	N/a

It is to be regretted that this treatment was not adopted in practice, nor does it appear that it was ever really applied to mortars as opposed to concretes, although it was hoped to use a related treatment in this current research programme. Returning to the use and definition of the term workability in the context of mortar, Plummer, 1977, used it correctly to encompass all of the working properties as opposed to just the consistence or flow and stated that it was an essential requirement that was readily recognised by the mason but for which there were no standard laboratory tests. He said that a mortar "*was workable if its consistency is such that it can be placed and spread with little effort, and if it has the property sometimes referred to as "stickability" or "stickiness" which causes* *it to adhere to vertical surfaces of masonry units immediately after placing"*, and that "*water retentivity, flow and resistance to segregation are factors affecting workability"*. He went on to say that this was easily recognised by the mason but quantitative estimates were hard to obtain and moreover varied greatly with observer. He classified mortars of low water retentivity, that is with a flow of less than 65% after suction as materials that would be judged as harsh by the operative and those with flows after suction of between 65 and 80% as of high water retentivity and likely to be judged as workable by the mason. His definitions are helpful and accurate, but do not tell the whole story, particularly in respect of his last statements concerning water retentivity.

Many other workers have also referred to plastic mortar behaviour similarly in terms of single property values, as for example Boynton, 1980, who stated that *putties ....continue to increase in plasticity* and Teutonico et al, 2000, who measured the flow of the mortars that they tested but did not relate the value to any other property.

The Portland Cement Association, 1976, suggested that *Workability is difficult to define because it is a combination of a number of interrelated properties. The properties considered as having the greatest influence on workability are: consistency (flowability), water retentivity, setting time, weight, adhesion and penetrability.* Isberner, 1969, proposed another useful and wide ranging statement, in that *The workability of a masonry mortar is difficult to define and equally difficult to measure. The masons appreciation of workability of plastic mortars depends on its (*sic*) ability to be spread easily, its ability to cling to vertical surfaces and its resistance to flow during placement of a masonry unit. In laboratory tests, workability is recognised as a complex rheological property that includes adhesion, cohesion, density, flowability, plasticity and viscosity. Although research continues to measure these individual* *properties, no one test method, per se, measures workability. In performing his task, the mason integrates these influences and arrives at a subjective determination of the mortars workability.* Beall, 1997, suggested a similarly helpful definition, in *Workability is not precisely definable in quantitative terms because there are no definitive tests or standards for measurement. Workability is recognised as a complex rheological property including adhesion, cohesion, density, flowability, plasticity and viscosity that no single test method can measure.* This is also a good definition but fails to define plasticity.

RILEM addressed the so called "complex workability-consistence-plasticity" issue for mortar with some rigour and accuracy in a paper issued by the mortar committee, RILEM, 1969. The paper stated that *The property of mortar called workability with components such as consistence (consistency), plasticity, water retention, cohesion, stiffening under suction and thixotropy determines the ability of the mason to do his work.* It then continued to give examples based on bricklaying and rendering. These were useful statements and remain valid today although again a definition of plasticity would assist. Bowler et al, 1996, stated that *a masons assessment of mortar workability includes its ability to hang on the trowel, spread easily to form an even mortar bed, adhere to vertical surfaces and allow placement of units without squeezing out of joints. This property has proved difficult to quantify and despite many attempts to harmonise test methods there are still a number of approaches.*

A recent CEN report, now a BSI published document, BSI, 2000, attempted to further elucidate these issues and to suggest how some of the contributing factors could be measured. The report stated ...*In contrast to the concept of workability as applied to concrete, workability in mortars is not just a question of adjusting the "wetness" of the* *mortar by adding more or less water. In masonry work the craftsman requires rather more of his materials in that he expects them to flow easily from the trowel and to spread on to the masonry unit evenly and without segregation.* The second statement is undoubtedly correct but it could be argued that the first sentence is rather dismissive of the rigour with which concrete may well be analysed for some applications, although the view taken is not uncommon. The document went on to attempt definitions of consistency and plasticity, stating that *It may be interpreted that consistence is a measure of wetness and could be measured using a penetration device but that plasticity requires a more dynamic assessment such as could be achieved by using apparatus which caused the mortar to move.* This approach represents an advance on most earlier thinking although is perhaps a little simplistic, and also fails to address the interaction between the two properties, with, as an example, the influence of the parameter described as plasticity on the consistence. Additionally of course even the penetration of the mortar by the dropping ball causes the former to move to a measurable degree.

Appendix 1 of this thesis proposes a rigorous nomenclature for the plastic properties, and suggests preferred terminology, in order to clarify this area.

There is a clearly a further key property of mortar in the plastic state that is rarely addressed and that is the time dependent change in the early plastic properties that is thixotropic or thixotropy related. An early reference, The Chalk Lime and Allied Industries Research Association, 1962, provides an excellent treatment of the phenomenon in the context of mortars containing lime but thixotropy is rarely mentioned in the majority of the literature, even though it is an important practical issue and is a factor in most mortar applications. As with the early views of Tattersall on the complex workability issues of concrete, the accurate but advanced analysis of this paper appears to have been overlooked in favour of much more simple, perhaps even simplistic, later treatments.

Recent changes in mortar composition and usage have meant that thixotropic behaviour is now an important characteristic of much mortar, far more so than has been the case with most mortars in the immediate past. This is because it is a time dependant phenomenon, with thixotropic thickening often developing with time and the increasing use of retarded factory made mortars has lead to an increase in the occurrence of the phenomenon. Many factory made mortars are retarded, for periods of up to 72 hours, perhaps greater, and during this period of time the development of a substantial degree of thixotropic stiffening is probable. Even dry silo mortars that are nominally unretarded usually contain some retardation to extend working life on the spot board. Interestingly, thixotropy would also have been a factor in historic mortars, albeit perhaps not identified as such, with lime putty based mortars being "matured" or "soaked", often for substantial periods of time, prior to use.

Thixotropy is considered further in section 2.3, the conclusions to the literature survey.

2.2 TESTS FOR THE PLASTIC PROPERTIES OF MORTAR

A large number of methods and procedures have been suggested for the testing of mortars but this section deals in detail only with those intended for use in connection with the plastic properties. These are dealt with in three parts. The first part is that concerned with the plastic properties at an early stage in the life of the mortar, immediately after and following cessation of mixing, continuing to a stage where thixotropic or other early time related phenomena are taking place, but before any discernable chemical set commences. The second part of this section is specific to water retentivity and related issues. The third part comprises the next stage in time, when setting mechanisms begin to exert a more profound influence and the fluid begins and then continues with the transformation phase into a solid. These two stages are not discrete. The material begins life as a fluid and as the first phase comes towards its mature stage, the second has increasing influence. As time proceeds, the second phase dominates more and more until it is predominant, then still more until the influence of the first phase is only minor and finally to a time when it ceases altogether. Thus the whole should really be viewed as one continuous process, starting as soon as mixing is complete and ending when the mortar may be described as hard. The adoption of classification into separate stages or phases is used in this work only as a convenience, and because the test methods for plastic properties in some respects tend to fall naturally into the two general categories outlined above.

Some workers in this field have used tests that were developed for other materials to test for the plastic properties of mortars, but without explaining whether this was due to a scientific judgement that they were more applicable, or because the test equipment was conveniently available, which appears often to have been the case. In one example, the CEN test for the setting time of cement was used, Marie-Victoire et al., 2000. As discussed below this may actually be a useful procedure for mortars, although it is not clear why it was used in the work cited above.

A British Standard or other slump test, BS 12350-2:2000 or similar, is sometimes used to determine consistence in mortars, Rankine 1998, but is almost certainly inappropriate as the amount of mortar used therein is so large that considerable amounts of bleeding and segregation may occur and these will distort the test result. The concrete flow table to BS EN 12350-5, 2000, has been used for testing retarded mortars where these are made in concrete plants and it is the only available piece of equipment. The Stanhope-Seta penetrometer, produced for use in the coated stone and food industries, and not called up in any mortar specifications so far as can be ascertained, was used in one well publicised research project, Teutonico et al, 1994.

Some test procedures not specifically designed for use with mortars can be used with varying degrees of success, as for instance the Vicat method for the determination of setting time of cement, BS EN 196-3:1995, and test results obtained using this procedure with mortars are discussed in section 4.4 and 5.1 of this work. Although the procedure seems to be of some use with mortars, and indeed was actually developed by Vicat for that purpose over one hundred and fifty years ago, Vicat, as reported by Livesey, 2002, in principle it does seem desirable to avoid a proliferation of test methods if at all possible.

The continuing use of a very old established test method is not restricted to the Vicat equipment. The so called viscometer originally developed by Emley nearly 100 years ago is covered by a current ASTM standard. It consists of an absorbent plaster block mounted on a turntable. The test specimen is placed onto this block and gradually raised and rotated until it contacts an upper disc. The torque applied to this disc is plotted and the points at which the material is no longer capable of plastic flow, the area under the curve and the time before the sample ruptures are said to indicate the plasticity and the resistance to suction or water retentivity.

This equipment is often said to measure the single property that is called plasticity in the context of the test, as originally defined many years ago in the USA by Johnson, 1926, who stated that *plasticity, as measured, was a determination of the relative resistance of* *the mortar to spreading when on an absorbent base.* This is indeed basically what the Emley equipment measures and it is still sometimes used by workers in the mortar field, even if they are specifically investigating the wider field of plastic properties. Thus Hansen et al, 2000, used the equipment for an in-depth investigation into the plastic properties of lime putties for mortar. He defined viscosity as consistence and workability as water retention and studied these properties individually by measuring the changes as water was added, although it is believed that the addition of water will result in a number of complex and interacting property changes, rather than change to a single property.

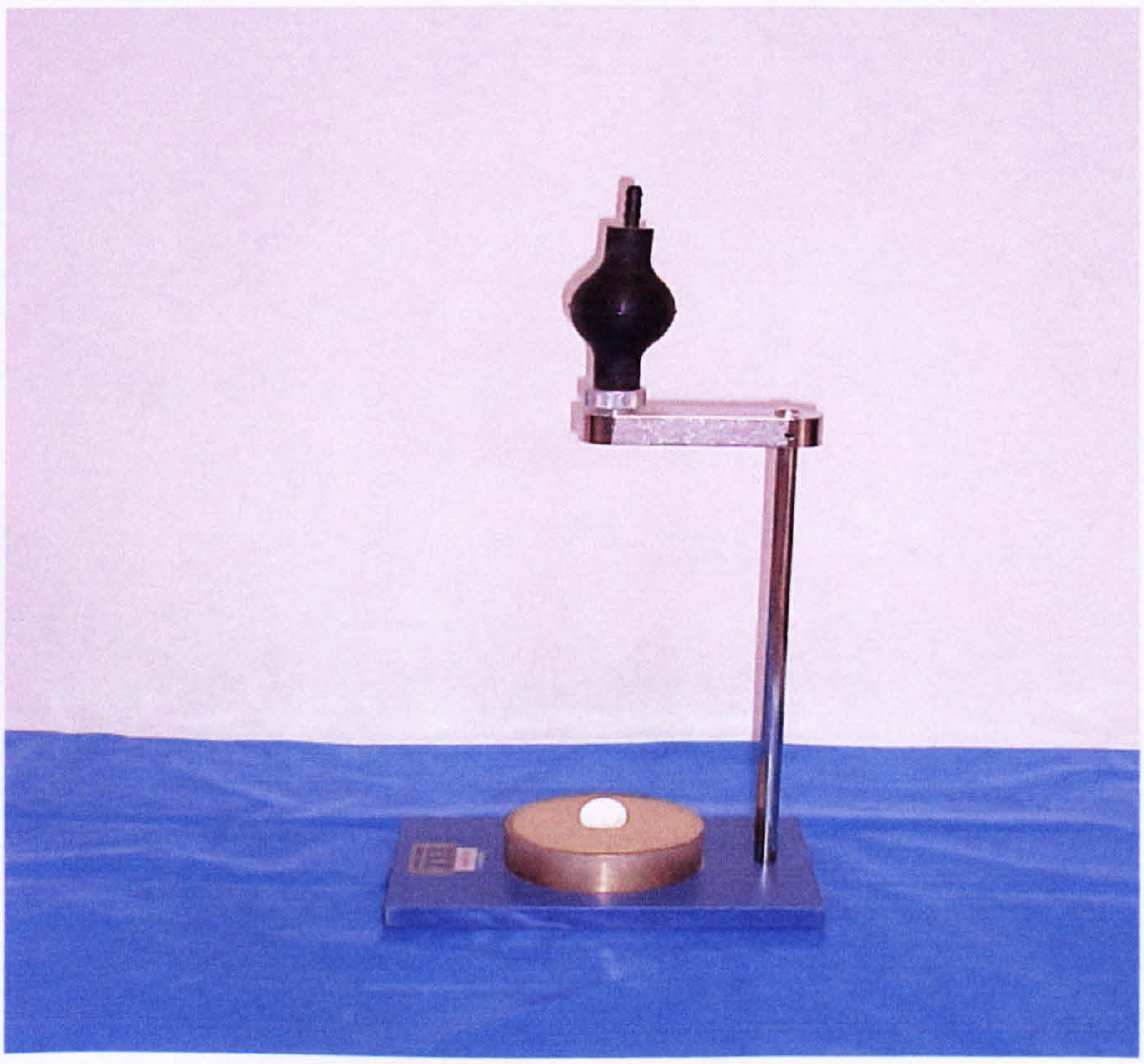
Hansen et al also produced graphs showing the amount of added water plotted against consistence, for two different lime types of lime putties, which they called consistence profiles. They showed that with the two limes studied, one required much more water to be added to achieve a given change in consistence, when measured by the Vicat plunger, than the other. They suggested that the lime putty with the much greater water demand, which had been aged for 16 years and had a smaller particle size, possessed a *much higher "cohesion" or physical bond*, which was *responsible for the "fatty" nature of this lime"*.

2.2.1 Test methods for consistence and related early life plastic properties

There are a number of test methods for the determination of consistence, flow or a related property, some of which have evolved little if at all from tests that were in use many years ago. Thus Collin, 1935, reported on a flow test that appears similar to the recent DIN test for flowing floor screeding mortars, that now given in EN 13454-2, 2003. Many of these tests appear to be measuring the same, or at the very least a closely related property, although users often tend to favour their own particular test in preference to others. Within the UK a small plastic ball was dropped into the mortar, to produce a value for the consistence by dropping ball as called up in the now superseded British Standard, BS 4551:Part 1: 1998.

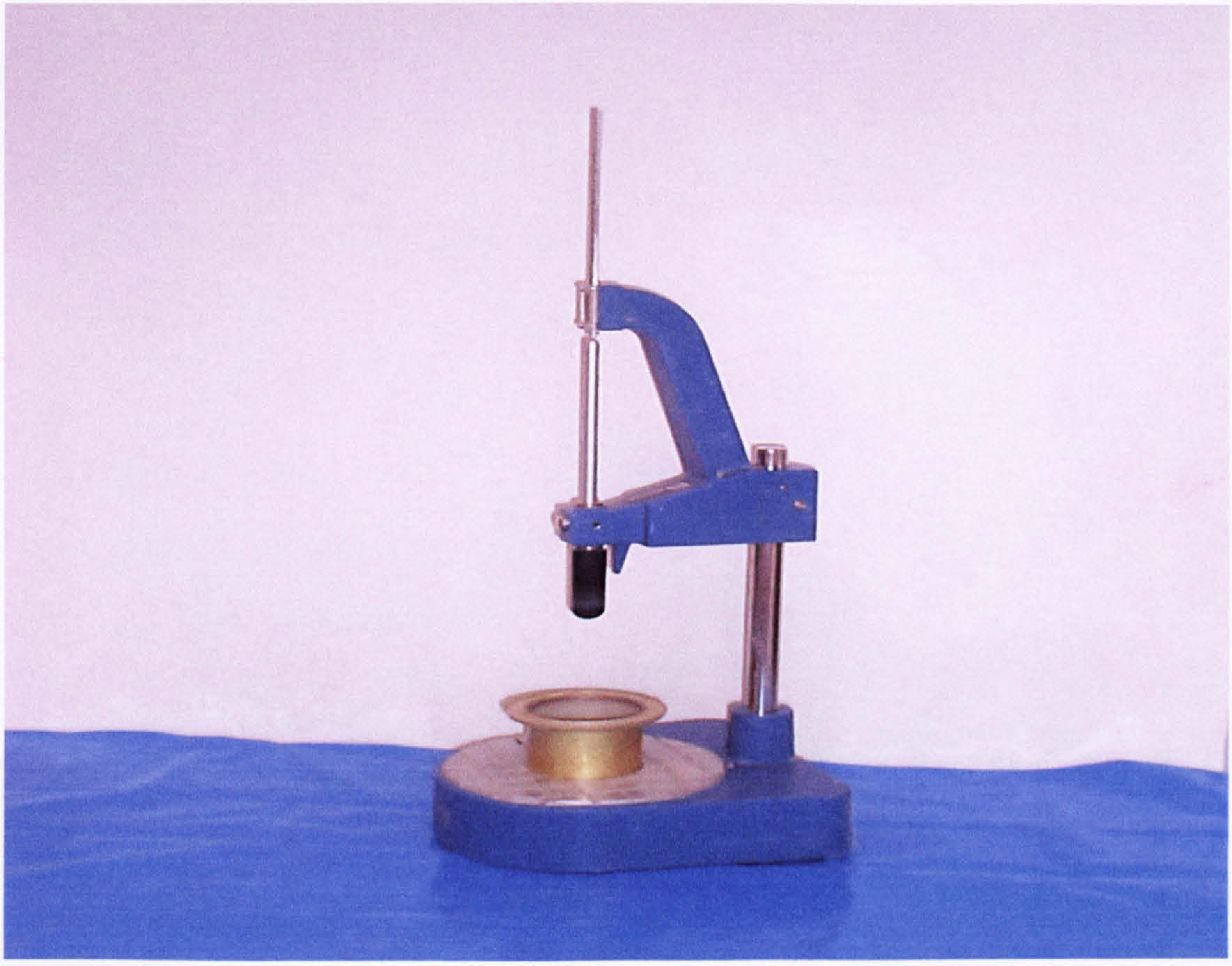
Fig 2.1 below shows this equipment with the mortar sample in the mould and the perspex ball resting in the mortar just prior to measurement of the indentation depth and thus of the consistence value, having just been released and allowed to fall into the test sample.

Fig 2.1 The dropping ball apparatus



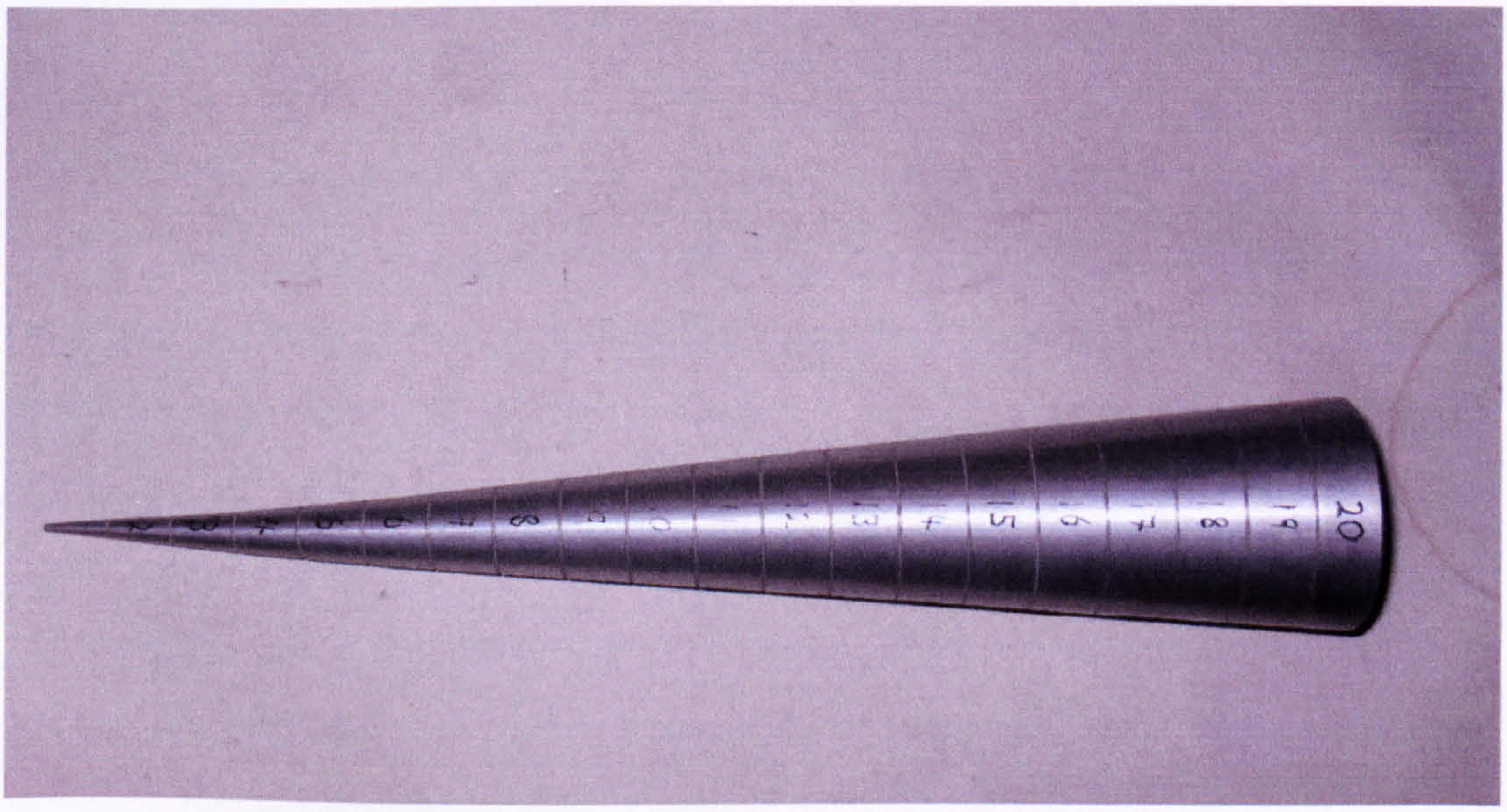
In Germany, instead of a ball a tethered plunger was used, as shown in figure 2.2, and this has now been adopted by CEN as a European test, BS EN 1015-4, 1999. The plunger and the ball are of similar cross sectional area, and fall freely under the influence of gravity, although the former has a greater mass. Both are in the same order of size as the original Vicat plunger, and not dissimilar to the plunger specified in the current cement testing standard, BS EN 196-3, 1995, and it is difficult to see the logic that led successive workers to continue to devise new equipment when existing methods appear to have been so similar. In the case of the dropping ball the value chosen as a standard consistence, a penetration of 10.0mm, is said to have arisen from the consensus views of bricklayers who were canvassed at the Building Research Establishment, BRE, 1970s, although that value is too low for the vast majority of unit types and weather conditions, both of which influence the required value.

Fig 2.2 The DIN plunger



Notwithstanding the number of test methods available, further ones have continued to be developed. In Russia and many Eastern European countries a large cone has long been used to penetrate the mortar under the influence of gravity, Polski Komitet Normalizacyny, 1973, although a RILEM paper, RILEM, 1982, stated that this method is less accurate than the flow table. This cone uses exactly the same principle as the penetration methods discussed above and as the Kelly ball used in concrete, Neville, 2000. A very similar cone penetrometer has recently been developed and is now used commercially in the UK with ready to use mortars on site but so far has not been covered in published papers as it is considered commercially sensitive. Figure 2.3 below shows an example of one of these cones, which as they are not manufactured commercially within the UK, was produced as a prototype in an engineering workshop.

Fig 2.3 The cone penetrometer



Work in Australia, Morgan, 1981, discussed a development of the concrete slump cone, to consist of a half size version for mortars.

The practical advantage of cone type penetrometers is that they are particularly easy to use in production and construction site environments, as they only require to be applied to a large sample mass of mortar placed in an open vessel of non-critical dimensions, so long as it is wide enough and deep enough to avoid pronounced edge or wall effects. Vessels such as wheelbarrows, ready to use mortar tubs and similar are found to be usable in practice. With these containers, there is probably a minor wall effect but where measurements are required to be merely comparative, as opposed to absolute, this is considered unlikely to represent a significant disadvantage. It must be remembered, however, that although the procedure is sometimes said to be superior to those mentioned earlier, any differences are only in terms of minor usability and practicality issues and quite fail to negate the overall theoretical inadequacies of these crude one point tests, as discussed further later in this chapter.

A proliferation of basically similar test procedures exists in the area of flow testing. The German flow table in accordance with DIN 4211,1986 is of similar dimensions to that called up in BS 4551, 1998, but the weights are different and the DIN table uses a glass plate, as opposed to the phosphor bronze one used in BS 4551. The BS table is based on an American one, called up in ASTM C-230, 2003, but differs in minor detail. The newly standardised table used in the European mortar testing standard EN 1015-3,1999 differs from that used in much of the work on masonry cement, PD CR 13933, 2000. The table used for many years in the UK for lime testing, BS 890, 1972, is different again, with a far heavier tabletop.

As reported by Plummer, 1962, the American National Bureau of Standards carried out work to develop a calibration method for flow tables, based on a standard paste that could be used on any type of table to arrive at a "standardised" value. This work was continued by Bowler et al, 1996, and was later the result of a substantial international programme of inter laboratory work, which culminated in the publication of the CEN report PD CR 13933, 2000. The overall conclusion of the work was that despite the substantial calibration work the method could not be recommended, as it was insufficiently reproducible, although further development work was perhaps justified.

In general, the concept of flow table testing leaves much to be desired. The equipment really requires to be mounted on a substantial concrete plinth to give reproducible results and shows great sensitivity to the firmness of the mounting and to the presence of any dirt or lubricating oil on the contact faces. Failure to observe these critical requirements has been shown in the RMC laboratories to produce poor results, with the flow tending to increase as the firmness of the mounting of the table and the cleanliness of the contact faces increases, presumably as a result of progressively increasing frequency of secondary and tertiary vibrations.

Hogberg, 1967, described mortar research using the Swedish test closely related to these flow table methods that is known as the Mo-meter. This consists of a vertical tube that is filled with mortar and dropped a number of times through a fixed distance, with the number of drops required to empty the tube being termed the Mo-value consistence. This is effectively the flow when constrained in a vessel so that the adhesion to and friction between the walls has a substantial input, probably more so than related effects between a flow table and the test mortar sample placed thereon. However, Bowler et al, 1996, reported that repeatability problems had arisen whilst this test was being appraised in the work that lead to the CEN report referred to above, and the test was therefore not investigated further in the current work.

A rather more complex determination of flow is provided by the modified Wuerpel apparatus, RILEM, 1982. This uses a dynamometer to record the work needed to deform a square of plastic mortar at a force imposed at constant velocity of 40mm/min., which is then recorded in the form of a graph of force plotted against deformation. The AFNOR method, NF P18-452:1988, also used a similar principle and a dynamometer. Although apparently a more sophisticated development of the flow table, this equipment has not become established in use and it is indeed difficult to see what advantage is provided by the added complexity.

Other workers have used much simpler tests, as for example Michoinova, 1999, who devised a test whereby mortar was allowed to flow down a tile that was inclined at an angle of 50 degrees, with the "length of the trace indicating the liquidity of the mortar". Again though, no evidence is presented to suggest that there is any specific attribute of the method that makes it superior to existing and better known and standardised existing test procedures.

There has been a limited amount of research carried out by various workers on comparing some of the methods for the determination of consistence/flow, but it did not prove possible to find one rigorous and complete piece of work as most workers had only considered a small number of methods. Thus Kampf, 1961, investigated slump, flow and penetration and whilst stating that each correlated well with water cement ratio believed that none represented an absolute index of workability. Rilem, 1982, reported work in Israel to have found that the flow table gave better results than the modified Vicat needle but that both were distinctly inferior to the craftsman's view. The same

RILEM report also suggested calibration factors for three different tests as shown in the table below.

Table 2.2, after RILEM, conversion factors for consistence

Property	Conversion factor for stated test
Dropping ball (mm)	USSR (Polish) cone/6
Dropping ball (mm)	54/Mo value
USSR (Polish) cone	324/Mo value

The RILEM report unfortunately goes on to say that there is more than one calibration graph, which therefore may demonstrate that the different methods are not measuring the same thing, which would seem to raise doubt as to the basic validity of attempts to define any precise numerical relationships.

Other workers have also carried out limited work on comparing the different methods. Anderson et al, 1983, showed a family of parallel curves for different mixes, but based on only a limited number of results. Morgan, 1981, carried out work on four different methods and concluded that the flow test was preferable. Bowler et al, 1996, reported a relationship between consistence as measured by penetration using the DIN plunger, (now an EN 1015 consistence test) and as determined using the BS 4551 dropping ball equipment. The most rigorous piece of work to compare different methods appears to be that carried out by Jessop et al, 1978. That work indicated a close relationship between the dropping ball test and a cone penetrometer, showing a graphical straight line, but the data was not presented statistically, nor was the raw data reported in the paper that would have enabled a contemporary statistical analysis of the reported relationship to be calculated. Nevertheless, these workers presented some 50 results to show an apparently reasonable straight line relationship. This tends to give some support to the hypothesis that all of the penetration methods are effectively measuring the same property, although judged on the basis of the totality of the work cited earlier in this context it seems unlikely that one or the other shows any real superiority. Notwithstanding this interim conclusion, it was decided to carry out a rigorous comparison between the most used methods in order to be able to propose or refute a definitive relationship. This work is reported in section 4.

2.2.2 Water retentivity and other related test methods

The Emley plastimeter, Emley, 1920, uses a combination of shear and suction to interact on the test specimen and thus simulates the effect of unit suction, whilst simultaneously providing an assessment of the plastic properties as the substrate is moved. Although the values in which the resultant property was reported were specific to the device, the result nevertheless does provide a basis for comparison and the test method has thus endured for over 80 years. Effectively the procedure consists of testing for plasticity after water retentivity, which appears to be a meaningful procedure and one relating to plastic needs although clearly completely lacking in sophistication in this form.

Water retentivity is a key property. It is often considered as a property in isolation from the other plastic properties and tested for by subjecting the mortar to a substrate of standardised suction, as for example in the old British Standard test, BS 4551, 1998. Some commentators associate the property with plasticity in a wider sense, as did Emley during development of his apparatus and more recently Plummer, 1962, as referred to earlier in section 2.1. An American patent for mortar, Lindgren et al, 1970, actually states that *The workability of the mortar is ....the water retaining ability,* which ignores many other important properties.

A current American test method, ASTM C91, 2003, tests for water retentivity by exposing the mortar to suction applied by a vacuum, an alternative to a physical substrate and a parameter that can be accurately defined. In contrast, using a particular type of brick as a standardised background of known and constant suction that may be used in many different locations over a long period of time is, if not impossible certainly very difficult to achieve, particularly where test methods are increasingly international in application and transcend national boundaries. Nevertheless this approach is regrettably used from time to time.

Recent work by Green et al, 1999, proposed a much more sophisticated procedure. He used a pressure cell to apply pressure via a gas to the plastic mortar, and then measured the amount of water expelled as a function of time. He argued for this approach on the basis of two premises, firstly that the existing tests use pressure as well as suction, which they clearly do as they place a weight on the test material and secondly that an applied gaseous pressure is effectively little different to the pressure exerted by the capillary suction where a filter paper is used. Although no results are reported on repeatability the method does appear to show promise and is certainly effective in removing a key source of variability that is associated with using a "standard" filter paper, brick or other test background.

2.2.3 Test methods for the stiffening and initial set phase

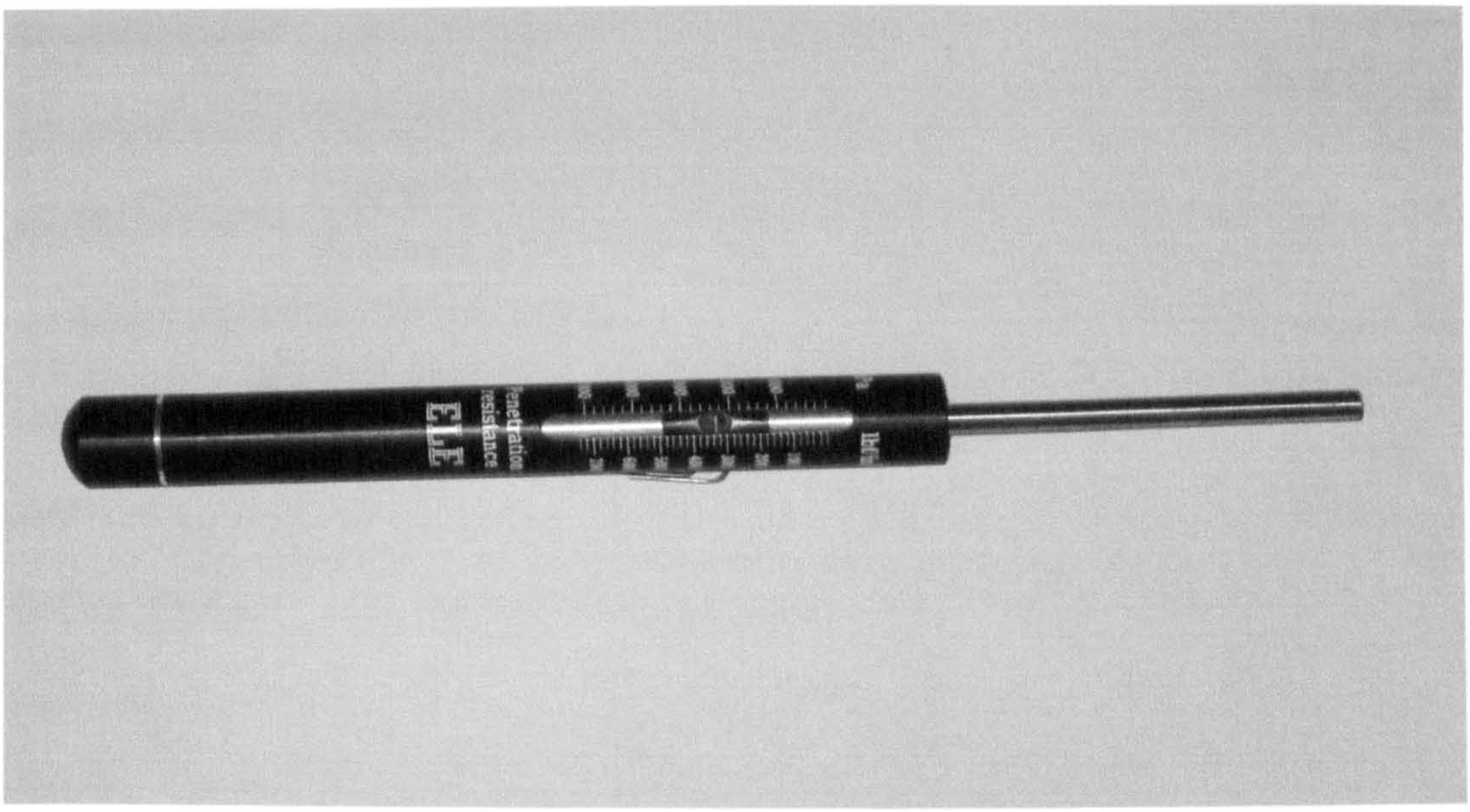
Prior to considering the above issues, it is important to realise that because there has long been a test for initial and final set, these terms may sometimes begin to be considered as two real properties or characteristics. They are not. They are purely arbitrary terms, and exist as a convenience. Moreover, they bear no fixed or finite relationship to a specific setting reaction in the context of Portland cement.

As discussed earlier, stiffening and setting arise at an early stage in the life of the mortar. If it is considered acceptable to again classify these processes in two parts, as a convenience, then the first part could be said to begin immediately after and following cessation of mixing, (within a few seconds only), continuing to a stage where thixotropic or other early time related phenomena are established, but before any discernable chemical set commences. The second part comprises the next stage in time, when setting mechanisms begin to exert a more profound influence and the fluid begins the transformation to a solid. These two stages are not separate, and the above descriptions are loose and simplified. The material begins as a liquid or fluid and as the first phase approaches its mature stage, the second has increasing influence. It is this second phase that is addressed in the testing of the parameter known as stiffening rate or workable life.

A very early worker in the field of stiffening and setting time assessment used a test procedure based on judging the resistance of mortar to the pressure of a thumb, Treussart, 1829. The ASTM procedure for investigating the stiffening and setting behaviour of the cement mortar phase of a concrete, ASTM 403, 1999, takes as the arbitrary value of setting a penetration of 3.5 N/mm^2 for the initial set, said to be the value at which a concrete is too stiff to be made mobile by vibration, as tested for by the penetration of a standard Proctor needle. A value of 27.6 N/mm^2 is taken as the so called final set. Other well known methods include the Vicat needle, BS EN 196-3:1995. The method of BS 4551, 1998, now effectively published as BS EN 1015-9, 1999, uses a similar penetration method. This test procedure is now known as the determination of workable life but the arbitrary value of 0.5N/mm^2 which is the level at which the test is completed and the time recorded is far too high to represent a usable mix, even one at the end of its workable life and it is suggested that a value of perhaps 0.1N/mm^2 would be in the right order in this respect. All of these tests apply a load sufficient to cause a rod to penetrate a fixed amount into the mortar. The Stanhope Seta penetrometer, which although primarily for the bituminous and food industries, described in BS 2499-3, 1993, has been used for mortar, applies the principle of using a known mass and measuring the penetration. This contrasts with most of the other penetration tests that measure the force required to reach a standard penetration.

Portable hand held penetrometers are also sometimes used and one such example is shown in figure 2.4 on the following page. Although originally developed for use in soil mechanics, and still retailed for that purpose, these instruments may nevertheless be used in a very approximate way on site, in the absence of correct equipment or ready access to proper testing facilities. All of these penetrometer tests apply a load sufficient to cause a rod to penetrate a fixed amount into the mortar.

Fig 2.4 A pocket penetrometer



Other methods have been developed that are effectively analogous to examining resistance to penetration by a non deformable body, but work by measuring the pull out load or the resistance to a turning moment. Thus the Windsor Probe or Windsor Pin, Al-Manaseer et al, 1999, measures the depth of penetration of a steel pin driven into the specimen by a spring loaded rig, the screw pull out test, BRE, 1997, the force to pull an imbedded screw out of a mortar joint and the proposed RILEM method as reported by de Vekey, for BRE, 2000, the energy required to drill into a mortar bed. All of these methods rely on a mechanical assessment, and in principle they or a similar technique could probably be adapted for stiffening rate or workable life, although they have generally been used for more mature mortars where a substantial amount of the final strength has been developed. Ferguson, 1995, tested the screw pull out test for mortars with a mean strength of some 5N/mm^2.

Interestingly however he reported that the test was still effective with mortar strengths of only 0.8N/mm^2, in the same order as a weak lime based mortar and approaching the order of strength associated with the latter part of the setting process for stronger mortars, although still a little too high. To address the right order of strength for screeding mortars, a mechanical deformation test was proposed for investigating the properties of the plastic screed, Beningfield, 1986, which relied on the deformation of an element of the screed by a plate. Unfortunately, however, the size of this plate precluded use of the technique for masonry mortars unless a relatively large sample size was used. Kreijger, 1967, believed that particle interference was an issue with all penetration tests and carried out a comprehensive programme of work on pull out testing. However it is apparent that there are substantial problems with all types of mechanical testing of the stiffening/setting area and this is discussed further in section 2.3.3.

2.3 CONSIDERATION OF THE PLASTIC PROPERTIES AND THEIR DETERMINATION

This section is divided into three parts and discusses, in order, testing for early life plastic properties, water retentivity and finally setting/workable life.

2.3.1 The early life plastic properties

The literature survey showed that although there were many methods of determining the consistence and the property loosely known as plasticity, in reality they were generally measuring the same property, or more correctly the same set of properties expressed as one, but at only one value of mix composition/water content. This is a fundamentally flawed concept, as may easily be demonstrated. If Mix A and Mix B are tested for cohesion using the flow table at a low dropping ball consistence value, A might prove to be more cohesive as judged by the flow table test, and could therefore be regarded as the better mortar. However, at a high consistence value, perhaps appropriate to use with a high suction unit in hot drying conditions, B might show the best cohesion, thus reversing the order in which they were originally ranked. This is because all of the tests described take a reading at only one preset value and are therefore clearly only single point tests.

This means that they are fundamentally incapable of fully defining or ranking complex fluid systems like mortars, as originally proposed by Tattersall, 1973, and as further discussed in section 2.3.4, which suggests that more complex and rigorous rheological procedures are essential if it is hoped to characterise a complex fluid, as opposed to a simple liquid like eg water, which would conform to simple Newtonian liquid behaviour and be capable of assessment using single point testing.

Even basic non-air entrained cement:sand mortars with relatively low time dependant properties prior to set are not properly characterised by the use of existing single point testing. As discussed earlier in section 2.1, the time dependant properties of mortars have become increasingly relevant in recent years, primarily as a result of the great increase in the use of more complex mortars, such as factory made materials containing admixtures that modify the plastic properties. All cement based mortars show some time dependant plastic behaviour, but this is often low in magnitude in the case of the simple cement sand mixes instanced earlier.

The inclusion of lime accentuates and brings about additional time dependant behaviour in the form of thixotropic stiffening as a result of particle size and shape modification and charge orientation, as reported many years ago, Chalk Lime and Allied Industries Research Association, 1962. More recently, the importance of thixotropy was acknowledged by Bowler et al, 1996. A great increase in the use of admixtures in mortars has occurred in recent years, either as purpose made retarders in all wet retarded ready to use mortars and in many dry silo mortars, as plasticisers that have some retarding action, or with other polymers as eg cellulose ethers, polysacharides or other viscosity modifying materials. This means that all of the mortars containing these materials will exhibit time dependant early life plastic property changes.

Air entrainment is also known to show time dependency, Beningfield, 1987, and its inclusion in virtually all mortars now means that time dependant changes have to be considered as a function of all mortar plastic property testing where accuracy is needed. Continuous instrumental monitoring methods, of the type that are intrinsically suited to the use of a rheometer, should enable all of the time dependant phenomena to be quantified and their interaction with the underlying plastic properties properly understood. The possible use of these techniques is discussed further in section 2.3.4.

2.3.2 Water retentivity and related procedures

The tests for water retentivity, generally based on application of a sample of mortar onto a background of known suction properties, (such as a closely defined blotting paper or standard brick), or using a standardised vacuum to apply suction, appear to be relatively satisfactory, although the use of any background, as opposed for example, to a defined gaseous suction or pressure, involves practical issues of ensuring that the background can be duplicated in any testing location, at any time.

Most of these procedures can play a useful part in characterising a mortar and are able to discriminate reasonably well, although sensitivity is not excellent, as can perhaps be seen by the fact that the water retentivity procedure in BS 4551, 2005, requires the result, which is reported as a percentage, to be rounded to the nearest 5%, and covers a range of only about 35% in total for the spread of values appropriate to mortars in general use. Thus although not now a part of the properties required by the latest European mortar standard EN 998-2, 1999, the test is of limited value and proves useful in practice. This is reflected by the fact that notwithstanding the introduction of the BS EN 1015 test series, 1999, the test is being retained as a British Standard, even though the remaining UK physical test methods were withdrawn following the adoption of the new European methods as BS ENs.

2.3.3 The stiffening and early setting properties

Currently standardised test procedures in this area appear to be unsophisticated and inaccurate, with poor reproducibility. Importantly, they do not relate to any fixed property of the mortar or concrete in terms of a specific setting reaction starting or finishing. They merely represent arbitrary snapshots or windows in time, chosen for historic reasons of convenience or some other unrecorded reason. As discussed earlier in 2.2.3, there are many of these procedures, Vicat, BS 4551, BS EN 1015, to name only a few. They are inaccurate for a variety of reasons. The way in which they act is not always clear, it is rarely analysed, and different methods that ostensibly measure the same property may actually act in different ways. It would appear that the automated Vicat equipment is designed to apply a fixed force, the manual Vicat a constant force and the stiffening rate a force at a rate that is variable, and is effectively determined by the operator. The Stanhope Seta measures penetration depth as opposed to the force required to achieve a fixed depth.

All of these tests are said to monitor the same property, often erroneously referred to as a fixed, finite value as eg initial set. In reality they are measuring the sum of a number of properties, all contributing to the final arbitrary value. The resistance of the cement paste to penetration is the fundamental property but sand grading, particle shape, amount of bleed and segregation as well as overall mix design all exert profound influences and cause a change in the measured value although the actual setting property has remained unchanged. This effect became clear in preparatory work and has also been reported by Newson, 1986. It is seen therefore that a test is required that is not affected by these issues, and it was hoped that one could be developed that was based on changes that did not require mechanical monitoring. Removal of this procedural aspect would clearly reduce the mechanical influences as eg the presence of large, irregular or particularly orientated aggregate particle influences, segregation, overall mix plasticity and similar.

2.3.4 The application of viscometry and rheometry to the assessment of the early life plastic properties

Prior to commencing work on the properties that are relevant a little later, when more pronounced stiffening and then setting becomes evident, the other topic of study to be covered by this research programme, the earlier life plastic properties, was investigated. As discussed in section 2.3.1, it became clear that a more advanced treatment was required and viscometry and rheology were clearly relevant. Perhaps the father of the modern rheological sciences, Bingham, 1922, stated that *A formal definition of plasticity in general is " a property of solids in virtue of which they hold their shape permanently under the action of small shearing stresses but they are readily deformed worked or moulded under somewhat larger stresses". "Plasticity is thus a complex property, made up of two independent factors, which we must evaluate separately".*

Much more recently, Tattersall concluded that a simple one point test, or even a combination of tests, was inadequate. In his very early work, Tattersall, 1953, showed that a cement paste alone required the use of at least 4 constants to describe its rheological properties. However, in later work, Tattersall, 1973, he stated that even the more complex system of concrete could be said to conform approximately to the two constant Bingham model, as he reported in his further research, Tattersall, 1975.

In order to appreciate the relevance of these statements, and indeed the relevance of the whole field of rheology to mortars and concretes, it is helpful to consider some basic principles of rheology. Rheological models may be said to derive initially from the simplest of them all, for a Newtonian liquid, where an applied stress results in a proportional reaction, and there is no thixotropy. This may be shown graphically as a straight line, passing through the origin, with the applied stress plotted against the strain or reaction to that stress.

A Bingham fluid is a little more complex, in that it requires a finite application of energy before it will yield so the line is displaced from the x axis and intercepts the y axis. These two models are as shown in the two graphs below in figures 2.5 and 2.6. These graphs are shown only as grossly simplified examples, as are those shown following them in the text as figures 2.7 and 2.8 and serve only to show a basic principle. In reality they would be more complex, and particularly in the case of the latter, if derived from practical test work would show considerably departures from theory due to experimental scatter, even if the most accurate computer driven equipment was used. This scatter is a feature of rheological testing and is discussed in more detail later in this section and also when the practical rheological work is presented.

Fig 2.5 The simplified theoretical behaviour of a Newtonian fluid

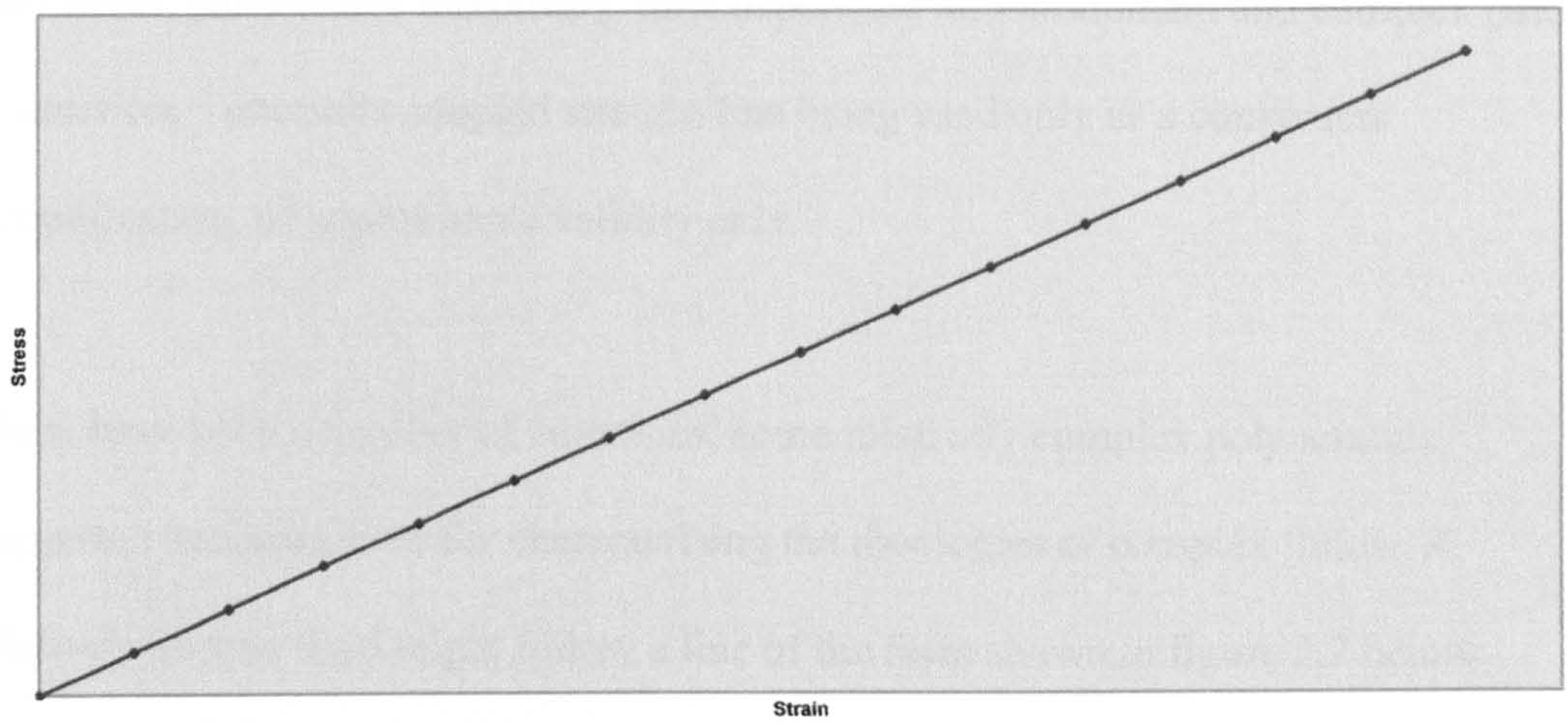
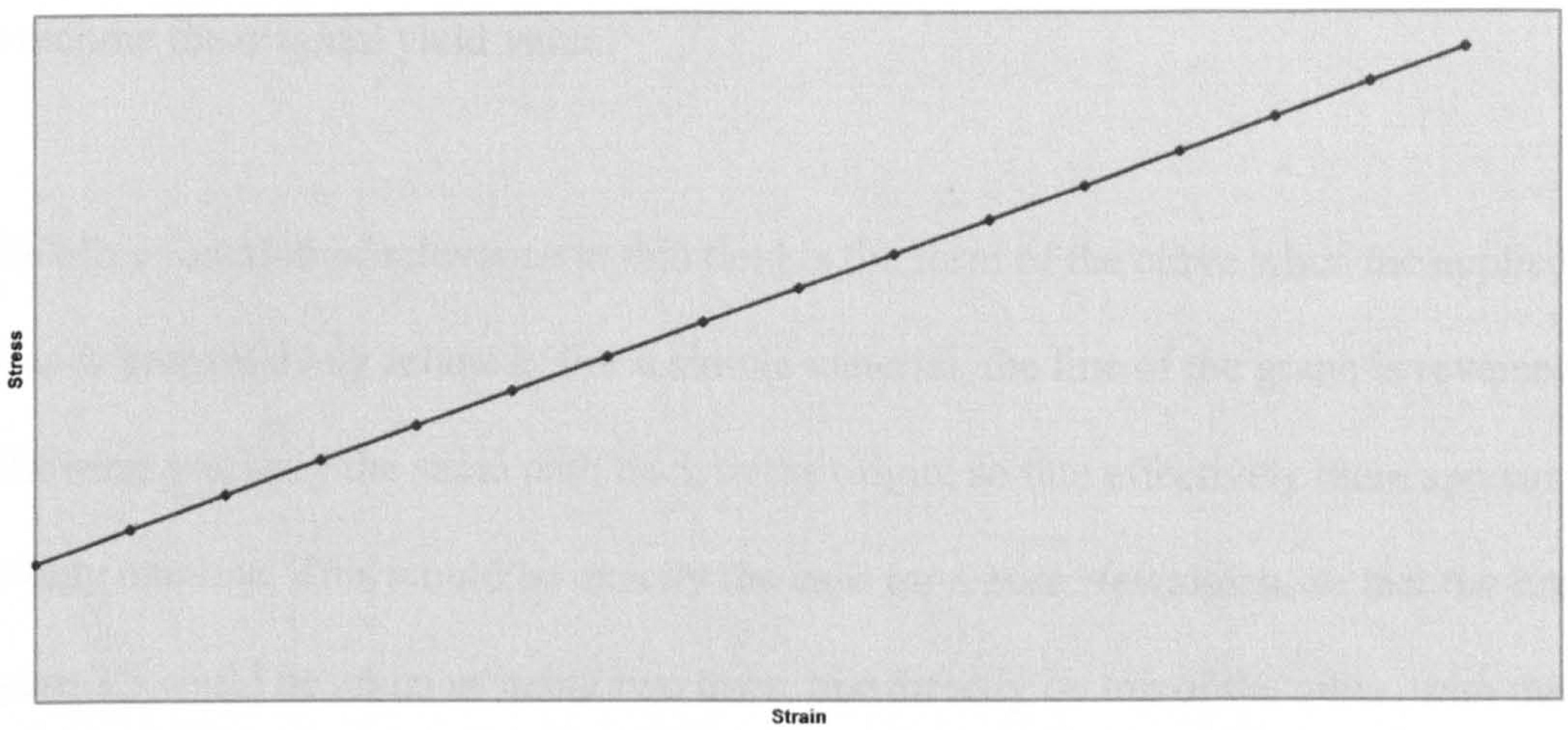


Fig 2.6 The simplified theoretical behaviour of a Bingham Fluid



The simple Bingham model is represented by the equation $F - F_0 = u\ d$, where

F = stress

F_0 = yield value

U = plastic viscosity, and

d = rate of shear strain

There are a large number of other more complex theoretical and empirical rheological relationships that are appropriate in varying degrees to different materials, and testing for best fit is often used to establish the best for a particular application. In reality, mortars and concretes follow curves, rather than straight lines as a function of many influences, particularly thixotropy, time dependant air entrainment and complex particle interaction, Tattersalls adopted straight line being used only as a convenient simplification, of approximate validity only.

There have been a number of equations, some relatively complex polynomials, suggested as appropriate for characterising the rheologies of complex fluids. A relatively simple fluid might follow a line of the form shown in figure 2.7 below although actually real examples would show the line displaced up the vertical axis a little, rather than starting from the origin, as a function of the energy required to overcome the original yield value.

The other function of relevance in this field is the form of the curve when the applied force is progressively relaxed. For a simple material, the line of the graph is reversed, following precisely the same path back to the origin, so that effectively there appears to be only one line. This would be exactly the case for a pure Newtonian, so that the line in figure 2.5 could be taken as being two lines, one directly on top of the other, with the line starting at the origin, gradually extending as the applied stress was increased and the strain response developed and then as the stress was gradually relaxed the line would return back to the origin along exactly its original path, thus appearing visually to be only one line. For a more complex material like a mortar, particularly one containing admixture and/or lime, the returning trace of the graph follows a different line, as shown in figure 2.8, in the manner of a hysteresis curve. This indicates the presence of thixotropy, as less energy is required for a given effect after some of the attractive energy responsible for the thixotropy has been redistributed.

Fig 2.7 Polynomial

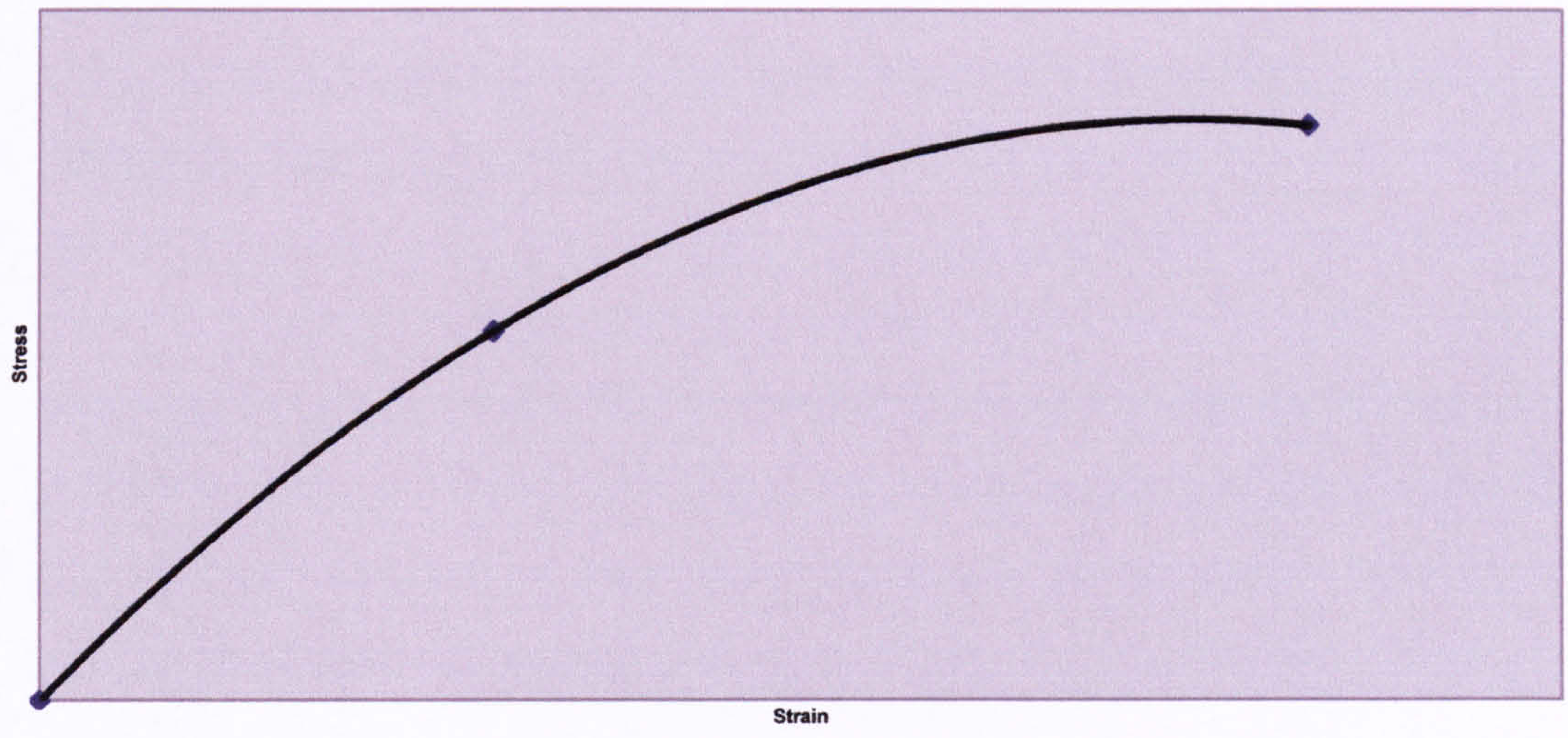
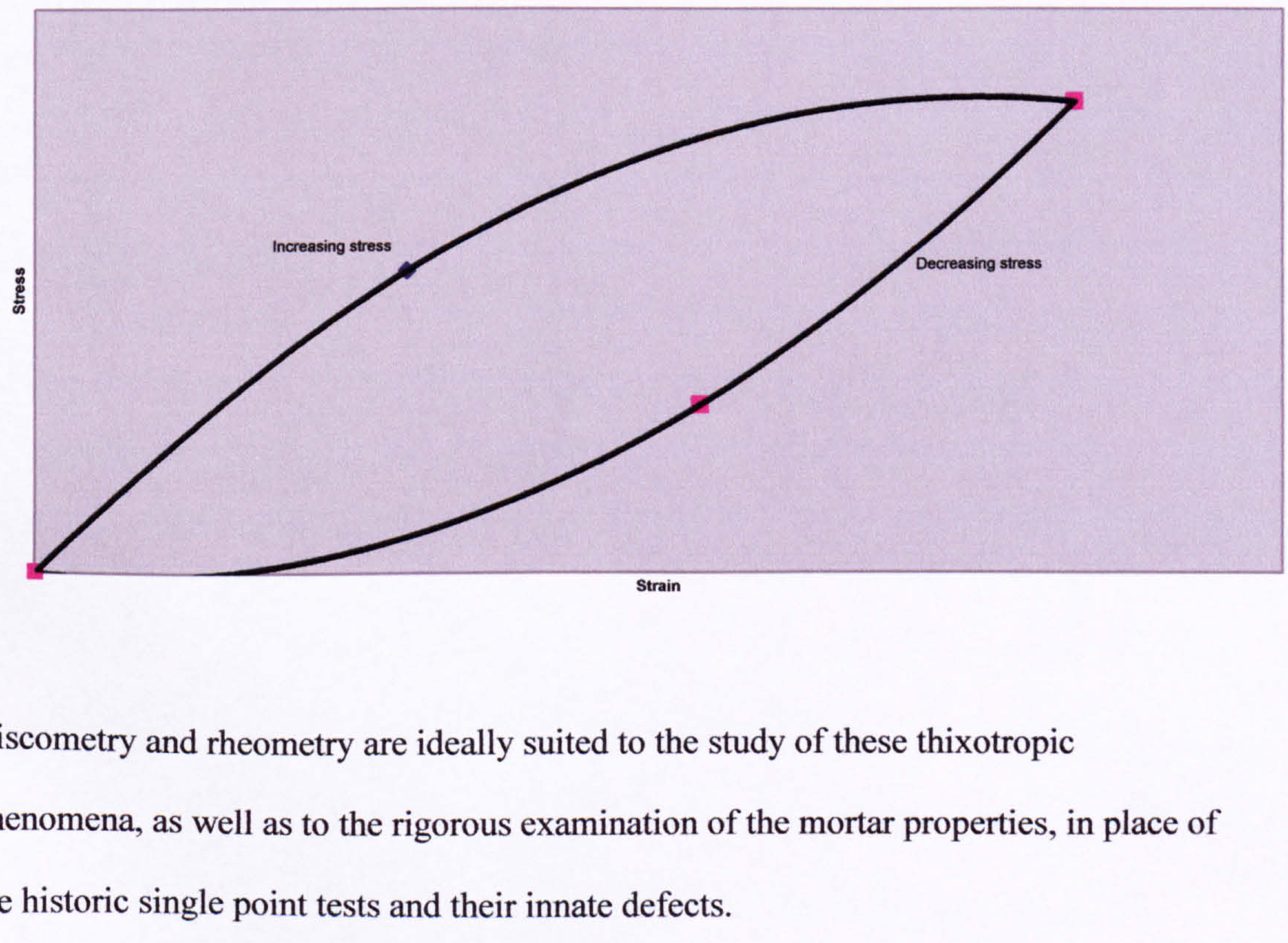


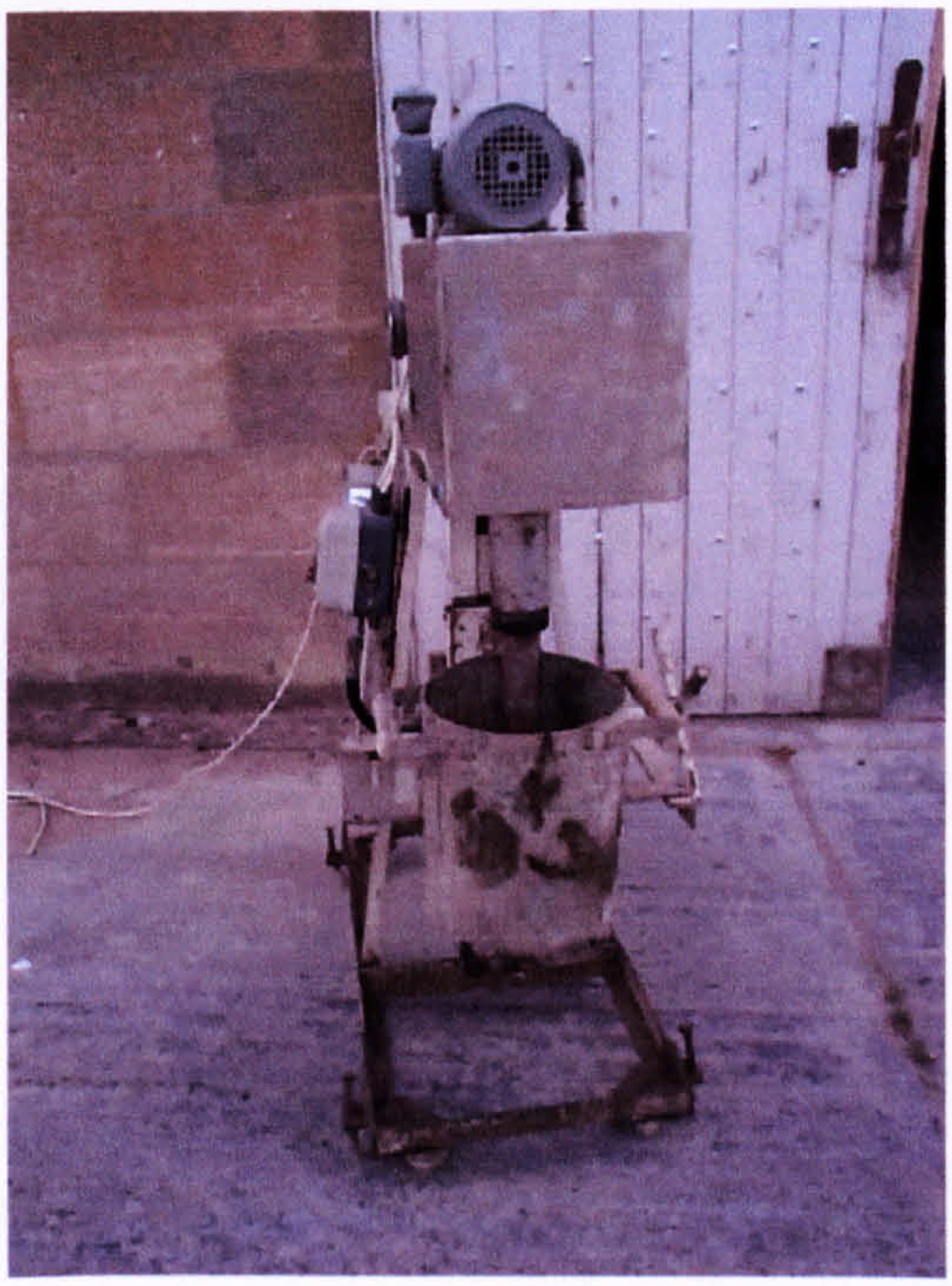
Fig 2.8 Quasi Casson with thixotropy



Viscometry and rheometry are ideally suited to the study of these thixotropic phenomena, as well as to the rigorous examination of the mortar properties, in place of the historic single point tests and their innate defects.

As outlined earlier, Tattersall was aware of these issues and went on to suggest that in plastic concrete, the slump approximated to the yield value, whilst the compaction factor and Vebe were measurements of viscosity, Tattersall, 1976. He devised equipment based on a small three speed mixer and measured the torque that was required to rotate the mixer blade at three different speeds, thus effectively applying three different shear rates and measuring the response, which he then treated as a straight line relationship. Tattersall then produced an improved apparatus based on an impeller and an infinitely variable speed control and this equipment is shown below as figure 2.9.

Fig 2.9 The Tattersall apparatus



The Tattersall equipment was a great advance on the status quo for the construction materials industry, but was of course unsophisticated, indeed basic, with manual operation and manual logging of the results. Other industries have long adopted sophisticated viscometers and rheometers that are so much more accurate and sensitive than manual plasticity/workability tests but also have the ability to output data directly into a computer. They may allow for the application of progressively increasing shear, in an almost infinite number of small increments, and then for the progressive reduction of that shear to enable thixotropic studies to be carried out. Some equipment has provision for a controlled rate of shear and a controlled shear stress, others for controlled strain. Sophisticated equipment allows for other modes still, allowing further parameters such as creep to be investigated. Still other equipment has provision for the application of oscillatory forces, thus enabling the effect of vibration to be studied.

In industries where rheology is of importance, as for example with slurries of chocolate and other fluid foodstuffs, oils, greases etc, rheological techniques have long been accepted and in some cases actually form part of the production/control processes.

In the field of self compacting concrete, the importance of a more sophisticated rheological approach is now just becoming apparent and appears to have been particularly lead by advances in Japanese developments, Urano et al, 1999, Noguchi et al, 1999, Takada, 1999, Tomasawa et al 1999. In SCC today the fundamentals of concretes are better understood and some of the desired plastic properties are now described in rheological terms. Thus Gaimster and Griffin, 1999, equated high flowability with low yield value and resistance to segregation was related to moderate viscosity. They also stated that simple addition of water would reduce both the yield value and the viscosity, whereas in most mortar as well as concrete viscosity is required to be maintained to some extent in order to achieve sufficient cohesion and adhesion. These workers also identified the importance of shear thinning and thixotropy, which as previously discussed are important issues with mortars as well as concretes.

Billberg, 1999, believed that evaluating the rheology of the concrete mortar was necessary in the optimal design of self compacting concretes, although as will be discussed later, it is now becoming clear that the properties of the mortar fraction of a concrete cannot be used to completely characterise the rheology of that concrete. They may be a guide, but the complete concrete must be investigated to fully characterise its rheology.

Work by Thompson, 2000, was carried out on a viscometer to determine the viscosity of different limes and gave values varying from 20,00 to 400,000 centipoise plastic viscosity for dolomitic and high calcium limes respectively but no value was given for yield, without which the contribution played by the consistence cannot be judged, as the more viscous material could have been at a much lower consistence.

Although recent work, some of it as cited above, utilised rheological values, their use is still in its relatively early stages even with concrete and they do not appear to have been adopted at all for practical applications in the field of masonry mortars.

Nevertheless, some work has been reported although this is of varying degrees of sophistication. Thus Yen et al, 2000, sought to optimise concrete mix proportions using mortar rheology, to use their own terminology, yet only used slump testing and theoretical packing calculations in their work, whilst Mantegazza and Alberti, 1994, also spoke of rheology in their paper, Slump Loss and Rheology of Superplasticised Mortar and Concrete with Different Polysaccharide Syrups, but slump and setting time tests were the only methods used in the work. Similarly, Petrou et al, 2000, believed that the rheological properties of the mortar influenced the aggregate settlement but calculated the yield stress from the slump value, which involves a number of assumptions and is clearly of nowhere near the same order of accuracy as a determination made using a rheometer. It does seem, however, that the importance of using proper rheological techniques for concrete is finally about to be generally acknowledged, as at a recent meeting of ISO TC 71/SC1:testing concrete, held in Istanbul, Turkey, British Standards Institution, 2004, both Bulgaria and Taiwan-China submitted proposals for work on two point testing of concrete.

Limited work has been found where rheometry has been used for mortars, although in some of this work the rheometer was only used to measure viscosity, as in the work of Toumbakari et al, 1999, and in general the work has been only with the mortar fraction of a concrete as opposed to a true masonry mortar, or with a specialist grout. Thus Hwang et al, 1998, investigated the effect on the rheology of concrete mortar of changing concentrations of pfa, Billberg, 1997 and 2000, carried out similar work with limestone fillers and Wallevik 1997 reported on preliminary work on the rheology of concrete mortar pastes using a coaxial cylinder viscometer in conjunction with concrete mortar thickening agents. Petrou et al, 2000, investigated the effect of mortar rheology on aggregate settlement but rather than determine rheological parameters directly made the not uncommon assumption that yield stress was the same as slump, which represents a most approximate treatment, as discussed in the following paragraph.

Although they only worked with cement paste mortars, Ferraris et al, 2001 carried out interesting work that is also of relevance to "real" masonry mortars. Firstly, they attempted to characterise the rheological properties of their mortars with simple tests, hoping to use these instead of rheometry. They failed however to derive adequate relationships and concluded that "*these simple tests were unreliable for measuring workability*", thus concurring with the views expressed earlier in section 2.3.1 of this thesis. This finding also appears to confirms the view that the work of several workers, including Yen et al, 2001, Mattegazza et al, 1994 and Petrou et al, 2000, all as cited above, was based on a very questionable thesis, namely that slump, aggregate packing or some other simple measurement could properly characterise one or more rheological parameters. Ferraris appreciated that sophisticated rheometry was essential in these fields, even though the equipment necessary to carry this out was very expensive, but confirmed that this appreciation was only recent, as demonstrated in an earlier paper of his, Ferraris, 1999.

Banfill, 1995, used a Viscomat rheometer to show that yield stress and plastic viscosity decreased with increase in moisture content, a not unexpected finding, and that this effect was less with a coarse sand, presumably because finer sands with greater surface areas and inter-particulate attractive forces have higher initial values for both of these parameters. Banfill also showed that although both measured parameters, viscosity and yield, decreased with increase in moisture, there was no valid correlation between the two, a correlation coefficient of only 0.29 being quoted. Once again this demonstrates another aspect of the invalidity of single point testing and the need for two point rheological tests.

As discussed in greater detail later in this section, Banfill referred in this paper to his earlier work, Banfill, 1994 as showing that temporary bonds and so called structuring in the test specimen should be broken down prior to testing. This is clearly a correct statement from the point of view of achieving reproducible results from the rheometry, because the thixotropy and structuring present in all mortars will be time dependant in its development and moreover will be gradually broken down as energy is applied in the course of testing. This often results in test variability, as these forces are broken down to varying degrees as a result of even subtle differences in elapsed times and test regimes, with elapsed times between mixing and any remixing and testing being critical, as are overall mixing times and mixing energy.

In addition to the chemical "structuring", well known in the field of non-drip paint and similar, it may easily be demonstrated that early loss of air also causes pronounced workability loss, but that much of this is reversible following re-mixing, as shown in figure 2.10 and discussed later in this section which shows work carried out in this programme that adds to that of Banfill, 1994 who stated "*work has shown that mortar shows irreversible structural breakdown.* That theory was developed further by Tatter sall and Banfill,1983, who proposed that several cement particles could become surrounded by a single all encompassing film of very early life hydrate and if energy was applied this could be broken down irreversibly. The theory was given added weight by later workers who used electron microscopy, Sujata et al, 1992 and Yang et al, 1995.

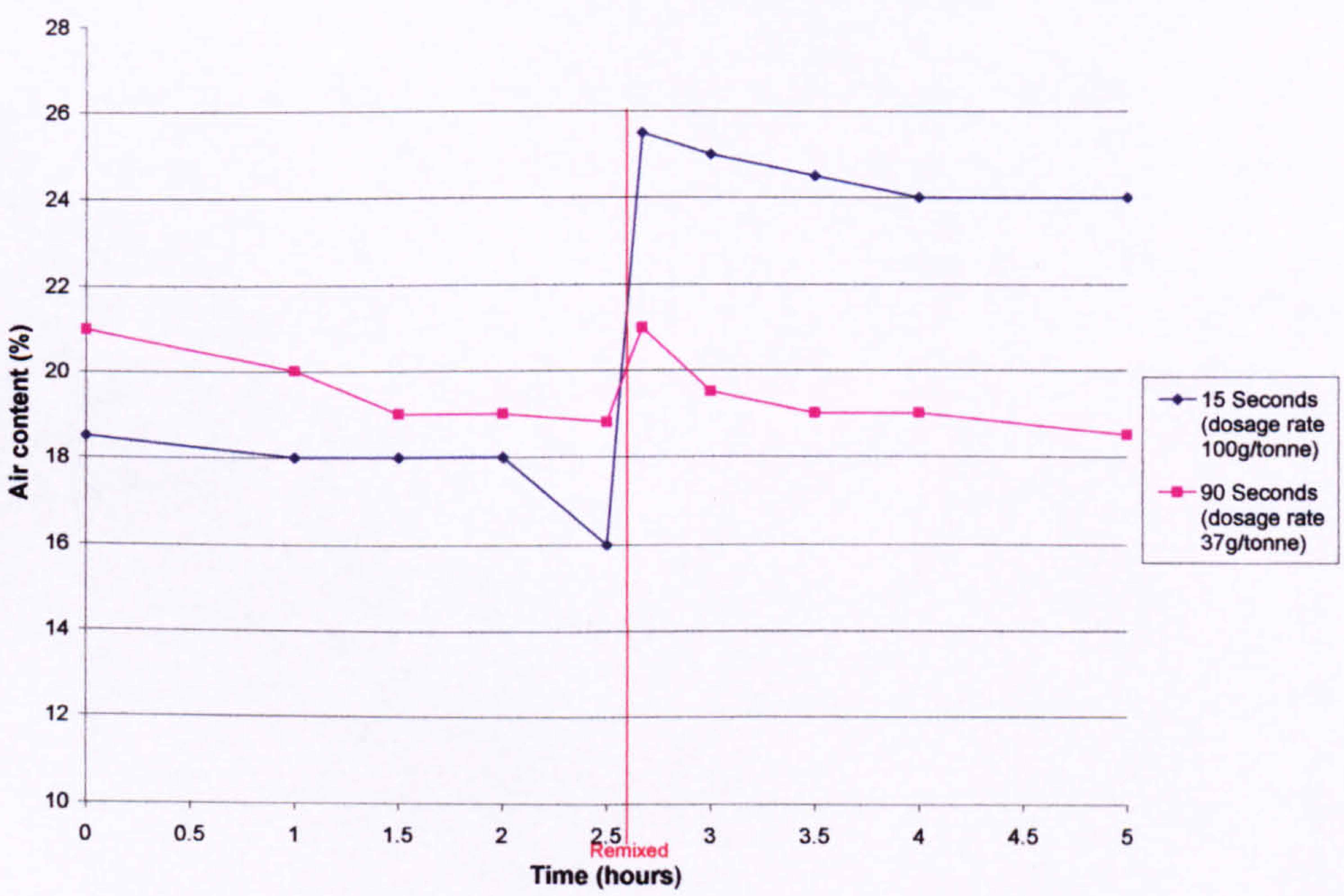
Although there clearly exists a great deal of data to validate the concept of irreversible breakdown, it is known that in practise, much of the initial loss is actually reversible air loss, with sometimes additionally some water loss. This is known to affect all retarded ready to use mortars to some extent, often to excess, so that after a period of standing the material has appeared to "stiffen" or "go off" in the view of the bricklayers. This is particularly noticeable after standing overnight, when the mortar may have lost so much workability or consistence that the bricklayers are unable to use it. However, it is common practice to then "knock up" or remix the mortar, by hand but as vigorously as possible, which then restores virtually all of the lost workability or consistence and results in a material that is usable once again.

This clearly shows that the permanent effects reported by Banfill and others are not the only ones, and illustrates that there are other reversible changes of considerable magnitude taking place at the same time. Of course, these mortars are all relatively highly air entrained mixes whilst other reported work was generally with non-air entrained mortars, but as discussed earlier the majority of mixes that are actually in practical usage are air entrained, generally to quite high levels, so that in practise on the building site are affected by two early life consistence reduction mechanisms. The first consists of the reversible air loss shown in the figure, the second the irreversible ones reported by Banfill, Sujata et al and Yang et al.

Some previously reported determination of the rheological mortar properties has taken place only after sufficient mixing energy has been applied to the mortar to not only re-introduce any lost air but also to completely break down the initial "Banfill type" stiffening associated with early life hydration membrane agglomeration. As previously cited, Banfill, 1995 recommends that the stiffening is broken down irreversibly, stating, *"The mortar should preferably be completely broken down in order to produce completely unambiguous results. This corresponds to the situation where mortar has been mixed in an efficient and energetic mixer".* Whilst this is clearly correct with respect to laboratory research work and its adoption will lead to less test variability, and thus to closer pure research results with less scatter it has to be said that it represents an artificial situation with respect to site practice.

Additionally, as a proposed testing regime it does not appear sound from a theoretical basis. In much, perhaps a majority, of the developed world, mortars are ready-to-use, either wet retarded or dry and delivered in silos or bags. In the former case, they remain on the building site unmixed for a period of up to two, perhaps even three days. In the latter, they pass through a very inefficient mixer with a total mixing time of perhaps twenty seconds. Most mortars then remain on a spot board or in a wheelbarrow prior to final use, when they may be mixed for a few seconds by the bricklayer using a trowel. Thus in all of these cases the mortar that is actually used in practice has "structured" and developed initial bonds. As discussed earlier in this section, these concepts were examined in the laboratory by mixing an air entrained mortar, allowing it to stand whilst monitoring its air loss, remixing and then again testing for air content. This is demonstrated in figure 2.10 below, for two dosage rates and mixing times.

Fig 2.10 Effects on air content of standing and remixing



It is therefore suggested that if it is desired to investigate the properties of mortars in a way that may be related to practical site conditions, the use of excessive high shear mixing in order to reduce test variability is generally not desirable. In particular, factory made mortars used on site are not unusually the subject of complaint for precisely the reason that they show pronounced thixotropic stiffening and structuring either in the mortar tub or on the spot board. Research work on improving the properties in these areas will clearly not be valid if testing is first preceded by the application of artificially high rates of shear which will tend to mask these undesirable phenomena. Thus the current projected programme considered the issue of comparability with site shear histories at all times and did not use especially high shear preliminary regimes unless otherwise stated, using instead only the standardised mixing procedure used in all of this work, consisting of the BS 4551 regime adopted in accordance with the discussion in section 3.1.3.

As discussed, there is some published data on the rheology of mortars, but there is little that uses sophisticated rheometry and none of the references cited successfully quantified the numerical values of yield value or plastic viscosity that were desirable as mix design objectives or enabled the properties of their trial mixes to be completely described in accurate rheological terms, although Wallevik stated by how much these parameters were changed by the inclusion of welan gum thickeners, but based on an assumption of the Bingham relationship.

The most valuable work found was that already cited of Banfill, 1995. He did quote values of yield stress of between 40 and 400N/Pa and plastic viscosity values of between 1and 5 Pa/s, but with such a wide range of values and no stated relationship to the overall acceptability or quality of the mortars giving rise to these values practical application is difficult. This work indicated that perhaps the variability of the method was high and it was therefore clearly necessary in any proposed programme of work to carry out repeatability work as a preliminary to any laboratory investigations with the rheometer.

However, although the range of values stated above may seem large, in the field of rheology, the numerical values for parameters as eg viscosity and yield are known to frequently vary through several orders of magnitude and this is one of the reasons why comparing absolute values in rheology, as opposed to eg the shapes of families of curves for comparative work, is so difficult. This is further reported in a comprehensive summary given in Tattersall and Banfill, 1983, where the results of different workers show reported values for plastic viscosity varying by two orders of magnitude, even when the same type of rheometer was used. Uzomaka, 1972, found yield values that were an order of magnitude greater than those reported by Murata and Kikukawa, 1973, even though the concretes that they were testing were of similar composition. Other published work by Vom Berg, 1979, showed that cement fineness and composition also had a profound effect on the reported yield, again of about two orders of magnitude just for the former.

There are many other examples of the complexity of this field. Thus Cusens and Harris, 1967 and 1973, used oscillatory shear in a cone and plate viscometer to examine structural breakdown and set commencement but similar work with the same type of equipment by Jones, Bridley and Patel, 1976 produced results that conflicted with those such that the second authors were not in agreement with the conclusions reported by the first authors.

Other issues relating to the complex interactions of virtually all of the mix design parameters also add complexity. As an example, as part of his valuable work, Banfill experimented with three different sands, which he referred to as fine, coarse and intermediate, and made tentative rheological conclusions based on these stated characteristics, thus attributing greater or lesser values to coarse rather than fine and so on. Unfortunately, as he himself acknowledged with the statement *"rheology is affected in a complex way by the particle size distribution of the dispersed solid"*, it is not easy to make simple changes in grading without these introducing complex interactions. Thus it is considered that, although Banfill produced sands in three progressively coarser, (or finer), gradings, as he made these changes he also profoundly changed the fundamental nature of the gradings. This is shown by reference to table 2.3 below, taken from Banfill 1995.

Table 2.3, after Banfill, sands used in rheological work

Sieve size mm/um	Sand designation		
	A	B	C
5.0	100	100	100
2.36	99	99.5	100
1.18	87.5	95.5	100
600	75	91.5	97.7
300	34.5	58.5	76.5
150	7	7.5	9.5
75	1	1	1

Whilst the descriptions coarse, intermediate and fine are not necessarily incorrect, the sands possess other features that could form an equally valid basis for their descriptions. Thus the coarse sand might be described as quite well graded, basing that description on a wide range of different particle sizes, each progressively filling finer and finer voids, as previously defined by Beningfield, 2003 amongst others as one basis for a "good" sand. Perhaps more importantly it is suggested that the fine sand could be defined as poorly graded, based on the fact that it is grossly single sized and is also deficient in fine fines. It is therefore suggested that the research findings associated with the work, whilst of excellent value, could be interpreted as being a function of well graded or poorly graded sands, rather than being as presented a function of relative coarseness or fineness.

This is only one example of an alternative way in which the materials could be ranked, and shows the complexity of work involving statements based on changing sand or aggregate types. In addition, experience in this area suggests that these types of changes bring about other secondary and probably tertiary changes that further complicate the simple aim of defining a material in fundamental rheological terms, changing one parameter, and then re-measuring the rheological terms to express in a quantitative way the effects of the change.

Notwithstanding the reservations expressed above, the book on the rheology of concrete by Banfill and Tattersall, 1983, although published some years ago was found to be a valuable source, and included much work on cement pastes, which whilst clearly different from masonry mortars nevertheless do possess many similarities. The authors pointed out that many of the cement pastes used in rheological test work had a lower water cement ratio than in practice in the concrete environment because workers wished to avoid segregation. Even so, it is probable that segregation does occur in many test regimes and leads to inaccurate results being reported. Cement pastes are not easy materials on which to conduct rheological investigations, partly for this reason but the literature refers to a number of other potential issues, particularly at the interface between the test material and the vessels in which it is tested. These potential problems can also occur at the interface with the test bob, vane, impellor or other device. Thus the existence of plug flow, where a plug of unsheared material rotates, has been reported by Dimond, 1975 and Tattersall and Dimond, 1976. This problem led to the workers referred to considering different surface finishes to the rotating and/or stationary surfaces, with grooves, slots and similar being adopted and even to the application of glued fine silicon carbide abrasive paper.

An unfortunate thread running through the whole area of rheological experimentation in the fields of concrete and cement pastes is that successive workers have used different test conditions and regimes but have often failed to ensure that these are standardised, are comparable with earlier reported work by others or even in some cases that they were comprehensively documented, thus making further, later comparison by others difficult if not impossible. In this context, the type and intensity of pre-mixing, if any, as previously discussed, is of key importance.

A further problem anticipated in the work currently considered was that because of the added complexity of mortars, with generally high air contents and often with relatively high amounts of complex plasticity affecting admixtures, it seemed likely that mortars could be expected to be even more complex in some respects than many concrete mortars.

2.3.5 A summary of the current shortcomings and a brief outline of the proposed work

In the area of the early life plastic properties, as anticipated the literature had shown that there were profound shortcomings. It had also failed to indicate any real problems in the areas in which it was hoped to proceed, although because these were new the precise areas had clearly not been covered. The major anticipated shortcomings were associated with the orders of magnitude through which some of the determined values for the rheological parameters to be investigated were known to vary.

Although in the earliest conceptual stages it had been hoped to be able to derive a relatively narrow range of numerical values suitable for the characterisation of various mortar types it had become apparent that whilst comparative work was undoubtedly feasible, the original objectives would be extremely difficult to attain with the equipment currently available. However, it also became clear that the use of the currently standardised and widely adopted crude methods for the determination and characterisation of the early life plastic properties was theoretically flawed and that accurate quantitative data on their accuracies and inter-relationships was not available, so that there was scope for more advanced concepts.

The literature indicated that rheological methods did appear to show promise and it was therefore resolved to investigate these. In the other general area investigated in this overall research programme, that of testing for the early life setting and stiffening rate properties, it was seen that a number of mechanical issues impinged on the test accuracy and it was decided to investigate instrumental methods that would be free of these influences and would be based instead on the measurement of chemical or physical processes that did not involve mechanical movement through the sample.

3 PRELIMINARY RESEARCH WORK

The preliminary work in this project consisted of the standardisation of all raw materials and work to confirm the core mix design and mixing procedures, as discussed below.

3.1 CONSIDERATION OF MATERIALS TO BE USED

Prior to commencement of the laboratory work consideration was given to the raw materials to ensure that they were standardised. For each of the key materials UK, European or other published standards or codes were sought to define the constituents to be used as the "standards" in this work. Where these existed, they were appraised and used if at all possible, to maximise the possibility of comparing the project results with other existing work in closely related fields. Standardisation also ensured that material parameters remained as constant as possible.

3.1.1 Investigation into the applicability of standard sand for use as the core sand in the project

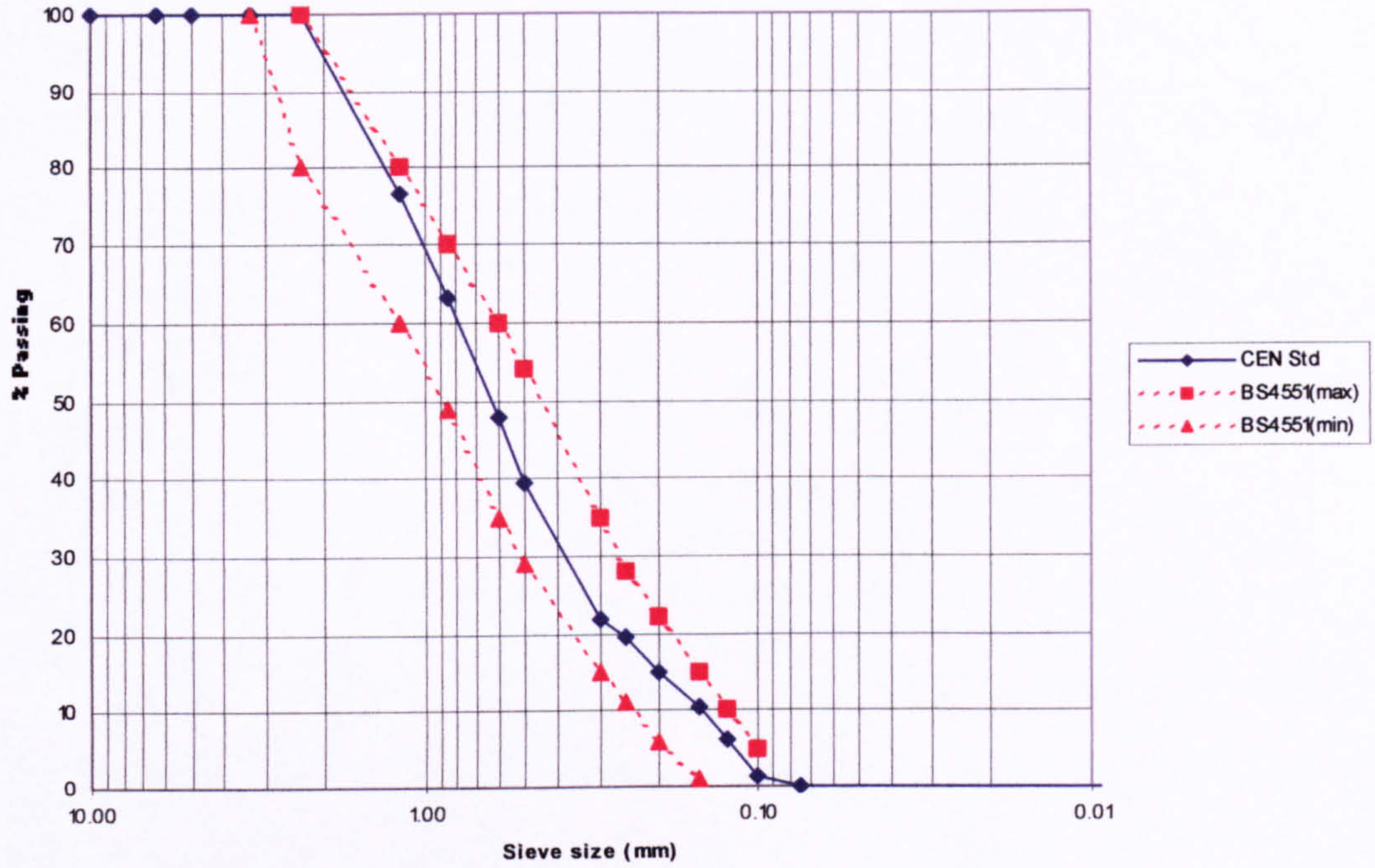
As well as standardising the grading of the sand, other parameters needed to be fixed. Because particle shape could clearly exert an influence on plastic properties, this was required to remain as constant as possible and it was thought that the use of spherical particles was probably the easiest way of achieving this. A stable chemical composition was also required, together with a chemistry that was as inert as reasonably possible, as much of the test mortar would contain admixtures and it was necessary to minimise any possibility of variable interaction with these, as might occur with some chemically complex aggregate types, as well as any other reactive mechanisms associated with these potentially "difficult" sands. The presence of clay species that would be difficult to characterise accurately in a research standard which required stable and non-complex properties was clearly undesirable, for several reasons. Firstly, clays react adversely with many admixtures by adsorption, which effectively begins to progressively remove the reactive grouping and diminish the effect of the admixture, thus changing the plastic properties.

Secondly, clays species impact on the free water, both by adsorption and absorption, thus progressively reducing consistence.

Thirdly, where present, clays are relatively complex to identify precisely and finally, clays are often present as mixtures of several different types with variation in the individual amounts of each type present being large. For these reasons a clean, natural siliceous sand was sought, with a rounded particle shape.

Consideration was given to sourcing such a material from a quarry, drying, sieving and then recombining to achieve a constant grading, but as standardised sands had been developed, these materials were appraised first. It was appreciated that they would still have some variability but as a condition of their acceptance as reference sands they were required to comply with stringent quality procedures such that variability would at least be minimised. BS 4551, 1998, states that where standardised sand is used for mortars it shall comply with the requirements of BS 4550, 1978, but this standard has now been superseded by BS EN 196-1, 1995, so that grading was considered. The definitions in the two standards varied somewhat, as can be seen from their gradings which are shown as figure 3.1 below.

Fig 3.1 The particle size distribution of some standard mortar sands



The only resulting theoretical issue identified was that the grading appeared to be coarse when judged in terms of current mortar sand gradings actually in use on sites. Nevertheless, preliminary mixes were made, using the standard grading shown in figure 3.1, with 1:3, 1:4$^{1}/_{2}$ and 1:6 mixes. Preliminary trials were then carried out on the plastic properties, using consistence by dropping ball test, and on the early life stiffening and commencement of set using the BS EN 1015 penetration equipment.

Unfortunately, as suspected, these tests showed the standard sand to be very poorly graded where plastic properties were concerned. There was a substantial and excessive amount of bleeding, even with the richer mixes and this meant that the accuracy of the dropping ball consistence test was degraded and that it became very operator dependant, as subtle differences in filling the mould, levelling and placing produced varying bleed levels and thus variable results. The penetration test was also clearly less accurate and pieces of relatively large sand could be felt intermittently presenting an erroneously high resistance to penetration as they were trapped underneath the penetrating plunger.

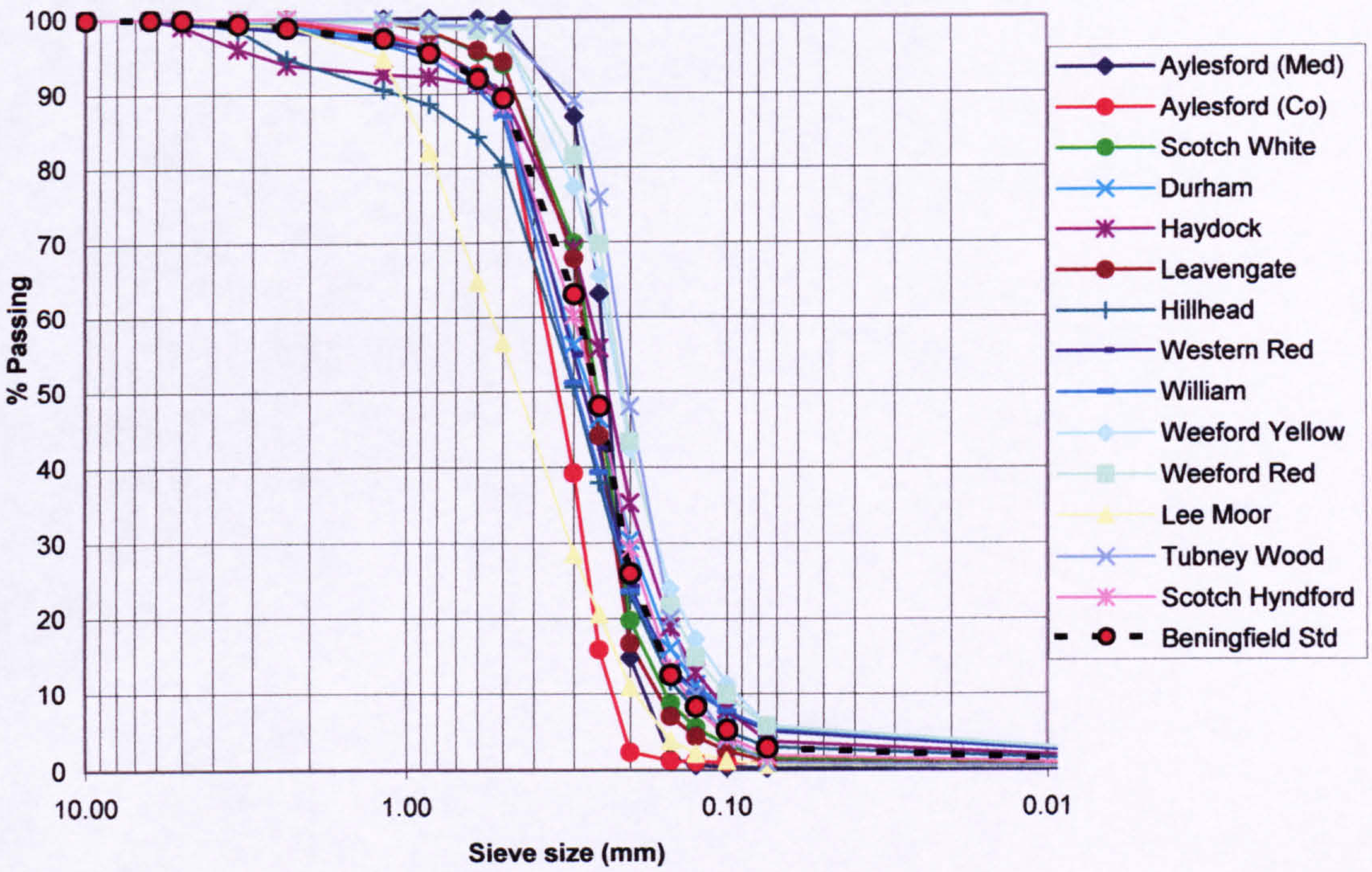
The applicability of this grading for use as a standard was therefore carefully considered and related to sands in actual usage, which were known to produce mixes with adequate plastic properties.

In order to approach the real situation a little more closely, a number of mortar sands were sampled from 14 major sand producing locations across Great Britain, representing a total gross usage in mortars of greater than 800,000 tonnes pa, or about 15% of all UK masonry mortar. It was considered that taking account of such wide usage would ensure that the final material was definitely representative of a usable grading. The gradings of these 14 sands are shown in fig 3.2 below, together with a further calculated empirical grading, which is the average of the 14, and is shown in the graph as "Beningfield standard".

Prior to adopting this sand as the "standard" sand, careful consideration was given to whether the concept of deviating from existing standardised materials was wise, having regard to the fact that a large number of laboratory standardised heavy building materials already exist and any addition must, from first principles, tend to represent a further and unnecessary proliferation, resulting in further complexity and cost, which must be deprecated. Nevertheless, it was decided that the decision was justified on technical grounds, as a suitable material just did not exist currently.

Preliminary appraisal of the proposed material was therefore carried out, using experienced personnel, and this showed that it had acceptable working properties.

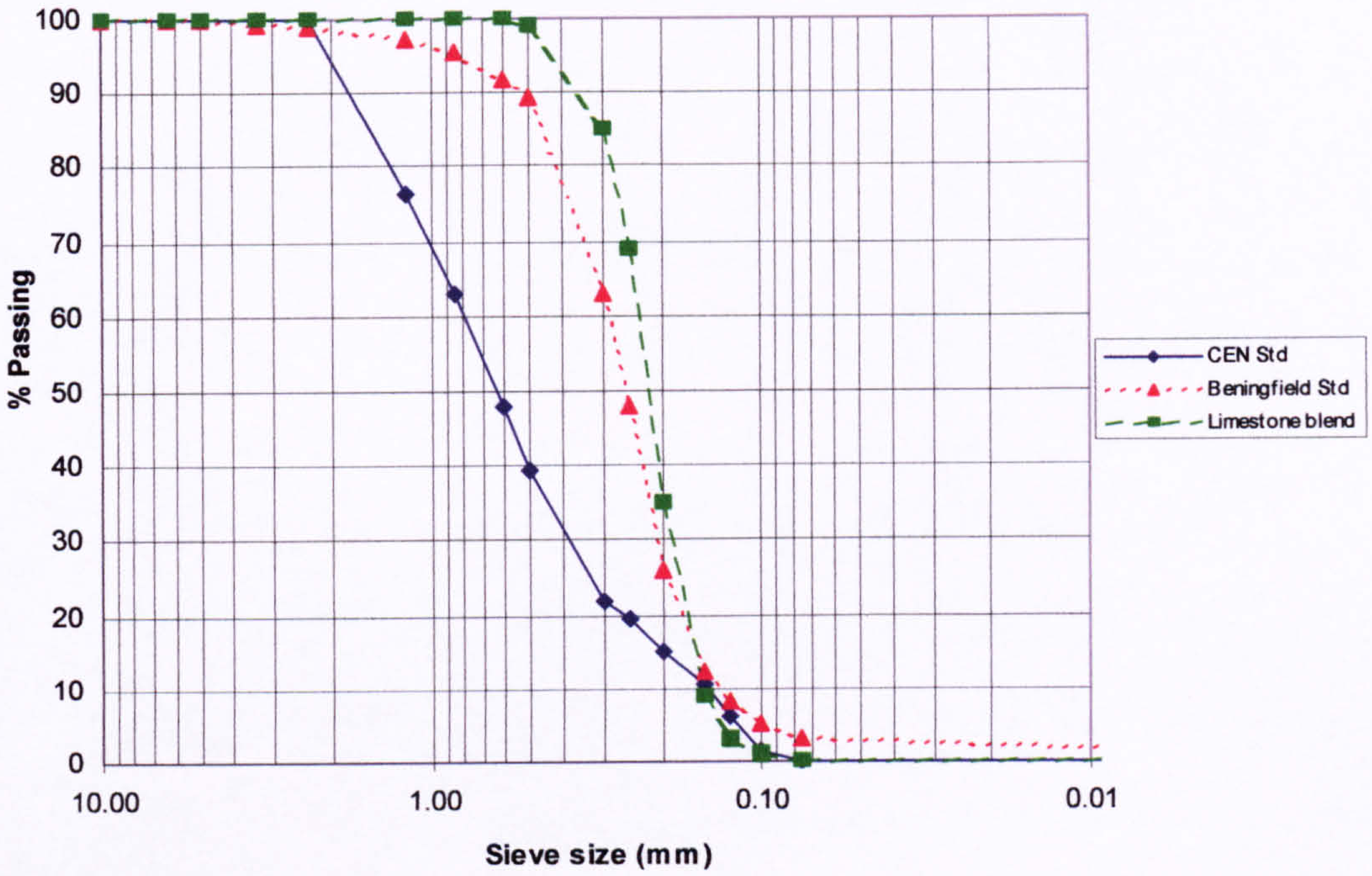
Fig 3.2 Particle size distribution for key mortar sand sources



This "standard " grading was then compared with the CEN standard sand, as shown in figure 3.3 below and it can be seen that the two exhibit fundamentally different characteristics when plotted on logarithmic graph paper. The straight line that is shown for the standard sand maximises the density, by filling the voids as much as possible, in the way that has often been described, as for example by Lees, 1981, as desirable for the production of the "best" mortar, using a so called "ideal" sand.

However, unpublished work by Pickard and Fowler at RMC Materials has also shown the importance of the 300 and 600μm particle size in enhancing the important plastic property parameters of plasticity, water retentivity and related properties that are so important to the overall requirements of a mortar. These workers suggest that these sizes are important because of surface tension and bubble size related issues. Their suggestions appeared to be validated in that the CEN standard was not actually usable in practice, but required the addition of the further fractions as they had suggested. The graph shown in Figure 3.3 also shows another line, which is shown as limestone blend in the key.

Fig 3.3 Particle size distribution for standard sands and limestone blend



This additional line arises as a result of further research that was carried out on a pilot plant/full scale basis at a newly commissioned dry mortar production unit. It is interesting to note that a very similar situation with respect to sand grading obtained in reality on an industrial scale at the quarry from which it was desired to produce large amounts of mortar sand for that new plant. The material in question was a crushed limestone and although this appeared suitable for mortar production, was not excessively coarse and complied with the current standard BS EN 13139, 2002, mortar produced from it had extremely poor working properties.

Because the production plant and available feedstock did not allow for economically viable changes to produce a better grading, the solution was to add fractions of natural sand where the grading possessed a shortfall. This was carried out on an empirical basis, with the sand fraction being added incrementally in fractions of 10%, the mortar being appraised each time to obtain an assessment of its working properties. As sand was added incrementally is was noted that the plasticity improved and the grading curve approached that of the standard produced for the mortar research and as shown graphically earlier in figures 3.1 and 3.2. Figure 3.3 illustrates this, which shows the final modified grading together with the "standard" that had been arrived earlier in the research programme by calculating a mean of the sands used in practice, as discussed earlier in this chapter. It is seen that the two were very similar and the blend that was successfully adopted in practice has proved most satisfactory in terms of user acceptability.

In light of the above issues and the demonstrable impracticality of the use of the CEN standard sand with respect to the plastic properties, it was therefore decided to use as a standard the empirical "standard" Beningfield grading devised earlier.

This revised standard sand was prepared by recombining a clean, washed, dried and fractionated material purchased from a specialist UK supplier of standard sands. This was not a straightforward procedure because the standardised fractions are not produced as fractions of the same size as those used in the research, which utilised a far greater number of sieves, with intermediate sizes required, so that assembling a given grading calls for some trial and error. When the grading had been arrived at satisfactorily, further preliminary work was carried out using this sand to produce mixes for testing of consistence by dropping ball and workable life (stiffening). These tests were satisfactory, with virtually no bleeding and segregation, and the re-combined standard was therefore selected for all of the main bulk of the future work.

The only potential problem with this solution was that the standard sand fractions were absolutely clean and pure, with no traces of the silt and clay contaminants that are present in the majority of the natural mortar sands actually in use on site. Whilst this cleanliness represented the most desirable research sand situation in virtually all respects, eliminating the possibility of extraneous reactions with admixture or binder, adsorption or other undesirable clay related problems, it did produce a material that was almost too clean and therefore not of perhaps the very best plastic properties, as the presence of even very small amounts of fine fines greatly enhances this aspect of a mortar sand. Nevertheless, it was decided that from the point of view of test integrity and reproducibility the chosen solution with the clean sand of very accurately known and reproducible properties was the best, albeit not that selected by many other researchers, possibly on cost grounds, as the standard sands were extremely expensive.

3.1.2 Standardised cement

The properties of the cement used clearly had the potential to influence virtually all of the work. The setting time was obviously relevant to the proposed work on the heat of hydration, as was the chemical composition, whilst the particle size, grading and particle shape all affect the plastic properties and their testing. Ideally, special research cement batches should have been utilised, with very tight control over these parameters, but this did not prove practicable and Cement Admixtures Association standard reference cement was used instead. Whilst not ideal, this was thought to be an excellent compromise. The material is assayed for key constituents, including tricalcium aluminate, a major setting time influencer and specific surface, another important parameter with respect to both the plastic property and the later life work. An abridged certificate is shown below, based by the author on a full certificate produced by the CAA and with acknowledgement to the latter.

Table 3.1 Properties of the standard cement

Property	Value %
Tricalcium silicate	55.6
Dicalcium silicate	16.7
Tricalcium aluminate	9.0
Tetracalcium aluminoferrite	8.1
Alkalis	0.67
Sulfate	3.3
Free lime	0.9
Specific surface m^2/kg	374
Compressive strength at 2 days N/mm2	29.9
Compressive strength, 7 days	47.2
Compressive strength, 28 days	57.5
Initial setting time, mins	100

It can thus be seen that the material is reasonably tightly defined. Moreover, all batches are taken from the same producing plant and this is specially selected to be one that exhibits minimum variation.

3.1.3 The adoption of a standardised mixing procedure

Having standardised the raw materials the next requirement was to standardise the mixing procedure. This was thought to require a fixed rate of shear/mixing energy to be applied, with the length of application or mixing time and the time of standing, if any, between starting and finishing mixing also to be standardised. All of these were thought to be important parameters, particularly with the time dependant systems and tests associated with the thixotropic properties of mortar. The procedure chosen consisted of 30 seconds dry mixing, a further 30 seconds mixing whilst water was added, followed by one minute of mixing. The mix was then allowed to stand for ten minutes and then given a final one minute mix. The mixer used had an axial blade rotation of 120 ± 25rpm and a planetary motion of 60 ± 15rpm.

The merit of the procedure was that a reasonable amount of mixing action was applied, almost certainly sufficient to obtain a mix that would generally be regarded as homogenous, but not enough such that high shear permanently destroyed substantial early chemical bonding. The 10 minutes standing and subsequent remixing was also thought to be in the right order of magnitude to allow any gypsum crystals that would form in the first few minutes to precipitate fully but then potentially to be broken by the final remix.

3.1.4 Testing using different aggregate cement ratios to confirm a standardised core mix design

Prior to commencement of the main body of the test programme consideration was given to standardising the aggregate cement ratio. It has been reported, Beningfield, 1987, that as a generalisation, masonry mortar mix design is based on the use of 1 part of binder to 3 of sand and this is the aggregate:cement ratio called up for the standard mortar in BS EN 196-1:1995. However, this mix is not widely used in practice, being richer than required for most applications, with the designation iii mix of 1:1:6

cement:lime:sand or I:6 cement:sand with air entrainment being the most common mix design, at least within the UK and the Northern European countries. Some workers have used 1:1:6 mixes for research, as well as air entrained mortars, Anderson et al, 1983 but it was considered desirable if at all possible to avoid any complexity whatsoever and choose a simple mix with a single binder for the majority of the work.

As the study of air entrained mortars would involve added complexity, particularly as entrained air is unstable and begins to reduce in amount immediately following mixing, thus producing a time dependant effect, it was decided to use non-air entrained mixes for the core work of the project, and preliminary work was carried out on 1:6, 1:4 and 1:3 mixes, with a view to adopting one of these as standards. It was found that the 1:6 was completely unusable as it bled profoundly, making measurements of consistence and flow virtually meaningless. Even the 1:4 exhibited excessive bleed. These findings were not unexpected, as in practice mortars leaner than about 1:3 are used in conjunction with admixtures and/or lime.

As a result of this preliminary work a 1:3 mix was adopted as the standard mix for the project. It could be argued that this was richer than would normally be used but it was nevertheless adopted in order to minimise bleeding and segregation and allow much more accurate assessment of the plastic properties. It should also be noted that this philosophy accords with that adopted nationally for many years with mortar testing in a number of countries, including Belgium, France, Germany, Italy, Portugal and the UK, as reported by Joisel, 1949. With the exception of the practical work involving bricklayers which is reported in section 3.2 below, or unless particularly stated, all of the work was carried out using this mix design.

3.2 CONSIDERATION OF THE PLASTIC PROPERTY TESTS AND THEIR RELEVANCE TO SITE PRACTICE

As discussed in the literature survey, many standard tests are in use to determine various of the plastic properties of mortars and a far larger number have been proposed but not standardised and adopted, although nevertheless being used to a limited extent. These tests have almost always been developed in laboratories or research institutes, environments that have unfortunately been generally separate from the reality of building sites where the mortars are actually used. It is suggested in the literature, Peroni et al 1981, that *none of the tests currently available yield results which correlate well with the opinion of a competent mason.* In other words, no test yet exists that gives a better result than the operative's judgement. There are some exceptions to this statement, in particular the now superseded British dropping ball test, BS 4551, 1998, which was developed in collaboration with bricklayers whose views were said to have been taken account of, but this situation appears to be an isolated one.

With the exception of that test, it proved difficult to find work relating the development of "scientifically based" tests to the on-site operative assessments that are undoubtedly made on mortars in practice.

In reality, mortars on a building site are rarely subjected to formal test, certainly not those that are site mixed, but it is certain that a degree of informal testing or assessment is used by the masons to appraise the mortars that they are asked to use. If they perceive the plastic properties to be inadequate, in terms of either the speed, ease or quality of output then the mortar fares badly in this unofficial test regime and may well be modified, perhaps by the unauthorised addition of undesirable admixtures such as domestic detergents. The intention of this preliminary part of the work was to consider whether the existing tests could usefully be used to interpret and relate to the practical reality of a masons assessment. Additionally, if this was not the case, to consider the feasibility of developing a method of interpreting a masons assessment of plastic mortar properties in a way that could be measured and used as an objective yardstick for comparison. If successful, it was hoped that this could remove or at least substantially reduce the subjectivity and lack of precision that has always been associated with this area to date.

To describe mortars, words such as "heavy", "sticky", "body" and "dead", to name only a few, are used by operatives and others but often with little uniformity in meaning or interpretation. It was hoped to quantify these terms and to relate them to measurable test parameters, with the long term aim of being able to use tests to compare different materials and systems objectively.

The separate objectives were therefore as shown below:

- To investigate and interpret plastic property terminology, and if possible to attempt to quantify the criteria used in appraisal and description.

- To rationalise and group these criteria into the lowest possible number of separate types/headings.

- To add to the criteria with any further properties obtained by a consideration based on first principles.

- If possible, to relate all of these criteria to measurable parameters and test procedures.

It was then hoped to be able to use these findings in the future work to interpret practical expressions and needs of mortar properties in a measurable way, and thus understand objectively how they could be met, or in what respect and by how far they fell short if that was the case.

3.2.1 Introduction to the investigation

Prior to commencement of the work, bricklayers were employed on a so called "day work" basis, that is with their income not depending on their productivity, which might otherwise have influenced their views. The bricklayers were initially questioned separately, without prior knowledge that this was going to occur, and examined as to how they formed a view on the mortar that they used and the way in which they informally quantified its properties in their own minds, or perhaps sometimes in discussion with a foreman or their labourer on site. It was hoped that they would be able to describe their methods of assessment and terminologies.

A logical basis for each of their assessed properties was then sought and the resulting list of those that seemed valid compared with a list of plastic properties derived from first principles. It was proposed that the latter would cover all of the interviewee's points, with the probable addition of some that they had not considered, as the list based on first principles was derived from a protracted period of experience, discussion and then brainstorming. The long term aim was therefore to be able to produce a final and definitive list of plastic properties, based on scientifically derived first principles but incorporating user experience and views. The last stage of this preliminary part of the project was to attempt to interpret the final list of properties in a way that would enable them to be quantified in future comparative research and development work. In this final stage, accepted tests were used if they existed but at the planning stage it was thought unlikely that these would cover every parameter, in which case procedures new to the testing of mortars were to be considered for development.

3.2.2 The classification of all the plastic properties

The interviews with the bricklayers produced results that were very helpful, generally comprehensive and clearly based on a substantial amount of consideration on the part of the interviewees.

The results of these interviews are shown in table 3.2.

Table 3.2 The results of the interviews with bricklayers to describe their views of plastic mortar properties

Bricklayer 1 Inexperienced	**Bricklayer 2** Very experienced, but not analytical in thought process	**Bricklayer 3** Reasonably experienced but analytical and articulate
Easy to play with when cut	Easy to spread	Needs to have a good amount of body
Aerated	Not too sticky	Should not be too sticky, to itself or to the trowel
Not too dry	Not too fluffy	Has the right amount of adhesion to the trowel
Fluffy		Cuts off cleanly when joint is finished
Not too Fluffy		Enables units to be pressed well into place
Not too heavy		
Has enough air		

In addition to these three bricklayers, further people were interviewed who were involved in the industry or in associated research fields in positions where they might have been expected to provide meaningful input to the process. Tables 3.3 and 3.4 below show interview results for 6 such people with involvement in different facets of the industry, all with a close knowledge and wide experience of mortars, two technicians and three researchers working in the field.

Table 3.3 The results of interviews with technicians to investigate their views of the plastic mortar properties

Technician 1, 5 years experience	**Technician 2** 3 years experience
Creamy	Workability
Light and spreadable	Spreadability
Cohesive	Cohesion
Not too grainy	Movement on placing
Not too heavy or podgy	Stability on placing
Light	Movement on pointing
Not too sticky	Stability on pointing
Easy to spread and place	Body
	Volume of material on trowel
	Creaminess or plasticity

The views of the three researchers working in the field together with those of one technologist/manager, were then recorded as shown in the table below.

Table 3.4 The views of four masonry experts

Researcher 1 25 years masonry research experience	**Researcher 2** 20 years mortar/admixture research experience	**Researcher 3** 30 years mortar experience	**Technologist** 25 years building materials technology experience
Should be thixotropic	Adequate workability	Fluidity	Spreadability
Low Poissons ratio	Ease of trowelability	Not too heavy	Not too sticky to trowel
Easy to strike off	Spreadability	Not too sticky	Non-excessive bleed
Lack of bleed under load	Cohesion	Non-excessive bleed	Retains flow on spot board
Plastic after laying (deformable)	Deformation under load	Cohesive	Retains flow in tub
Develop bond rapidly	Density	Adhesive	Cohesion
Optimal properties with all unit suctions	Air content	Not too-thixotropic	Smoothness on trowel
Maintain initial workability for sufficient time	Viscosity	Easy to spread	Close joint with trowelling
Chemically compatible in wet state	Water retention	Easy to deform	No fissures in joint
Develop early stiffness and adhesion	Amount of bleed	Stiffens at correct rate	Not too heavy
	Finishability	Finishes acceptably	Good water retention
	Colour		Good adhesion
			Sufficient body on trowel
			Light
			Good bed length possible
	Optimum and stable air bubble size and distribution		Stable after placing
			Resistant to joint pull out when brushed

The contents of all of the questionnaires were then considered and an attempt made to incorporate every comment into as succinct and comprehensive a final definitive listing as possible. The problem was then also considered from first principles and a final list produced, as shown in table 3.4 below, which incorporated both the practical empirical approach and that based on first principles.

Table 3.5 An interpretation of the finally derived plastic mortar properties

The perceived properties	Colour
	Amount of bleed water
	Presence of any visual grading anomolies
	Noticeable light or other floating particles
The initial plastic properties	Harshness/smoothness
	Cohesion
	Optimum weight
	Adhesion to trowel/units
	Max. amount that is stable on trowel ("body")
	Ease of spreading
	Ease of furrowing
	Ability to deform and flow when unit is plumbed and levelled
	Ease of finishing
The more time dependant properties	Rate and degree of stiffening in a mortar tub or similar
	Rate and degree of stiffening on spot board
	Rate and degree of stiffening when laid
	Ease of joint finishing
	Resistance of joint to tearing when cleaned

It was believed that this list was reasonably comprehensive and covered all facets of the so called workability or plastic property area.

The final stage of this exercise was to attempt to classify the properties into a group of measurable parameters that were covered by test methods. In the absence of existing test methods for any of the properties, the possibility of developing appropriate ones would be considered. Table 3.6 below shows all of the above properties, together with the test/parameter that was believed to define each.

Table 3.6 The plastic mortar properties and proposed objective criteria

Property	Test or criterion
Colour	Visual observation or spectrometer
Amount of bleed water	Grading, air content, mix design
Visual grading anomalies	Grading
Light or floating particles	Impurities in sand
Harshness/smoothness	Grading and shape, air content, mix design
Cohesion	Flow at fixed consistence, *but see [1] below*
Weight	Air content *(assuming no aggregate anomalies)*
Adhesion in plastic state	No accepted test known to the writer
"Body"	Air content & flow @ fixed consistence, *but see [2] below*
Ease of spreading	No known mortar test
Deformability	No known mortar test
Ease of finishing	No known mortar test
Stiffening in tub	Air stability, water loss, thixotropy
Stiffening on spot board	As above, and weather dependant
Stiffening when laid	As above, plus water retentivity
Resistance to tearing	Aggregate shape, thixotropy, water retentivity

[1] Although cohesion is said to be measured in this way it is suggested that at best this is an imperfect test regime, as both of the properties involved interact and show interdependency. Additionally, it was

thought unlikely that their relationship was fixed, which meant that a measurement taken at one value of consistence gives rise to a flow that is valid only at that value.

[2] *The comments as* [1] *above are also valid here, but in addition there appear to be influences from other rheological properties that are not covered by any of the tests shown.*

This list was then simplified somewhat by removing duplicated tests to give the known tests/requirements for which there appeared to be a need as shown below:

Visual observation

Grading and particle shape

Air content

Flow at fixed consistence or other related/improved parameter

Thixotropy and other time related behaviour

Water retentivity

A test or tests for the properties affecting spreading, deformability and finishing.

In addition, the imperfect nature of the assessment of cohesion by flow at fixed consistence should be appreciated and addressed if capable of solution.

It was considered that this preliminary work on plastic mortar appraisal did indicate that the concept of using bricklayers opinions in a quantitative questionnaire based format had some validity, and it could be replaced by more objective appraisal using laboratory developed test methods, as listed above, so long as tests could be developed in places where the work had shown there was a current shortfall. It was clear there were areas within the field of plastic property testing that were not adequately covered by existing test procedures, in particular those involving the more complex rheology of the system.

It was also clear that the current methods of test for the plastic properties of mortars included many that appeared to measure the same property, and that there were no existing test methods for some of the properties that had been identified. Additionally, one existing procedure, the determination of flow at a fixed consistence needed further analysis. Ease of spreading, deformability, finishing characteristics, stiffening behaviour and thixotropic behaviour were all properties identified as needing characterisation, using test methods as yet unidentified. The future work requirement could thus be considered as follows:

- A preliminary trial to validate the final list of plastic properties and their use in practical appraisal.
- An investigation into current methods of test for plastic properties, particularly flow, consistence and related properties.
- Analysis of exactly what flow at fixed consistence represented.
- A search for a way of measuring ease of spreading, deformability and early life finishing characteristics.
- An investigation into methods of test for thixotropic behaviour.

To this list may be added a need for a procedure for the further and better consideration of the plastic properties at a slightly later stage in the life of the material, where thixotropic stiffening begins to interact with the very early part of the setting reactions, and later life setting commences, the time during which the so called stiffening rate is characterised. This would be considered in phase two of this overall project.

Prior to investigation of the main issues outlined above, one further preliminary trial was carried out to attempt to further validate the basic principle of an approach based on the final property list shown as table 3.5. to appraise a test mortar. In this work, three bricklayers were again retained on a day work basis and given the task of laying bricks and blocks to produce full size walls, using mortars produced in the laboratory and characterised as far as possible using the current test methods where these existed. The aim of this trial was to check that the bricklayer's productivity, achievable quality in areas like joint finish and overall opinion was in agreement with the suggestions arising from the laboratory appraisals.

3.2.3 Trials to confirm the interim test procedures

The initial mortar used for this work was produced in the Readymix dry silo plant in Doveholes Quarry, Derbyshire and its composition is shown in table 3.7 below

Table 3.7 The composition of the mortar used for the test verification trial work

Aggregate kg		**Cement kg**	**Air Entrainer kg**	**Retarder kg**	**Water Retainer kg**
Lime stone	Silica Sand	CEM 1	PMA 950 Sodium Alkyl Sulfate	PMR 852 Sucrose	PMW 551 Methyl Cellulose
43.57	43.77	12.61	0.005	0.019	0.028

The units used were two brick types of high initial rate of absorption, one brick of relatively low initial absorption, aggregate concrete blocks and lightweight autoclaved aerated concrete blocks. It should be noted that the actual total absorption of units is not the key factor in early life plastic mortar unit to interaction, but rather it is the initial rate of absorption that is the major determinant of performance. The British Ceramic Research Association, (now CERAM), 1980, published details of a simple test to determine this, which consists of partially immersing a unit in a shallow tray of water and weighing the amount absorbed after a period of one minute. Table 3.8 below shows the initial rate of absorption of the units used as determined using this test procedure. Also shown is a proposed view of a relationship between the numerical test value and the magnitude of the rate of absorption defined in descriptive terms. It is appreciated that these assigned values may be criticised on the grounds of subjectivity but they were used to assist in a more rapid understanding of the work and only as a means of complementing the numerical values determined by test. They may be considered as subordinate to the quantified test values.

Table 3.8 The properties of the units used for the test verification work

Unit type	Initial rate of absorption $kg/m^2/min$	Description of absorption
"Multi cream" stock brick	3.6	High
"Old English Rose" rustic	2.3	Medium to high
Class B engineering brick	0.2	Very low
Dense concrete block	0.5	Low
Very lightweight aircrete block	7.5	Very high

An attempt was made to characterise the mortar in two ways, by using the questionnaire and appraisal devised earlier, and by the use of BS 4551 and other standardised test results. The results of these procedures are shown collated in table 3.9 and 3.10 below, with the bricklayers being asked to assign a value of from 1 to 10 for each property, with one representing the worst result, ten the best.

The two bricklayers were in good agreement, (even though they had not had the opportunity to discuss their opinions with each other), as shown in the tables.

Table 3.9 The bricklayers assessment of the properties of the mortars determined by the use of the final version of the questionnaire

Property	**Bricklayers assessment**	
	Bricklayer 1	**Bricklayer 2**
Colour	Acceptable	Acceptable
Amount of bleed water	10	10
Presence of any visual grading anomolies	10	10
Noticeable light or other floating particles	10	10
Harshness/smoothness	10	10
Cohesion	10	8-9
Optimum weight	9	9
Adhesion to trowel/units	10	8-9
Max. amount that is stable on trowel ("body")	10	8-9
Ease of spreading	10	10
Ease of furrowing	10	10
Ability to deform and flow when unit is plumbed and levelled	9	10
Ease of finishing	9	9

Rate and degree of stiffening in a mortar tub or similar	9	8
Rate and degree of stiffening on spot board	8	9
Rate and degree of stiffening when laid	9	10
Ease of joint finishing	10	9
Resistance of joint to tearing when cleaned	9	9

These results were not really satisfactory because, as may be seen from the table, the operatives assigned a value of 10, the highest available, to many of them and the ability to use this procedure to characterise and discriminate between mortars with properties in this order was thus seen to be poor. This was not seen as cause for a complete rejection of the methodology used, because it was thought to be at least partly due to the fact that the mortar was of optimised properties and at that high level of performance, discrimination was poor. Previous ad hoc experience had shown that at lower levels of product quality, operatives appeared more able to discriminate, but it was nevertheless clear that as mortar performance reached high levels there was a need for more test data.

The standard test methods were then considered, with consistence and flow both being measured by the methods of BS 4551 and BS EN 1015, for two relatively extreme conditions of consistence, one appropriate to the autoclaved aerated concrete and one to the dense, low absorption brick, as shown in table 3.10. Once again the test sensitivity did not appear to be good and it was certain that the laboratory test procedures failed to integrate with the questionnaire based appraisal. It was seen that in general these test procedures did not really fully characterise the good mortars used, and would certainly have failed to provide a detailed characterisation of those properties that the practical work had shown to be desirable. The exception to this statement was the test for water retentivity. This was seen to be of some worth, as the value recorded was one that would be regarded as very good, and this was born out by the practical appraisal. It also accorded with theory and expectation as the mortar was a sophisticated formulation, the result of a substantial programme of work to optimise its plastic properties. The remaining tests would perhaps have been of some value in that the flows at the consistence values tested were relatively low, thus pointing to a material with good cohesion, but relating this one parameter to the overall expectations of the bricklayers would leave many areas of product requirement completely unexplored.

Table 3.10 The properties of the mortar used in the trials tested in accordance with BS 4551

Plastic mortar property	For the low absorption brick used in the trials	For the high absorption aerated concrete blocks used in the trials
BS 4551 consistence, mm	8.5	11.0
BS 4551 flow, mm	130	145
BS 4551 water retentivity	90	80

Following on from the initial testing and appraisal of the material, the trials proceeded with the objective of studying the time dependant characteristics of the mortar when used to lay the units of differing initial rates of absorption. The criteria used here were simply for how long could the unit still be adjusted and how long could the joint then be left before final finishing. This work is shown in table 3.11 below, based on practical trials using a bricklayer and bricks and blocks that possessed initial absorption characteristics that tended towards the extremes. It should be borne in mind when considering these results that the mortar contained a retarder so that the time periods shown for the denser, lower absorption units are much greater than would be expected for non retarded mortars, but of course mirrored the situation that obtains on a large number of contemporary construction sites.

Table 3.11 The time dependant characteristics of the mortar when used with the units of differing initial rate of absorption values

Unit type	**Time to first point** (mins)	**Time to second point** (mins)
High absorption aircrete	4	$8^1/_2$
Absorbent brick	$1^1/_2$	$6^1/_4$
Dense concrete block	185	233
Dense brick	269	298

This part of the trials was successful in some respects as the masons proved adept at identifying these values, with the procedure working well, although effectively the unit properties were also the variable, as different types were used with one mortar.

Additionally, the trials showed that there appeared to be a relationship between the bricklayers views of the working life and the water retentivity, albeit on the basis of this very limited amount of work, in that this mortar of high water retentivity also gave rise to a long open time on site.

Further work was then carried out using the two new European test methods given in BS EN1015-9, which are described as tests for the determination of workable life and correction time. Although these procedures were published specifically for use with thin-layer mortars, there did not appear to be any reason why they should not be applied to general purpose mortars that were designed to be used in mortars of normal, that is nominal 10mm, thickness.

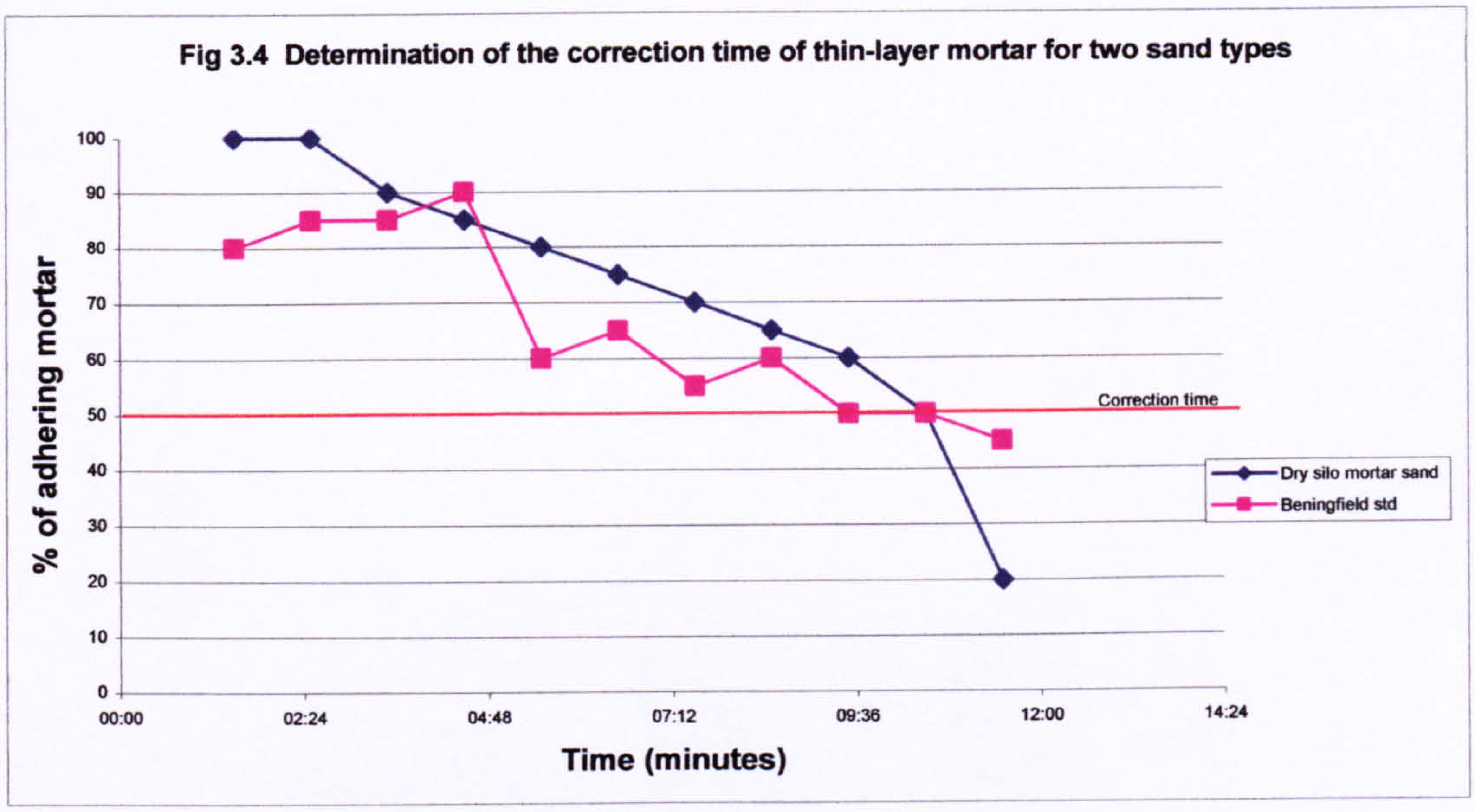
The first procedure was based on determining the flow of the material initially and then carrying out further determinations at fixed time intervals of 15 minutes until the flow had decreased by 50%.

The second procedure consisted of pressing small 50 mm cubes that had been made by cutting a standard block and conditioning at 70 ^{0}C to constant mass and then storing at 20^{0}C and 65% relative humidity for 2 days prior to testing, onto a layer of mortar applied to a substrate composed of a full size block placed flat on the bench. Blocks were then removed progressively, one at a time, at successive time intervals of one minute and the adherent area, which was easily identifiable as a pronounced grey stain and a smooth surface, as opposed to the original textured one, was reported.

The first procedure, based on the flow table, was simple and easy to use but clearly only addressed the hydration/drying process. The second was much more difficult in that it required dexterity and some skill but it also took some account of the water retentivity of the mortar when applied to a background that provided suction. It was thus arguably in some respects more relevant to actual site conditions but if the achievement of this situation has resulted in an unacceptably high variability then it could be argued that a simpler but less variable test would be preferred, particularly in the case of a CEN reference test as opposed to eg a pure research test.

The results of correction time determinations using the procedure based on adherent area for two 1:3 mortars, one based on the "standard" Beningfield sand defined in fig

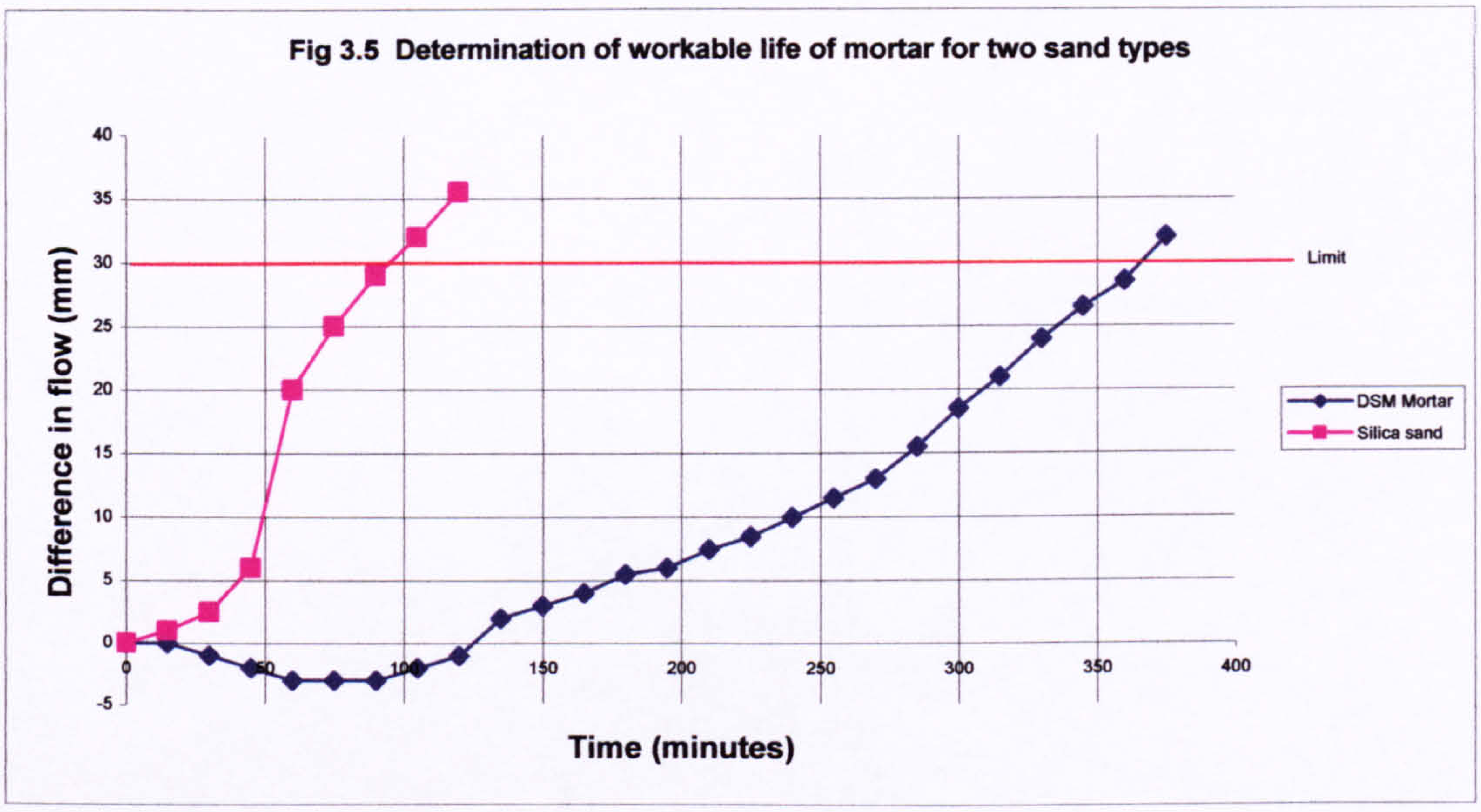
3.2, and one on the sand used for dry silo mortar production, also as described earlier in this section, are shown below in figure 3.4.



This method was surprisingly easy to use but protracted in that it required the preliminary cutting to produce the small cubes, followed by washing, oven drying and then conditioning. The execution of the actual determination, which involved manually pulling the small cube away from the surface, was easy although in the earlier stages it was thought that imparting a slight, subconscious twisting motion could perhaps result in a minor increase in the apparent adherent layer. In the event, this did not prove to be an issue.

It may be seen that each sand type followed the same general form and the values for the time at which the flow value reached 50% were in surprisingly good agreement, having regard to the manual nature of the majority of the procedure. The dry silo material showed a more progressive, less erratic trend, but on the basis of just one series of determinations it would be unwise to draw any conclusions from this.

The alternative method of BS EN 1015-9, that based on progressively decreasing flow values, was then appraised and the results for this are shown in figure 3.5 below.



This method was quick and simple and required none of the laborious preparatory work of the adherent area method. However, it did not really measure the same parameter as that method. The change in flow is a function only of the properties of the mortar in isolation and takes no account of the substrate. It is thus of value where a quick and easy absolute value is required and should show good reproducibility, although there was insufficient resource to test this hypothesis. In contrast, the adherent area method takes account of the water retentivity of the mortar as well as the early life stiffening and in that respect is more allied to a specific site situation, especially if the appropriate units are used. It is, however, probable that it will show a reduced level of reproducibility, in particular because of variability in unit suction from one laboratory to another.

The time for which the joint remains workable enough to finish thus appeared to relate to the properties of the mortar in isolation, the unit initial rate of absorption and the mortar water retentivity, but these factors alone cannot fully define the totality of the mortar properties and requirements. It seemed also that some rheological property or properties in advance of the appraisal and test procedures used to date in these trials were relevant, particularly in the very early life when the mortar is still very plastic and deformable. It was believed that resistance to deformation, thixotropy and ease of finishing were important properties in this context, together with the early commencement of setting that takes place in the stiffening phase that is encompassed by the current test procedures.

In conclusion, the work showed that water retentivity was of value, as was one or other of the correction time tests, preferably that based on area of adhesion, but neither the properties arrived at from the process that used the questionnaires nor those determined by any of the standard testing regimes provided a comprehensive assessment of the mortars.

Based on these trials and as discussed earlier in the project, it was clear that more work was required in these areas. In particular, the methods of measuring consistence, the validity of using flow after consistence to measure cohesion or some related property and the more detailed rheological properties, including deformability, thixotropy, and time dependency need further investigation. These points confirm the earlier conclusions reached in section 3.3.1.2.

4 AN INVESTIGATION INTO ALTERNATIVE TEST PROCEDURES FOR EARLY LIFE PLASTIC PROPERTIES

Although, as previously discussed in the literature survey, it is often suggested that there is a unique attribute to some of the single point tests, it was becoming evident that in general they were measuring the same combination of plastic properties, although to different sensitivities and with differing degrees of accuracy. This is reflected to a certain extent in BS EN 1015-4, 1999 which states that *Normally there will be a linear correlation between the plunger penetration value, measured according to this test method, and the flow value measured in accordance with BS EN 1015-3, for the same type of mortar with increasing water content, but the slope will differ with different types of mortars.* Despite this suggestion, there was little comprehensive published work to show how the various single point test methods actually related to each other or how their sensitivities varied. It appeared to be worthwhile to investigate these issues rigorously in order to form a view as to which of the available procedures was the better overall and whether there were valid relationships between them.

4.1 BS 4551 DROPPING BALL

The dropping ball test, shown earlier in Figure 2.1, was originally developed in the Building Research Establishment, (then the Building Research Station), in the 1960s and is effectively just a simple penetrometer. As it uses gravity and a ball of known mass to apply the energy, the force should always be constant. The ball falls freely, thus eliminating any of the possible frictional errors that are a possibility with most other penetrometers, for example the Vicat and the newly adopted European DIN plunger test.

The only adjustment possible with the dropping ball apparatus is the length of fall of the ball and once set this remains fixed. So long as the operator uses the equipment properly, allowing the ball to fall freely without touching the hand, then the apparatus itself and the test are remarkable free of potential inaccuracy and/or variability. The only issue with the test, which is an issue with all mortar tests where plastic mortar samples have to be placed in the apparatus, is the potential variability associated with filling the mould and consolidating the sample.

Although the British Standard, BS 4551, 1998, gives a reasonably closely prescribed procedure, the accuracy of the test is clearly dependant on the amount of compaction used to fill the mould, the way in which it is struck off, the presence of any bleed water and the way in which this is dealt with, if present. The standard attempts to control these issues, carefully defining the number of increments with which to fill the mould and the way in which it should be struck off, but some experience is nevertheless needed to produce excellent results, which indicates the potential for good repeatability but with reproducibility that will almost certainly be less satisfactory. Because the sample has a relatively large surface, the potential for inaccuracy in this area is large.

Newson, 1986, reported that there was a relationship between dropping ball value and water content and showed a graph in the form of a straight line but this was based on only a limited number of results. In order to investigate this relationship in detail and also to determine the range of moisture contents over which the method was usable, a programme of work was carried out. The standard sand that had been developed as described in section 3.1.1 was used initially, together with the standard reference cement and the 1:3 cement:sand mix adopted for the earlier work.

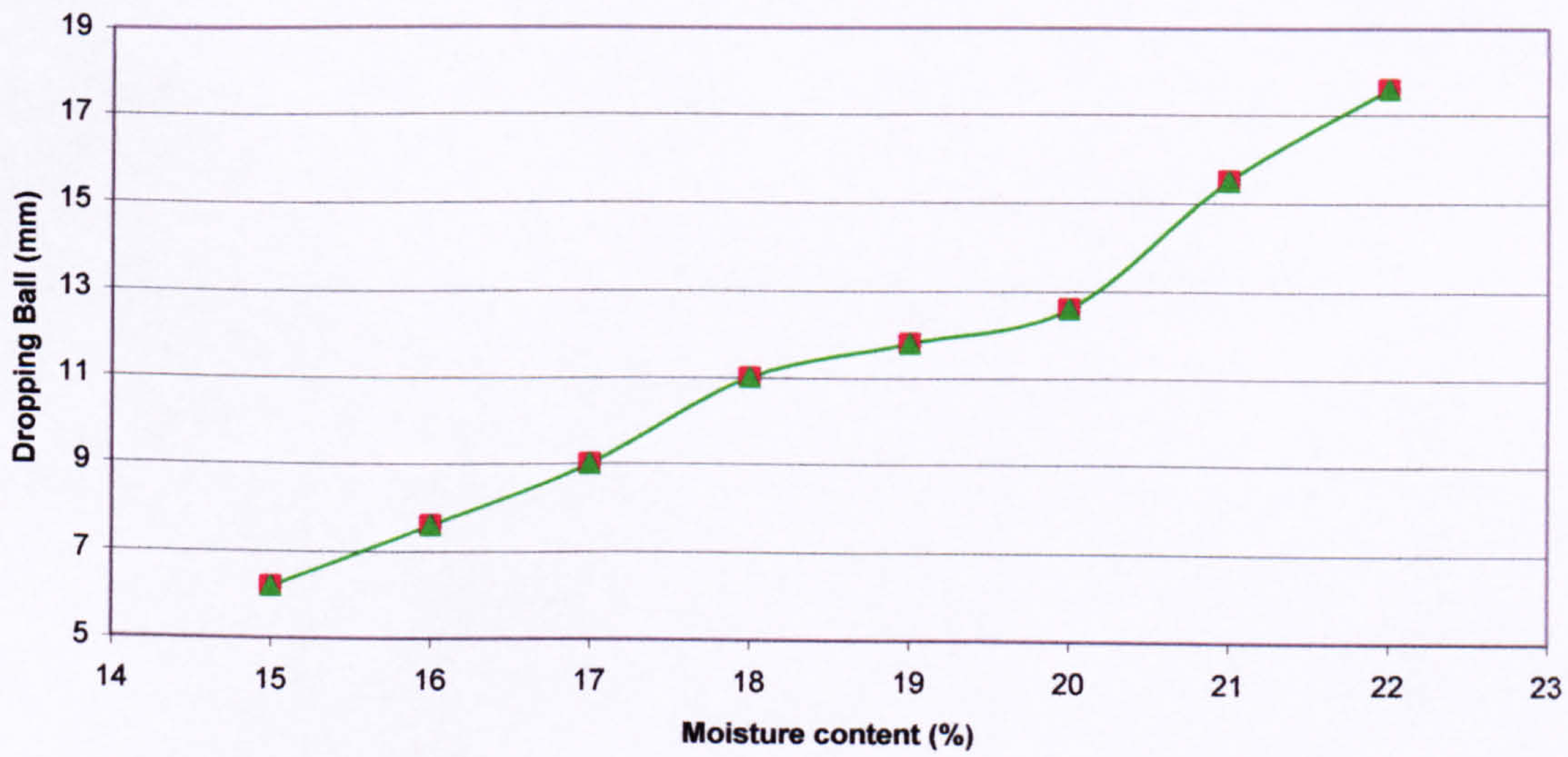
The work was carried out by producing a standard mix at a low moisture content, determining the dropping ball value, adding an increment of water, mixing and re-testing and repeating this procedure progressively until the approximate upper limit of test usability had been reached. This procedure was carried out as rapidly as possible in order to minimise inaccuracies caused by the material beginning to stiffen and set, thus potentially producing an erroneously low penetration value for the dropping ball. The alternative procedure, of making a different mix for each moisture content was considered but discarded because it was judged that this could have potentially introduced even greater errors as a function of the need to continually re-weigh and re-batch and mix the successive mixes to close tolerances. The addition of a retarder was also considered but the vast majority of these also exert a plasticising effect, which would have introduced another variability, particularly as repeated mixing with a plasticised mix would almost certainly have been associated with further plastic property changes of an increased magnitude.

In order to check on whether the total time that elapsed throughout a complete test run was not so great that the mortar was actually beginning to set a little and thus produce a low result, a duplicate determination was made with a mix made up to the highest moisture content. This work validated the chosen technique and showed the methodology selected, that of progressively adding water, to be valid.

The results of this dropping ball consistence trial, shown in figure 4.1, appeared to indicate that the relationship was not straight line, as suggested by Newson, 1986, but rather that there was an initial curved or curvilinear relationship, followed by a flattened area, which then again became curved. This was thought to be due to the material reaching a transition stage. Perhaps a little simplistically, it could be said that from a wet solid, with inter-particulate interaction, the material enters an interim phase, and then becomes a liquid with solid particles in suspension. More conventionally, the material begins a transition from a pseudoplastic solid to a liquid. During the transition phase the water is displacing the air so that there is little change to the consistence value until the process is complete.

This mechanism is known to exist in other fields as eg with limestone slurries used in the manufacture of cement. In the case of mortar, as there is known to be in the order of 6% entrapped air present in the mix types under investigation, it seems reasonable to assume that this is replaced by water when the system becomes wet enough for liquid water to freely enter the voids.

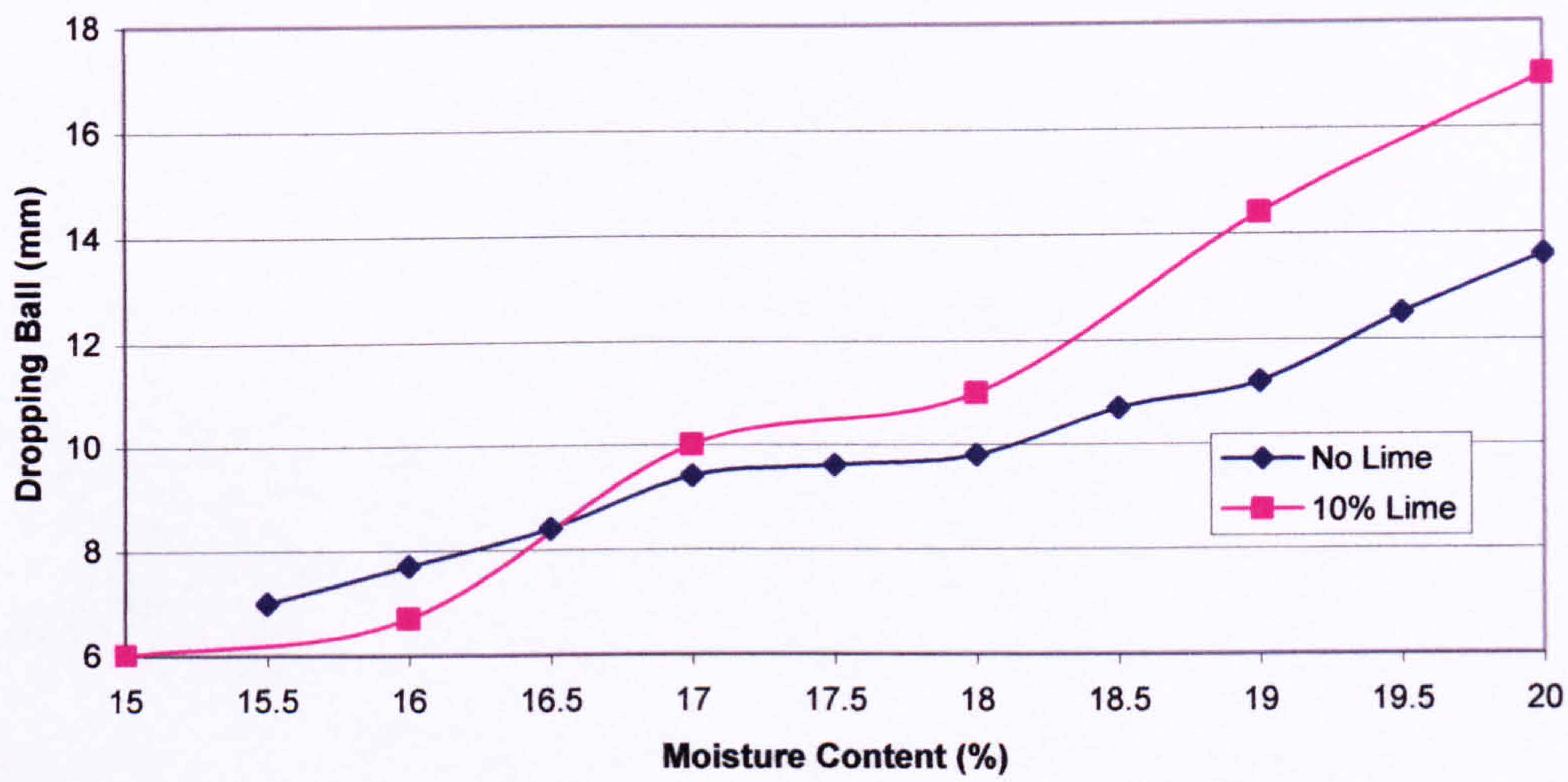
Fig 4.1 The effect of moisture content on dropping ball value using Beningfield "standard" sand blend



Because this phenomenon of the "dip" in the graph corresponding to the postulated transition zone did not appear to have been previously reported in the field of mortars, indeed a simple straight line relationship having been proposed, as outlined above, the work was repeated using different mix types, with a cement: lime: sand mix used in addition to the "standard" cement sand mix.

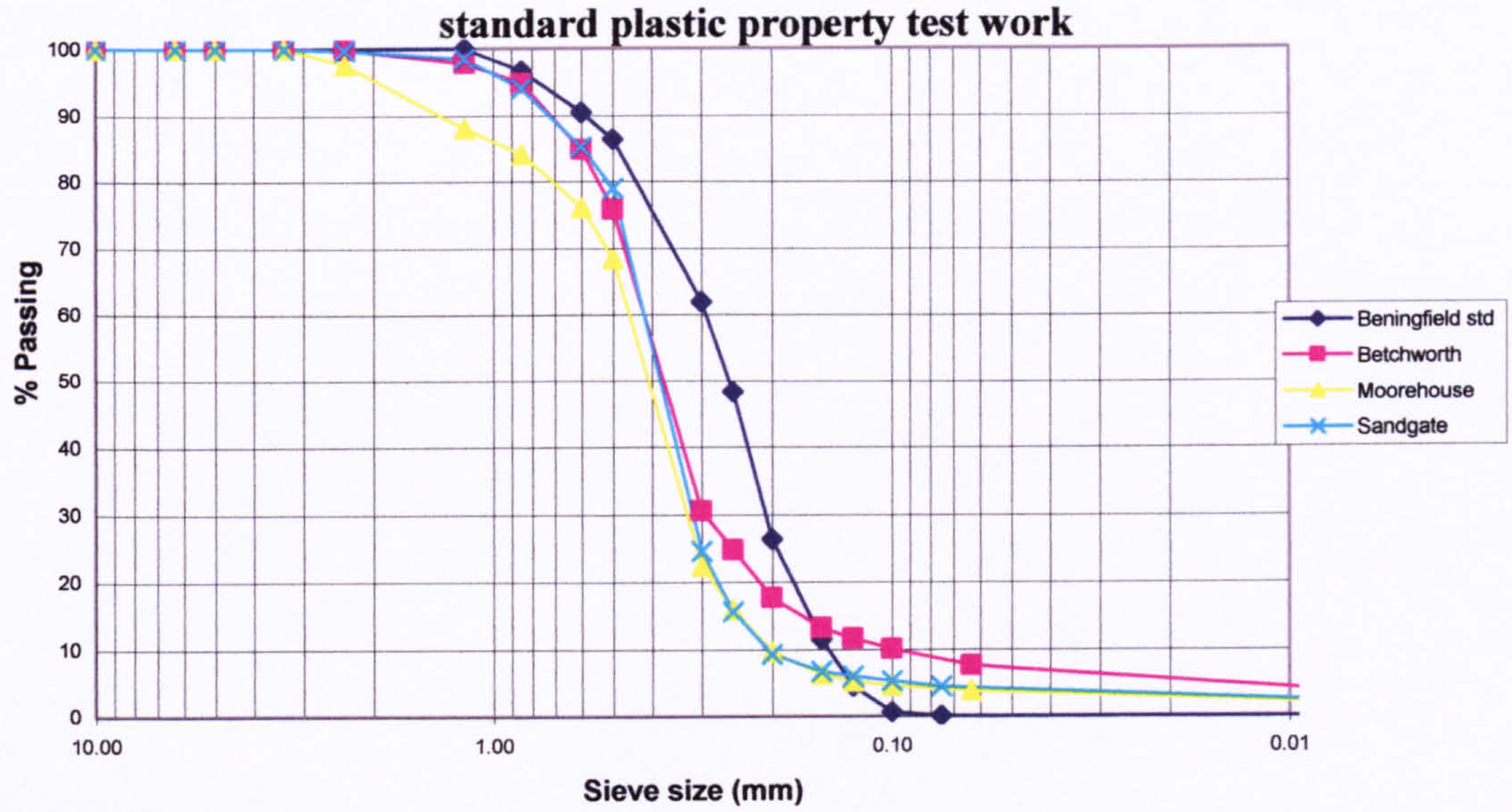
The results of this work are shown in figure 4.2 and show that the effect was still present, even with a change in mix type.

Fig 4.2 The effect of moisture content on dropping ball value, using Beningfield "standard" sand, with and without lime



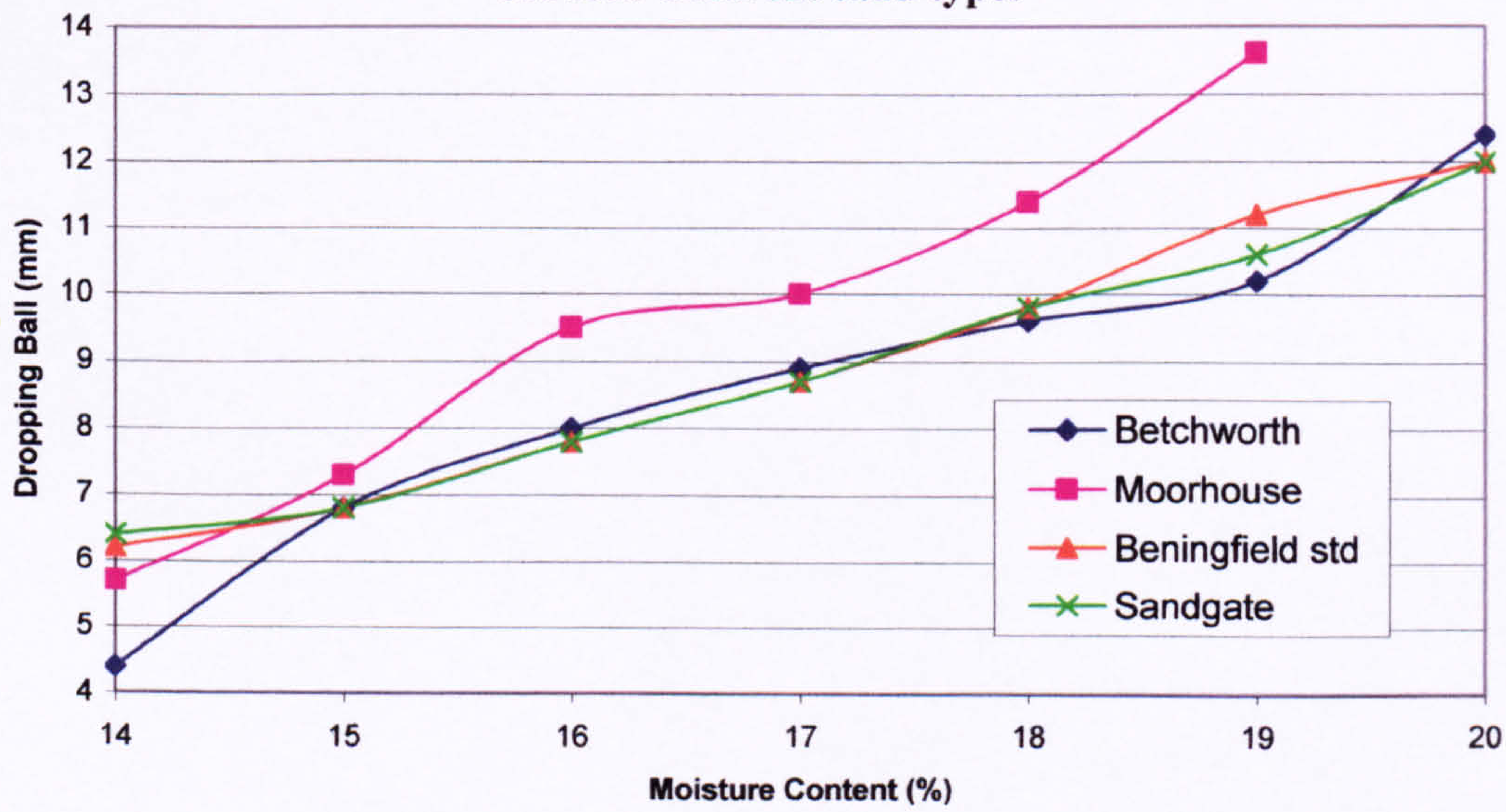
Once again it appeared that there was a transition phase, and it is believed that this is a function of water filling air voids, as opposed to acting to displace solid particles and effectively tending to a volume increaseIn case the phenomenon was associated with the particular sand grading used, which seemed not unlikely, the work was repeated using four different and quite dissimilar sands in addition to the "standard" blend, (shown in the graph legend as NB blend). The other three sands chosen were Sandgate, a very clean washed mortar sand, Betchworth and Moorehouse. These sands were chosen to be as representative as possible of those known to be widely used commercially for the production of both factory made and site mixed mortars. The gradings of all four of the sands are shown in figure 4.3.

Fig 4.3 The particle size distribution for the four sands used in the standard plastic property test work



The results of this further work are shown in 4.4, from which it may be seen that the phenomenon of the transition zone occurred with the full range of sands tested.

Fig 4.4 The effect of moisture content on dropping ball value using the four different sand types



This work also enabled the workable range of the dropping ball test to be assessed. It was concluded that the procedure worked reasonably well between values of about 8 and 14mm penetration. Below a value of approximately 6-7 the error associated with filling and compacting the mould became unacceptably high, above a value of about 16 the presence of excessive bleed water meant that the sample was no longer homogenous and the zone tested was no longer truly representative of the whole, being finer and wetter. These figures related to a moisture content of about 15-19%. They also related well to the consistence values that are used in practice. The original test development carried out at the then Building Research Station, now BRE, suggested a value of 10mm penetration as a "standard" value, based on the views of the bricklayers who were employed to validate the test but more recent experience tends to lean towards slightly wetter mixes. As an example, the full time craft trained bricklayer at Ceram in Stoke on Trent regards a value of 11.2 as a good standard.

4.2 DIN PLUNGER

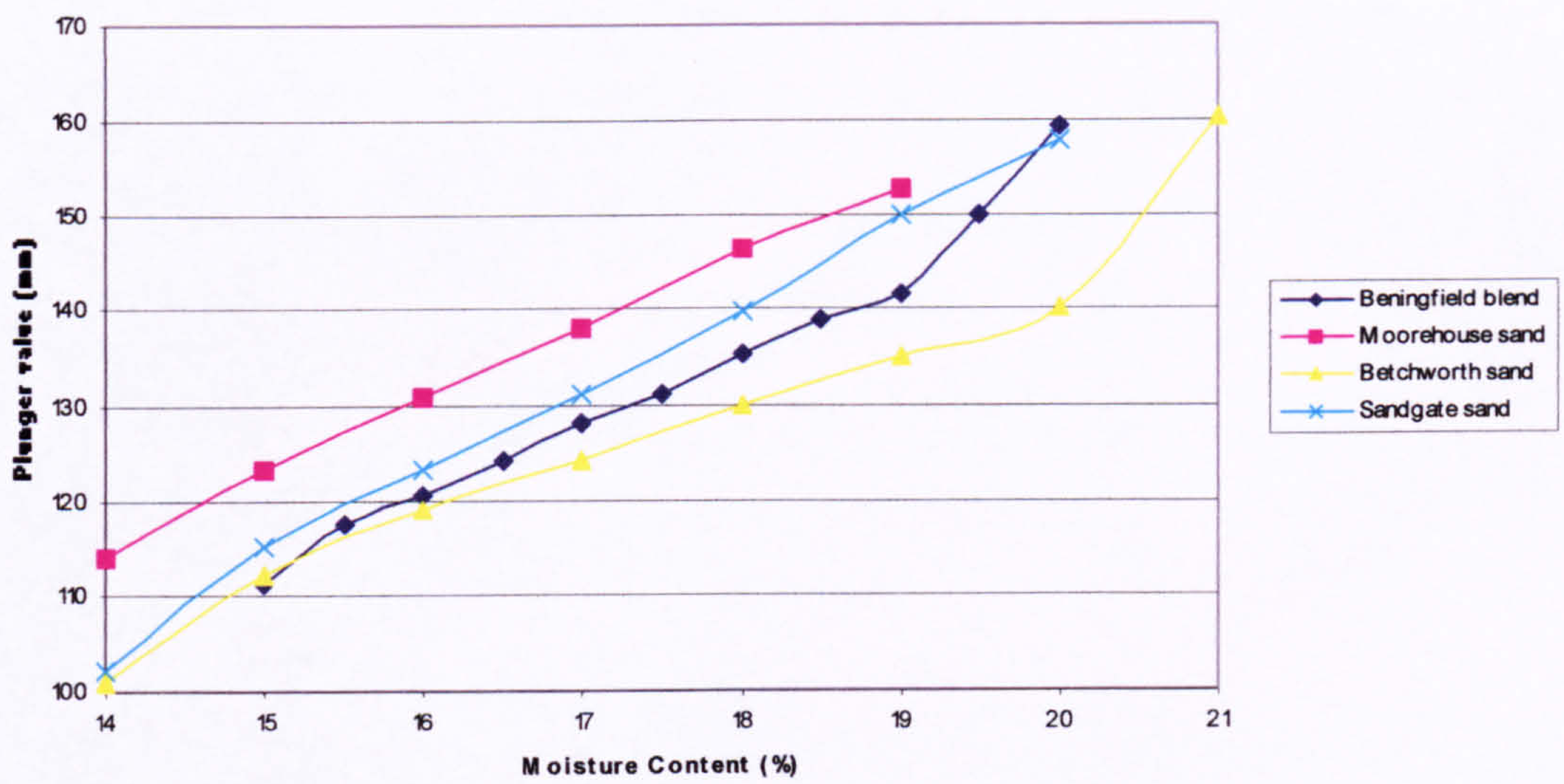
The DIN plunger specified in BS EN1015-4, discussed in section 2.2.1 and shown earlier in figure 2.2, has now superseded the dropping ball test in the EN1015 suite of European mortar test methods.

Although it possesses the practical benefit of having a tethered penetrating element, the DIN plunger has the clear disbenefit that there is friction between the sliding weighted part and the main body. Moreover, this friction has the potential to vary, increasing with corrosion, dirt build-up and similar and then reducing as the equipment is cleaned and lubricated.

At the time of writing the method has not become widely accepted in European testing and indeed it is understood that it is little used even in the country where it was originally developed. Nevertheless, trials were carried out using the four different sand types, in the same manner as with the dropping ball test, to determine the relationship between the measured value and moisture content.

The results are as shown in figure 4.5 and it may be seen that the graphs again show the transition zone previously identified with the alternative types of apparatus, thus leading further credibility to the view that it was a basic function of the mix designs and the solids particle/aqueous phase interactions therein.

Fig 4.5 The effect of moisture content on DIN plunger value for the four different sand types



Notwithstanding the disadvantages associated with the frictional element, as discussed earlier, it was immediately apparent that the plunger equipment was more accurate and less operator dependent than the dropping ball test. The method of filling the mould, which used a conventional tamper, was much less sensitive to operator technique than the dropping ball mould. The equipment also used direct measuring with its own integral scale as opposed to relying on a separate measuring device. The equipment had a range of 100 to 170mm penetration, which represented the total scale, and which covered a moisture content of from about 15 to 20%.

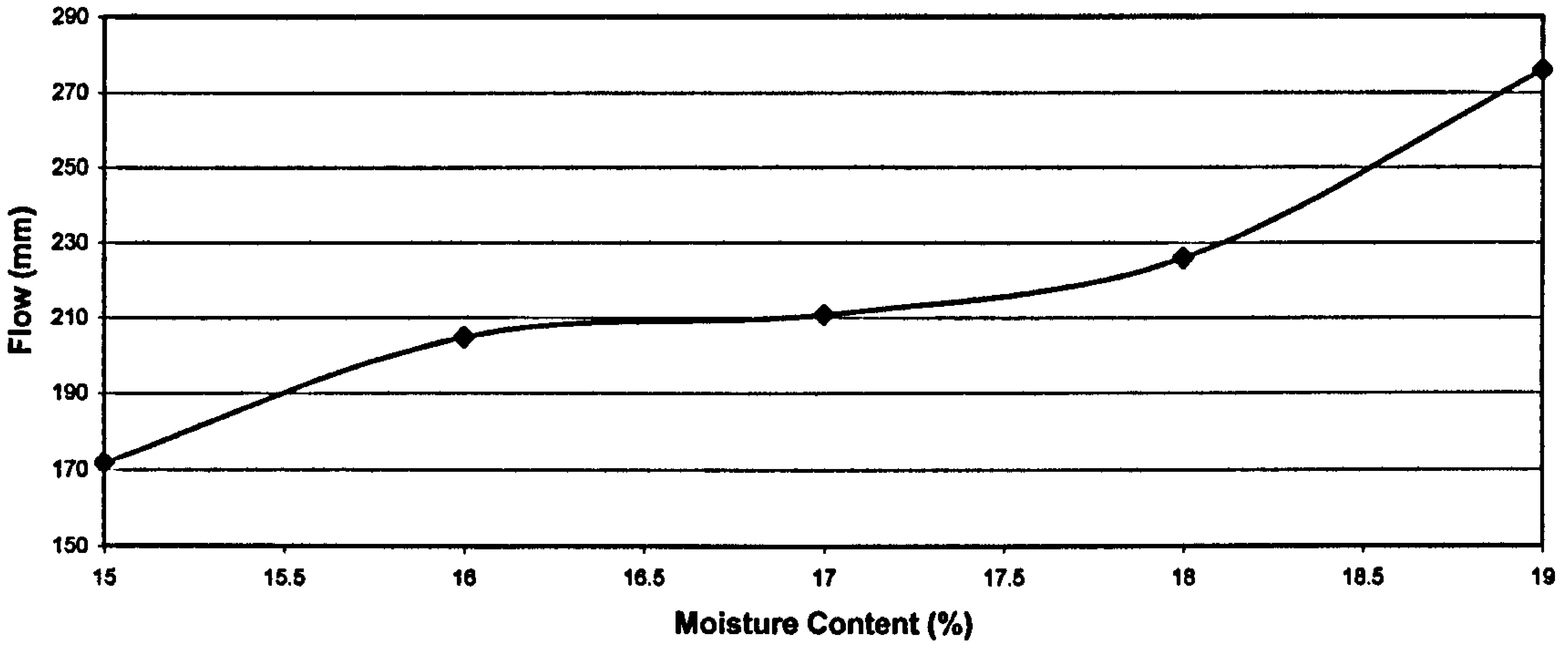
4.3 BS EN 1015 FLOW TABLE

This flow table has been recently adopted as a standard although it differs only slightly from the earlier used BS 4551 table, which was itself based on the ASTM table. Although previously used within the UK to determine the property described as plasticity in British parlance, in continental Europe flow tables have long been used to measure consistence and it was in this context that the method was appraised.

The flow table showed a very similar relationship with moisture content to the two earlier used methods with the standard sand. Because the relationship followed the form of that found with the other two methods so closely a limited amount of work only was carried out with other sand gradings and the result shown in figure 4.6 is for just the standard sand. This work showed that the relationship is again of the same form as that for the dropping ball and the DIN plunger, with the characteristic transition zone in evidence.

It was believed that this finally confirmed the transition zone phenomenon, as discussed earlier in this section, and in the absence of an alternative theory the reasons for the existence of this behaviour will be taken to be those postulated there.

Fig 4.6 The effect of moisture content on flow table value using Beningfield blend sand



The flow table was relatively easy to use although as it used manual operation of the handle and shaft as opposed to the electrical one sometimes found it did require an experienced operative.

4.3.1 Changing the role of the flow table and describing it as a test for cohesion

Although the new British Standard BS EN 1015-3 is defined as a test for consistence, in the old British Standard BS 4551, which was withdrawn in January 2005 before being re-issued without consistence tests, the test is described as one for cohesion. The standard states *Since the more plastic and workable mortars are more cohesive, their flow at a given consistence is less than that of mortars of lower cohesion.* This statement is an excellent example of the subjectivity that is endemic in the field of plastic property description, as discussed in the literature survey and its conclusions. In addition, whilst undoubtedly correct in a narrow sense, as discussed in detail earlier in section 2.3.1 a relationship that is given for one consistence may be entirely erroneous at another, which of course illustrates the overwhelming disadvantage of a single point test.

Nevertheless, at that one consistence, the procedure was found to be of value in comparing different mixes, with those having the lowest flow at the fixed consistence in general possessing more cohesion and less propensity for bleeding and segregation. Thus the flow table may be used in comparative work on cohesion that is required to be valid at just one consistence but its usefulness in this context is restricted to just that narrow circumstance and extrapolation may well be incorrect.

4.4 VICAT PLUNGER

This equipment, which was discussed briefly in section 2.2.1, is the oldest established that is in current usage for mortar testing and it is still specified for the preliminary determination of the consistence of a cement paste. It is very similar to the DIN plunger, except that the latter has a greater cross section and it is difficult to see the logic behind the more recent development of the DIN equipment, particularly as it has not superseded the earlier test, which means that two effectively very similar tests continue to exist.

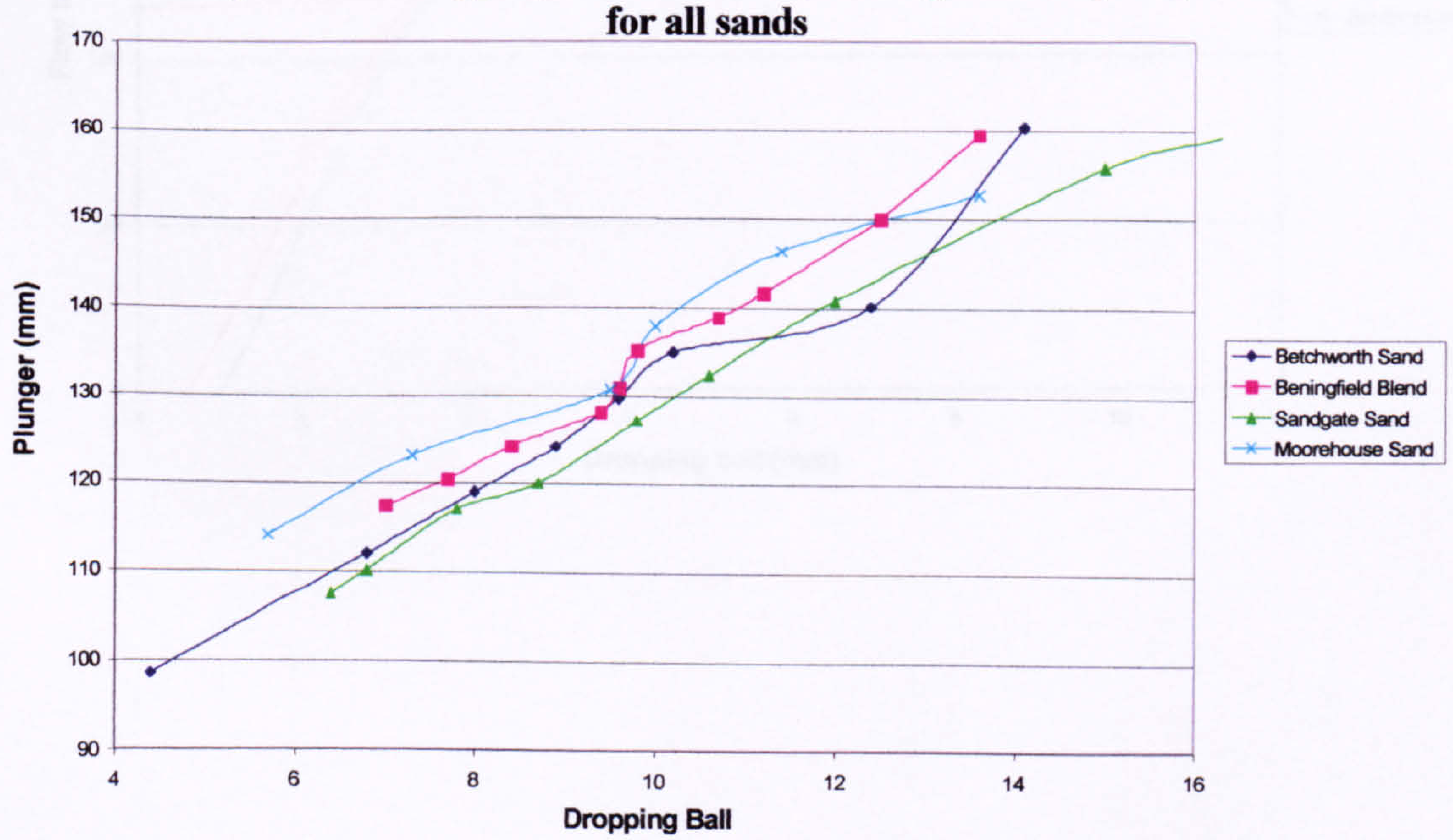
However, although the Vicat apparatus is used for work with cement pastes it did not prove to be usable for mortars. The equipment is used with either a needle or a plunger, the latter for use in the early life of a standard cement paste. Even with the widest accessory available and with all of the weight removed from the top of the plunger shaft the equipment penetrated virtually to the bottom of the mortar in the mould and was thus not really usable. Work with this equipment in the area of the early life plastic properties was therefore abandoned and it is recommended that it should not used for masonry mortars.

4.5 A COMPARISON OF METHODS FOR THE DETERMINATION OF CONSISTENCE

The relationship between the different tests was then considered together with their sensitivities, relative accuracies and applicability. As suggested earlier in the work, it was believed that all of these tests were measuring the same property, or more correctly the same mixture of properties, with accurate quantification of the relative importance and change of each sub parameter being quite impossible.

The hypothesis that they are all measuring the same property was investigated by considering their inter relationships and a plot of the dropping ball value against the DIN plunger is shown in figure 4.7. This work showed a good relationship for all of the four sands used in the investigation.

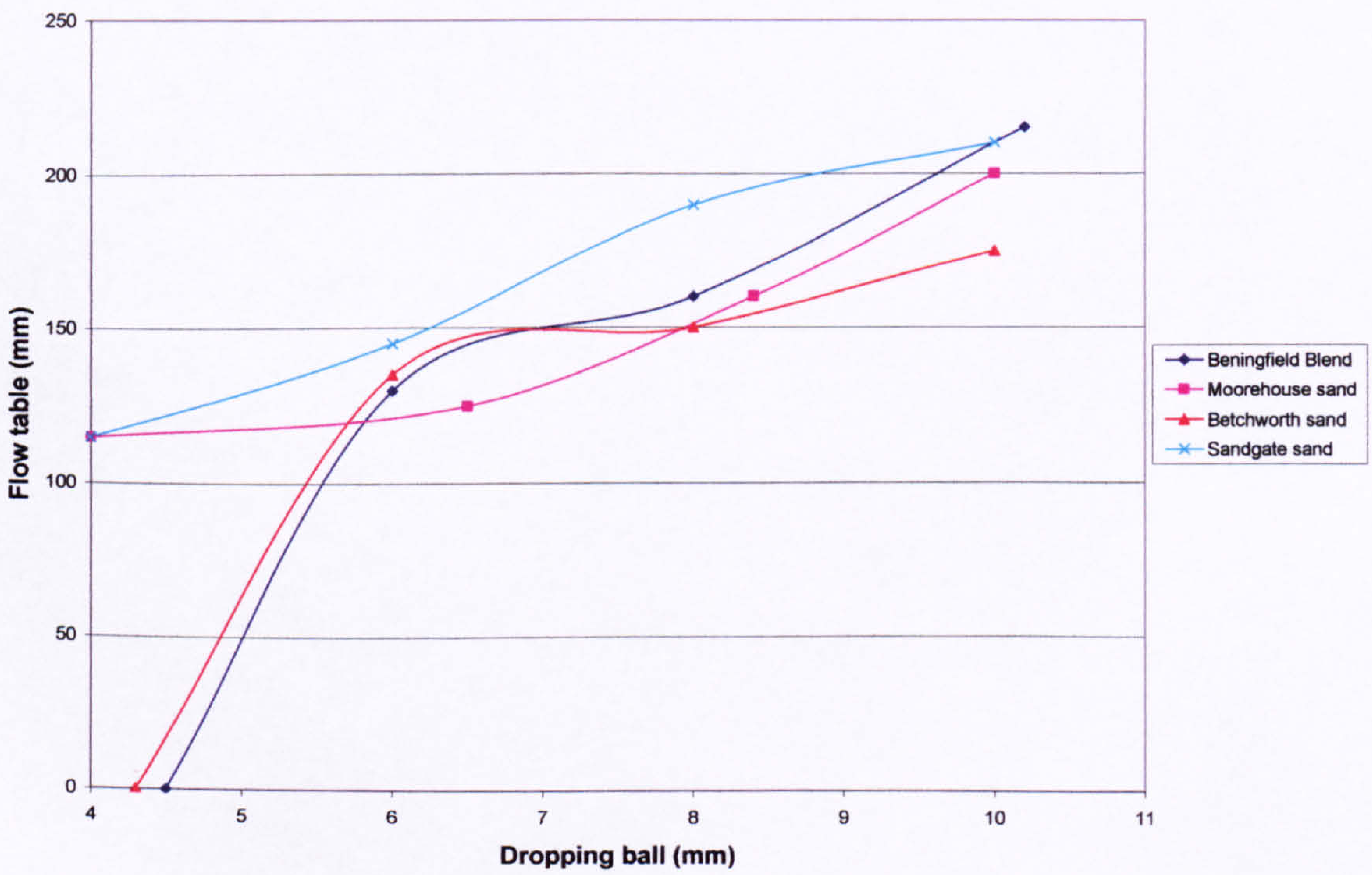
Fig 4.7 The dropping ball value compared against the plunger for all sands



The relationship thus demonstrated was also seen to show the clear dip and plateau effect that was a feature of the individual relationship of penetration against moisture content and within this zone it is seen that as the moisture content increases first the plunger is more able to discriminate, then the dropping ball.

The flow table was also tested against the dropping ball, although the relationship hear is a lot more complex, possibly because the mix segregated somewhat towards the end of the number of drops. More work needs to be carried out in this area.

Fig 4.8 The dropping ball compared against the flow table for all 4 sands



The relative properties and attributes of the four test methods investigated may be summarised in table 4.1 below.

Table 4.1 A comparison of plastic property test methods

Test method /property	Dropping ball test	DIN plunger	Flow table	Vicat plunger
Range, moisture content	15-19%	15-20%	Dependant on mix design. Can be restricted	Work did not proceed, due to the inappropriate nature of the method for masonry mortars
Sensitivity	Fairly good	Very good	Fairly good	
Operator dependence	High	low	Low	
Ease of use	Requires experience	Easy to use	Reasonably easy to use	

As can be seen from the table the dropping ball and flow table were usable over a wide range but the former suffered from a high degree of operator dependence. The wide shallow mould was difficult to strike off in a reproducible manner and the effects of this on surface finish and bleed were more pronounced. This could be exacerbated when the measuring device was placed on to the filled mould and adjusted into place, with this movement tending to cause more bleed, particularly with mixes of higher consistence. The placement of the measuring device was itself a further source of error, as was the lowering of the measuring foot onto the top of the ball without disturbing the latter. Clearly the errors cause by these effects could be additive, potentially leading to a relatively high variance.

In contrast, the DIN plunger was easier to fill reproducibly, with less surface finish effects and a better means of measurement. So long as mechanical effects are minimised by careful maintenance or perhaps by a re-design of the release mechanism, possibly to incorporate electro-magnetic release, this equipment was judged to be superior to the dropping ball apparatus. In practical work it is more effective and appears more sensitive, being preferred by operators who are given the choice of test.

The flow table was relatively easy to use and quite reproducible but requires careful setting up on a heavy concrete plinth and thereafter some maintenance. It also lacks a measuring system, relying instead on manual use of callipers or ruler. Mixes possessing poor cohesion and those that segregated readily were not well suited to the flow table and the former may flow over the edge of the table at relatively modest moisture content levels that would not cause problems with either of the alternative procedures. Additionally, air entrained mixes may give anomalous results, as air is knocked out of the mix during the test.

It was interesting to note that the plateau or transition zone effect that was first noticed with the dropping ball work degraded the accuracy of all of the test methods in a range of from about 9 to 11mm, just the area where the greatest sensitivity was required, that is in the middle of the critical brick and block laying consistence band. Nevertheless, it was clear from this work that of all of the single point tests the DIN plunger was the best by a considerable margin, both in terms of precision, convenience and the time taken for an operator to achieve familiarity and competence. However, it is still the case that in general, single point testing is crude and unsatisfactory and in order to obtain a comprehensive view of plastic properties rheological techniques are a necessity. This conclusion is developed further in sections 6.1 and 7, but prior to that additional work the mortar properties and the testing thereof at a slightly later time period, when stiffening and then setting commenced, were addressed as discussed in section 5 below.

5 AN INVESTIGATION INTO TESTING FOR STIFFENING AND EARLY LIFE STRENGTH DEVELOPMENT

Following on from the period in time when the plastic property tests discussed in the earlier sections are relevant, the mortar begins to stiffen and set, as discussed in detail in sections 2.2.3 and 2.3.3. Existing accepted procedures for the testing of the stiffening rate, as it is known in the recently superseded British Standard BS 4551, 1998, or the workable life as it is referred to in the new UK standard BS EN 1015-9, 1999, consist of methods based on mechanical penetration. As discussed in the literature survey there are a number of these, but in the current research programme it was envisaged that only the most used would be investigated and the results of this work are presented in the remainder of this section 5.

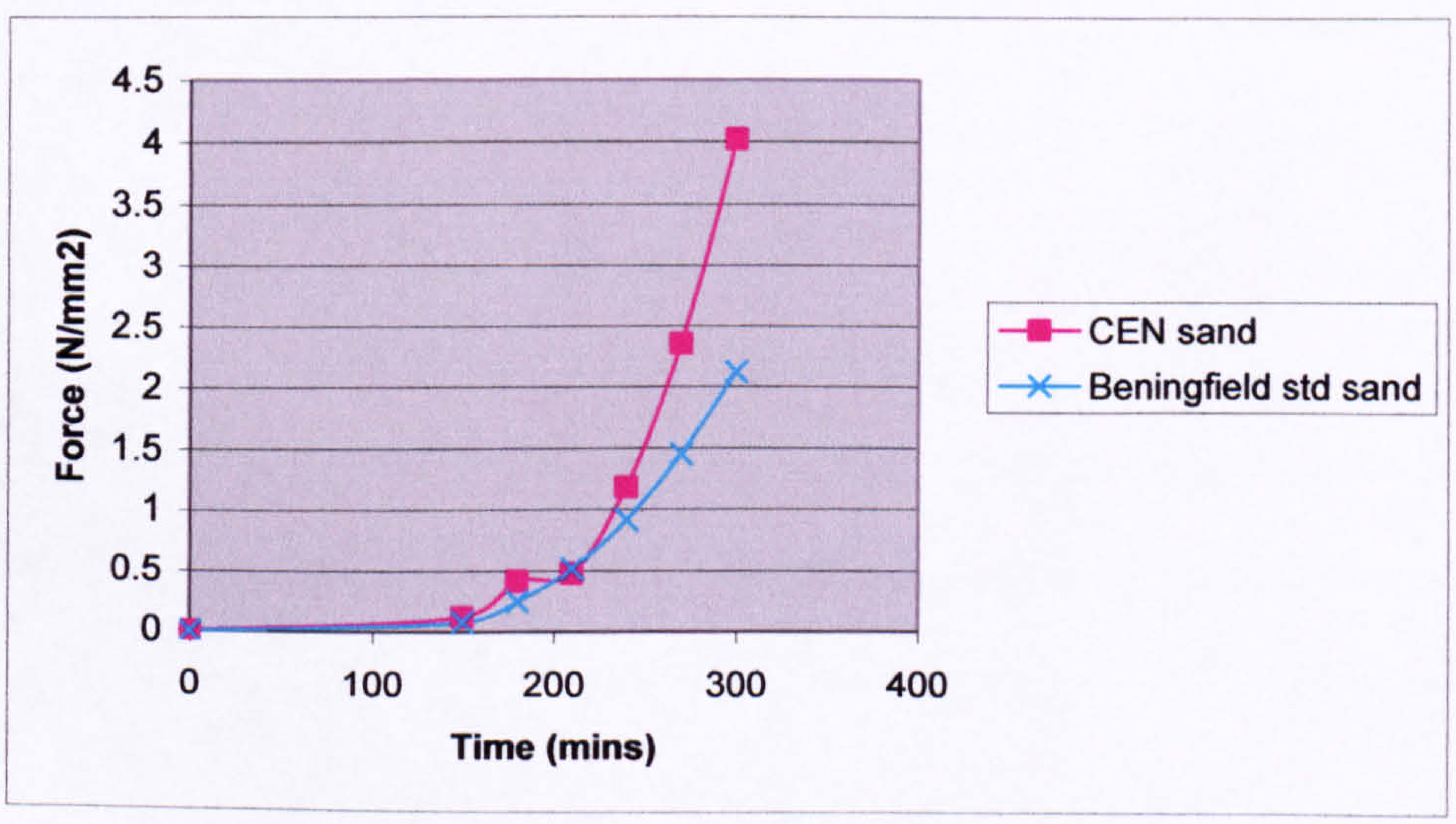
5.1 BS EN 1015 WORKABLE LIFE APPARATUS

Because experience with this test over many years had not been positive, with lack of precision associated with mix composition being clearly in evidence, it had already been decided to attempt to develop a better method, based on non-mechanical procedures.

Nevertheless, a small programme of work was carried out with this equipment to investigate the effect of changing the major variables, in particular sand grading, which was thought to be of particular relevance to this procedure as it was used with a wide variety of sand gradings, from the coarse materials used in floor screed mortars, through relatively coarse rendering mortars to masonry mortars that could potentially be based on very fine sands in some cases. Figure 5.1 below shows that the sand grading does exert an influence on the workable life, with the coarser sand impeding the penetration of the plunger and resulting in a higher reading. This was most noticeable during operation. As the plunger was depressed the increased resistance of large particles could be felt quite obviously and could be seen as the scale reading registered a higher value, and then "slipped back" again to give the lower and it is believed more accurate and representative value, as the large particle was either passed or rotated to present a narrower face in plan.

It should be noted, however, that there may be a minor input to the setting time effect caused by the very slightly lower water:cement ratio perhaps very slightly accelerating the set. Although Neville, 2004, makes no reference to any such effect it is believed nevertheless to be present to a very minor extent.

Fig 5.1 The effect of sand grading on the workable life determined in accordance with BS EN 1015-9, using CEN sand and Beningfield standard sand



It had also appeared in routine laboratory work that aggregate cement ratio affected the value of workable life determined using this procedure with the richer, more plastic mixes allowing an easier passage for the penetrating plunger and thus providing a lower penetration reading. Unfortunately, the effect was masked by the more pronounced effect that this variable has on setting time, as it is known that rich aggregate/cement mixes set and develop early strength more rapidly, Neville, 2000. This area would also be investigated more accurately using instrumental techniques, in the second half of the overall test programme, if these could be successfully developed.

During the work numerous mechanical inadequacies associated with the design and operation of the equipment were observed and these are discussed in detail in section 6.2.

5.2 VICAT NEEDLE

The Vicat apparatus is very old established, simple and robust. It was initially used with masonry mortars as well as with cement pastes but latterly was replaced in the UK by the stiffening rate test, later adopted by CEN as the workable life test. For the testing of cements it utilises an all purpose frame into which a variety of plungers and needles may be fitted, thus providing the flexibility to vary both the weight and the cross sectional area over which it is applied. A plunger of relatively great area is used for the determination of consistence of a cement paste mortar (although as discussed earlier in section 4.4 this did not prove practicable for use with masonry mortars as the equipment penetrated too far into the test specimen). After a mix of standardised initial consistence has been produced the plunger is replaced with a needle and the initial and then final set of the cement paste mortar determined.

For this research programme, the setting time needle was used and the standardised 1:3 mix used earlier in the programme was made up to a dropping ball consistence value of

10mm. The equipment was tested against the BS EN 1015 workable life apparatus and the results of this work are shown in Table 5.1 below.

Table 5.1 A comparison of the Vicat needle and BS EN 1015-9 test methods for initial/final set and workable life

Elapsed time (mins)	BS EN 1015 penetrometer value	Vicat needle value
0	0	0
30	10	0
60	11	0
90	12	0
120	10	0
150	11	0
180	17	0
210	16	0
240	18	0
270	21	Initial set exceeded
275	23	Final set @ 2.5mm
290	n/a	Final set @ 0.5mm
300	25	n/a

As may be seen from the table, the Vicat needle is less sensitive than the BS EN 1015 penetrometer, which is perhaps to be expected as it is used primarily for cement pastes. From first principles, as the area of the needle or plunger increased a greater sensitivity could be expected, together with a greater accuracy as effectively a larger sample of the heterogeneous test material would be sampled.

Unfortunately the data from the BS EN 1015 test was not available in absolute terms, which precluded the interpolation of the two points for initial and final set that are addressed in the standard, although this technique is adopted later in the work, as shown in section 11, figure 11.8. Where this interpolative technique has been used in the main laboratory involved in the work it has, however, been found satisfactory.

5.3 STANHOPE-SETA APPARATUS

This apparatus had been included in the original research programme partly because it had been used in the comprehensive Smeaton project, Teutonico et al, 1994, but discussions failed to reveal a scientific reason for its inclusion. Further talks with the manufacturer of the equipment led to the conclusion that it was not really used with cementitious construction materials. The equipment is a penetrometer but rather than measuring the force required to penetrate a given amount, as for example is the case with the BS EN 1015 apparatus, it measures the depth of penetration resulting from the application of a fixed force, in a similar manner to the Vicat equipment. Following on from the test work carried out on each of these pieces of equipment, a theoretical consideration of the methodology and design led to the view that it was merely another form of penetrometer, with no unique features and it was therefore removed from the test programme.

5.4 A COMPARISON OF THE MECHANICAL METHODS FOR THE TESTING OF STIFFENING AND EARLY AGE STRENGTH DEVELOPMENT

Although both of the test methods examined used the essentially similar concept of mechanical penetration and were thus subject to the many inaccuracies associated with this type of procedure, as discussed in greater detail in section 6.2 below, there were some practical differences. The sample size was greater for the BS EN 1015 procedure, which should potentially result in greater accuracy. The area over which the test is carried out is also larger, which gives rise to the same benefit. As the mortars became stiff the slender Vicat needle was affected more than the plunger in the BS EN procedure. The latter equipment was also more robust although the distance measuring washer would jam on occasions and could potentially give an erroneous result. Overall there was not a great deal to choose between the two, with the more robust BS EN apparatus with its larger sample being judged the best on balance.

Of course, both pieces of equipment suffered from all of the defects associated with mechanical test methods for the determination of this parameter, as discussed in detail in section 6.2.

6 AN ANALYSIS AND DISCUSSION OF THE TEST PROCEDURES FOR THE PLASTIC PROPERTIES

The research showed that the existing procedures described in the current British and European standards had some merit but, as expected, the existing tests possessed deficiencies and an assessment of mortar properties based solely on them would be imperfect. In addition, the existing tests were generally not convenient to carry out, requiring for example heavy equipment, monitoring for inconveniently long continuous periods of time or frequent attention to cleaning and preparation of the apparatus. These deficiencies are expanded on separately, for the two classes of plastic property tests, in sections 6.1 and 6.2 below.

6.1 THE EARLY LIFE PROPERTY TESTS

These are the tests used for characterising the mortar immediately after mixing, up to and including the initial laying of the units. They may be broadly considered as tests for consistence and/or cohesion, together with the specialist tests of water retentivity in the UK and Europe and the Emley plastimeter or viscometer used in the USA. The research described in section 4 showed that there was a relationship between the consistence/cohesion tests and that they were indeed all measuring the same property, as suggested earlier in the conclusions to the literature survey that are presented in section 2.3.1. They all possessed the shortcoming that the properties of consistence, "wetness" in unsophisticated terms, and cohesion could not really be separated, with each exerting an influence on the other and preventing a clear expression of one without the influence of the other. Determination of consistence first, followed by cohesion, has been suggested as a way of overcoming that fundamental issue but has two apparent drawbacks. Firstly, any comparison is only valid at the consistence value at which it is made, and may even be reversed at a different value. Secondly, from a consideration based on first principles it is difficult to understand the theoretical basis upon which exactly the same test, for example that for flow, can measure two properties that are said to be completely different, in this case flow and cohesion, whilst using the same units of measurement and expression.

In addition to these conclusions based on a theoretical analysis and also on the test programme, the practical work using the bricklayers also revealed shortcomings in the existing plastic property tests, as discussed in section 3.2

6.2 THE SHORTCOMINGS OF MECHANICAL PENETRATION TESTING FOR THE STIFFENING AND EARLY SETTING

There are many errors in the mechanical penetration methods described earlier, the key ones are as discussed below.

6.2.1 The bleeding/segregation error

As time proceeds, segregation and related bleeding is likely to occur in the majority of mortar mixes. This is substantially independent of the stiffening and setting time phenomena and means that two mortars with absolutely identical behaviour in the setting area will be erroneously recorded as having dissimilar properties, because higher values will result from material that has segregated as the probe is impeded more by the segregated layer.

6.2.2 Plasticity

Two mortars that are identical in composition and properties but with differing plastic properties as a function of eg the inclusion of an admixture, perhaps cellulose ether, which is widely used in factory made mortars, will produce dissimilar readings, even though their setting behaviours may actually be identical. The test thus to some extent registers plasticity as well as setting behaviour, particularly in the early stages at around the beginning of the development of initial set.

6.2.3 Grading

The presence of relatively large particles of sand gives rise to gross errors, as the penetrating element is obstructed and has to force the particles downwards and/or sideways.

6.2.4 Shape and surface texture

Flaky and elongated particles will provide more or less resistance to penetration depending on their orientation and heavily textured surfaces will also tend to produce higher readings.

6.2.5 Consistence

As consistence increases, so penetration resistance decreases, giving rise to a further potential source of error.

6.2.6 Operator error

Many manual tests, particularly the current BS EN test, are dependant on the operator judging the rate of application of load. This may vary greatly between operatives, and even within the work of a single operator there may well be a variation.

6.2.7 Mechanical error

The BS EN 1015 apparatus exhibits variable friction as the head of the equipment slides down the vertical shaft. There is an additional source of potential error as the washer on the equipment slides up the shaft. The design specification and engineering tolerance are such that the washer often has a tendency to jam, to a lesser or greater extent, thus producing an error source of variable occurrence and magnitude.

Overall, the errors in the existing procedure discussed above may be classified in one of three categories, errors as a function of mix design interacting with penetration, operator dependency, and mechanical errors arising from equipment variability and intrinsic design inadequacy. If equipment could be designed to overcome these three sources of error the potential for a successful procedure would be considerable and this issue was investigated and a test proposed and developed as described in chapter 8. Prior to this work, however, the earlier life plastic properties were also considered in more detail with the aim of suggesting ways in which more advanced rheological techniques could be used to better measure and characterised them, as outlined in section 7 below.

7 RHEOLOGICAL APPROACHES TO THE MEASUREMENT OF THE EARLY LIFE PLASTIC PROPERTIES OF MORTARS

The shortcomings of the existing tests for consistence/plasticity, for use in the assessment of the early life plastic properties having been suggested in the conclusions to the literature and confirmed by testing, the next stage of this project was to proceed with the rheological work for which there appeared to be a need. The first work was carried out using a Tattersall viscometer, as shown in Figure 2.9 in improved Mk 2 form and as described in section 7.1 below.

7.1 RESEARCH WORK WITH THE TATTERSALL VISCOMETER

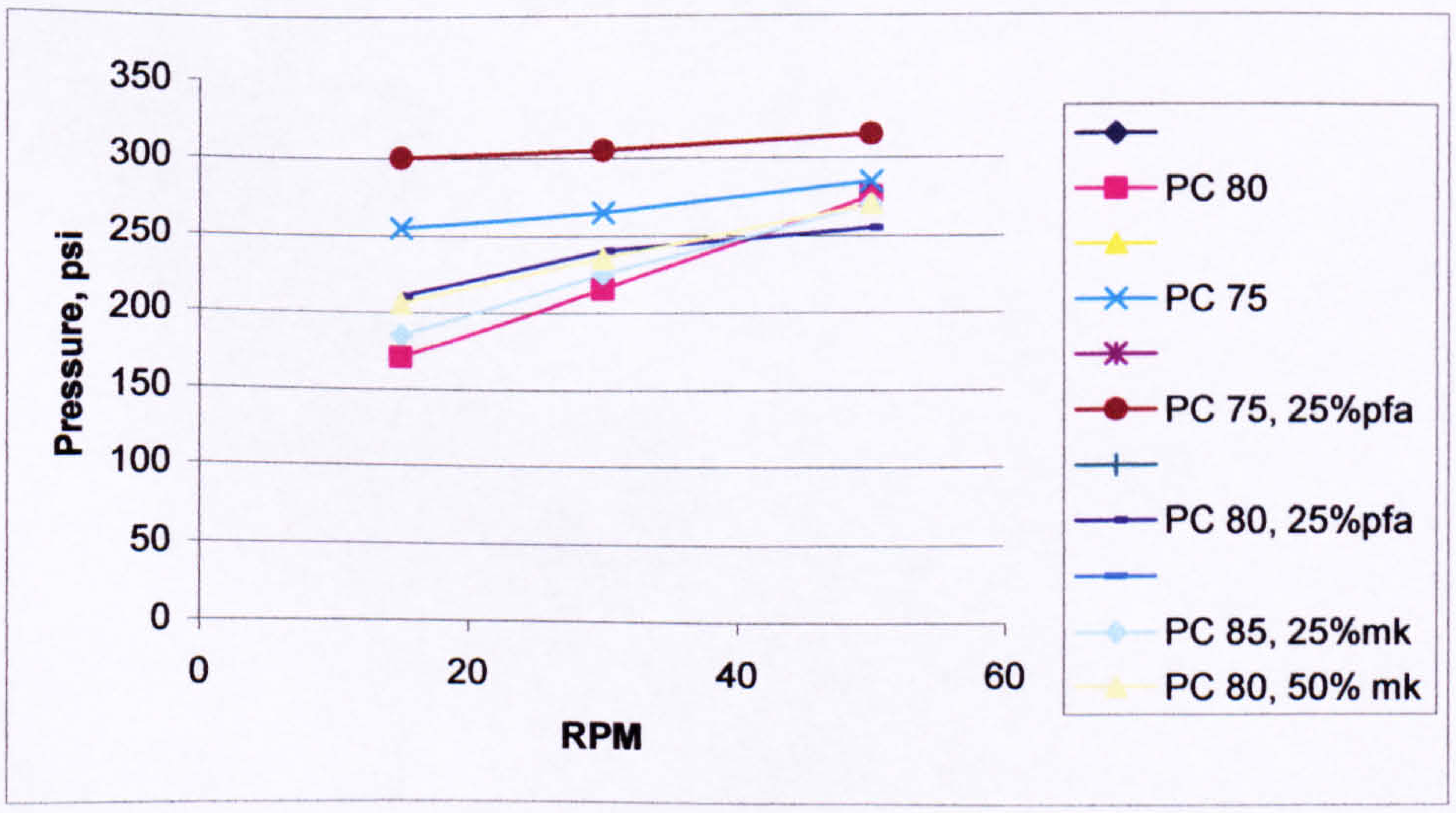
Tattersall's work represented a quantum leap forward from the status quo of single point testing, and his ideas were visionary for the construction materials industry, but with the benefit of hindsight it is now clear that his equipment was crude. Mechanical losses, electrical power variation, oil thinning as the gears and other mechanical components became warmer as the machine ran and variable friction in the drive/geartrain all may cause variations in the readings obtained. Edge effects have the potential to cause further inaccuracies.

The test vessel is of appropriate size for use with concrete but when used with mortars the bowl is larger than necessary, with the potential for bleeding and segregation that will give rise to a test specimen that is not homogenous and thus cannot be considered able to conform to any valid rheological treatment. Additionally, use of the equipment also really means implied acceptance of the proposal by Tattersall that the Bingham relationship may be assumed. Although Tattersall pointed out on several occasions that this was really only a convenience and that in reality the relationship had to be curved or curvo-linear, his use of the straight line Bingham form was only as a simplification to aid his procedures. Modern computer programmes have now been developed with the ability to curve fit to greater accuracy, using polynomials and thus to produce truly accurate relationships although this does not for one moment decry Tattersalls vision and work.

Notwithstanding the reservations expressed concerning the approximate nature of the fundamentally assumed relationship, and the mechanical shortcomings of the equipment, early work was carried out at RMC by Martin, using the Tattersall equipment shown in figure 2.9. Unfortunately, the results obtained, shown in figure 7.1 below, were disappointing. It is seen that the repeatability is poor and although there did appear to be a family of curves neither the absolute values nor the interrelationships appear reliable. It may be that the equipment used was problematic, or the operation in error, but for whatever reason the results were unfortunately deemed to be not promising enough for the work to be proceeded with.

Fig 7.1 Results of the work with the Tattersall rheometer

Note. *See appendix 1 for a full description of legends and test methodology*



7.2 THE HARKE AND BROOKFIELD RHEOMETERS

These instruments represent a quantum leap forward from the Tattershall equipment and possess attributes that are completely lacking in the latter. The mixing action of the Tattershall mixer has the potential to produce disordered, turbulent flow when used with materials of high consistence, which clearly mortars are, whereas rheometers are designed to produce ordered, laminar flow. The basic mathematics of rheometry, and indeed of many fluid flowing systems, are invalid when applied to turbulent systems and are only applicable with controlled systems, with turbulence reported as able to distort the result by 100% or even more, Schramm, 1994.

As they are directly linked to a computer and are microprocessor controlled, the Haake and Brookfield instruments are able to apply energy incrementally in any of a large number of modes whilst simultaneously measuring the response to that input, although the Haake is much less flexible than the Brookfield in this respect.

With both systems, the linked computer is then able to process the data and export it to spreadsheet or graph production software. Alternatively, when used in production control in some industries the computer software can even be linked in to microprocessors controlling the production process.

There are disadvantages with rheometry and influences that degrade the accuracy. Thus turbulent flow must be avoided, as previously discussed, as must plug flow, which occurs when a plug of test specimen revolves around, rather than the desired situation occurring which is of laminar flow between adjacent sheet-like elements of the test sample. The test specimen must be homogenous, and segregation, which is potentially a factor in nearly all mixes, is clearly detrimental. Additionally, rheometry is very temperature dependant and any investigation that may be required to be closely comparative needs to be carried out in a constant temperature water bath, cabinet or similar.

Finally, although rheometry is a powerful comparative technique, producing absolute values that may be used for valid comparisons between laboratories and different equipment this is difficult and involves the use of materials of known, standardised parameters that are costly and not widely available. Notwithstanding these issues, in order to progress in the field, a Haake viscometer was first sourced and this was then followed by a Brookfield at a later date.

8 AN INVESTIGATION USING THE HAARKE VISCOMETER

This equipment is often described as a rheometer but is sometimes said only to be a sophisticated viscometer as it is unable to apply a controlled stress, only working with a controlled rate of stress and then determining yield stress by interpolation. Yield stress is a critically important parameter. In concrete, it is related to slump. As the slump cone is raised and then removed, the force of gravity is exerted on the concrete which reduces in height until it is in equilibrium with the yield stress and further vertical consolidation then ceases. In mortar, yield stress is of importance in determining the amount of energy required to deform the bed as the material is spread and levelled. It also relates to the energy required to level the brick or block by pushing or tapping plumb and level. Concrete, particularly self compacting concrete, had been found in early unreported work at RMC to require to have relatively low yield stress but moderate plastic viscosity.

This early work had exposed a further issue with the Haake in that the maximum size of the aggregate with which it may be used is constrained by considerations of particle interference and edge effects. Because rheometric phenomena require laminar sheets of material to flow relative to each other, or at least to interact without external influence, it is necessary to avoid interference from edge effects and other vessel interaction. In practice, this gives rise to the empirical statement that the gap between the inner and outer walls of the test vessel needs to be not less than about six times the maximum particle size for angular particles, somewhat less for rounded ones. This means that in general, with the normal instrument configuration, only cement pastes or very fine aggregates can be studied, with mixes containing relatively large fine aggregate particles often proving to be outside the scope of the equipment. The other basic requirement of a twin cylinder apparatus of the Haake type is that the ratio of r_1 and r_2, where these are the radii of the inner and outer cylinders respectively, approaches unity as closely as possible. Unfortunately to achieve this requirement results in a very large size of apparatus, with a concomitant increase in both the cost and in the likelihood of an increase in bleeding and segregation.

The problems of cylinder radius and maximum particle size have been known for some time but it was hoped that use with mortars of very small maximum particle size would reduce the major inaccuracies sufficiently to enable meaningful results to be obtained. Thus although the Haake viscometer possessed some disadvantages it was nevertheless hoped that it would be of use in the early stages of the work on cementitious pastes and fine mortars, although as outlined earlier, the application of the equipment to mortars containing coarser sands may be problematic.

Early work with the Haake was concerned with preliminary testing to obtain familiarity with the equipment. It was easy to use in the form purchased which was with Rheowin software. This software had 13 different equations embedded in it, from the basic Newtonian, through Bingham to more sophisticated polynomials like the Casson relationship. Initial work was carried out to confirm the accuracy and repeatability, using two "concrete mortar" mixes, as the equipment was also planned to be used for work on self-compacting concrete. The mixes were designed to be as basic as possible with fine sand all passing the 250μm test sieve, in order to ascertain whether reasonable results could be obtained using the very easiest of test conditions although proprietary admixture was used, of the same type as would probably be chosen for inclusion in the self compacting concrete, at two levels of addition. In addition, some slag replacement was incorporated, as this is representative of current practice. The mix design used is shown in table 8.1 below.

Table 8.1 The mix design for the mortar used in the Haake repeatability work

Material	Weight g
Portland cement	180
Ground granulated blastfurnace slag	180
Sand	1400
Advaflow 300 admixture	1.8 mixes 1-8, 0.99 mixes 9-15

Note The water content was not recorded but all mixes were made up to a consistence value of 10mm dropping ball.

The results of this preliminary repeatability work, carried out by Chinery at the sponsors laboratories, demonstrated good repeatability, as shown in tables 8.2 and 8.3 below.

Table 8.2 Repeatability work with the Haake viscometer, moderate admixture addition

Test no	Yield stress	Plastic viscosity
1	28.46	0.730
2	28.88	0.615
3	25.25	0.850
4	25.87	0.669
5	32.85	0.627
6	28.00	0.642
7	31.39	0.695

8	27.74	0.802
Mean	28.56	0.704
S D	2.39	0.085

This work clearly yielded promising data, with reasonable repeatability, with the chosen paste mix design. Because the inclusion of admixture at the level chosen, appropriate to the levels used in some of the self compacting concretes being developed might have produced a paste of "good" properties from the rheological point of view, that is one with good dispersion and minimal segregation and bleeding, a further series of trials was carried out at a lower admixture dosage rate. This work, shown in table 2.6 and also by Chinery at RMC, showed that the test variability was greater but it was still judged to be acceptable.

Table 8.3 Repeatability work with the Haake viscometer and a reduced rate of admixture addition

Test no	Yield value (Pa)	Plastic viscosity (PaS)
9	62.24	0.645
10	56.53	0.655
11	53.09	0.936
12	48.50	0.754
13	50.70	0.733
14	39.11	0.947
15	46.86	0.683
Mean	51.00	0.765
S D	6.83	0.127

The fact that the Haake viscometer was unable to work with particles of a larger diameter than about 150μm, due to the small gap between the sensor and the bob, which means that it is effectively limited to use with pastes only, as opposed to real mortars or concretes, is not always seen as a disadvantage, as some research is carried out on paste rather than concrete because of the obviously much easier practicalities of smaller sample size, less weight, smaller apparatus requirement and related practical issues.

Work in the sponsors laboratories by Rigby used the Haake VT500 viscometer but with modifications to the central bob after Billberg, 1999. These were made because it is known that with a concentric cylinder viscometer a layer of solvent (water) rich material can collect adjacent to the central bob and cause slippage to occur, with the material intermittently but regularly sticking to the bob and then slipping away. The bob was therefore modified by machining 0.5mm x 0.5mm grooves into the surface and trials carried out to determine the relationship between the Tattersall equipment and the Haake. This work was disappointing and did not show a reliable relationship, indicating that one or both of the procedures was problematic.

The issues surrounding the Tattershall equipment have been discussed earlier in this section and it is considered that they have a major influence on the lack of comparability, although of course the Haake was only working on a fine cement mortar, little coarser that a cement paste, and a number of influences mean that achieving accuracy and repeatability with these mixes is very difficult. Although it would seem at first sight that working with a cement paste or a fine "cement mortar" would represent a somewhat easier case than a full scale concrete this is actually not the case, due to the influence of segregation, bleeding and the dominant effect of cement agglomeration and partial breakdown as a function of applied energy which can be somewhat unpredictable.

Further work still failed to confirm a relationship between the yield value of cement pastes in the Haake viscometer and full scale concrete mixes tested on the Tattershall equipment, with a correlation coefficient in the order of 0.45, clearly inadequate. In addition to the cement paste issues, it appears that the presence of aggregate in the concrete has an effect that is dominant overall with respect to the totality of particle interaction and rheological properties, thus leading to the requirement to work with a rheometer capable of handling larger particles. This conclusion was also arrived at by Banfill and Tattershall, 1983.

The Haake VT550 Viscotester rheometer with Rheowin software was then subjected to a rigid appraisal. The software was good, the equipment was easy to use and gave good repeatability. Unfortunately, as discussed, the maximum test specimen size is constrained and although it can apply a constant rate of shear it cannot be used in constant shear stress mode. This means that yield point is not determined directly, only calculated using regression analysis and best fit. The equipment is also unsuited to use with creep and elastic recovery work, for the same reason.

These issues led to the decision to upgrade the instrument, as discussed in the next section.

9. AN INVESTIGATION USING THE BROOKFIELD RHEOMETER

Following on from the early work with the Haake, the price of much more sophisticated rheometers was declining rapidly, with equipment that would have been priced at well in excess of £100,000 becoming available at a far lower price. This led to an examination of the available equipment and an appraisal of the Brookfield apparatus. The Brookfield SST 200 Soft Solids Tester can operate in controlled shear stress as well as controlled shear rate modes and can measure yield directly. It can also be used in conjunction with sand of up to about 5mm particle diameter, perhaps up to 10mm with low shear rates, assuming approximate sphericity and can be used for investigations into creep. The software is powerful and reasonably easy to use. These factors led to the acquisition of a Brookfield RS rheometer.

This equipment can be used with a cup and bob in which case the maximum sand particle size is about one sixth of the clearance between the two, due to the potential edge effects discussed earlier, thus permitting work with the particle sizes instanced above. In reality, this renders cup and bob geometry only suitable for pastes.

Alternatively, it may be used with a vane, in which case theory permits the assumption of "infinite sea theory" to the specimen size, which assumes no edge effect. Sample sizes can be up to 20cm in diameter, which realistically permits maximum particle size of up to 20mm, when run under moderate shear rates. It was configured in this manner for the programme of work that is presented in the remainder of this section.

Prior to commencement of an investigation into the measurement and study of mix types and variables using the rheometer, consideration was given to the objectives of the work, in terms of first principles but also in terms of the issues identified in section

3.2.1. It was concluded that for the use of rheological techniques with mortars to be validated practically, the procedure would have to be capable of identifying and quantifying the influence of mix variables and ideally to measure the properties in table 3.6 of section 3.2.1for which no suitable test currently existed. Although it was clear that rheological techniques could not produce direct numerical values for the properties for which no satisfactory test currently exists, these, taken from the table, being "body", ease of spreading, deformability, and ease of finishing, it was believed that they would certainly be of great value in helping towards a solution. It was therefore decided to proceed with addressing the rheological procedures and examining the effect of relevant variables on the determined values.

9.1 THE EFFECT OF VARIABLES

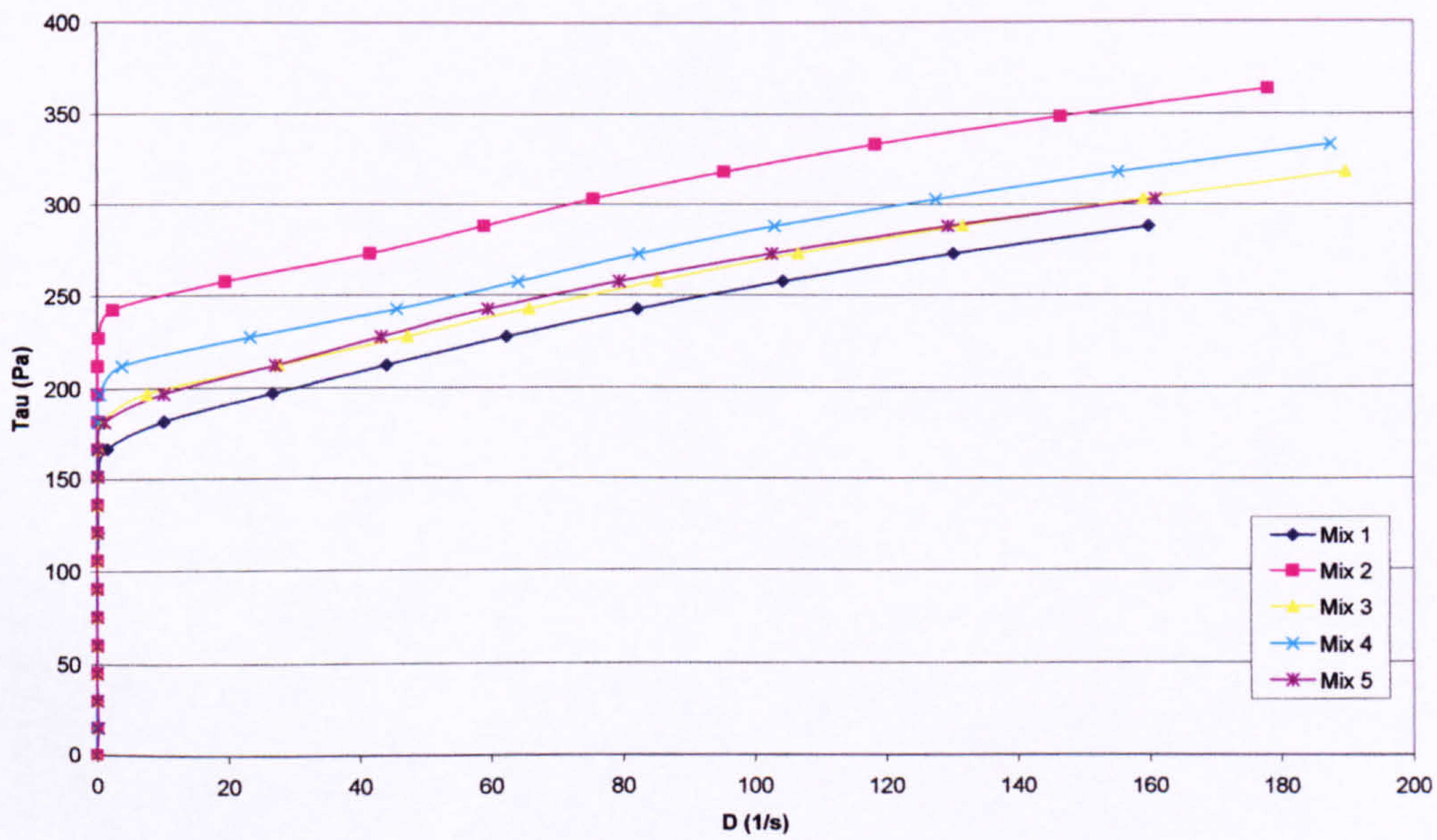
The variables that were of interest were as shown below:

- Consistence
- Air content
- Water content
- Sand grading
- Air entrainment
- Lime
- Cellulose ethers
- Non-cellulosic viscosity modifiers
- Time dependant behaviour and thixotropy

It was decided in the cause of clarity to investigate each variable separately, as described in the remainder of this section, even though a multi variant technique could still be used, utilising inputs from these primary variables.

Prior to addressing the effect of these variables, preliminary work was carried out to obtain some familiarity with the equipment and to ensure that it was capable of providing acceptable repeatability. This consisted of repeat determinations of yield stress, each made on a separate and freshly made mix of standardised 1:3 mortar, at a consistence value of 10mm dropping ball, using the previously adopted standard "Beningfield" sand blend. The results of this work are shown below.

Fig 9.1 Results of the repeatability work for the Brookfield Rheometer



This work gave a range of values for yield of from about 175 to 240 Pa.

The repeatability was initially judged to be less than that to be expected of many scientific procedures and at first glance could be considered to be quite disappointing. However, with rheological work, notwithstanding the sophisticated equipment that is often used, repeatability is frequently associated with orders of magnitude rather than the much closer absolute values associated with the majority of well accepted test procedures. This lack of absolute precision was also shown in the work with the Haake that is reported in tables 8.1 and 8.2 and is discussed in sections 2.3.4 and 7.2. Rheological techniques can only be used in a meaningful way if these principles are clearly accepted.

Although every effort was made to standardise variables, there are many reasons for variability that are always present and the main ones are discussed below.

- Raw material variation. In order to minimise this effect, sufficiently large batches of all sands, binders and admixtures were procured to complete each phase of the programme.
- Raw material segregation. Handling of all raw materials was minimised and where necessary was carried out with due care, particularly in the case of dry sands, where careful and gentle hand re-mixing was carried out prior to taking increments from bulk for test. Sands were always fully homogenised before being sampled or used for batching.
- Temperature variation. All materials and equipment were held for at least 24 hours before use and used in a controlled temperature environment. The optimal temperature control solution for samples undergoing rheometric analysis, namely a fully temperature controlled water bath was unfortunately not available.
- Segregation and bleeding of the test specimen. This issue represents a major potential source of error and is probably present to some extent in the majority of mortars. The problem is exacerbated where investigation is required of a progressive effect that may result in increasing lack of homogeneity, for example where the assessment of materials that includes those with poor working properties is required to validate an effect.

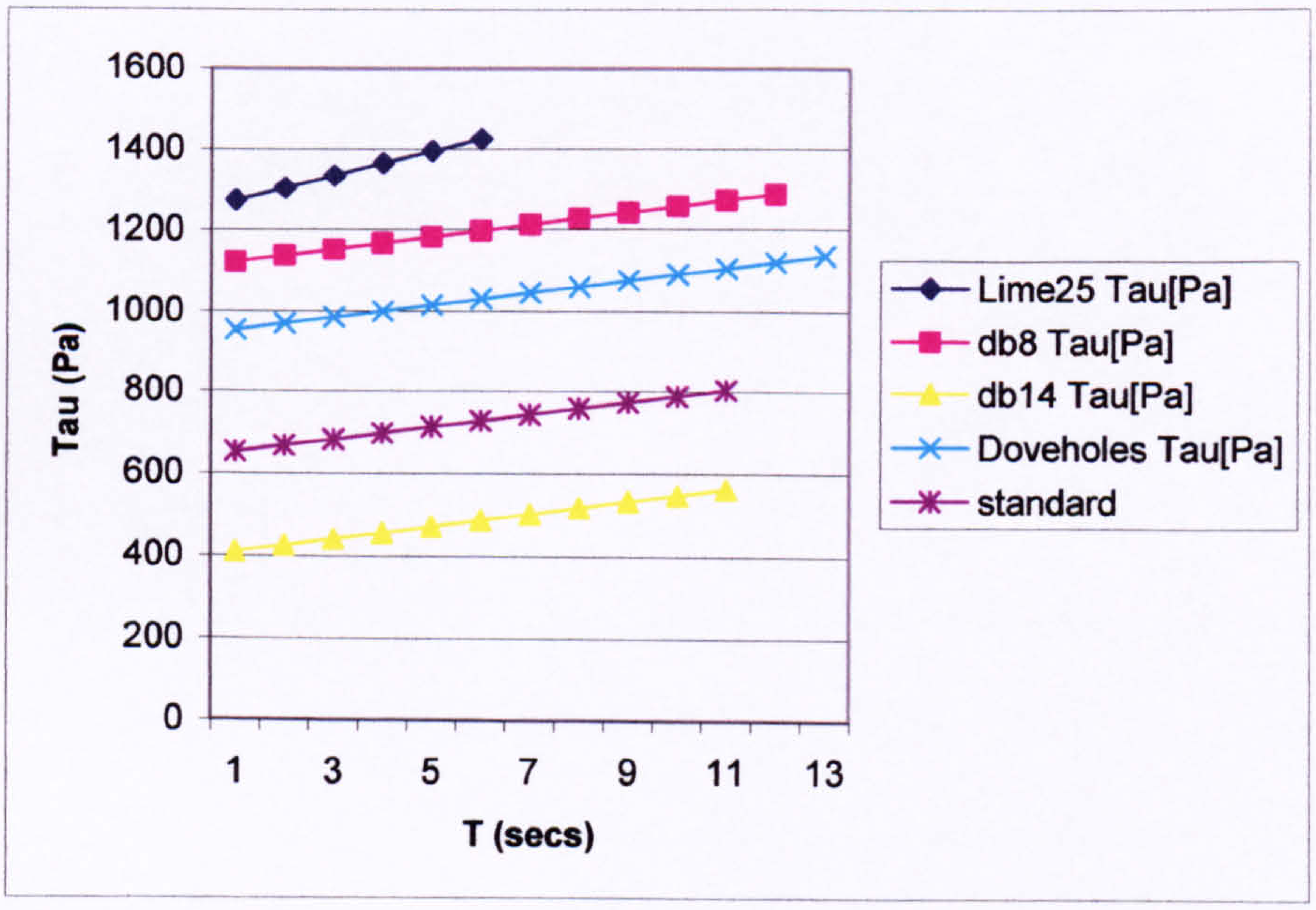
- Agglomeration. Cement particles are prone to agglomeration and both agglomeration and the subsequent breaking down of agglomerates when present, as they invariably are, by the action of the impellor or bob, will give rise to scatter of results.
- Plug flow. This may occur where major agglomerations are present within a suspension. These agglomerates may reach a size of several millimetres, yet act as a single particle and hence degrade accuracy.
- Minor influences. Any mechanism that causes or influences segregation, including bleeding, agglomeration, non-homogeneity, temperature and setting, particularly "false set", can affect rheological results. Therefore test procedures must endeavour at all times to minimise these issues.

Notwithstanding these problems the work was proceeded with for the following reasons. Firstly, there is clearly a sound basis for the adoption of the scientifically valid test regime of two point testing, in preference to the theoretically invalid one point tests currently in general usage, as discussed in section 2 of this work. Secondly, it was hoped that this completed project could form the foundation for a second project, which would refine procedures further and hopefully use even more sophisticated equipment, to build on the basic rheological work envisaged in this first instance.

The trials then investigated whether the procedure could differentiate between the yield stress for a "good" and a "bad" mortar. It was realised that the whole concept of classification of mixes into categories such as "good" and "bad" could be criticised as subjective and unscientific; it is possible that one observer might view a mortar as good, another as average or even poor. In order to address this issue, consideration was given to as many plastic properties as possible and the results of the preliminary trials with users closely borne in mind.

As discussed above, the rheological testing of mortars traditionally characterised as having poor working properties, is problematic. Therefore mix selection did not extend into the extremes of unsuitability. The yield stress of a selection of mortars that were classified with as much care as possible into good and bad are shown in figure 9.2 below.

Fig 9.2 The yield stress (Tau) of "good" and "bad" mortars



The "bad" mixes were those shown as lime 25, 25% by mass of lime produces a mortar that is too sticky, db 8, a mix that was too dry, and Doveholes, a mix with a very coarse sand. This work showed that the "bad" mortars had yield values in the approximate range 950 to 1300, the "good" materials in the range 400 to 640, an average of about 520.

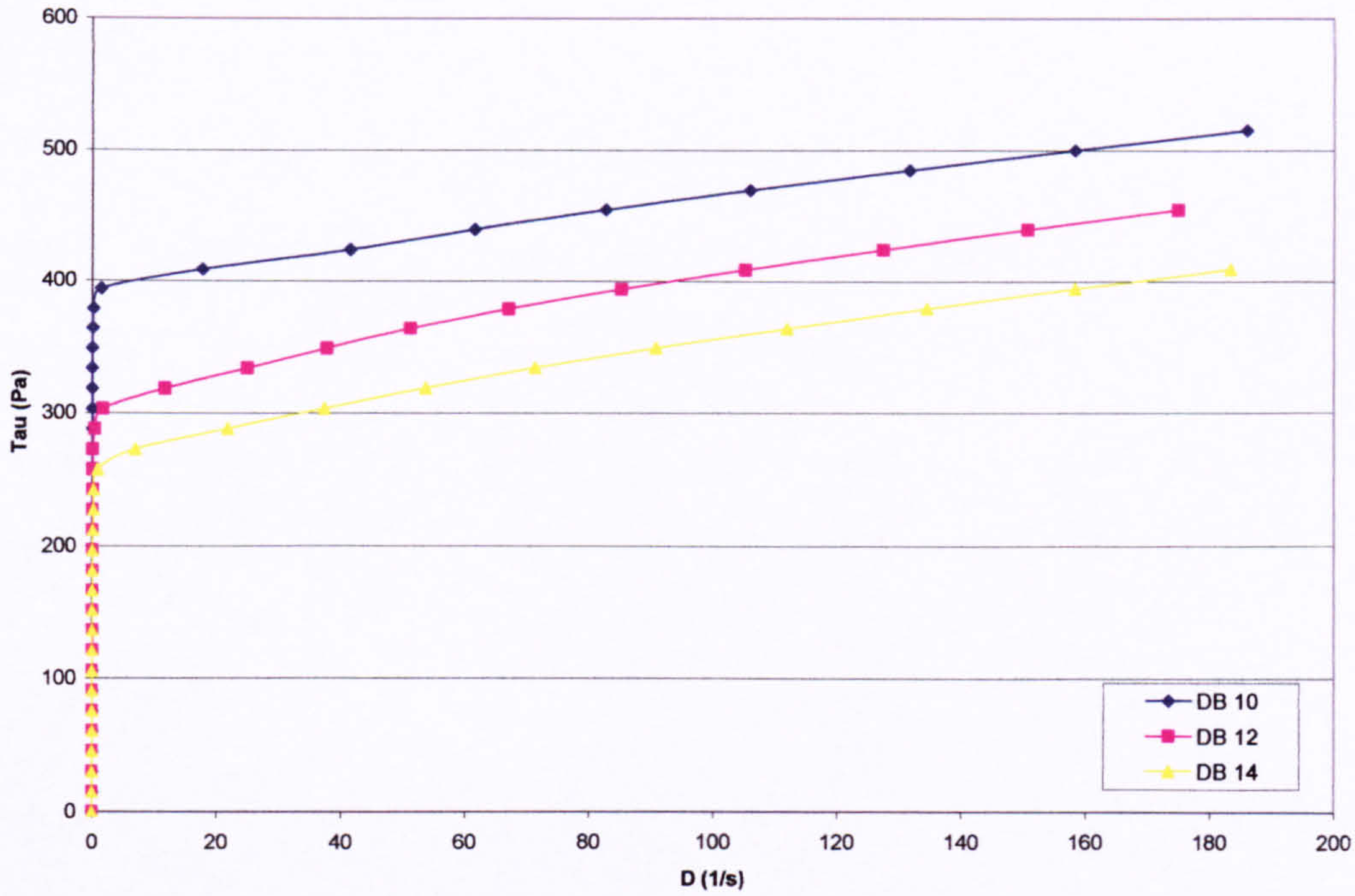
Although less scatter and a closer relationship to the mean in each case would have been desirable, these findings were taken as confirmation that there was clear enough discriminatory behaviour to validate a continuation of the project, with the values for "good" and "bad" forming two separate populations. Overall, the preliminary work confirmed that the equipment had reasonable discriminatory ability and that the yield stress changed in a way that seemed to be logical, so the remainder of the variables were then addressed as discussed below.

9.1.1 The effect of consistence

The effect of increasing the consistence was then considered and as already discussed in section 2.3.4 and reported by Gaimster and Griffin, 1999, this is said to decrease the yield value, a clearly logical phenomenon.

To investigate this parameter, mixes were made with the same moisture content but with a different dropping ball value. This was achieved by mixing material with an excess of air entraining admixture for progressively increasing periods of time. This showed a decrease in yield stress with an increase in consistence and appeared to confirm the reported link between plasticity and yield stress. The finding also appeared logical, as the mortar became more plastic and easier to deform, so that the yield value decreased. These findings are shown graphically in figure 9.3 below.

Fig 9.3 The effect of consistence value by dropping ball on yield stress

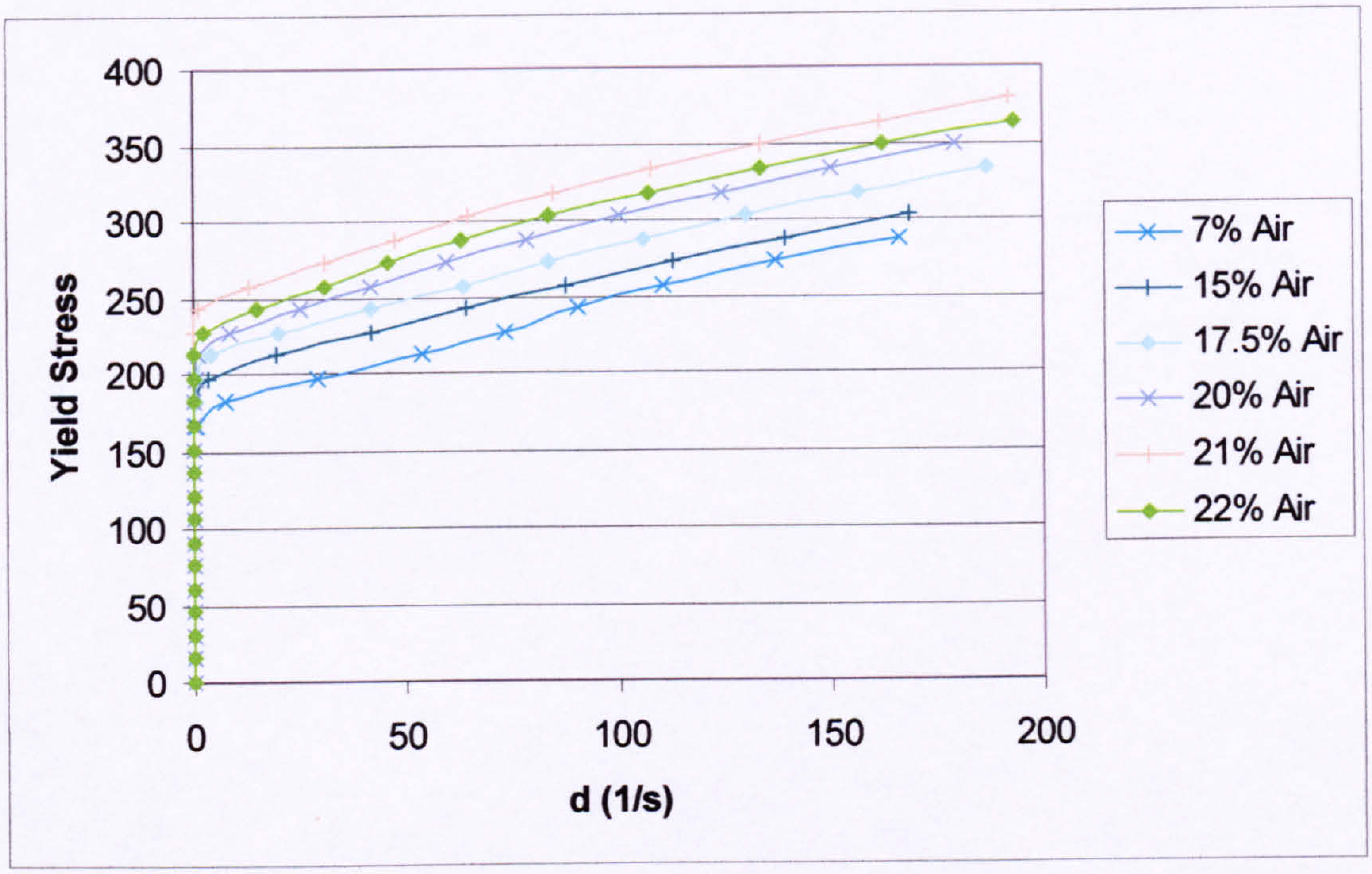


This initial rheological work had shown that the yield stress changed in a way that seemed logical but a fuller rheological characterisation was thought to be one based on determination of the yield stress and also of the plastic viscosity at that yield and the Brookfield apparatus had the potential to produce results in this form. The data capturing software was therefore addressed to produce appropriate output and the remaining work reports the way that each of these values, the yield and the viscosity, changed as a result of an input change in the particular variable that was being examined. The effects of each of the variables examined are described below, with the "standard" 1:3 mix and "standard" Beningfield sand being used in all cases.

9.1.2 The effect of air content

The effect on the yield stress of changing the air content from 7 to 22%, but keeping the consistence value fixed, was then investigated, as shown below in figure 9.4.

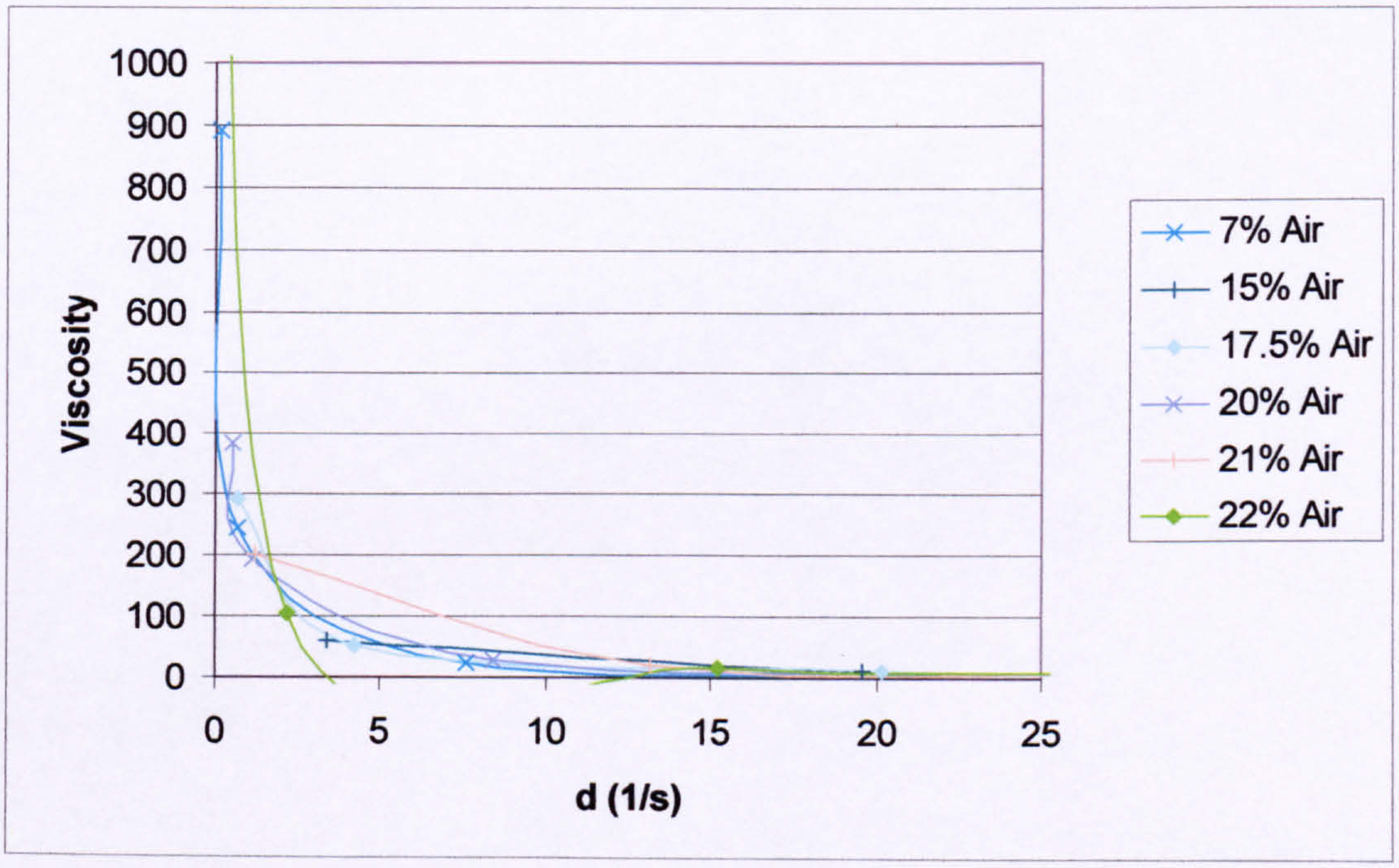
Fig 9.4 The effect of changing air content on yield stress



This showed an increase in yield as air content increased. At first sight this result was a little puzzling, as it had initially been postulated that addition of air would always tend to make the material easier to work and deform. Traditionally the amount of entrained air is increased for mortars of inadequate working properties in terms of anecdotal assessment on the building site and the inclusion of this entrained air always seems to enhance the plastic properties of the mortars in question making them easier to work.

However, further consideration confirmed the logic and probable validity of the finding, in that an increase in air entrainment at a fixed consistence level is observed to give rise to a material that whilst "creamier" and easier to move with the trowel is nevertheless more cohesive. Furthermore, air may crudely perhaps be considered as added fines, which will clearly make the material more cohesive and "sticky". Additionally, it is proposed that the excess air entraining agent present in the mortar but not actually part of the entrained air system, that is the "excess" surfactant not actively bonded and entraining air, will produce a cohesive feel to the system as the electrical charges associate, partly with each other but also with the other species present in solution and suspension. The effect on the viscosity was then determined and this is shown in figure 9.5 below for fixed consistence.

Fig 9.5 The effect of changing air content on viscosity



This work showed that as the air content increased, so did the viscosity.

Overall then, the work on air content showed that an increase was accompanied by an increase in both plastic viscosity and yield stress, so long as the consistence is kept constant. Although this finding was unexpected, it is seen to be perfectly logical and when analysed, to relate to practise. Anecdotally, adding air entrainment could at first sight be judged to be likely to reduce yield and viscosity, making a more mobile and fluid mix but it is important to remember that the present work has been carried out at a fixed consistence. Practical experiences leading anecdotally to suggestions of reduced yield and viscosity always tend to be accompanied by an increase in consistence, and it is suggested that it is this, which not only hides but reverses the real effect, that of increase in these values at constant consistence.

At this stage of the programme the vane in the rheometer was changed for the next larger size. This is routine, a series of determinations is typically commenced with a conservative vane size, one that does not exceed the torque capacity of the motor and sensing equipment, and this is then increased after preliminary work to provide a greater surface area and a corresponding increase in accuracy. Work then proceeded with the remainder of the test programme, with assessments of the effects of a number of parameters as follows.

9.1.3 The effect of water content

It was thought that an increase in water content would give rise to a decrease in both yield stress and plastic viscosity, as previously discussed in section 2 and reported by Gaimster and Griffin, 1999. A brief investigation showed this to be the case. The result of this work is shown in figs 9.6 and 9.7 below.

Fig 9.6 The effect of water content on yield stress

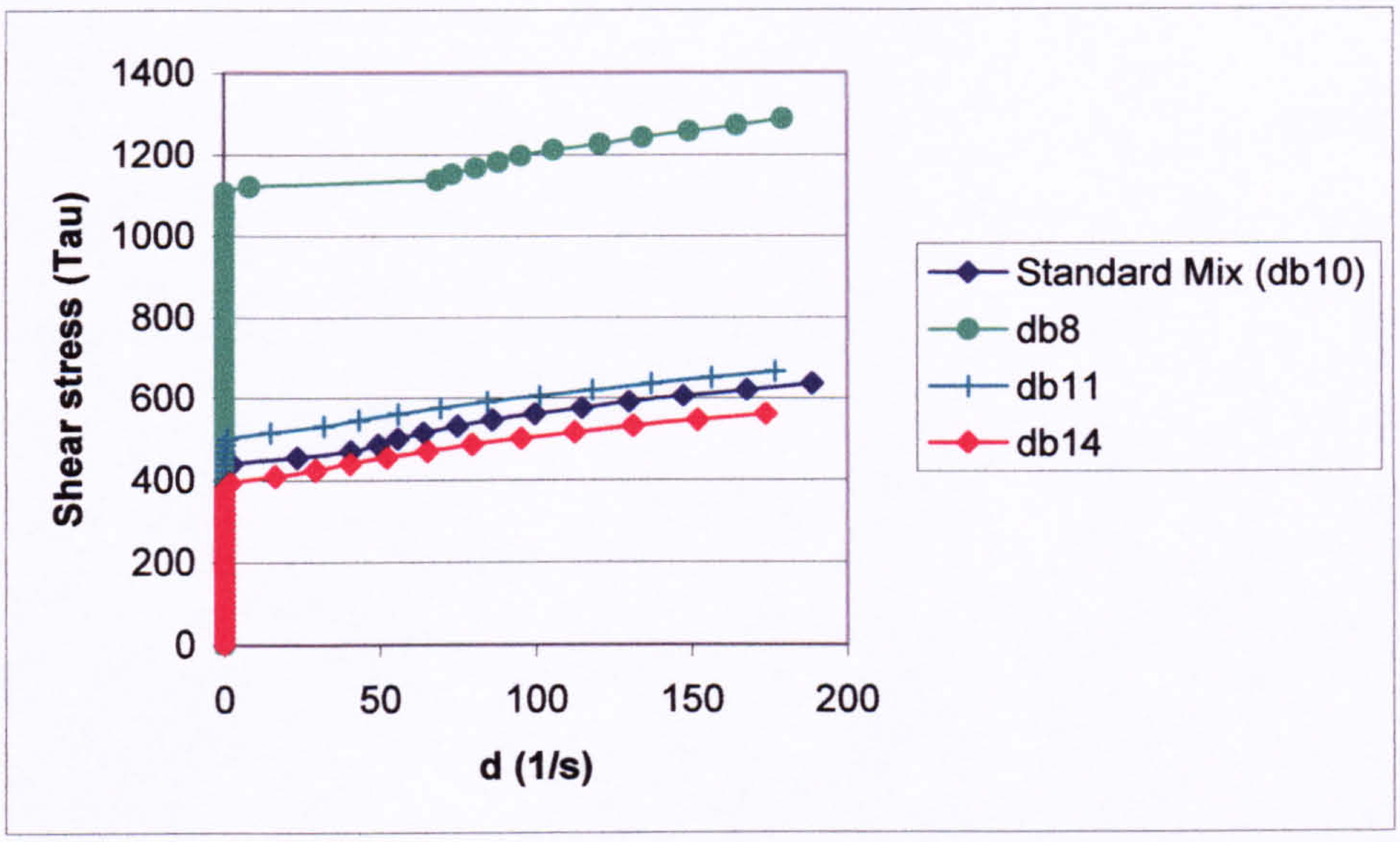
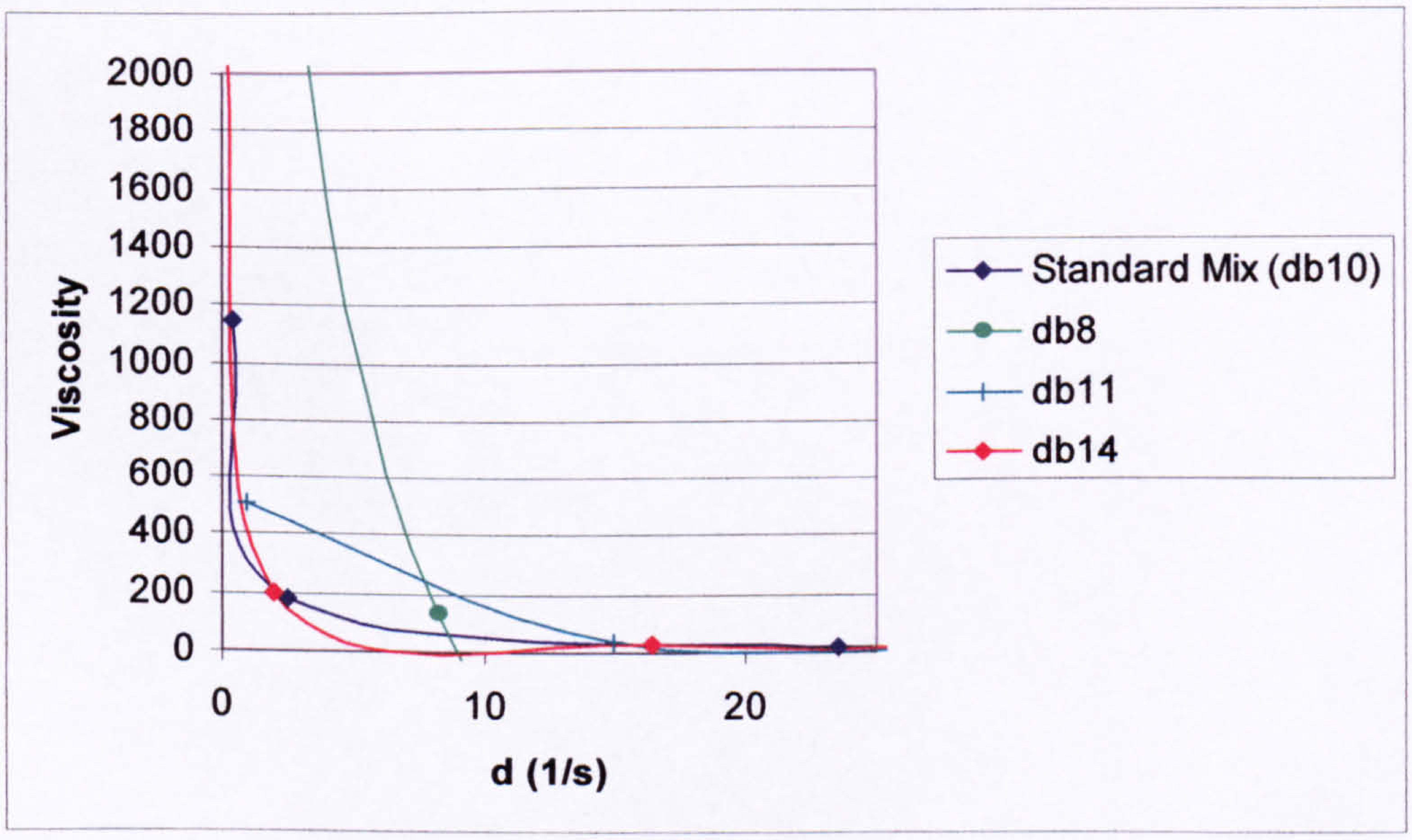


Fig 9.7 The effect of water content on viscosity



As indicated above these findings were as expected, water has a reduced yield stress and is less viscous than cement or mortar paste, and these results were taken as further evidence that the procedure gave rise to logical results.

9.1.4 The effect of sand grading

Four sands were examined in this work, a very coarse limestone from Doveholes quarry, the coarse CEN standard sand described in section 3.1.1, the "Beningfield standard" sand and finally a very fine mortar sand from Aylesford. The results for these materials are shown in figs 9.8 and 9.9 below. In fig 9.8 it is seen that yield stress shows a progressive decline with increasing fineness, whilst fig 9.9, which shows the results for the work on viscosity shows similar trends, although with not such pronounced discrimination, after the first few seconds of testing.

Fig 9.8 The effect of sand grading on yield stress (Tau)

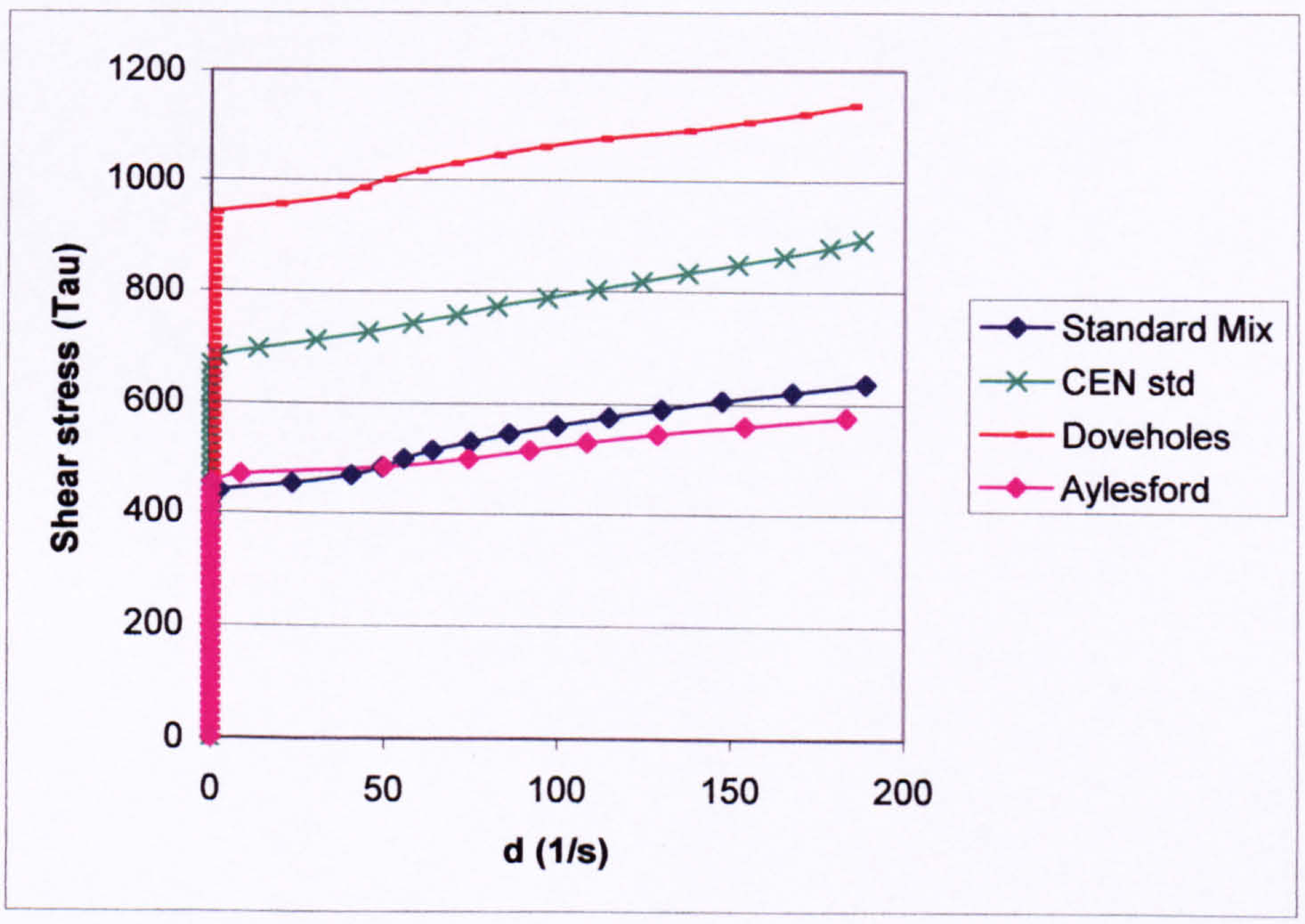
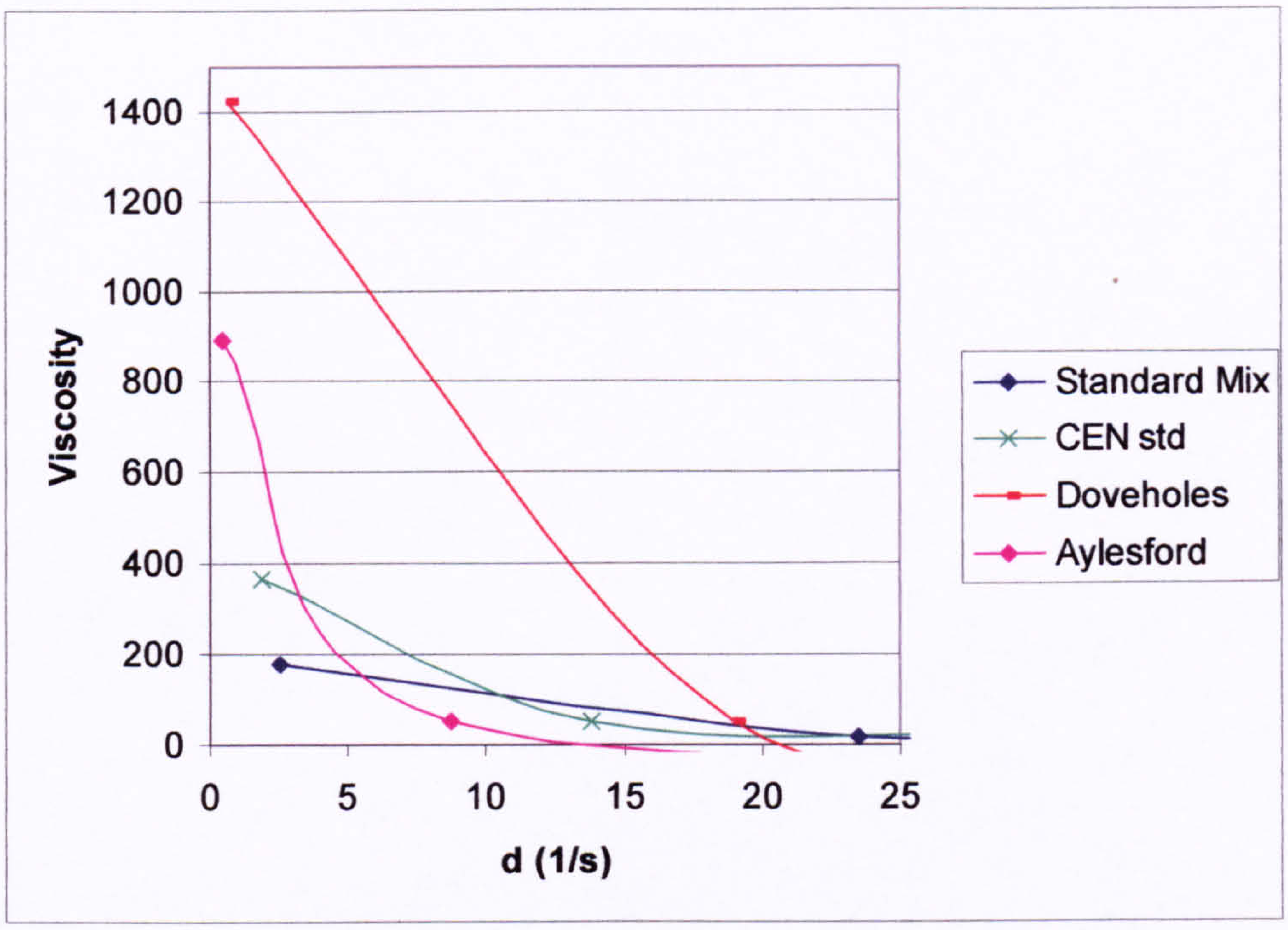


Fig 9.9 The effect of sand grading on plastic viscosity

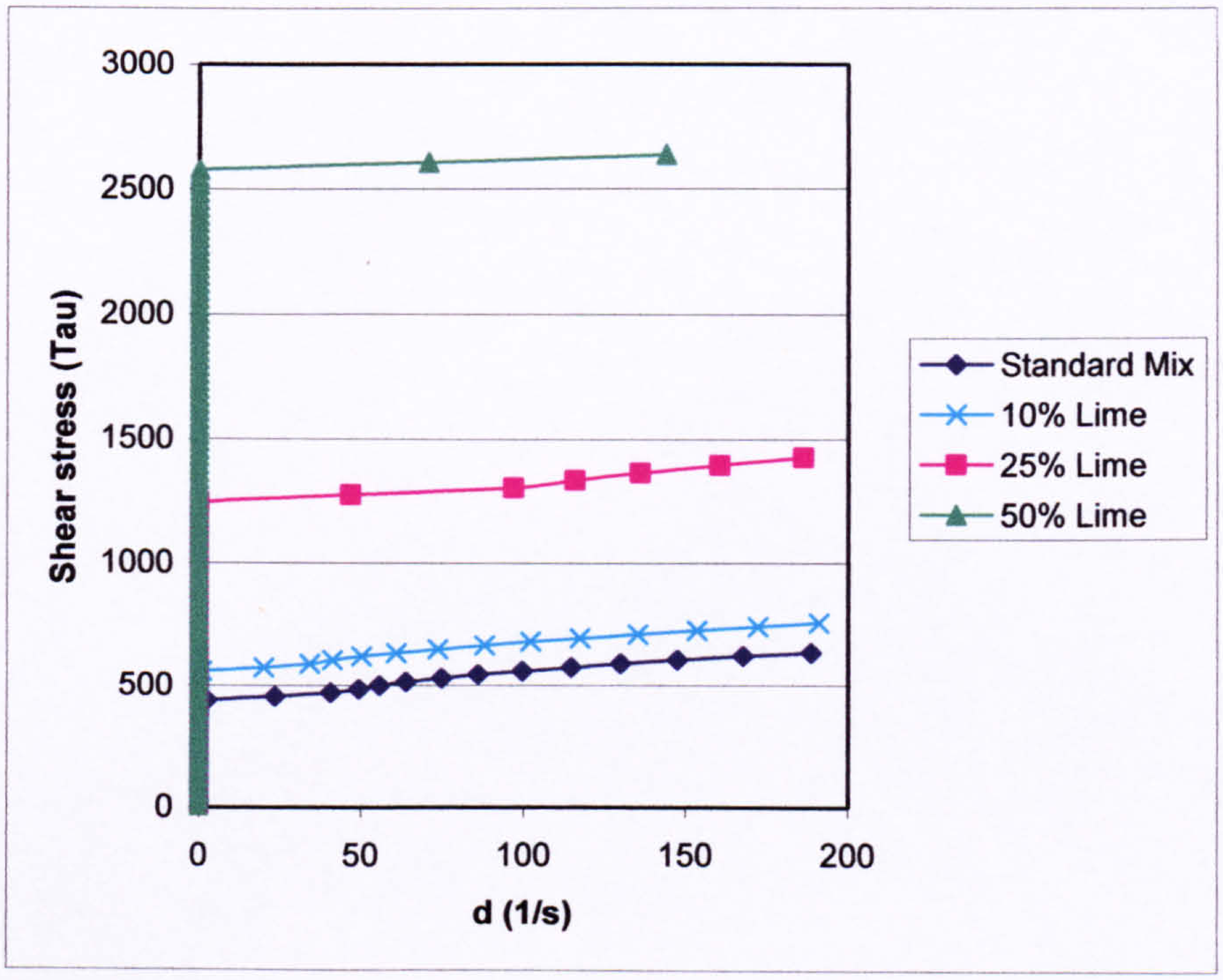


Again, these results are not unexpected. The very coarse materials appeared when judged, albeit subjectively, using a palette knife and a trowel, to have a high yield stress. In addition, they possessed a property that was certainly analogous to viscosity, and may perhaps be described reasonably accurately in that way, that was high. Even when the initial yield stress had been overcome, they gave considerable resistance to movement.

9.1.5 The effect of lime

Because the addition of lime to a mortar causes an effect on the plastic properties of considerable magnitude, it was expected that the rheometer would show marked trends in this part of the work and that did prove to be the case. Figure 9.10 below shows a substantial increase in yield stress as lime is added in progressively larger amounts, although it should be noted that the lime additions used at the higher addition rates were greater than those that would normally be expected on the building site, in order to ensure that the effect could be easily measured.

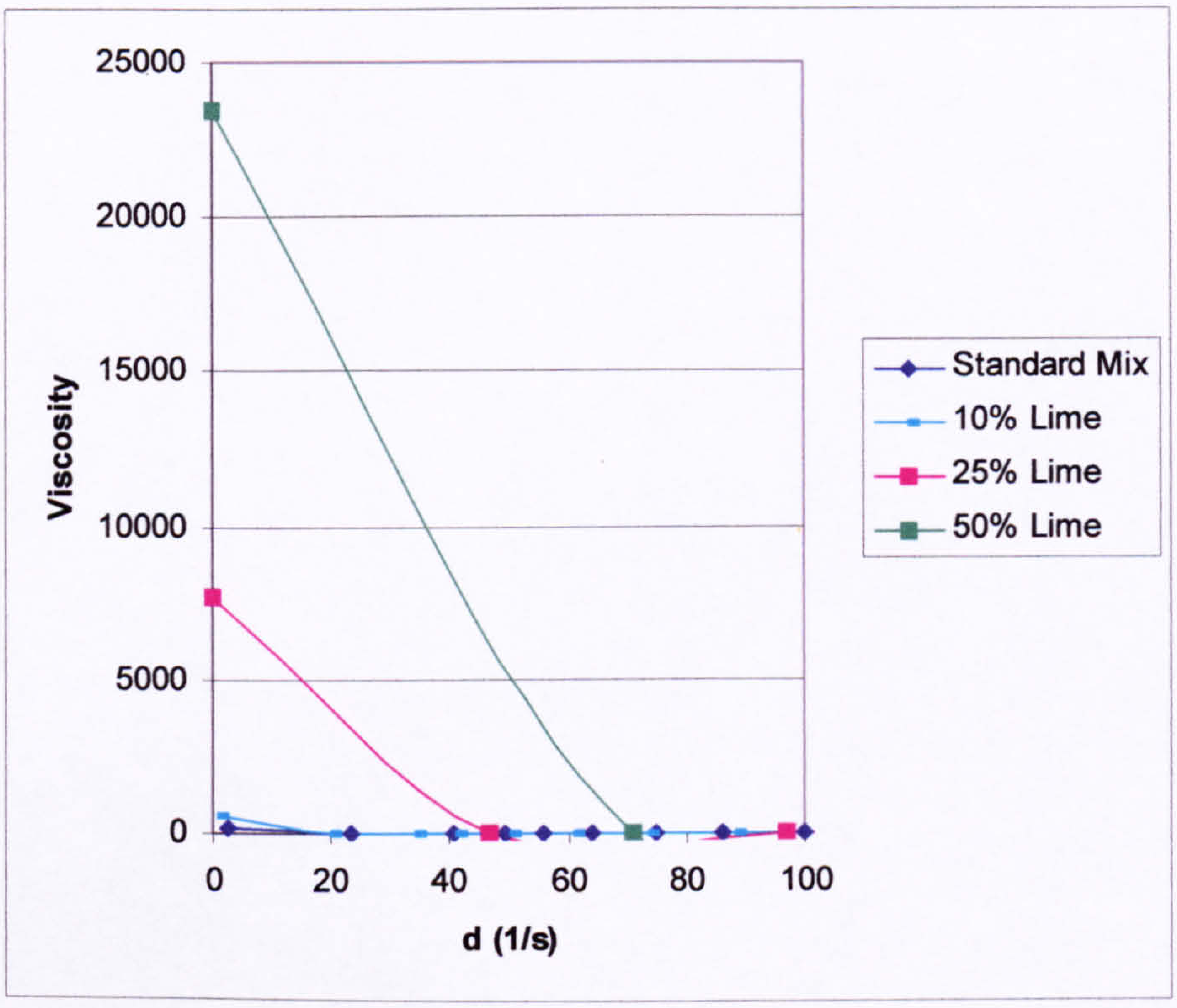
Fig 9.10 The effect of lime content on yield stress



The effect of lime content on the plastic viscosity is shown in figure 9.11 below, from which it may be seen that viscosity also increases markedly with an increase in lime.

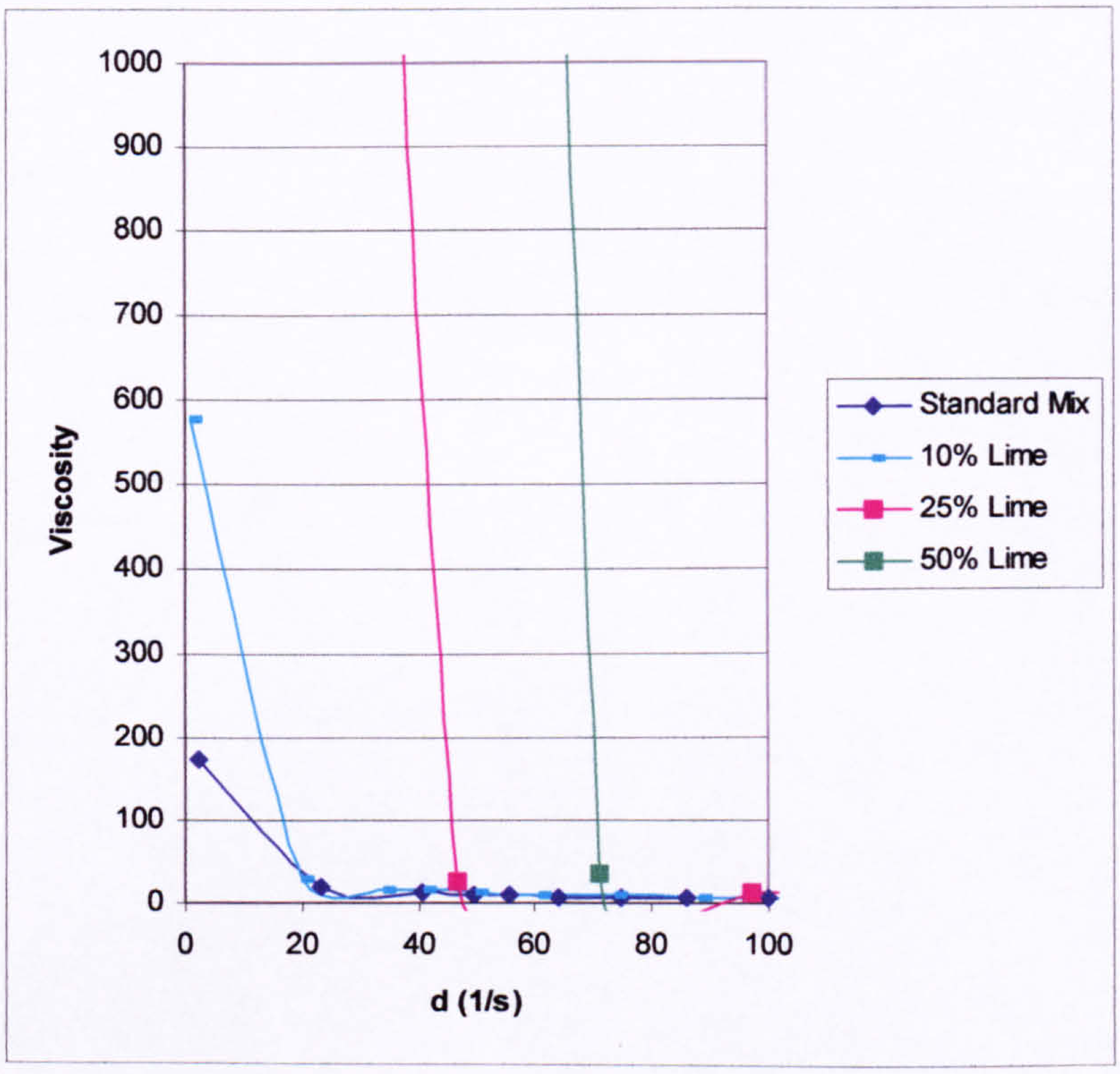
It is seen therefore that an increase in lime content makes a material that is more difficult to move in the first instance, ie has an increased yield stress, and is also more difficult to maintain in motion. The one property that is not really adequately covered by the two rheological parameters that were monitored was the tendency to segregate and bleed, although it is invariably reported that in practice this is reduced as the lime content increases. It is believed that in general an increase in viscosity will lead to a decrease in segregation, as it was suggested long ago by Powers, 1939, that bleeding was a special case of segregation and it appears logical, although not definitely proven in this work, to expect segregation to decrease as the system becomes more viscous.

Fig 9.11 The effect of lime content on viscosity



The change in viscosity brought about by increasing the lime content is so pronounced, extending through several orders of magnitude, that it is not easy to represent both extremes with one graphical scale so figure 9.12 below is included, to be read in conjunction with figure 9.11, which shows the effect plotted using a different scale. It can now be seen that the viscosity varies from about 180 to about 23,000 Pascals.

Fig 9.12 The effect of lime on viscosity with the x axis rebased



9.1.6 The effect of cellulose ethers

Cellulose ethers and the extent of their current usage illustrate some of the changes in current mortar technology and the need to adapt procedures and consider new ones. It is estimated that in excess of I million tonnes of mortar production per annum in the UK now incorporates these materials, making an investigation into their input into the rheology of mortars of key current interest. They are present in the majority of factory made mortars, and dry silo mortars and the most sophisticated dry bagged mortars invariably contain them. They are complex materials, with very long carbon chains and viscosities in solution of from about 2,000 to 80,000 centipoises. They modify the mortars in various interacting ways, many of which are still not completely quantified.

Their long chain molecules may be simplistically regarded as "molecular sieves" that provide by the means of a barrier, a reduction in the loss of water into high suction backgrounds as for example some brick or block types. Because they greatly increase the viscosity the mortar shows less segregation and bleed. The charged form of the materials also results in internal interaction to "structure" and align dissimilar charges so that they can enhance thixotropic effects.

All of these phenomena mean that cellulose ethers affect the plastic properties in complex ways and mortars containing them are difficult to characterise using only basic techniques. The effects of the inclusion of a typical cellulose ether are shown in figures 9.13 and 9.14 below.

These results demonstrate the effectiveness of rheometry in this field of study. They showed that the yield stress increased a little, whilst remaining in the same order of magnitude, but the plastic viscosity increased markedly with an increase in cellulose ether content. This finding was in accord with anecdotal user views, where mortars containing large amounts of cellulose are referred to as "sticky" or "podgy", although showing much reduced tendency to segregation and bleeding. This illustrates the potential for modifying the viscosity alone, for example where segregation is a problem, without markedly increasing the yield stress, although clearly the amount by which this may be done is constrained by the need to avoid excessive stickiness.

Fig 9.13 The effect of cellulose ether on yield stress

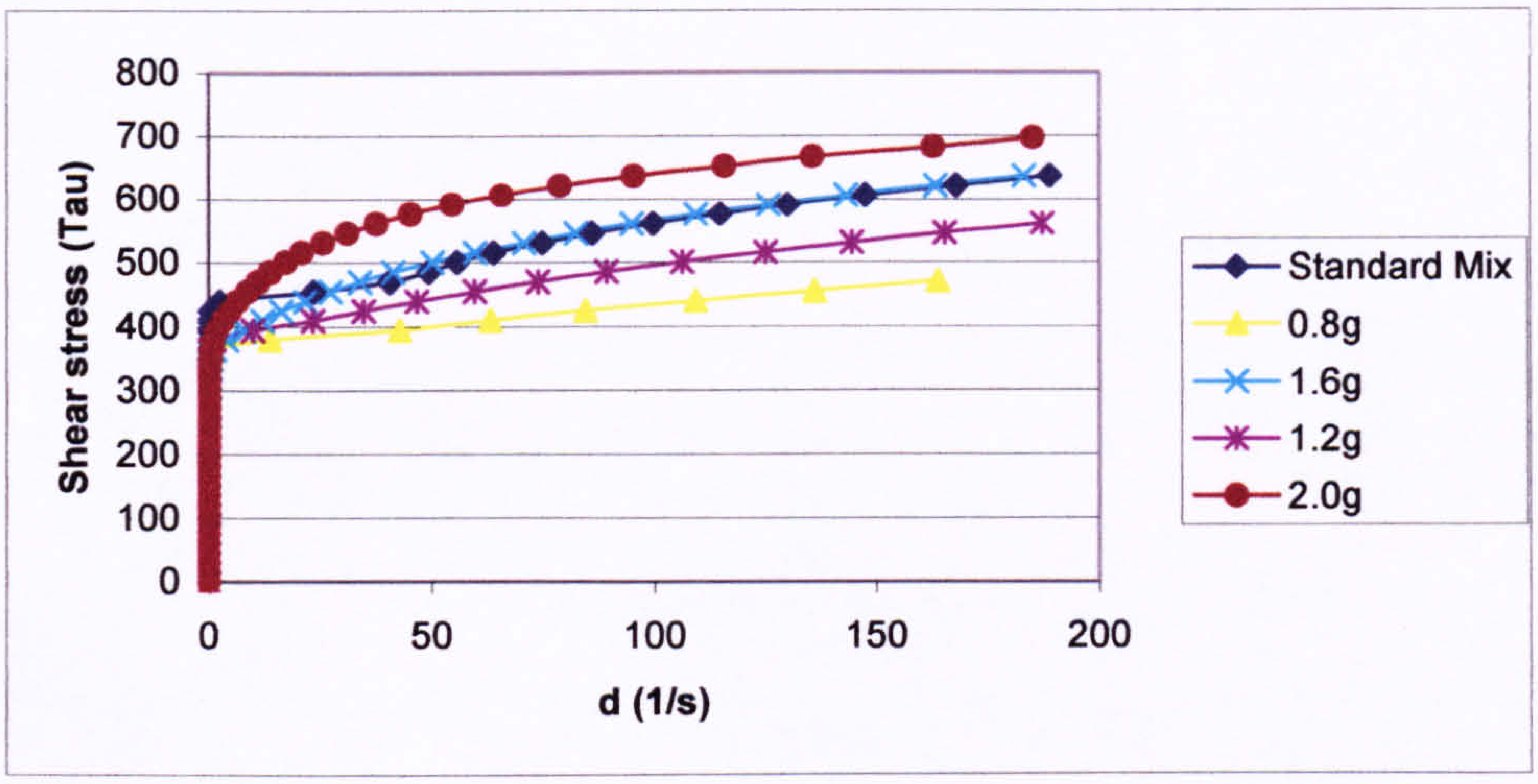
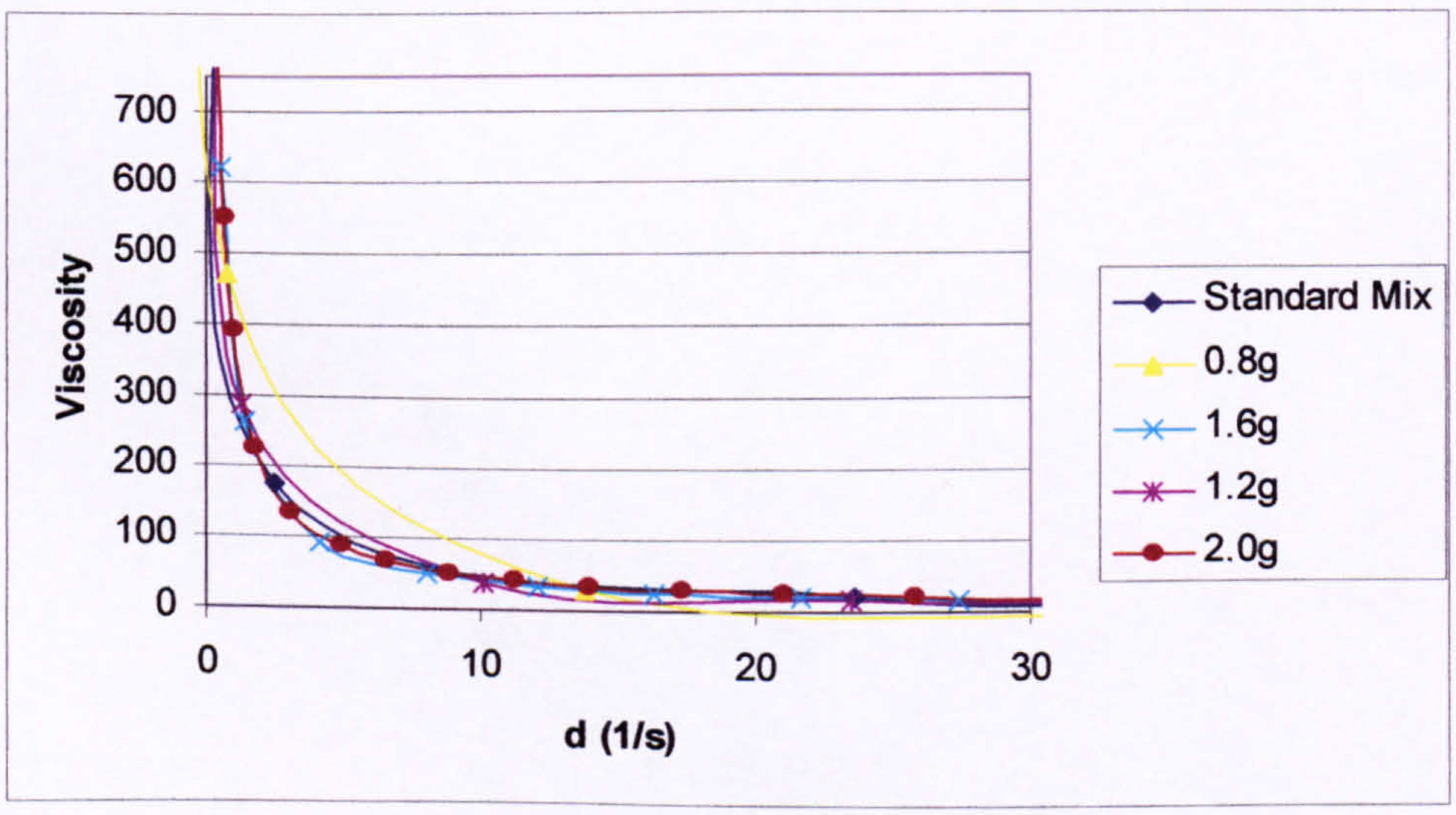


Fig 9.14 The effect of cellulose ether on viscosity

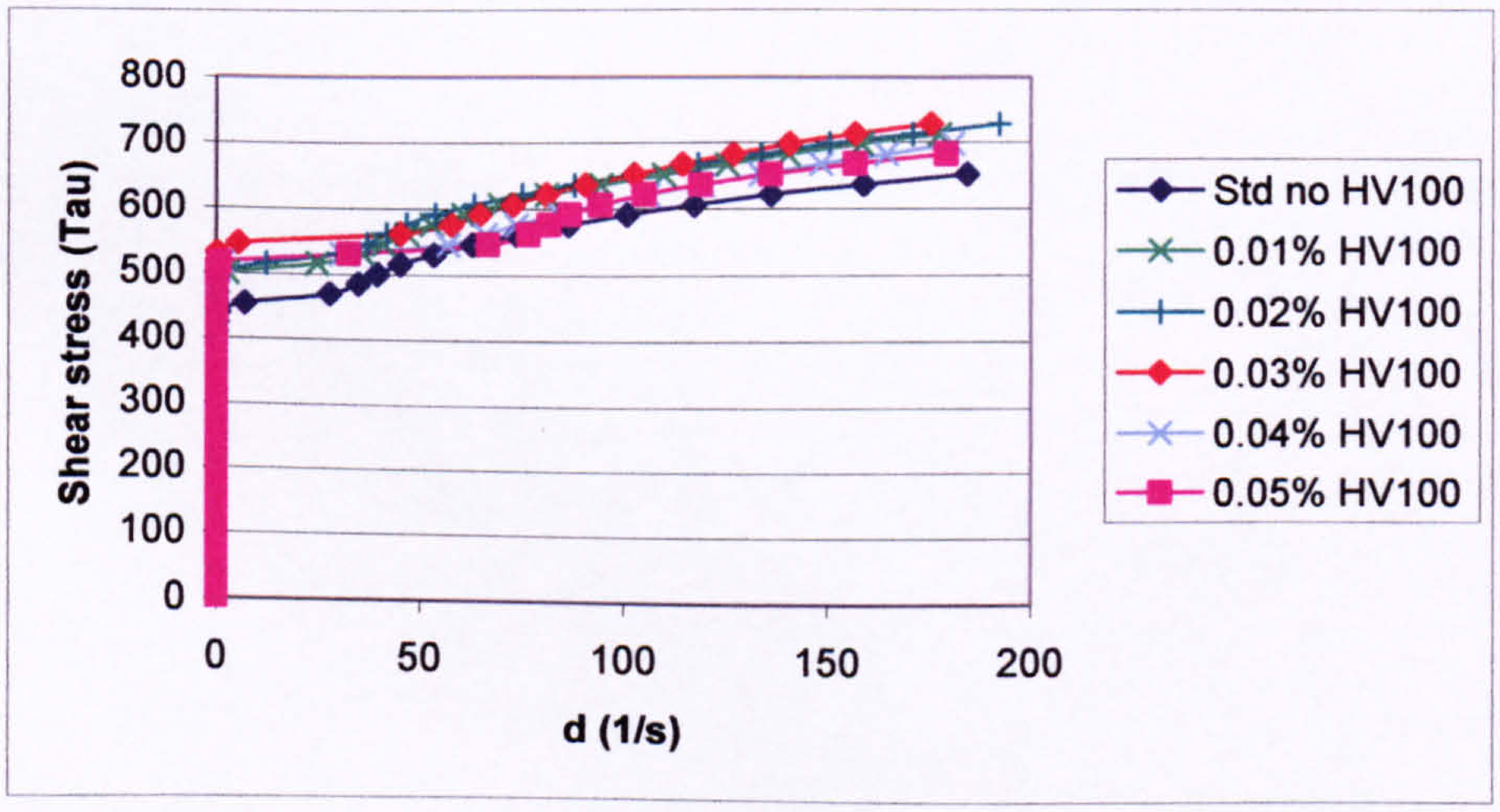


9.1.7 The effect of non-cellulosic viscosity modifiers

Cellulose ethers are generally regarded as costly materials when placed in the context of the building materials industry and are also often very pure, as their key markets are areas like foodstuffs and pharmaceuticals where these attributes are generally required.

For use in the field of construction, however, this purity may not really be needed and a variety of alternatives are available that provide adequate performance and quality without excessive cost. These alternatives are of several types. One class of materials that are still relatively pure but may be more economic are based on acrylic copolymers. These materials function by swelling in aqueous solution in the presence of alkali and are thus appropriate for solutions/suspensions containing Portland cement. They are now widely used in dry mortar and plaster formulations, sometimes in combination with cellulose ethers. Trials were carried out on one such material, produced by a multinational chemicals company and marketed as Viscalex. Figure 9.15 shows the yield stress behaviour of mortars modified with this material, figure 9.16 the plastic viscosity profile.

Fig 9.15 The effect of Viscalex acrylic copolymer on yield stress



It may be seen that this material modifies the viscosity but has only a minor effect on the yield stress. The way in which this material behaves may be shown with greater clarity by plotting the two parameters against addition rate as shown in figure 9.17 and this clarifies the minor change in yield stress but the pronounced increase in viscosity.

Fig 9.16 The effect of Viscalex acrylic copolymer on viscosity

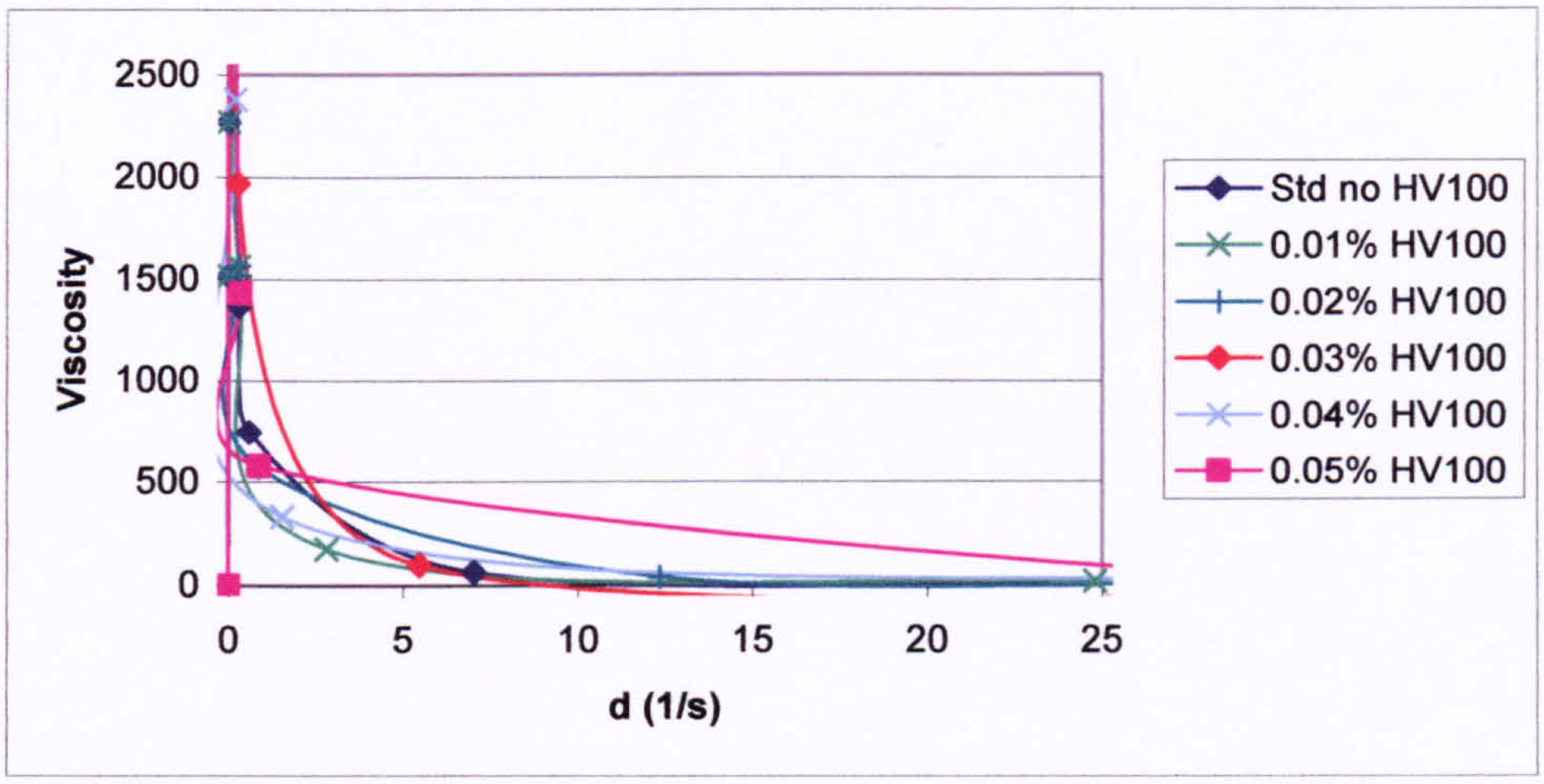
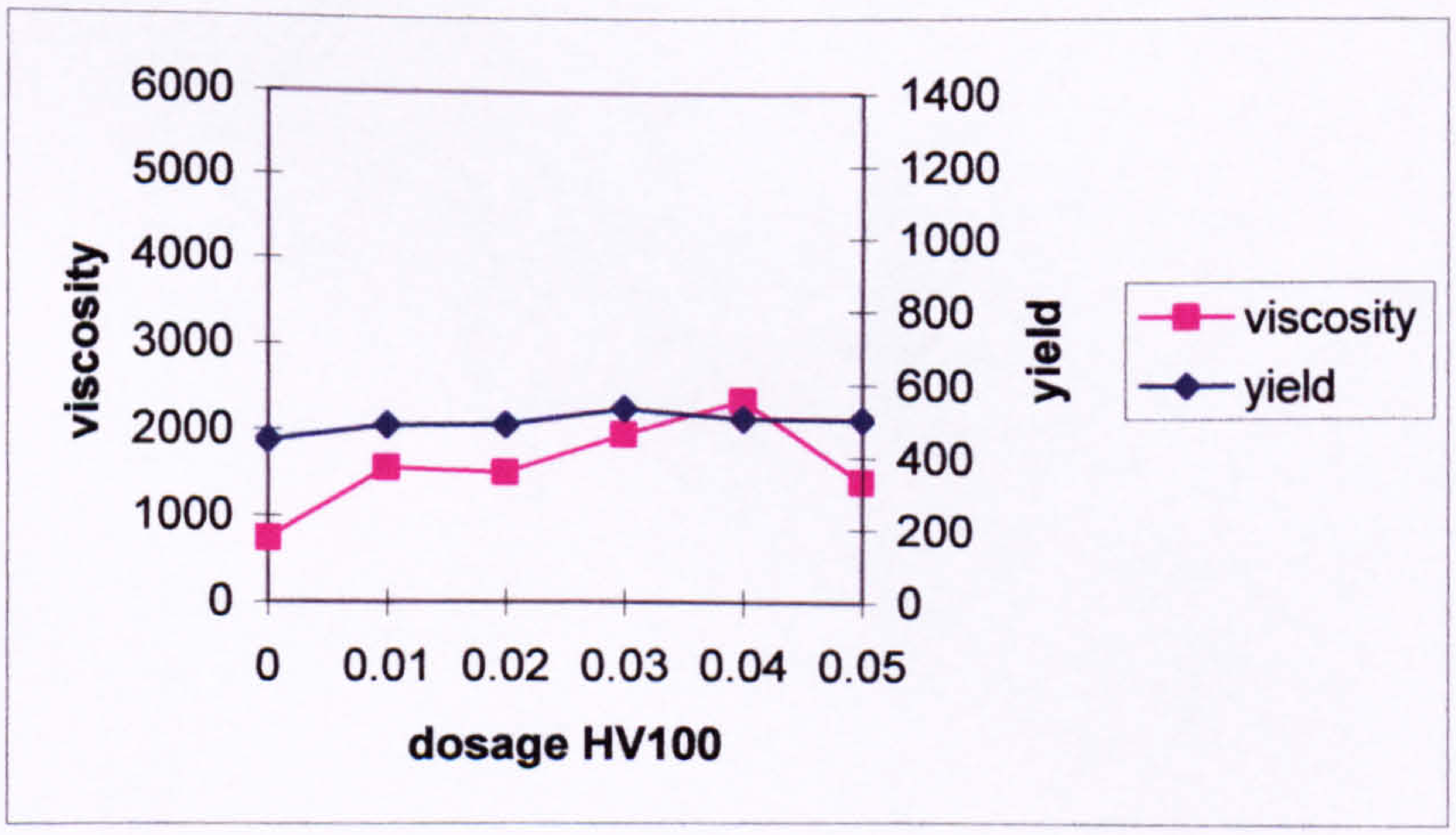


Figure 9.17 shows an interesting effect in that there appears to be an optimum addition rate in terms of the cost effectiveness with regard to viscosity at an addition of about 0.01% by mass. It is unclear whether the 0.05% value for viscosity is valid or an outlier and unfortunately it was not possible to repeat this determination.

Fig 9.17 The effect of Viscalex acrylic copolymer on viscosity and yield stress



There are still further types of viscosity modifiers that could be regarded as being even more remote from the very pure and relatively expensive cellulose ethers and one such is the group of materials known as polysacharides. These have long chains, with a molecular backbone similar to cellulose or starch, which may themselves be considered as polysaccharide materials.

Many polysaccharides are made by a process of fermentation or bacterial decomposition and one of these types that may be used in mortars is xanthum gum. This is a long chain polysaccharide composed of a cellulosic backbone and sidechains of three sugars, glucose, mannose and glucuronic acid. It is produced as a slimy gel arising from bacterial attack on cauliflower, broccoli and similar vegetables. It is used in convenience and pre-cooked foods and sauces as well as animal feed products as a viscosity modifier. It also acts synergistically with some other natural gums so that the overall effects of each are amplified. The results for yield were the first in the research programme to exhibit lack of a clear trend, as shown in Figure 9.18 the xanthum gum appears to have little or no effect on the yield stress

Fig 9.18 The effect of xanthum gum on yield stress

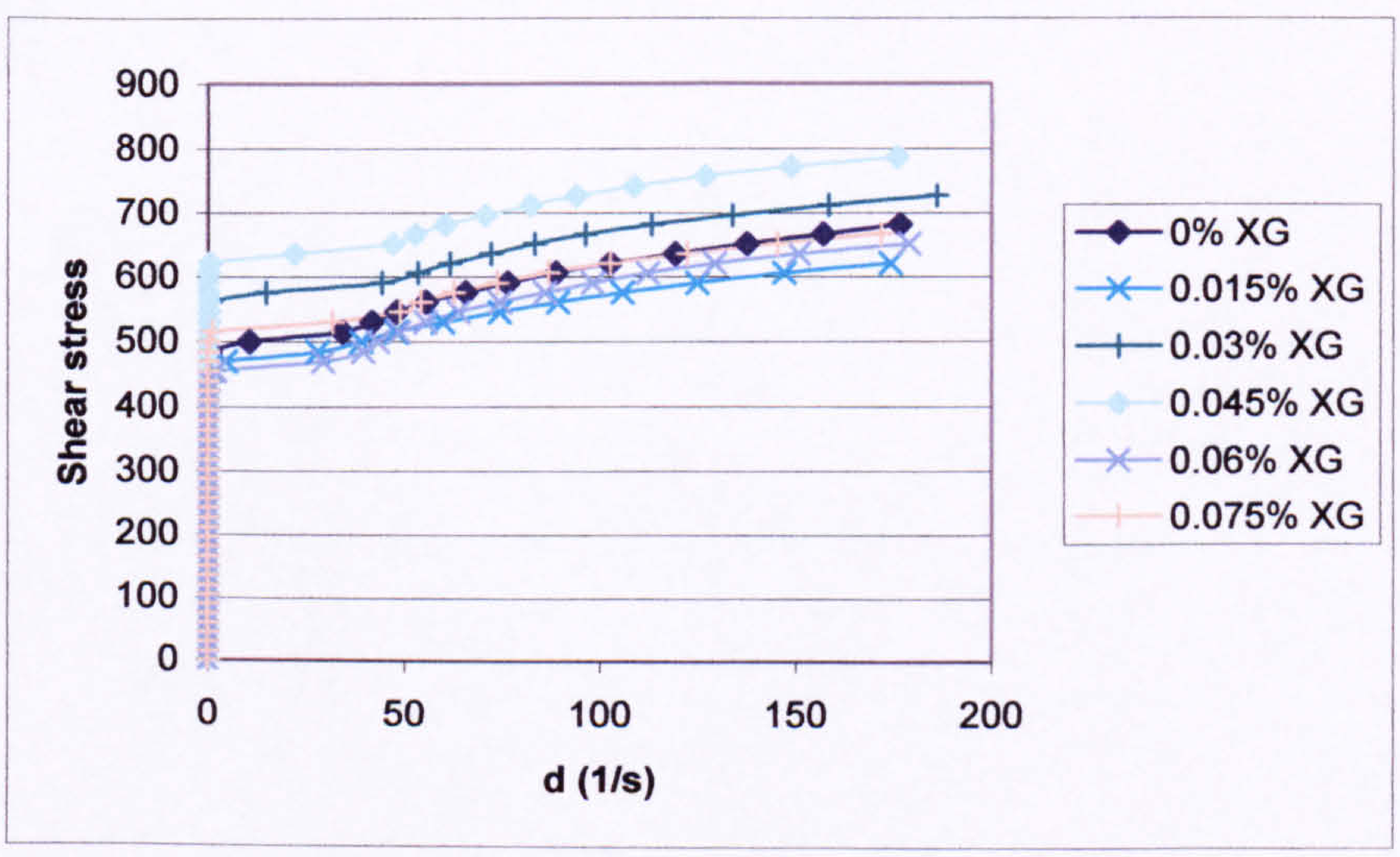
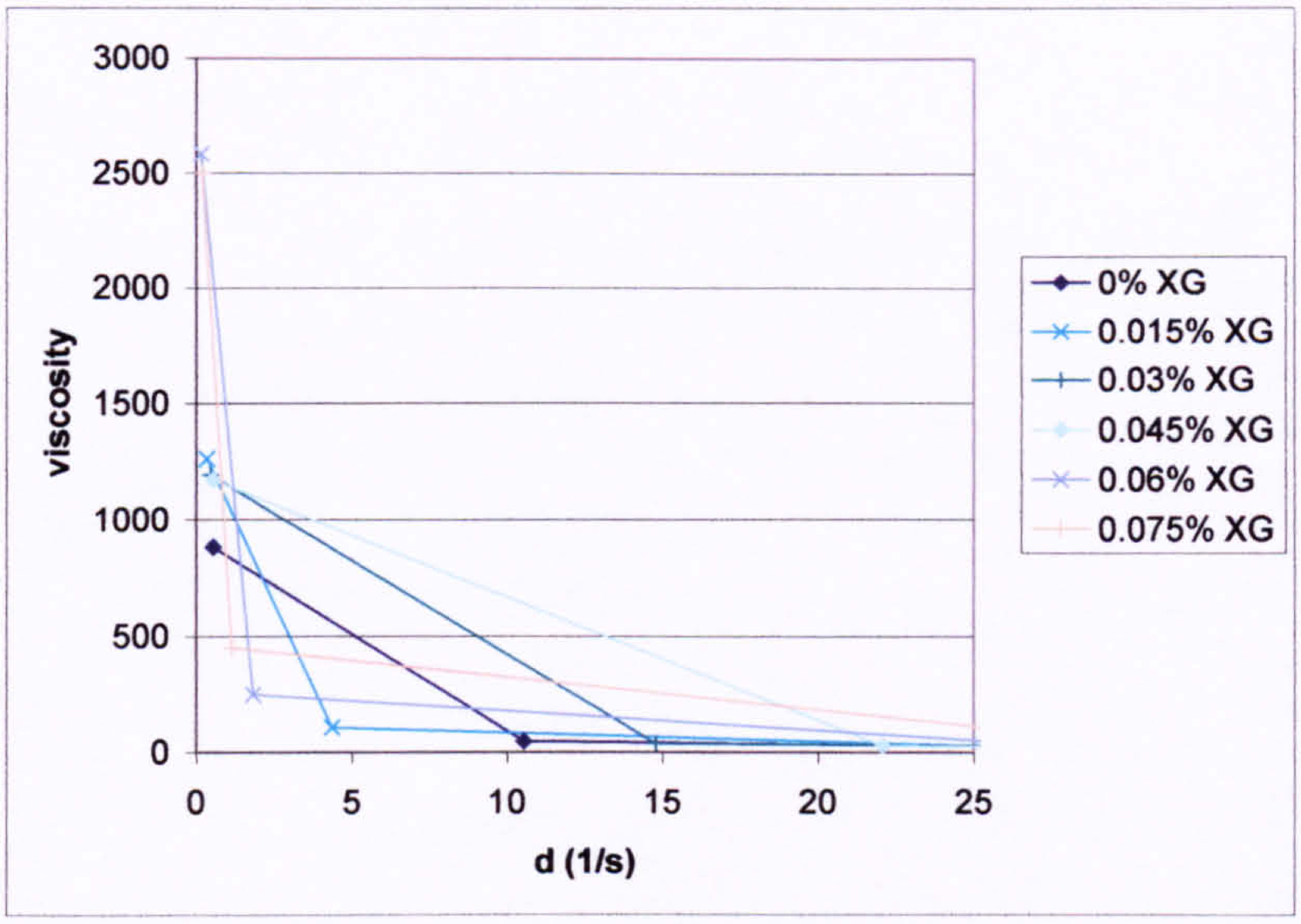


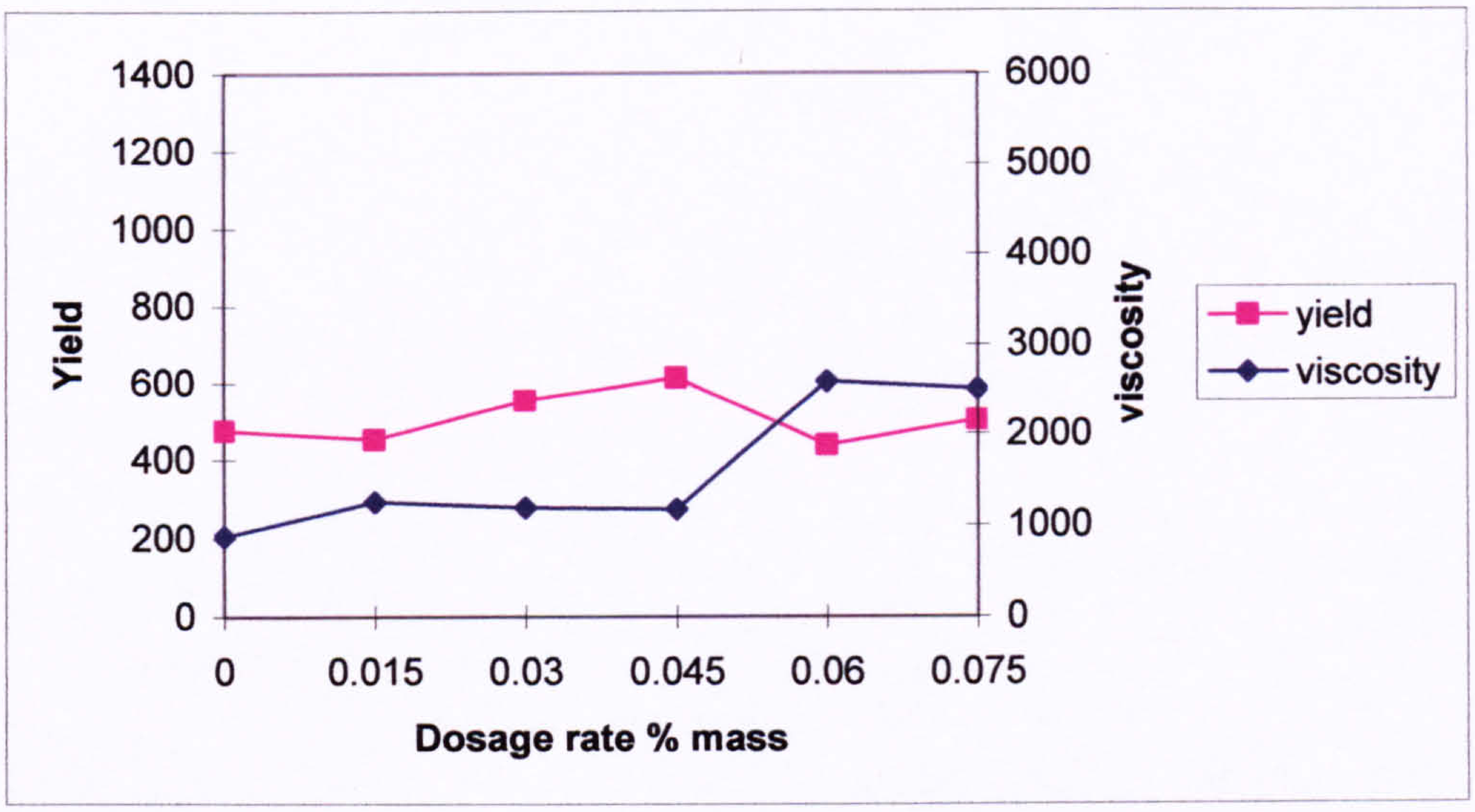
Figure 9.19 below, however, shows that the material has a profound effect on the viscosity, with increased gum levels progressively increasing viscosity.

Fig 9.19 The effect of xanthum gum on viscosity



The effects here are not easy to interpret and the overall effect of the xanthum gum may be better illustrated by plotting the variation of yield stress and viscosity with dosage and figure 9.20 demonstrates this.

Fig 9.20 The effect of xanthum gum on yield stress and viscosity



It is seen from figure 9.20 that the yield remains in the same order but the viscosity increases, in the same manner and order of magnitude as with the use of acrylic copolymer, although to a smaller extent than with the lime trial mixes. It should be noted though that the lime was used at a much higher addition rate.

9.1.8 Time dependant behaviour and thixotropy

As discussed earlier, thixotropy is present in virtually all mortars, and certainly in all of the more advanced formulations that contain one or more admixtures. The field of thixotropy is large and its study represents a specialist science, the detailed investigation of which is outside the scope of this work. However, the major thixotropic influences relevant to the project are discussed below.

In this work the thixotropic mechanisms are divided into four groups for convenience. These are not based on the scientific first principles of the phenomenon, but rather on the observed effects.

They are listed below.

- Early life cement related reactions often associated with retardation
- Admixture related effects
- Clay swelling and orientation phenomena
- Lime crystal orientation/flocculation phenomena

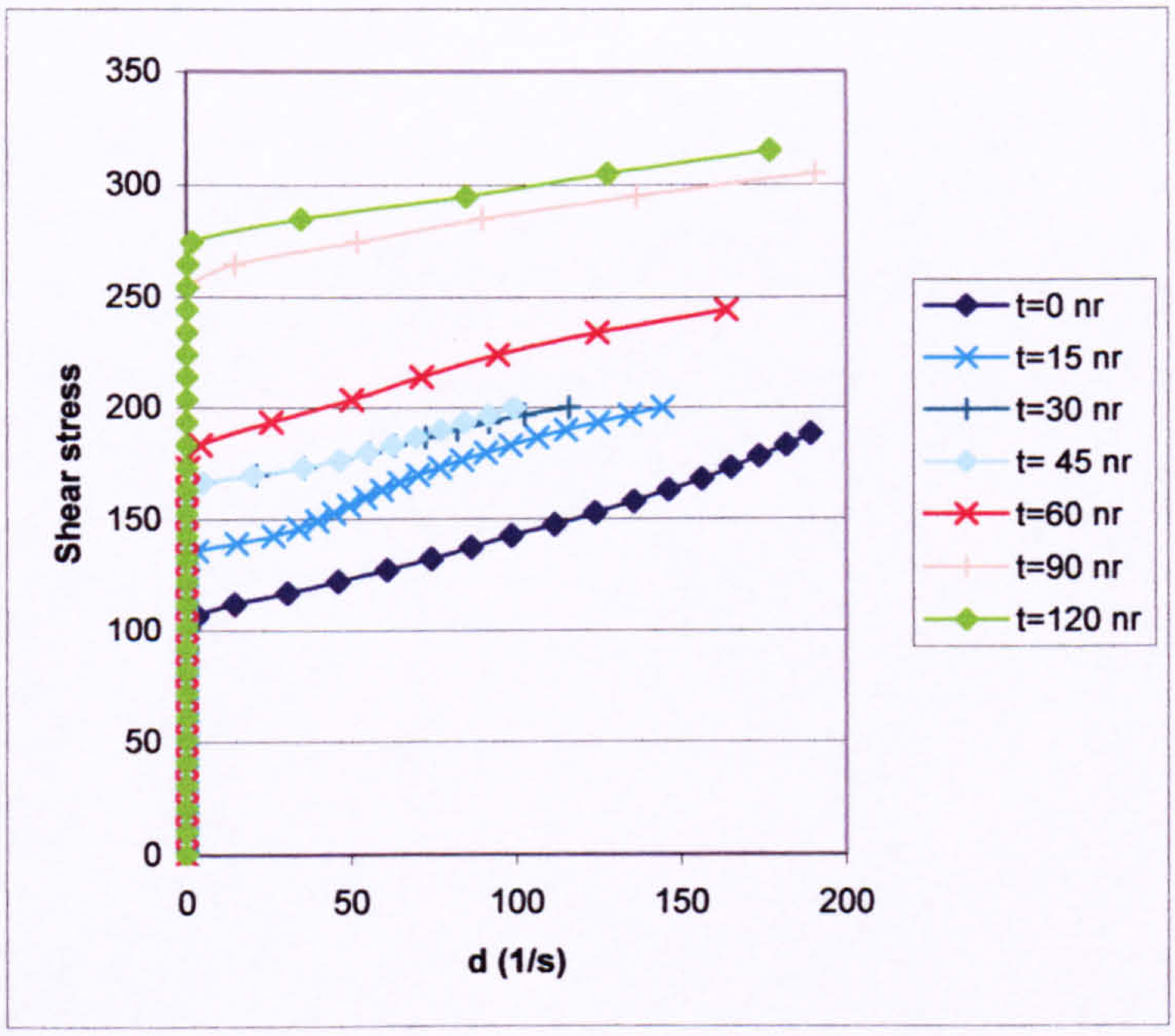
The early life effects are associated with gypsum hydration and supersaturation, with effects relating to false set sometimes producing a marked drop in plasticity within a few minutes of mixing, which can be overcome by application of energy in the form of a short period of hand or mechanical mixing

Whilst in theory it is possible for cements to contain some anhydrous gypsum, particularly where grinding has taken place in hot conditions, in practice modern grinding mills generally monitor temperature and use water injection and cooling to counter excess temperature. Other grinding and homogeneity issues can also theoretically cause some early life rheological changes but in the present programme of work one batch of standard material was used for most of the rheological work and the properties of this material were therefore both constant and known.

Nevertheless the possibility of errors occurring as a result of this issue is recognised in the BS EN 1015 series of mortar test method standards which all require as a preliminary in the mixing procedure section that "before testing, the batch shall be gently stirred by hand using a trowel or palette knife in 5-10 seconds to counteract any false setting etc., but without any additional mixing of the batch". This was adopted where appropriate throughout the work.

The initial work on time dependant/thixotropic behaviour was on the simple 1 to 3 mix that had been used as the standard in the earlier work. Not unexpectedly, this showed that yield stress increased with time, as shown in figure 9.21 below, where t is the elapsed time in minutes after mixing and the suffix “nr” indicates that there was no retarder added to the mix.

Fig 9.21 The effect of thixotropy on yield stress

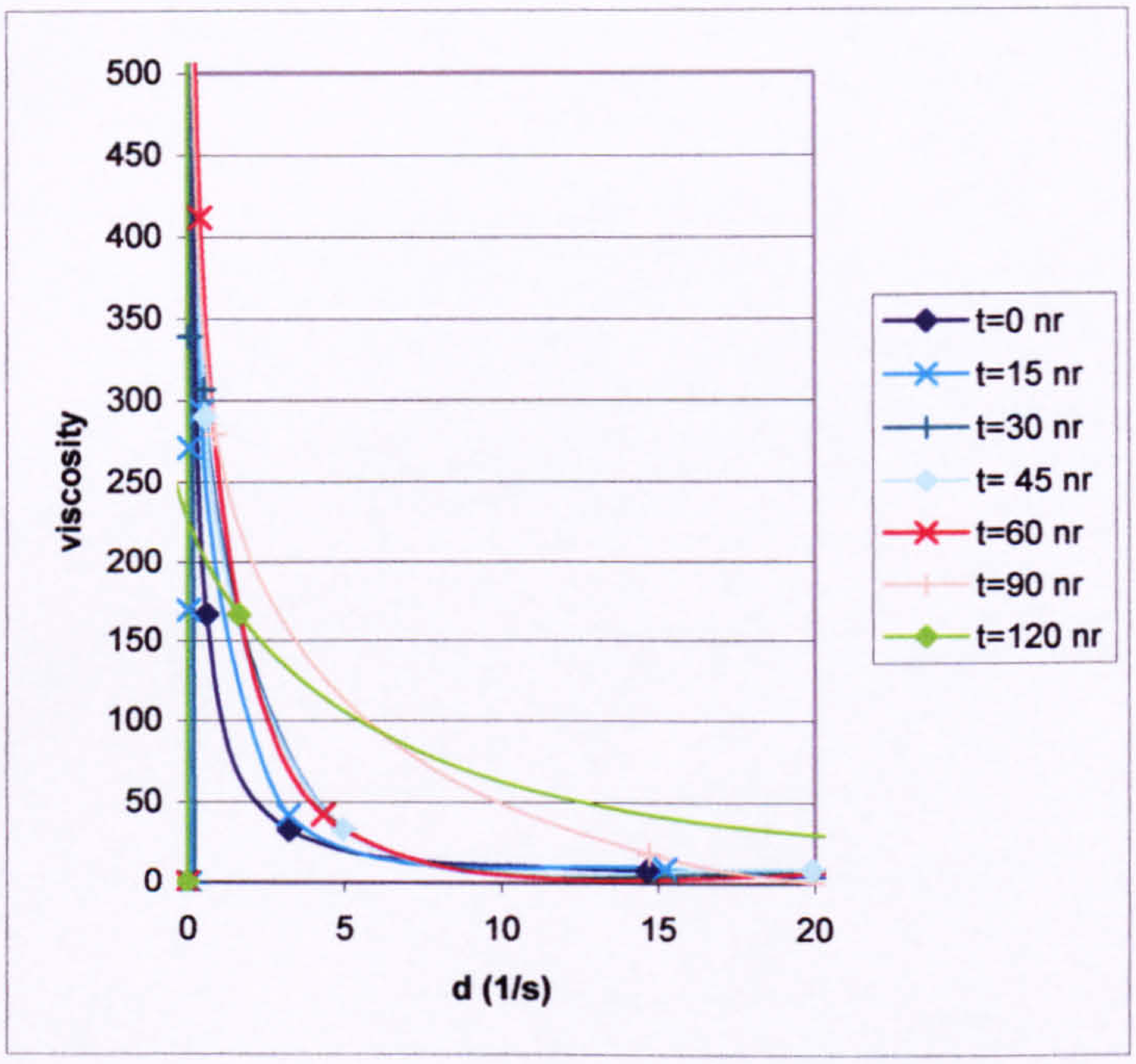


The area of great interest in this finding is in the early stage, where the yield stress is seen to increase considerably in the first 15 minutes. This has a great deal of relevance to real situations that occur on the building site, where so called stiffening in the early life of the material, before set has proceeded so far that it is unusable, is often a very real issue, Beningfield, 1997.

The finding that the rheometer was able to identify and quantify this effect should be of value in future work into this phenomenon.

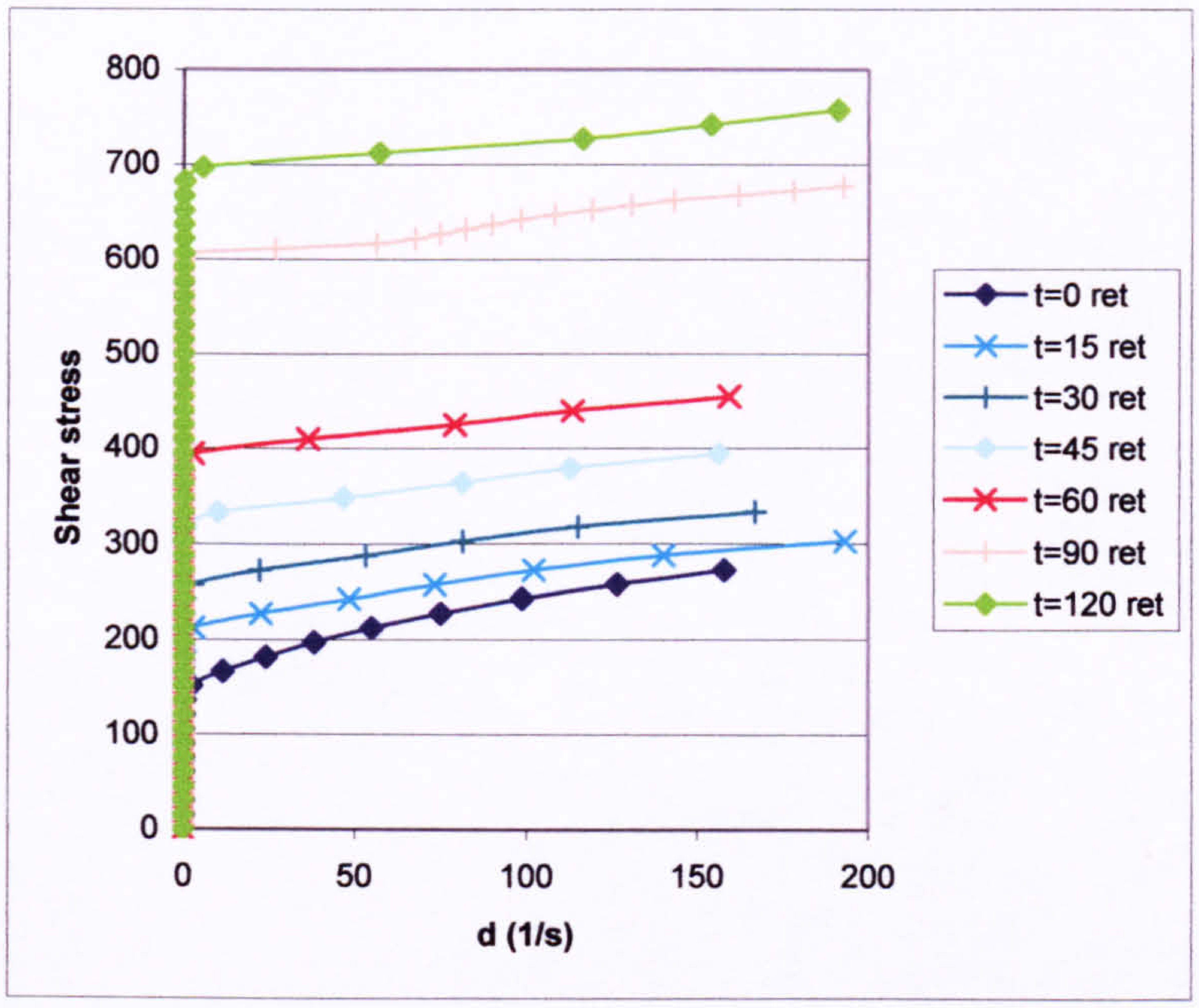
The influence of thixotropic phenomomena on the viscosity of a non-retarded mix was also determined and is shown in figure 9.22 below, from which it may be seen that this also increases with time, as the thixotropic behaviour develops.

Fig 9.22 The effect of thixotropy on viscosity



Although the findings reported in figures 9.21 and 9.22 were related to thixotropic phenomena, it is clear that there could also have been some influence of very early set or set related mechanisms. Accordingly, additional work was carried out in this area but with a retarded material, as shown in figure 9.23 below, where the value of t is the time in minutes and the suffix "ret" indicates that the mix contained retarder.

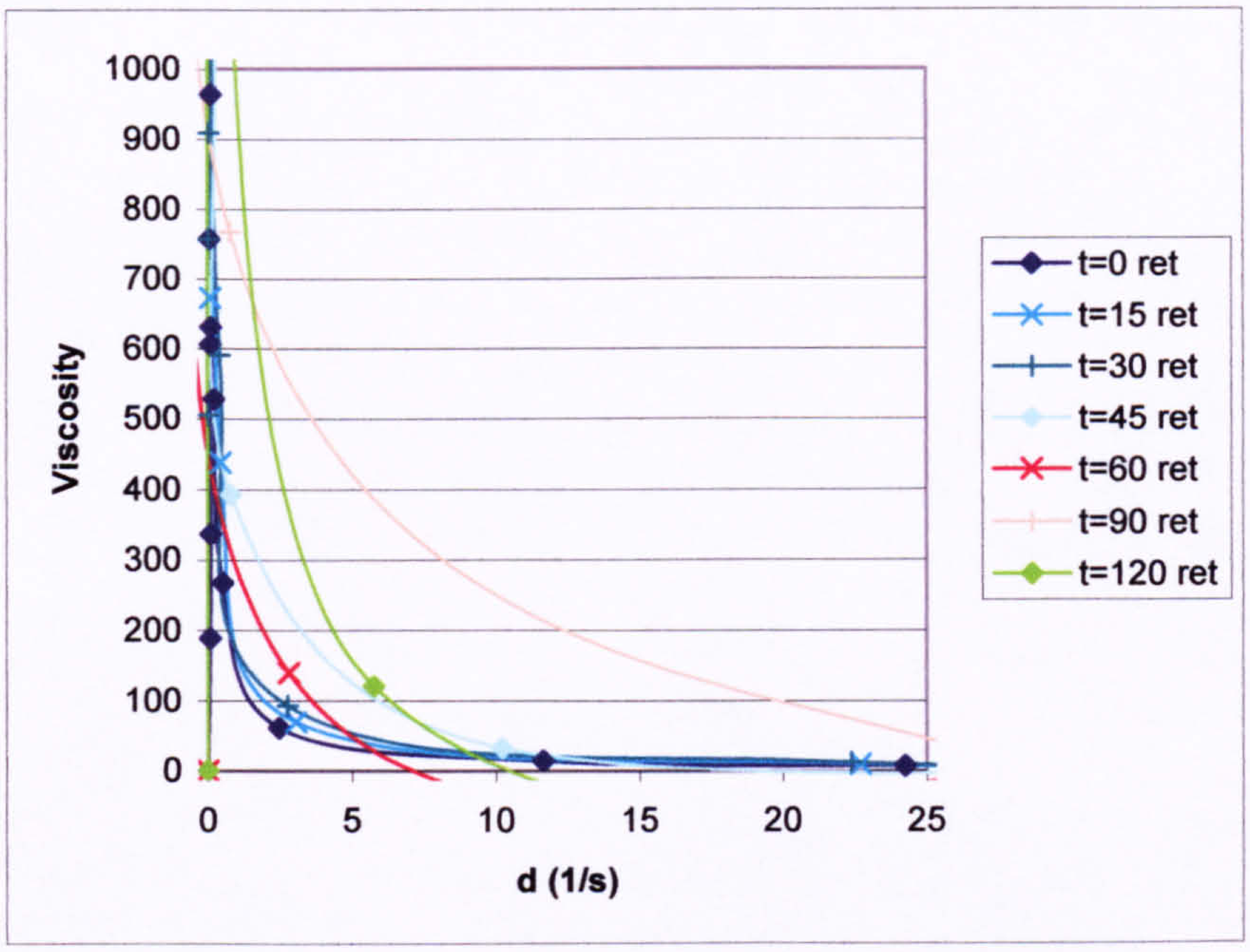
Fig 9.23 The effect of thixotropy on yield stress for a retarded mix



This work provided very interesting results. The yield value increased, and continued to increase progressively, even though the system was retarded for a considerable time such that even initial set would not take place for at least 24 hours, and the maximum elapsed time within the test programme was only 2 hours.

The data for viscosity showed a similar trend, as shown in figure 9.24 below, where again t is the time in minutes.

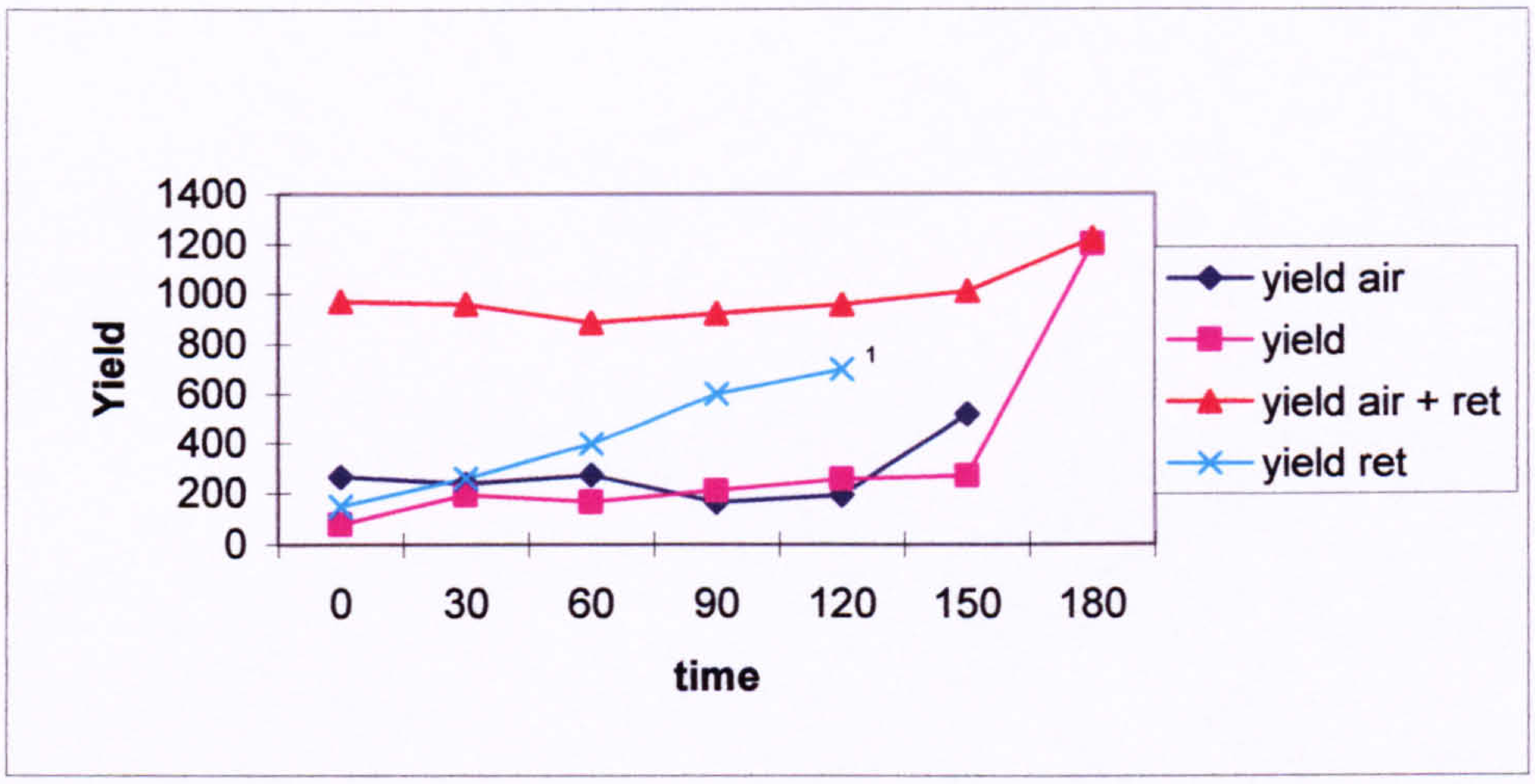
Fig 9.24 The effect of thixotropy on viscosity for a retarded mix



It was believed that this behaviour was caused by the retarder producing a secondary, rheological effect, in an analogous way to the manner in which air entraining agent was also found to act, as discussed in section 9.1.2 and as demonstrated in figures 9.4 and 9.5. The work in this section and the earlier one both show how in addition to the primary effect for which these admixtures are developed and used there are also substantial secondary effects.

This may well provide at least a partial explanation to the complex plastic behaviour of some of the most recently developed specialist factory made mortars, where problems sometimes arise in the areas of reported premature stiffening and other problematic early life behaviour. To investigate this area further, and to establish whether synergistic behaviour could be amplifying the individual effects, trials were carried out with a common admixture blend of air entraining admixture and retarder and figure 9.25 shows how this blend affected the yield stress behaviour.

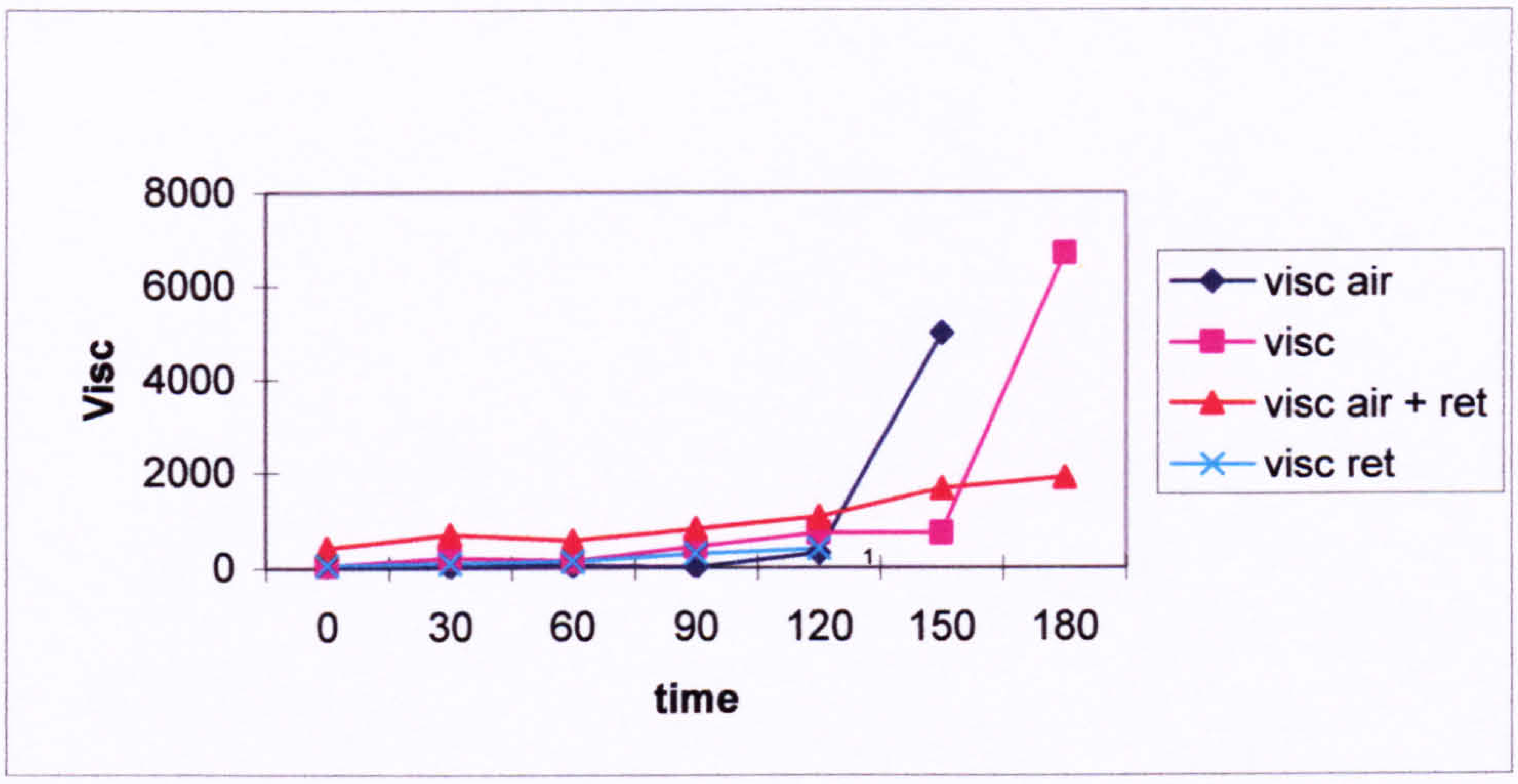
Fig 9.25 The effect of retarder and air entraining admixture on yield stress



[1] *Test discontinued at this point*

This work appeared to show that, as postulated, the effects of the two admixtures were indeed synergistic and it is thought that this may explain some of the issues that arise with multi component admixture systems in mortars. The plastic viscosity behaviour was also studied in this work and the effect is shown in figure 9.26 below. This showed a small initial viscosity increase, which confirmed the earlier findings presented in section 9.1.2, with the mix containing air as well as retarder having a higher plastic viscosity, although in the figure the effect is somewhat masked by the large increase in viscosity for the non-retarded mixes as they begin to set.

Fig 9.26 The effect of retarder and air entraining admixture on viscosity



This completed the work into the thixotropic effects associated with the two early life issues, those relating to early cement reactions and admixture phenomena, but left outstanding work on clay and lime related thixotropic behaviour. Some initial mixing work was carried out with clays but it became clear that this was a very large field of study. Not only were a number of different clay types potentially present in naturally occurring sands, but isolating these from natural sands or obtaining them as pure materials proved to be difficult. Washing, decanting and drying natural sands to concentrate the clay fraction involved large quantities of water and protracted settlement times, and the resulting clay product was then not of one type but was a mixture of clays. Other workers, Yool, 1998, have used the alternative route of obtaining pure clay minerals from specialist suppliers. Although bentonite and kaolinite are readily obtainable, problems were experienced sourcing a range of pure clays that were reasonably representative of those that occurred naturally in mortar sands and actually gave rise to practical mortar issues.

Regrettably, work on this aspect of the project could not be continued further, due to operational constraints, although it was planned to return to this area in the future.

Practical considerations unfortunately also precluded further investigations into the thixotropic behaviour of lime based mortars. As discussed in section 2.3.1, it has long been known that these mortars exhibit pronounced thixotropy and it is hoped to re-examine this issue in the future when operational issues permit.

Apart from these aspects of clay and lime interaction outlined above, the work planned in section 9.1 was considered to have completed the investigation into the effect of the most widely occurring mix variables on the plastic mortar properties, and the findings may be summarised below as follows.

- Rheological techniques can differentiate well between "good" and "bad" mortars.
- Increase in consistence results in a decrease in yield stress.
- Increase in air content increases both yield stress and viscosity.
- Increase in water content decreases yield stress and viscosity.
- Increase in sand fineness decreases yield stress and viscosity.
- Increase in lime content markedly increases yield stress and viscosity.
- Inclusion of cellulose ether increases yield stress by a small amount but markedly increases viscosity.
- The use of non-cellulosic viscosity modifiers produces very similar effects to those obtained with cellulose ether.

9.2 THE REMAINING PLASTIC PROPERTIES

After the effect of the key mortar variables had been addressed, as reported above, the work considered those plastic properties that were identified during the preliminary work that were found to be of interest but for which there were currently no acceptable/standardised test procedures, as discussed in section 9.2 below.

a) Ease of spreading

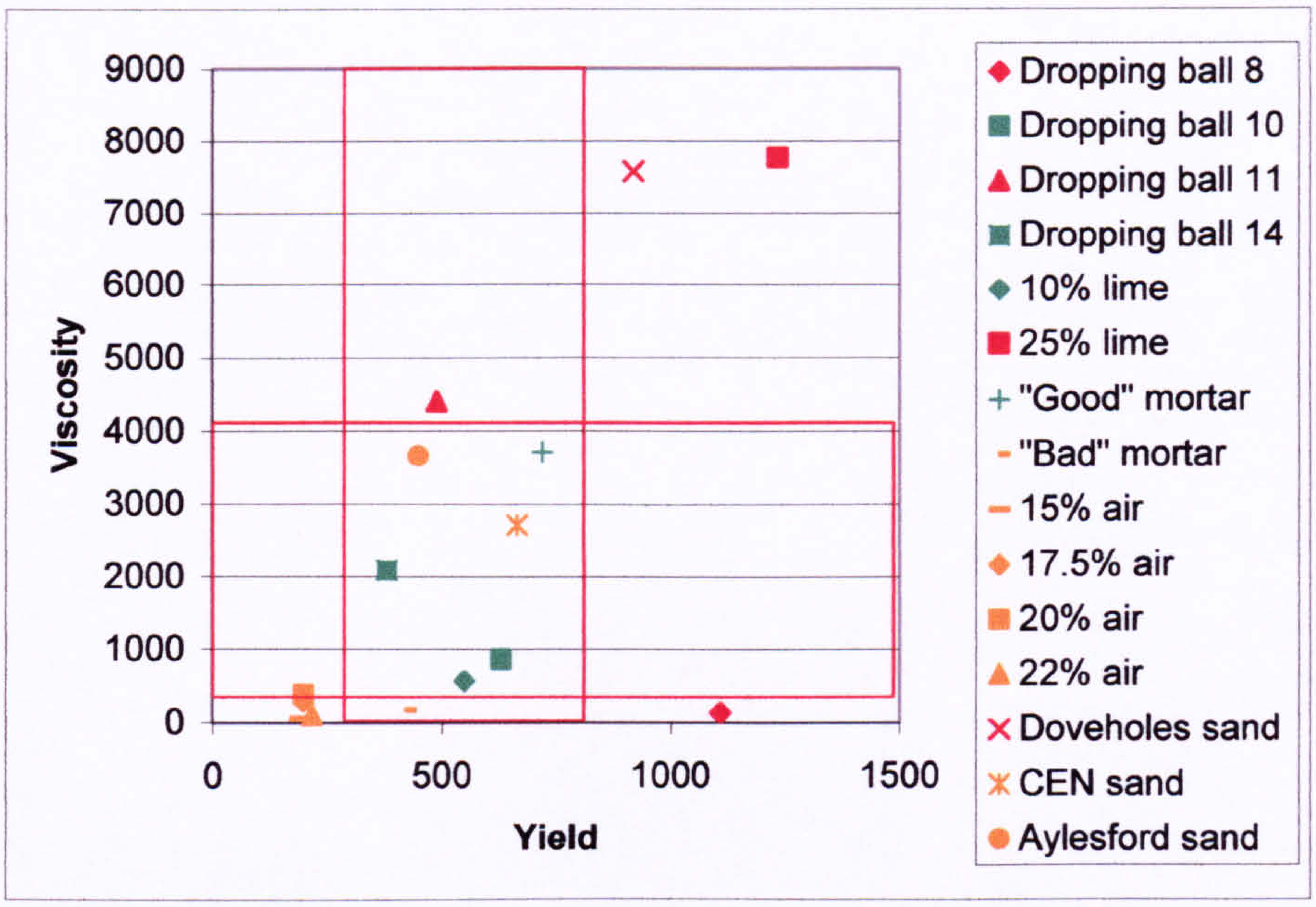
b) Deformability

c) Ease of finishing

The other property, stiffening in the mortar tub and on the spot board, was a complex multi cause phenomenon. It was partly a function of thixotropic effects, but other factors, as for example segregation, air loss and initial stiffening/setting behaviour also exerted an influence. As reported in section 3.2, these properties were of great importance. They were perceived by the operatives in practical usage but did not form the subject of any standardised test regimes and so far as could be established had so far not been quantified in the laboratory.

The research work using the rheometer had shown that yield stress and plastic viscosity were key parameters and it had appeared throughout much of the practical work that they were closely linked to ease of spreading, deformity and finishing, if not the key determinants of those secondary or tertiary properties. Excessively high yield stress produced a mortar that was more difficult to move initially, whilst excessively high viscosity produced a mortar of excessive stickiness that was difficult to spread. If the yield stress was too low, the weight of the units would not be supported without reducing the consistence, which would lead to excessive viscosity and difficulty in use. This may be presented graphically as shown in figure 9.27 below.

Fig 9.27 Suggested control limits for yield stress and viscosity



Arising from this work therefore, it is suggested that tentative limits may be placed on both parameters, yield stress and viscosity, and the following statements are proposed.

- As yield stress exceeds about 750 the mortar becomes difficult to move initially
- As it falls below 300 the mortar is unlikely to support the weight of an applied unit without unacceptable deformation
- A viscosity range of between 500 and 4000 appears satisfactory. Above that the mortar is too sticky, below it insufficiently viscous, which will tend to segregation and the so called "lack of body" identified during the practical appraisals carried out in section 3.2.

Following on from the work reported in section 9.1 above on the effect of primary variables, a far more empirical approach was investigated and this is discussed in section 9.3 below.

9.3 PRACTICAL APPLICATIONS OF RHEOMETRY TO MORTAR TECHNOLOGY

The work reported above in sections 9.1 and 9.2 clearly showed the value of rheological comparison and confirmed the ability of the procedure to discriminate between mortars where relatively minor compositional changes had been made. Because the values produced are absolute, they should enable comparisons to be made of work in different laboratories nationally or indeed worldwide.

9.3.1 Using rheological profile comparison

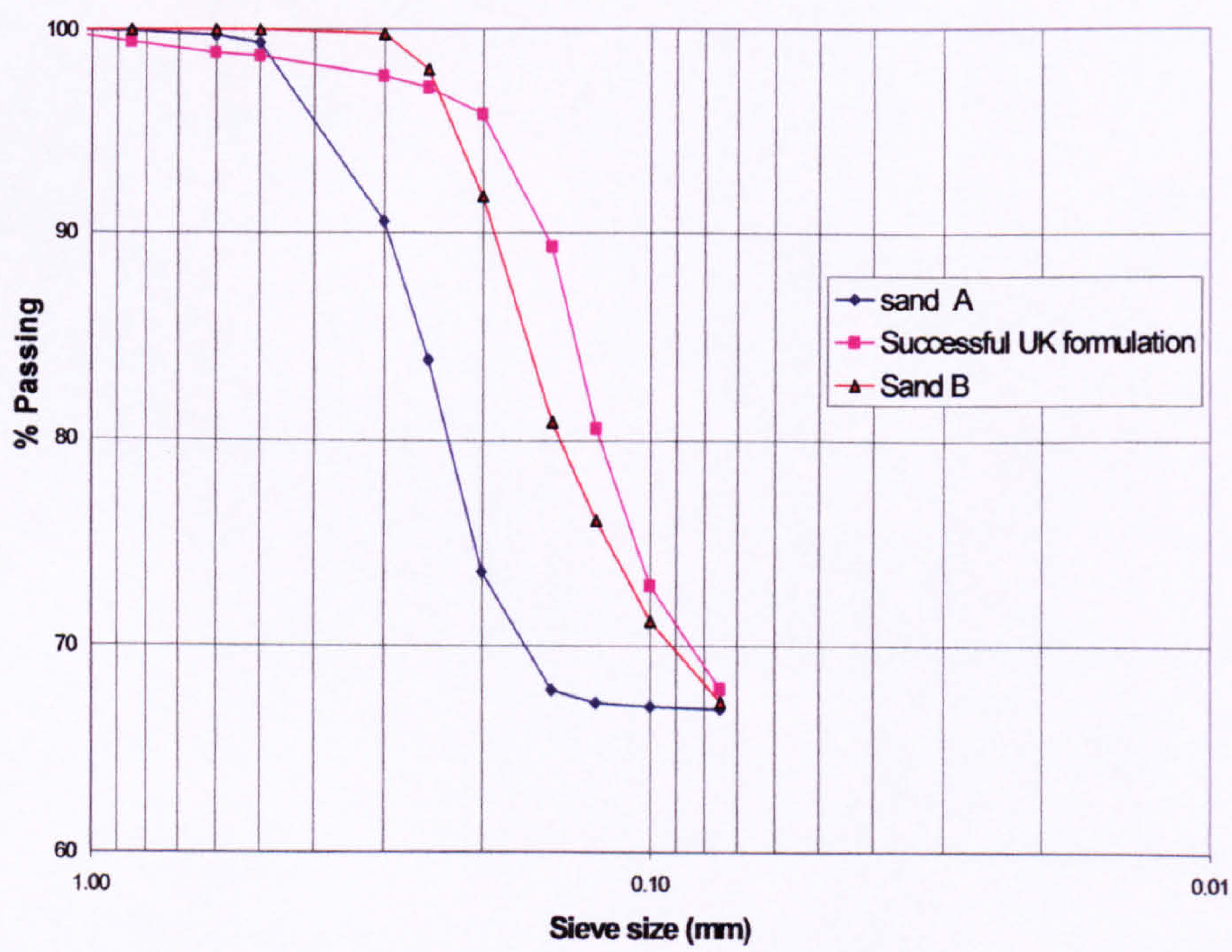
As may be seen from the comparative work, rheological techniques may be used to compare numerical values for key parameters, in particular yield stress and viscosity. This is appropriate in many cases but in some practical instances, particularly with complex systems, it is probably more meaningful, and certainly more practical, to superimpose the graphical profiles of two or more materials. This will often facilitate a simple visual analysis, although clearly not in accurate quantitative terms.

To investigate this technique, the procedure was used to attempt a comparison of alternative formulations of a sophisticated calcium sulphate based flow applied thin layer levelling screed. This class of materials possess complex rheological properties, partly as a function of their multi component constituents, which in this case included gypsum, lime, fine limestone filler, air entraining plasticisers, cellulose ethers, and acrylic viscosity modifiers.

In this work, an attempt was made to duplicate the properties of an existing and successful floor screed, using a proposed sand grading A, as a material that was said to be suitable, but which was dissimilar to the successful screed. In addition, a sand B was sourced that possessed a very similar grading to the successful screed. The grading of three trial screed formulations made using these alternatives is shown in figure 9.28 below.

After the three formulations were made up they were tested on the rheometer using a comparative technique.

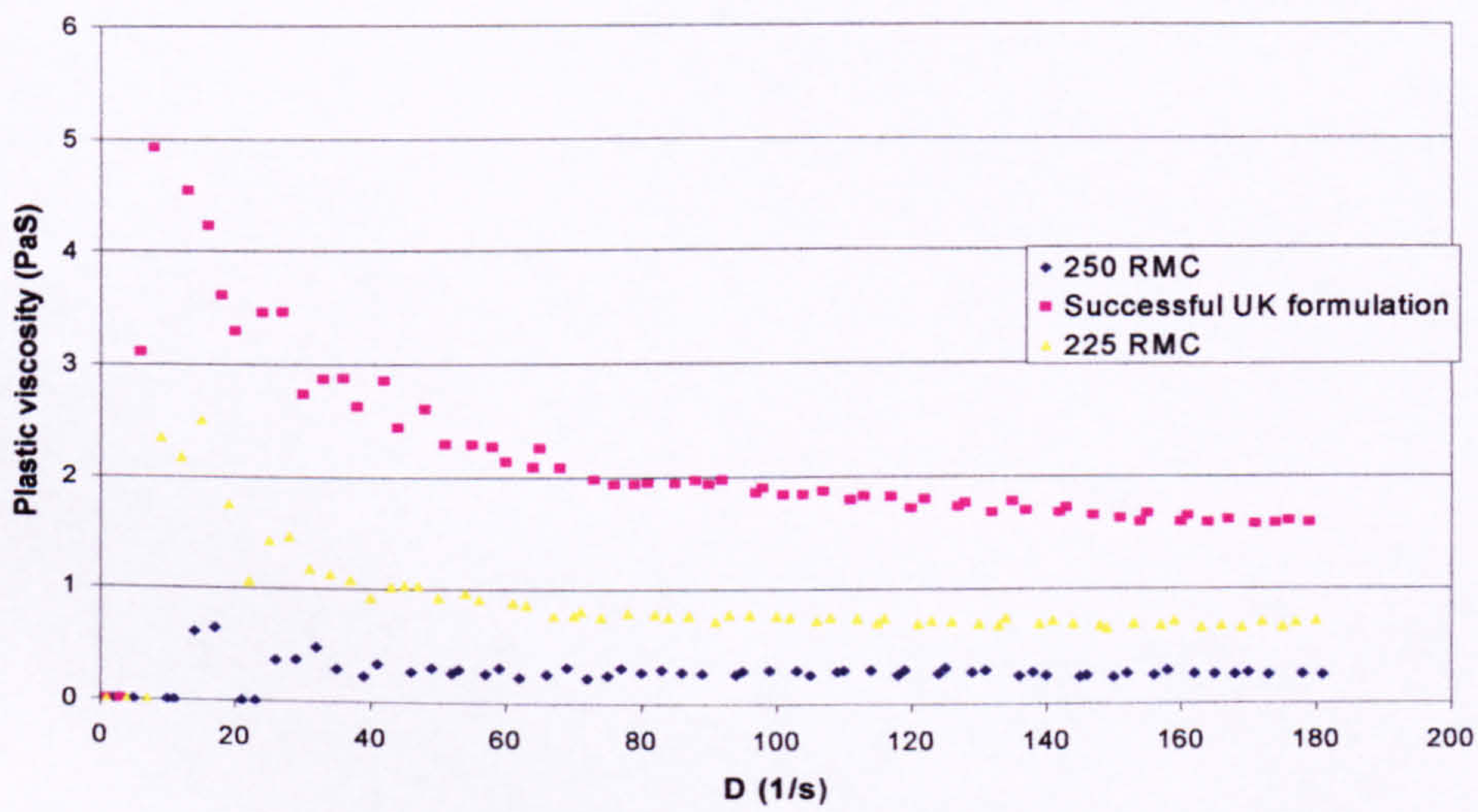
Fig 9.28 Comparison of the three sand gradings used in the thin layer screeds



It can be seen from the graph that the successful formulation had a high initial viscosity that then decayed rapidly, showing its thixotropic nature. The formulation made using the first sand to be recommended did not possess this type of behaviour, its low initial viscosity and constant time/viscosity profile showed it to lack this property. When reformulated to use the second sand, however, the profile exhibited was seen to be very similar to the successful material. Further practical work and operative appraisal resulted in the second trial formulation being adopted. This work was carried out at RMC by Fowler.

It is seen, therefore, that comparison of rheological profiles, as well as the use of absolute values, is of value.

Fig 9.29 Variations in plastic viscosity for calcium sulphate thin screed formulations



This work concluded the rheology test programme. The work had shown that absolute rheological values could be used to facilitate the classification and specification of the plastic properties of mortars and that comparative profiles could be of value in product development and comparison. As suggested in figure 9.27, it is believed that acceptable maximum and minimum values may be selected for yield stress and viscosity, between which mortars of satisfactory properties are found, and outside which problems of acceptability are likely.

It was also believed that the rheological parameters worked with related to some of the mortar "finishability" and "spreadability" issues identified in the initial practical trials and reported on in section 3.2.

All of these concepts are developed and discussed in more detail in the overall conclusions to the work which are section 13 of the thesis.

Following the finalisation of this work on the early life plastic properties, work then began on the second phase of the overall project, the development of a new test for the stiffening and early setting behaviour of mortars.

10 THE DEVELOPMENT OF A NEW TEST METHOD FOR STIFFENING AND EARLY AGE STRENGTH DEVELOPMENT

As discussed in the preceding chapters, the existing tests for the above characteristics that were in common usage all relied on mechanical penetration and are thus all affected by the plastic mix properties as well as the strength development that they should really be measuring. A further problem associated with the existing test procedures had gradually become more and more pronounced as retarded mortars were developed. This revolved around the simple logistical issue of the staffing and laboratory requirements needed to monitor the setting characteristics of retarded ready to use mortars. As the retardation times of these materials progressively increased in line with market demand, from the initially ubiquitous 24 or 36 hours to 48, then 72 and occasionally even longer, (where mortars were made on a Friday for use on the following Monday), test mixes could only be made on Mondays and Tuesdays of the week. Any mixes made later in time would result in setting taking place at the weekends when staff were not present.

Additionally, any slightly anomalous setting time behaviour even on the part of mortars produced in just these those first two days of the week would sometimes give rise to the need for staff to be present during anti social hours. Thought was thus given to the possibility of devising a non-intrusive test that would allow a measurement to be taken without the need for any mechanical disturbance and that would have the additional benefit of using remote measurement or logging in the absence of staff.

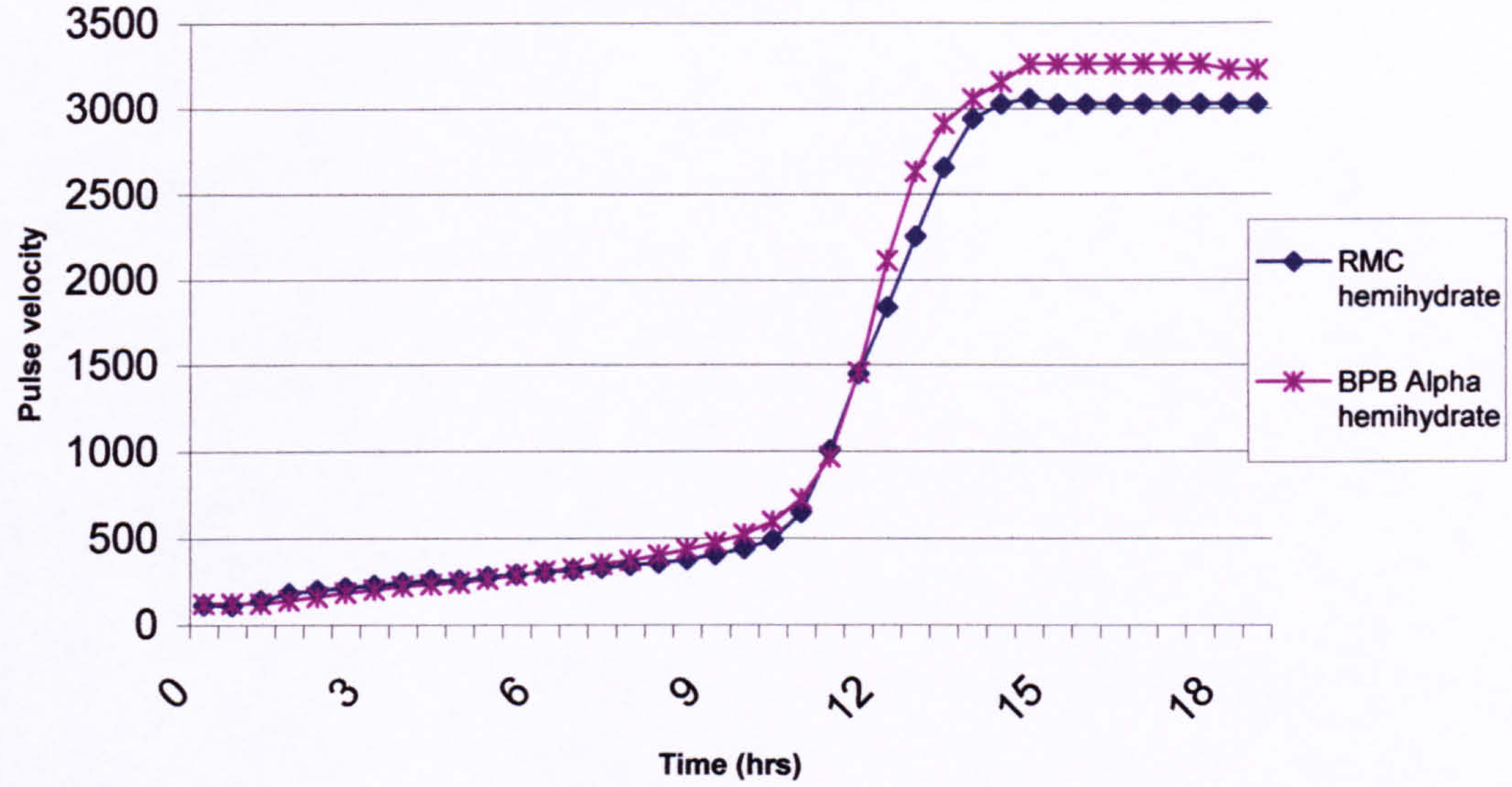
A number of possibilities were considered, including use of some of the tests used in concrete and commonly referred to as non destructive, together with some already in use for the later life testing of mortars.

The tests discussed in the literature survey in chapter 2, and appraised in chapter 6, were all dismissed for various reasons. Many were influenced by the mechanical properties of the mortar mix, a function of consistence, cohesion, segregation and interlock and sand characteristics. Others, based on pure non intrusive procedures like ultrasonics and gamma radiography were complex and expensive and also often complicated by surface/interface effects associated with the vessel containing the plastic mortar. Impulse response and impact echo testing had been used for concretes, but were heavily influenced by the presence of cracks, as reported by Pearson, 2003.

None of these methods were deemed worthy of further investigation, with the possible exception of ultrasonic pulse velocity, which had been investigated in mortars at early ages, Casson et al, 1982. This work did show that upv changed from an early time in the setting process, because the pulses are transmitted at a higher velocity as more solids form and there is less free liquid to attenuate the waves, so the procedure thus showed some potential.

Upv is sometimes used in the field of plasters and in this application is able to differentiate between two slightly different calcium sulfate binders, of the type used in plasters and also in some floor screeding mortars, as shown in figure 10.1, which is reproduced by kind permission of BPB Formula Ltd of Newark who carried out this test. In the figure, a gypsum hemihydrate floor screeding binder in successful usage by RMC is shown contrasted with a proposed new formulation based on the alpha allotropic form, and developed by BPB Ltd, with the aim of matching as closely as possible the setting and early strength development of the existing material.

Fig 10.1 Ultrasonic pulse velocity plotted against time for two different calcium sulfate cements



As may be seen from the figure, this technique appeared to enable a good comparison to be made between these two materials. Unfortunately, however, the presence of air voids influences the results of the procedure. With flow applied floors screeds, which do not contain intentionally entrained air and because they are placed at very high consistence values contain virtually no entrapped air, the procedure may have potential. However, for the vast majority of mortars entrained and entrapped air are present in substantial amounts. Moreover, this air exhibits some time dependant instability so the use of the procedure would not be wise.

Consideration was then given to whether or not any simple chemical processes that gave way to or were associated with early life stiffening and strength development could be monitored non intrusively. Physico chemical properties like change in weight, colour and density were all considered but were readily seen not to be applicable.

Measurement of electrical properties was initially considered, in the same way that capacitance or dielectric properties may be used to construct a calibration graph that enables moisture content to be read. A brief consideration lead to this being rejected but that is not to say that the concept would not work. It does not appear to have been reported in the literature as a potential procedure.

The exothermic nature of these early setting reactions was then considered. It was known that in this respect they could perhaps be broadly related to the exothermic reactions associated with another common building material, lime, when that hydrated, although clearly the hydration of cement is much slower, far more complex and indeed dissimilar in many respects. However, the sponsoring company's laboratories had tested the early life hydration characteristics of lime by measuring the exotherm produced when water was added to lime in a simple vessel. Because the lime tended to form lumps during this process, partly a function of the powder not wetting out completely, it was necessary to use a stirrer, but this would not be needed for work on mortar as the system is already wetted out and of course generally contains surfactants, a further help in this respect.

The progress of many different types of chemical reaction has been studied by measuring the heat involved, using various types of calorimeter, and these techniques are basic and have been known for many decades. In the field of construction materials and cement hydrates, the analytical technique of differential thermal analysis (dta) relies on the exotherms to characterise and measure many compounds. The use of heat measurement to investigate the properties of cements is also reasonably well established, although generally not long enough or well enough to have yet become widely standardised. The Bremortar test, developed by Southern, 1989, utilises heat of hydration to estimate the mix proportions.

The earliest reports of the use of a crude form of calorimetry in conjunction with cements date back many years, Carlson, 1937 and Carlson et al, 1938, but these were more in relation to differential thermal analysis and chemical characterisation than to the properties of the final mortar or concrete.

These calorimeters used traditional isothermal principles, where the heat flows from the material that is evolving the exotherm and the temperature is maintained as a steady state, with the heat flow being used to generate an e.m.f that is then measured. Lerch, 1946, used the technique to study the very early setting reactions that involve the gypsum and the tricalcium aluminate (C_3A). He reported that there appeared to be three distinct exothermic peaks. The first occurred within the first minutes and involved the initial gypsum hydration/reaction, the second took place within the first few hours and the third at approximately 12 hours as the gypsum became completely depleted, all of these times being for simple unretarded cement pastes. Lerch termed the first peak the heat of immediate hydration and considered the later two to represent the heat of hydration itself.

The literature, Neville, 2000, refers to the heats evolved in the four major exothermic reactions as follows:

$2C_3S + 6H \rightarrow C_3S_2H_6 + 3Ca(OH)_2$ + 502 J/g

$2C_2S + 4H \rightarrow C_3S_2H_6 + Ca(OH)_2$ + 260 J/g

$C_3A + 6H \rightarrow C_3AH_6$ + 867 J/g

$C_4AF + 7H \rightarrow C_3AH_6 + CFH \quad + 419J/g$

The rate at which these reactions proceed, and the time that each takes, varies widely. The first two shown, those involving the C_2S and the C_3S are slow, the former particularly slow so that only perhaps 10% has reacted in the first 10 days, whilst with the C_3S perhaps 50% has reacted in that time. These reactions are not of immediate relevance to the proposed technique and it is the remaining two that produce the early exotherms that are of interest in the context of the proposed test, with 80 or 90% of these reactions being completed within the first day, and a substantial amount within a few hours. This means that the work reported on exothermic methods is actually concerned with just these two reactions, of the four major ones that comprise hydration. However, that was not seen as a disadvantage as it is only the very early behaviour that is of interest, as opposed to the longer term strength development

Other workers followed Lerch in the use of isothermal techniques to investigate cement chemistry. Thus Copeland et al, 1960, Stein, 1961, (two references), Stein, 1963, 1965, Verbeck, 1965, Kondo et al, 1969 used the technique to study cement hydration and there is recently published work, Roberts, 2005, which used a sophisticated high resolution isothermal calorimeter to study gypsum/clinker interactions. Some later work relates to admixtures, Previte, 1971, Kantro, 1975 and Kondo et al, 1977, Wilding et al, 1984 and Vernet et al, 1992. A very comprehensive ASTM bibliography, 1996, refers to these and to further general references in the field. The work reported above was generally on cement pastes and all used isothermal calorimetry with steady state heat flow, which tends to utilise expensive equipment, with prices of perhaps £40,000 for some types of apparatus.

As an alternative to studying the exotherm using steady state isothermal conditions, adiabatic methods may theoretically be used, where all of the heat of reaction is maintained in the sample, by using optimal insulation and measuring the temperature rise. Unfortunately, it is not practically possible to ensure that there is no heat loss whatsoever, even if extremes of insulation are used, so in practice all that can be achieved is the partial condition, which is known as the semi-adiabatic state.

The use of semi-adiabatic methods does mean that the precise kinetics cannot be studied, but these are of little interest in the majority of practical, operationally orientated research, so in reality this reservation is only relevant where detailed study of the chemistry and kinetics of the reactions are required. This was not the case with this proposed research, where the ease and relative cheapness of the semi-adiabatic technique presented a great advantage.

There appears to be only a small amount of published work in this field. There is the early work by Carlson et al, 1938, referred to above and work by McCoy, 1963, Gragg et al, 1972 and Costa, 1979. Later work is scant although there was research by Malek, 1992. In general, these workers used single specimens, although Gragg et al used a multi sample apparatus.

Although it was also known that cement set retarders had been studied by examining their effect on the heat of hydration, much of this work is commercially sensitive, having been carried out by an admixture manufacturer, and had therefore not been reported on, nor could it be quoted. Casson et al, 1982, had carried out some preliminary work with calorimetry in conjunction with other procedures like upv and penetration and it appeared that promising relationships existed. Some work on retarders has been published, Egan, 1988, that compared isothermal and semi-adiabatic procedures and used a very simple form of the latter, based on a vacuum flask containing a single sample.

Livesey et al, 1991, compared the different techniques and tested the Langavent method recently adopted in a French standard, AFNOR, 1988, and in Spanish standard UNE 80 118, 1986. He concluded that simple semi-adiabatic techniques were promising although his research findings were primarily orientated towards the application of the method to predict temperature rise in mass concrete structures.

The only relatively recent work appeared to be that by Sanchez de Rojas et al, 1993, which used the Langavent method to investigate pozzolans and appeared to do so quite successfully.

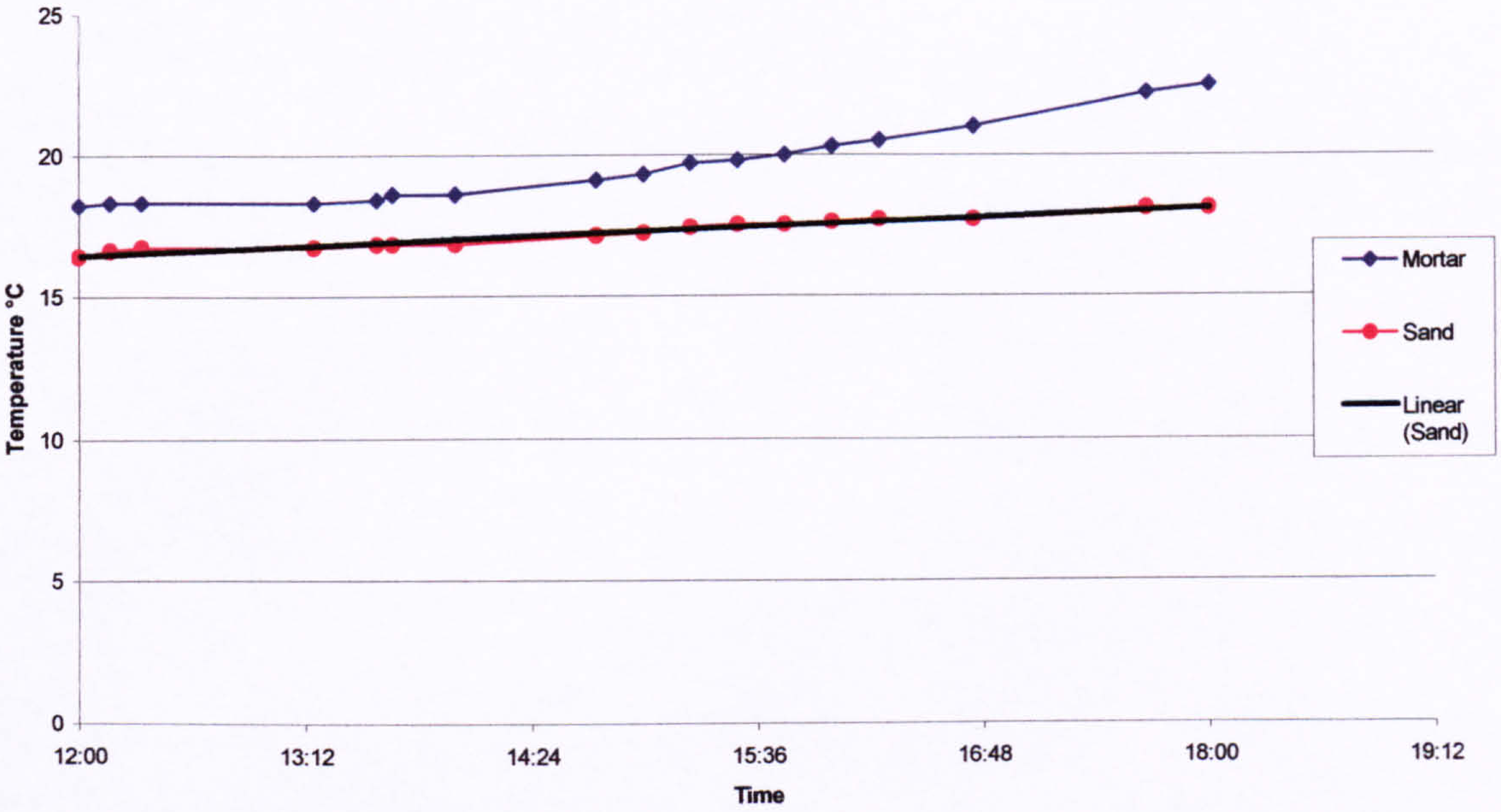
Although the technique was clearly still in its infancy, and when used was generally in the isothermal form with the resulting expensive apparatus, it was decided to investigate the feasibility of monitoring the early life exotherm and developing a simple but multi specimen method using a basic semi-adiabatic method, as discussed below in section 11.

11 DEVELOPMENT OF THE HEAT OF HYDRATION TEST

Initial work on the new procedure was carried out using a 1:6 mix, with the standardised sand used in the earlier work and the standardised reference cement, using a beaker and a thermometer, but this proved inadequate because although a minor temperature increase could be observed if great care was taken, this was quickly dissipated as heat was lost to atmosphere. The procedure would clearly also be badly affected by drafts cooling the outside of the beaker. An additional issue was associated with the small sample size of 400 grams, which although theoretically adequate for the purpose of measuring the temperature in practice evolved insufficient heat to allow accurate measurement to be made with the equipment available.

The work was then repeated with a plastic cup being used instead of the glass beaker. This had a larger capacity and better thermal properties. The amount of heat evolved was also maximised by substantially increasing the cement content by the use of a 1:3 mix in place of the original 1:6. This provided a mix of much better working properties, and also in a practical sense integrated with the preliminary findings in section 3.1.2, that a 1:6 gave problems associated with segregation and bleeding, but a 1:3 was generally acceptable in this respect. The richer mix, larger sample size and better insulated test vessel yielded sufficient data to produce a graph which showed a clear point at which the heat produced that was evolved by the exothermic reaction resulted in the start of a temperature increase. This first graph is shown as figure 11.1, which also shows the trace for the feed of a second probe into the data logger. This probe was in a similar vessel that was filled with sand that was adjacent to the first sample and acted as a blank as the air conditioning in the laboratory was malfunctioning, resulting in an unplanned change to the ambient temperature that had the potential to distort the results.

Fig 11.1 Heat of hydration plotted against time, using a simple sample container



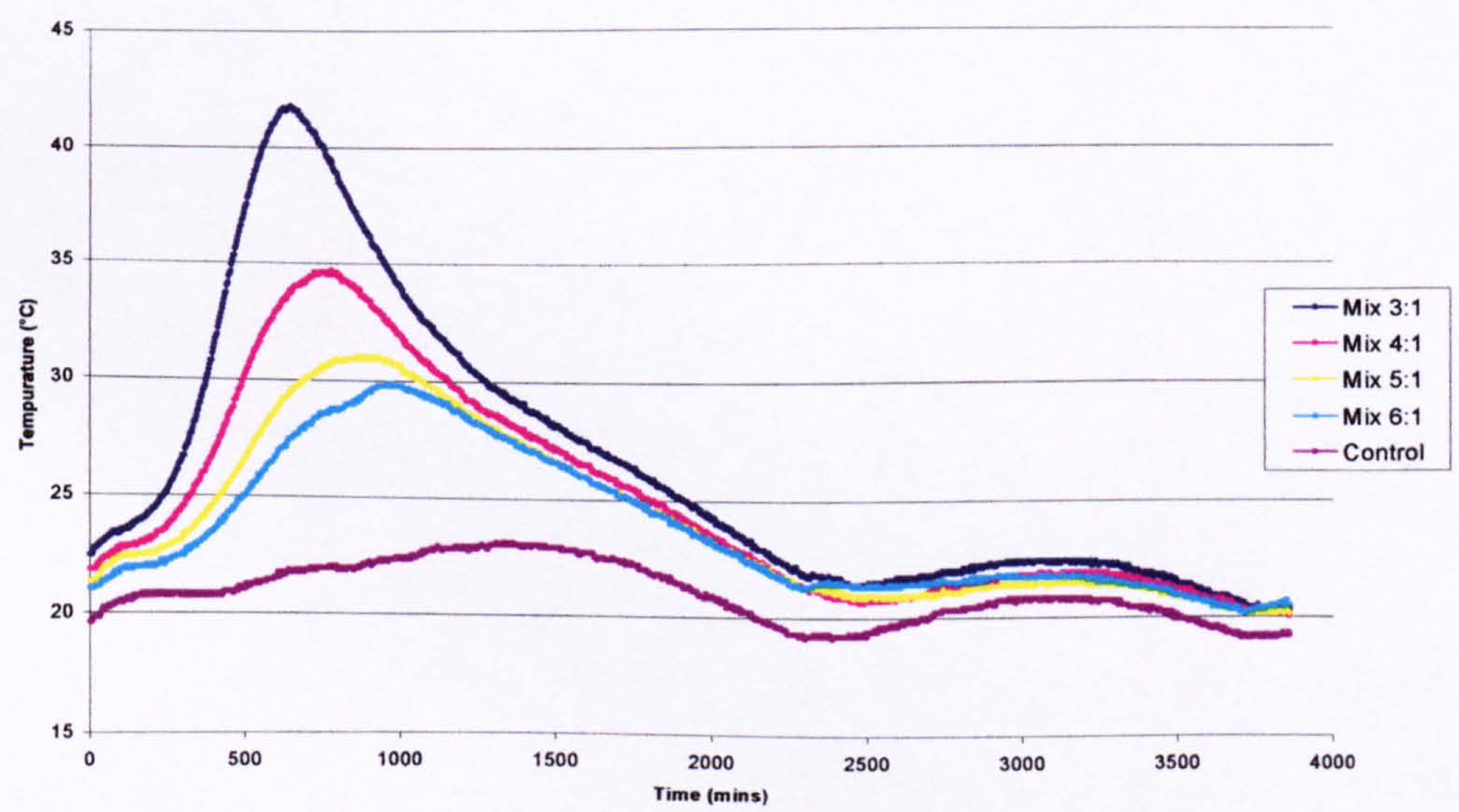
Although as can be seen from figure 11.1, this gave rise to a measurable temperature rise, the magnitude was small leading to a concomitant lack of accuracy. A thermos flask was then used as the vessel in which to place the mortar test sample and this proved much more successful but unfortunately each test resulted in the sacrifice of a flask, as the material set therein unless monitored with great care and washed out immediately the test finished. Fragility was also an issue, as was the improbability of achieving reproducible thermal performance from successive deliveries of flasks, so this option was therefore ruled out as a future routine test.

At this stage consideration was given to placing the complete apparatus in a water bath to minimise temperature variation, as was done by Gragg et al, 1972 with his proposed equipment. However, because the objective was to develop a simple and uncomplicated piece of equipment the air conditioning fault was rectified and the research proceeded with the aim of attempting to achieve sufficient accuracy using this resource, rather than the more complex system enclosed in a very closely temperature controlled environment.

Following from the work shown in figure 11.1, a further enhancement was then made whereby an insulated expanded polystyrene container replaced the plastic cup/thermos container. This also permitted a larger size to be used.

This was more successful and produced a reasonably defined point where the setting/stiffening appeared to start taking place, as shown in figure 11.2 below, which also used a small number of alternative aggregate cement ratios.

Fig 11.2 Heat of hydration plotted against time with an insulated sample container



These trials were carried out with a relatively unsophisticated Comark data logger. This suffered from the inadequacy that it produced output on a small paper roll and that output then had to be manually entered into a computer. Additionally, the hardware and printout mechanism were vulnerable to mechanical defect. To overcome these issues, a further data logger, a Grant squirrel meter/logger type 1025 was acquired that fed directly into the computer. Both of these loggers are shown in figure 11.3 below.

Figures 11.3 the data loggers used for the heat of hydration work

The work described above was carried out at the usual research laboratories but at a smaller regional laboratory work was also carried out using an old multi point chart recorder with 6 different pens, each of which could be manually filled with coloured ink to produce up to six coloured traces, thus potentially enabling a number of samples to be tested simultaneously. The sample size was also upgraded at this laboratory, to 1 kilo, using sample vessels improvised from plastic drink bottles cut horizontally about 80mm up from the base. A further upgrade was then made in the form of a purpose made piece of equipment which enabled all of the 6 probes to be utilised, which meant that 5 samples could be tested simultaneously, with the sixth probe being used to measure the ambient temperature and thus provide a zero for comparison, to enable the exact start of the heat evolution that it was desired to measure to be identified. The equipment was further upgraded to improve the insulation, thus amplifying the heat evolved to aid its measurement.

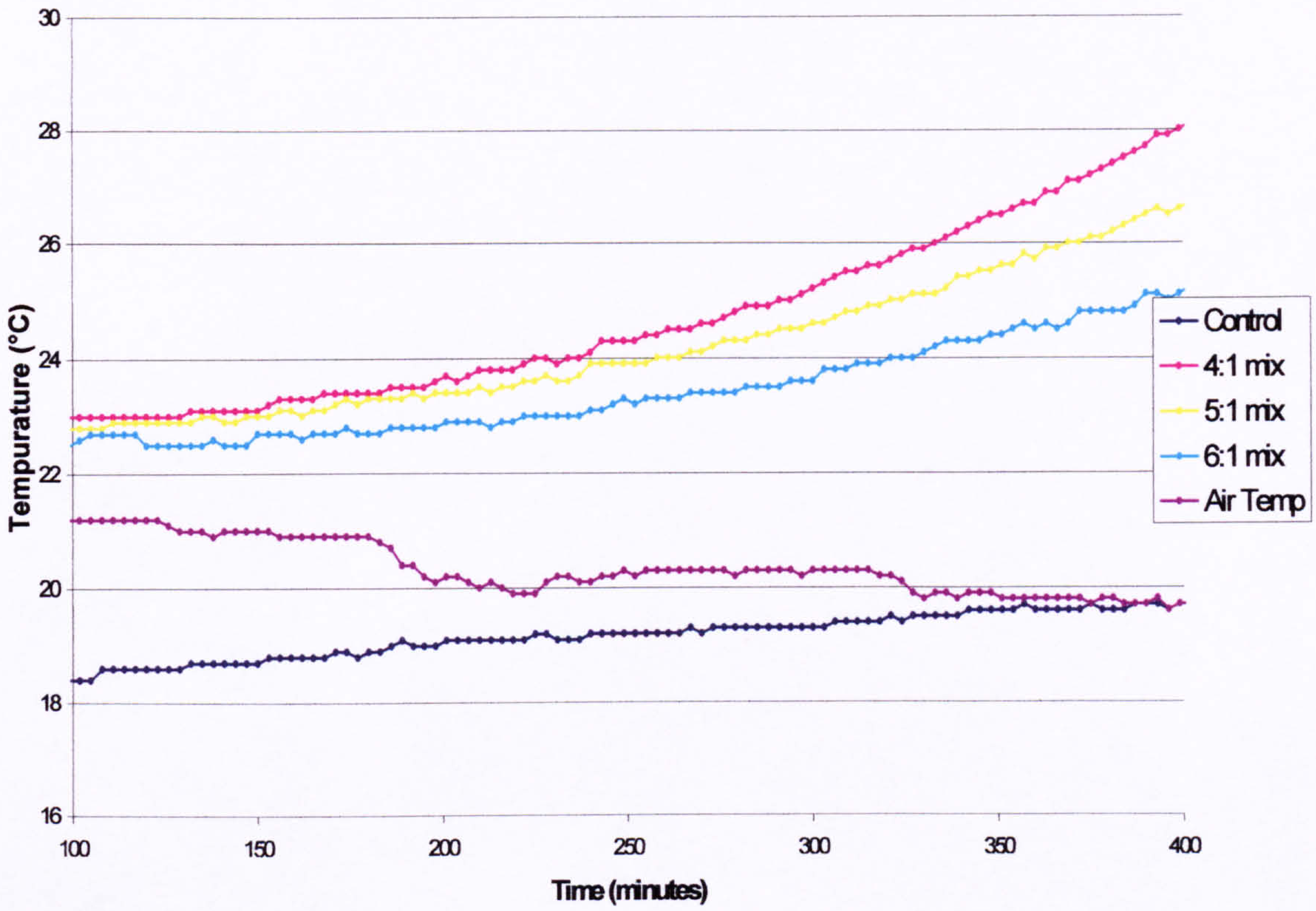
This equipment did produce usable results, and was used for some time as a very basic research tool, but unfortunately the early apparatus suffered from a number of inadequacies. The insulation was basic expanded polystyrene and was fragile, abrading and crumbling as the samples were taken in and out and the apparatus was thus completely redesigned, with some other improvements also being made. PT 100 platinum resistance temperature probes with an accuracy of ± 0.2°C were used in place of thermocouples which were stated to be accurate to only ± 0.5°C and the chart recorder was dispensed with to be replaced by the improved data logger.

A completely new piece of equipment was then built, using far more robust components. The internal insulation was much more rigid closed cell material and the apparatus had provision for the simultaneous testing of 10 specimens. This equipment is shown in figure 11.4 below

Figure 11.4 The heat of hydration equipment

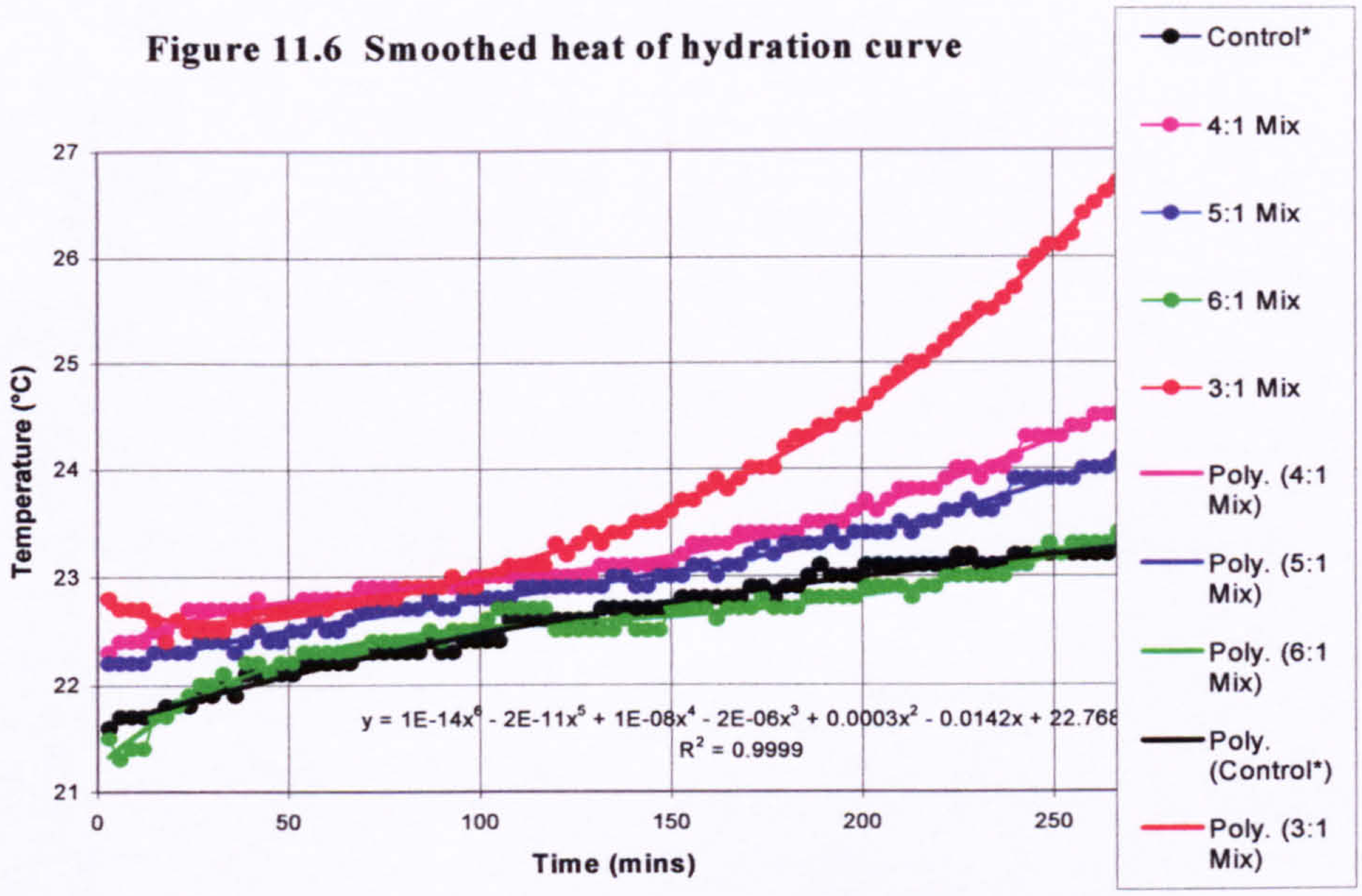
Unfortunately, the graph derived from the temperature trace was of a rather erratic, slightly jagged form, as shown in figure 11.5 below.

Fig 11.5 Early undamped trials with the new equipment



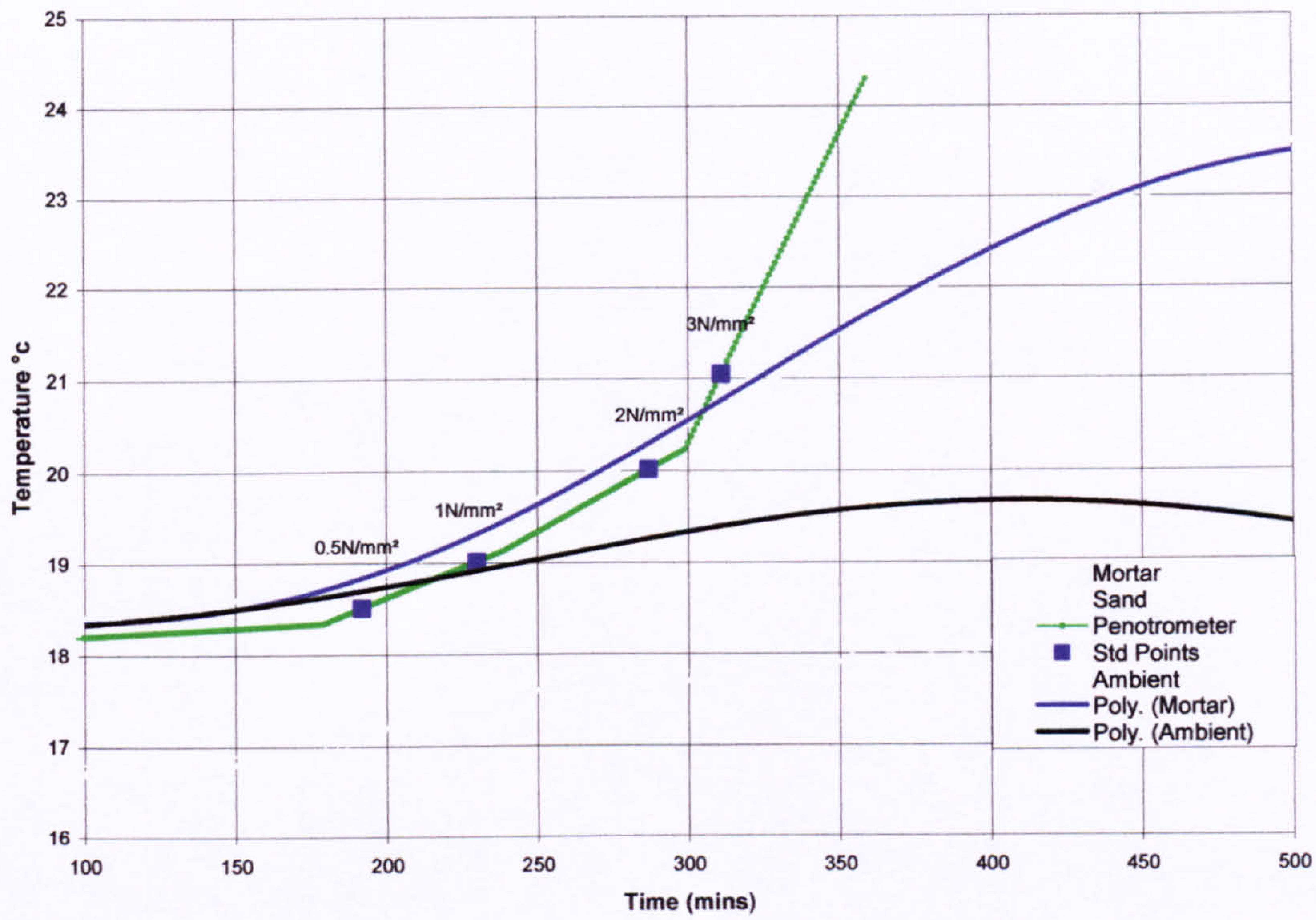
It was then smoothed a little, whilst still retaining its precise form, by using the computer to calculate the equation of the line, and a relationship in the form of a sixth order polynomial, was deemed to represent the situation. The form of the graph thus produced is shown below in figure 11.6, for different aggregate cement ratios.

Figure 11.6 Smoothed heat of hydration curve



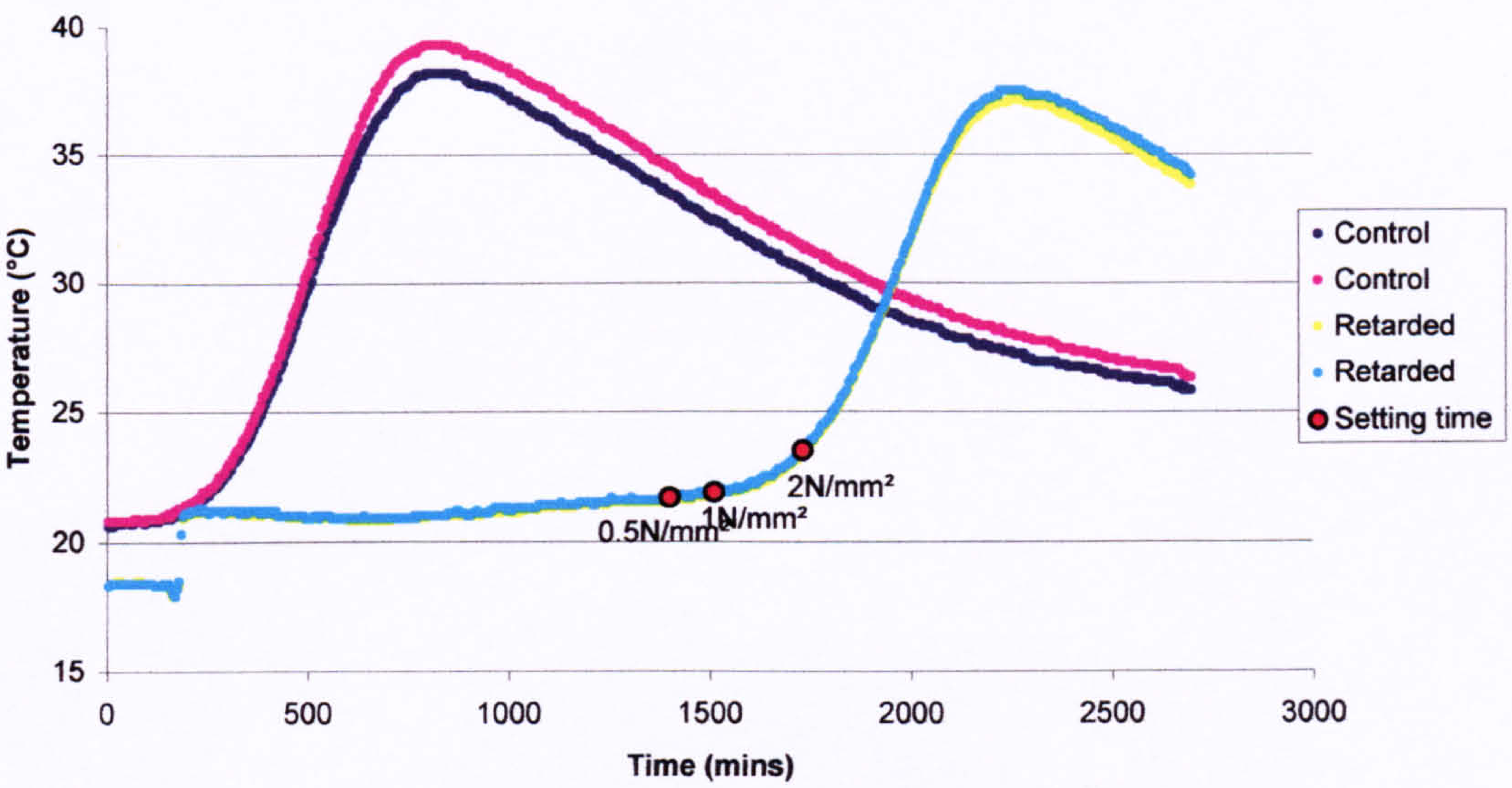
The results were then considered in the context of exactly how the procedure compared with the existing penetration methods in a quantitative way. The most recent and current British test method, BS EN 1015-9, 1999, states that a value of 0.5 N/mm^2 shall be taken as the prescribed limit of resistance, although higher values of 1.0 and 2.2 have been used with this procedure in unpublished work by the sponsoring company. As discussed earlier in section 2.2.3, these values are really arbitrary, chosen for convenience and bearing no fixed relationship to any finite part of the stiffening or early hydration process. Nevertheless, they have proved useful for comparison purposes and the data obtained using the heat of hydration were therefore plotted against those obtained by penetration, using approximately linear interpolation, as shown in fig 11.7

Fig 11.7 The relationship between the heat of hydration values and stiffening rate



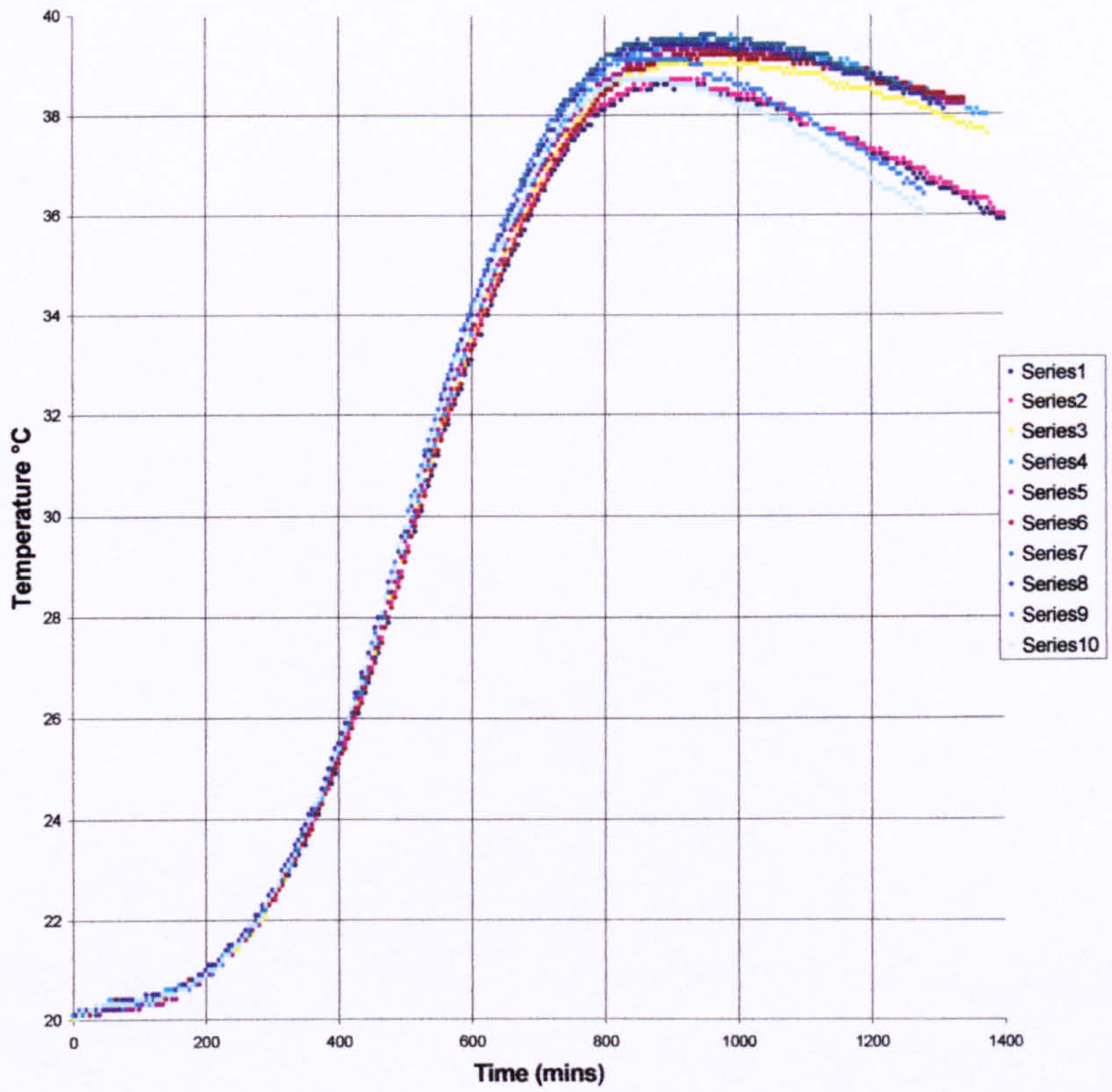
Because the linear interpolation used clearly failed to properly represent the actual form of the setting time relationship, the curve was smoothed by deriving the line of best fit and this form was used in future comparisons carried out in all future work. Figure 11.8, produced primarily to illustrate the relevance to retarded work and the repeatability also shows how this was utilised in practice.

Fig 11.8 Further trials to confirm the repeatablility with retarded and non-retarded systems



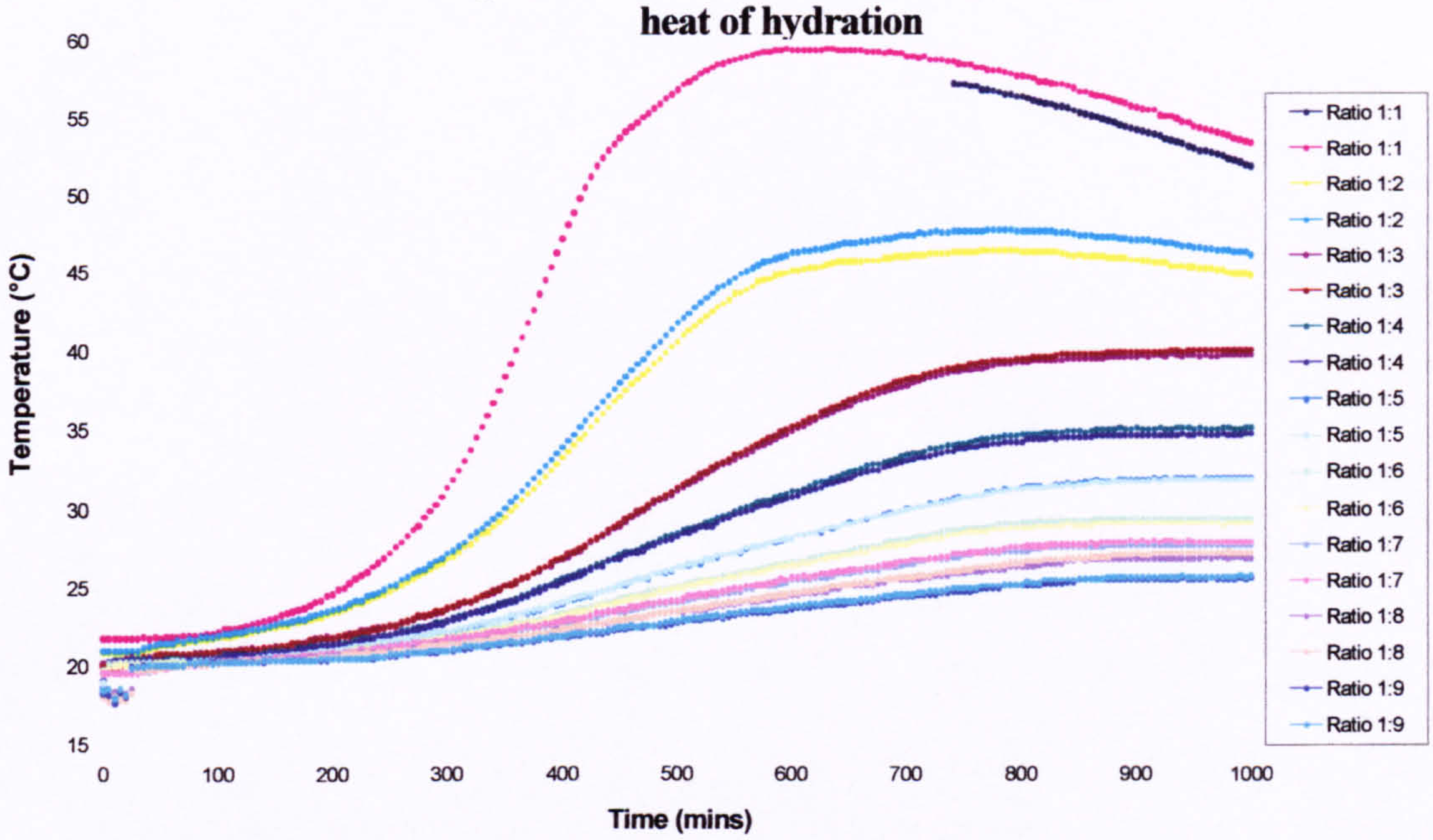
Before the revised equipment was accepted further trials were carried out to investigate whether the initially promising repeatability could be confirmed and figure 11.9, for a series of ten separate retarded mixes batched to the same weights, confirmed the good repeatability.

Fig 11.9 Repeatability trials



The effect of changing the mix design was then investigated in more detail, using a series of mixes with aggregate cement ratios varying from 1:1 through to 1:9. These results are shown in figure 11.10 below, and the effect of the cement content on stiffening and setting time, reported by Neville, 2000, for concrete may be clearly identified in the range of mortars tested, with a reduction in time as the cement content increased. These results were produced from duplicate determinations, as shown in the figure and it can be seen that the repeatability of the method was confirmed, with excellent agreement within the duplicates.

Fig 11.10 The effect of cement content on heat of hydration



The work to date had shown that in the early stages of the temperature monitoring, at which time there were undoubtedly rapid reactions involving saturation of the solution with calcium hydroxide and gypsum reaction to form ettringite, temperature was evolved that did not really constitute part of the formal setting process, but rather was a function of those very early reactions, and others not outlined above, that occur when water is added to Portland cement. This initial phenomenon led to the perceived need to carefully re-visit and standardise the mixing procedure, in order to minimise variability at this time in the test. Although the standard procedure of batching, mixing, standing and re-mixing confirmed in section 3.1.3 had been followed at all times it was thought worthwhile to re-examine this whole area. Investigation confirmed that there were standardised procedures, but that as suspected there were various conflicting regimes, as shown in table 11.1 below.

Table 11.1 Standardised mortar mixing regimes

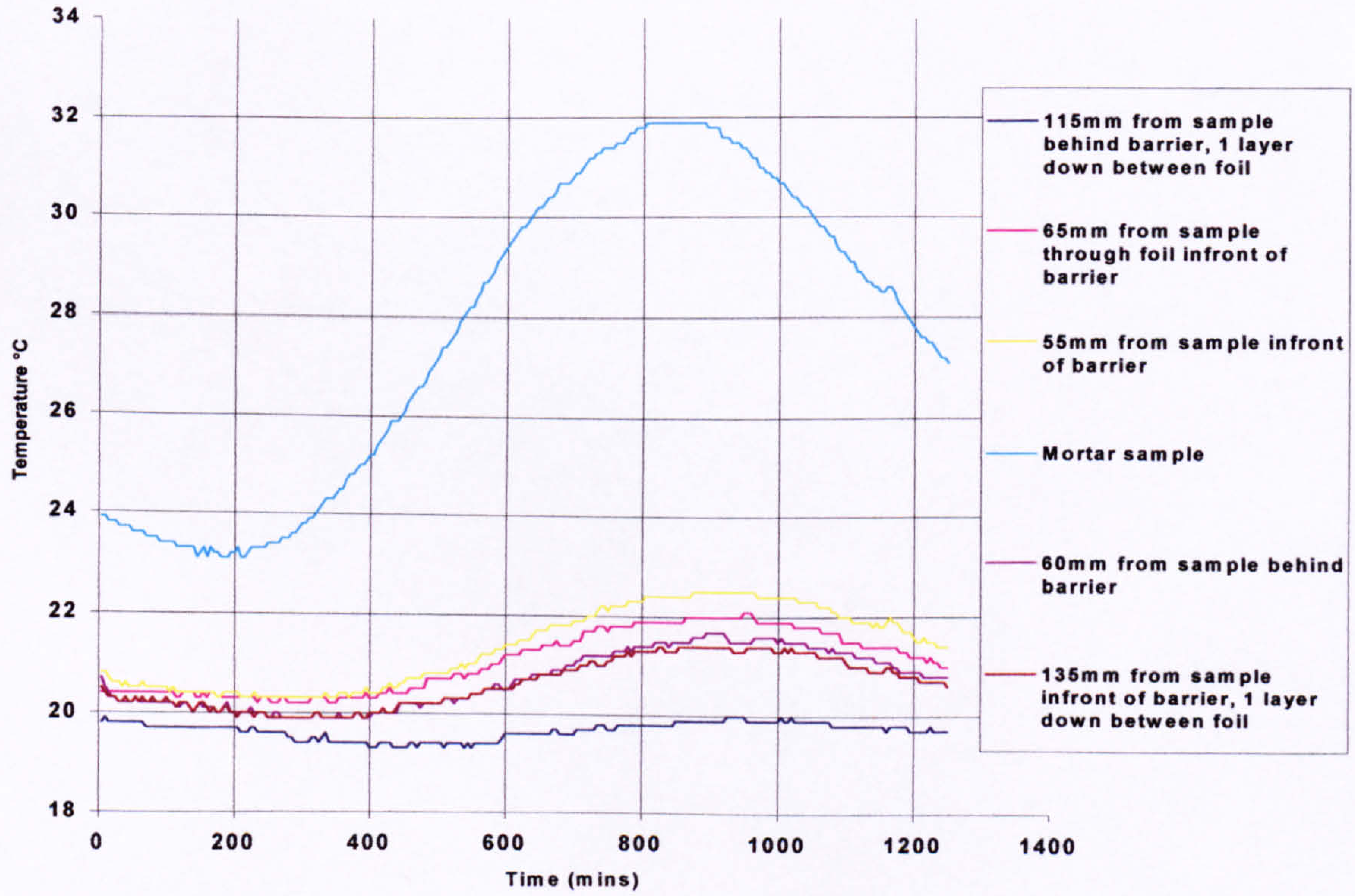
	BS EN 1345-2:2002	BS EN 196-1:1994	BS EN 1015-2: 1999	BS 4551: Part 1: 1998	ASTM E 149
Initial mixing (seconds)	5-10	60	15	30	15
Further mixing (seconds)	90	30	0	30	20
Standing time (seconds)	15	90	0	600	600
Final mixing (seconds)	90	60	75	60	60
Total elapsed time (minutes and seconds)	3.20-3.25	4	1.30 *	12.00	11.35

- An additional 5-10 seconds stirring is used immediately prior to the actual test procedure which follows the mixing

Having considered the different mixing regimes shown in the table it was decided to confirm use of the BS 4551 procedure, even though it would shortly be withdrawn and rendered obsolete by the mandatory introduction of BS EN 998, 2003 and the BS EN 1015 series, 1999. *(Note: since completion of this section the new standards have been introduced, and the original conflicting standard scheduled for withdrawal).* This decision was made on the grounds that the technique was a familiar one that worked well, it was very similar to the well established ASTM one and also it resulted in the standing time during which the very early life reactions took place being maximised, after which the equipment could be zeroed, which would aid the readability and discrimination of the method.

It was believed that the method had now been validated in principle, it worked with different aggregate cement ratios and with retarded mixes and showed sufficient promise to be worth proceeding with. Efforts were now made to improve and refine the apparatus prior to investigating further its applicability in the presence of different variables and using it to obtain accurate information on stiffening/setting times and figure 11.11 shows work on the effect of sample position in the apparatus.

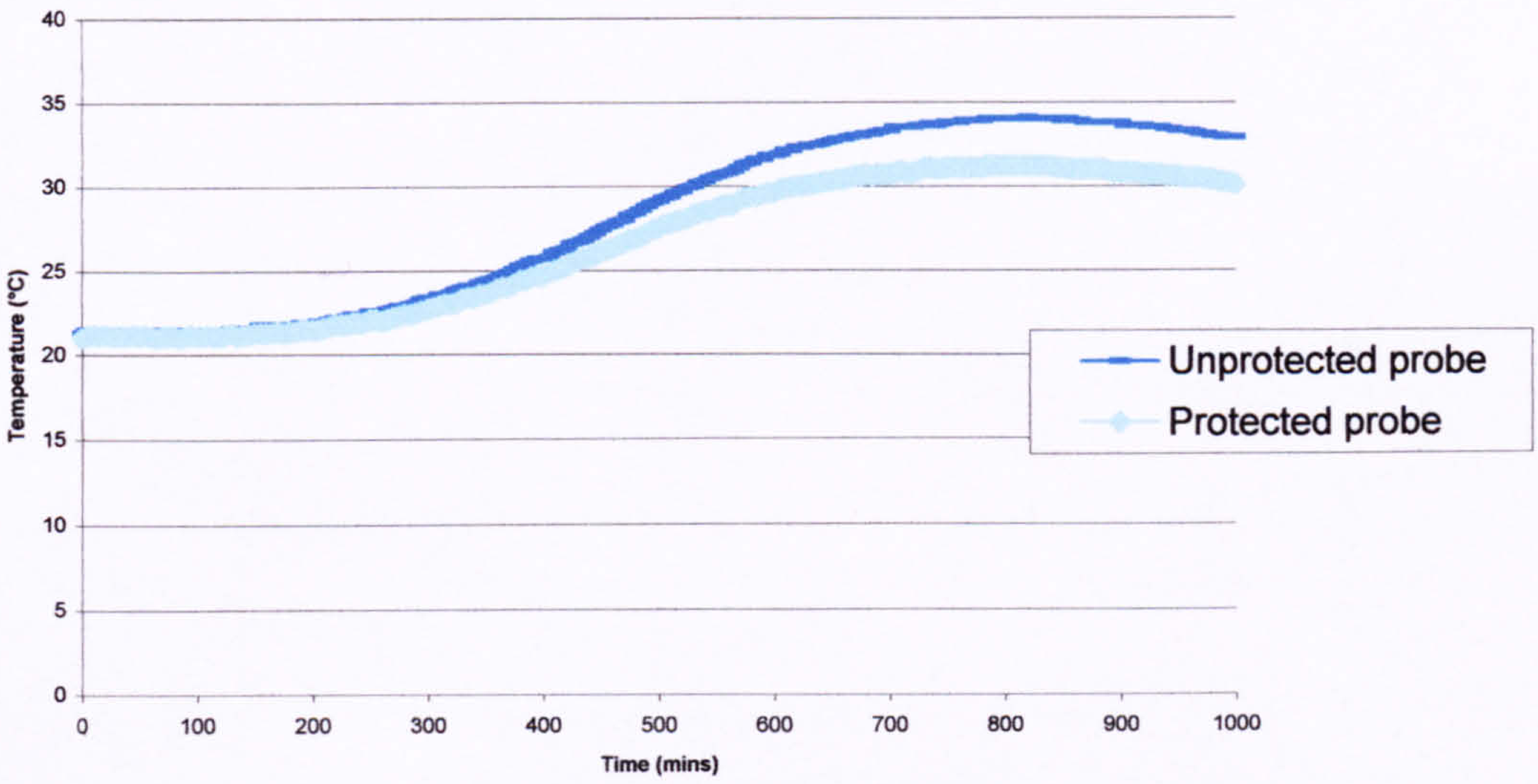
Fig 11.11 The effect of internal position in the apparatus



This graph shows the results of the investigation of the first issue, whether the heat evolved by one sample was interacting with any of the others. If it was, it would clearly degrade the accuracy. In order to check on this point one sample of mortar was placed in the apparatus and temperature probes were then situated at varying distances away, placed between different layers of the foil that comprised the foil backed insulation of which the equipment was constructed. As seen in figure 11.11, there was heat loss and related conduction from the specimen that appeared to cause the different heat increases shown. It seemed probable that this was associated with the layers of aluminium foil that ran through the equipment so it was dismantled and re-assembled without foil and a further trial carried out.

This further work, shown in figure 11.12 also shows the effect of another variable, that of changing the material used to protect the temperature probes. In the figure, one probe is used without protection, placed directly in the mortar, the other is wrapped in foil to ensure that it may be re-used, as probes without protection were seen to stick in the mix and were quite often badly damaged on removal, even when they had first been smeared in Vaseline. As an additional issue, coating the probe with Vaseline was itself considered undesirable because of the difficulty of ensuring that this was done in a reproducible manner, thus giving rise to the possibility that a greater thickness of Vaseline would produce a greater insulating effect.

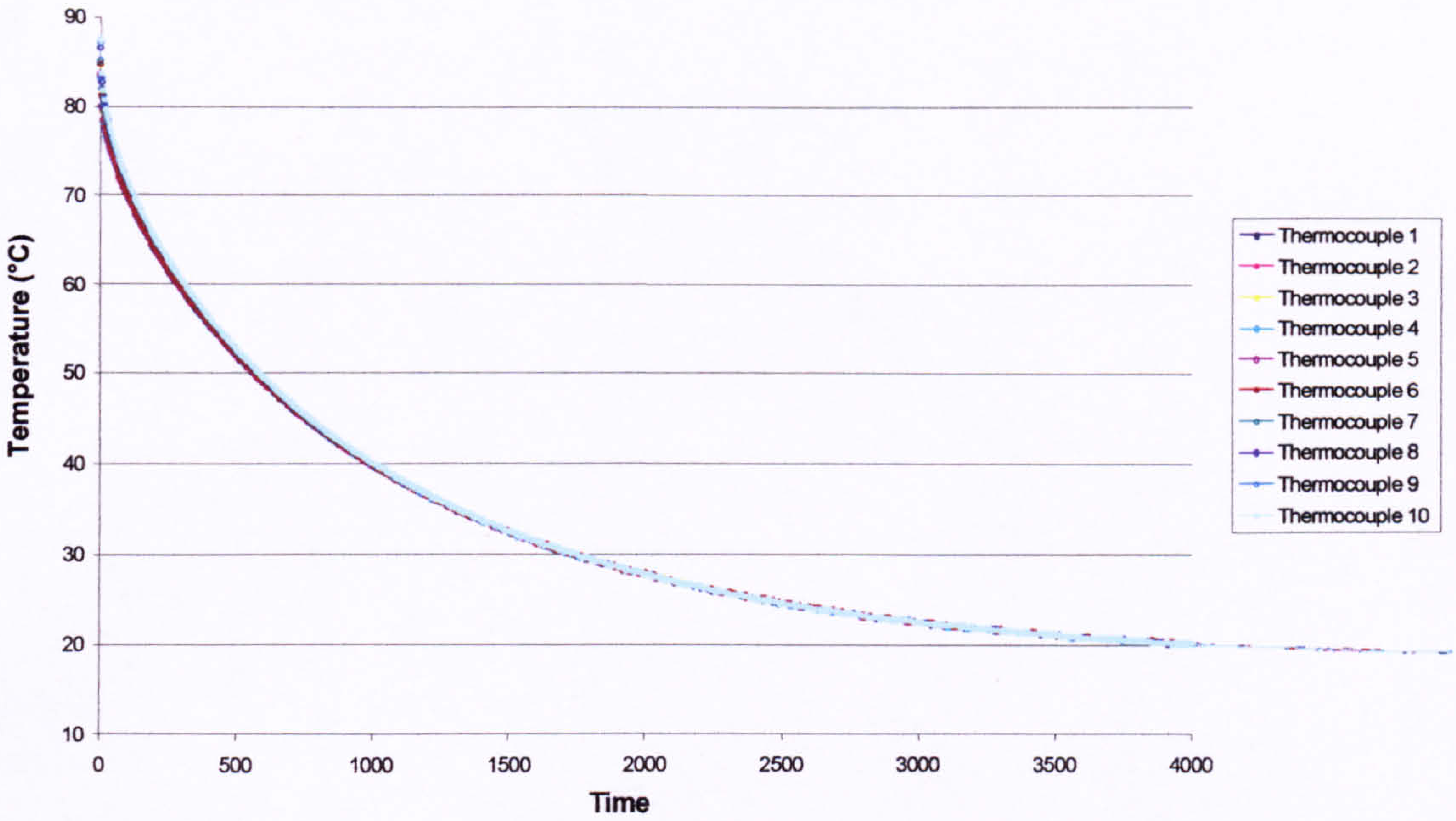
Fig 11.12 The effect of protective systems for the temperature probes



The use of foil was an improvement but again it was potentially operator dependant as it proved difficult to ensure that precisely the same amount was used and wrapped on in exactly the same way. This lead to the final evolution in this area in which the coating was replaced by the use of disposable plastic pipettes, the inside diameters of which coincided with the outside diameter of the probes and which were available extremely cheaply in 500 lot amounts from laboratory suppliers. These probes insulated a little better than the vaseline or the foil but provided a robust and non-operator dependant system.

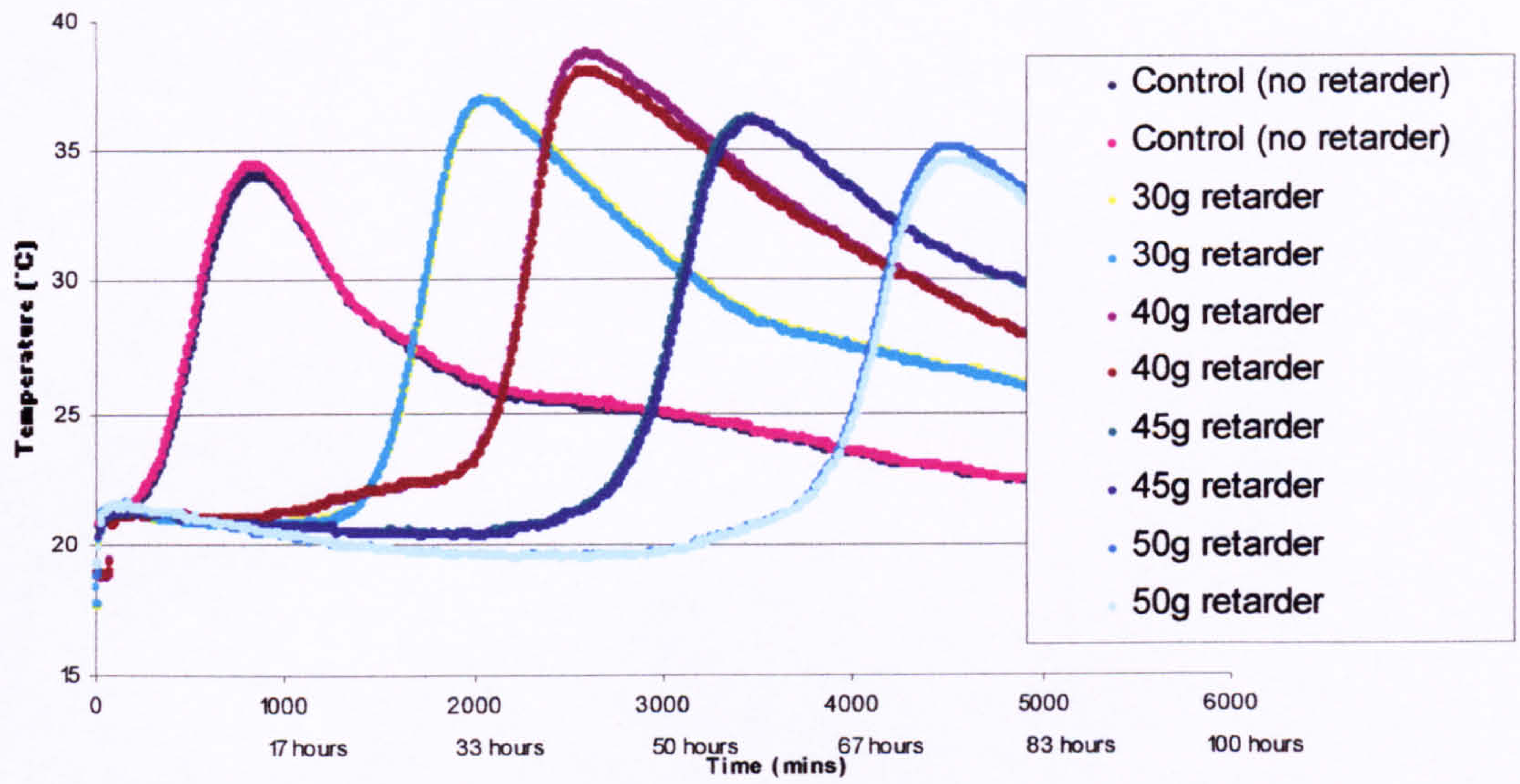
The next issue to be addressed was the accurate calibration of the temperature probes. Without calibration to achieve a common zero, comparison of different test samples was clearly not valid. This zeroing proved to be simple and figure 11.13 shows this work for ten probes, using water at just below boiling point as a simple calibration material.

Fig 11.13 Calibration of the resistance thermometers



The position of the sample in the test apparatus was then again investigated and figure 11.14 shows this work, for duplicate samples with differing retarder addition rates. Each mix was tested twice, once in the centre of the box and once at the ends. This work also further validated the repeatability of the method, which as can be seen was again shown to be excellent. It also showed that there was a "position in the box" effect, and this would have to be borne in mind if absolute work was ever considered but appeared to be hardly of sufficient moment to cause a problem with comparative work.

Figure 11.14 The effect of specimen position in apparatus



This again showed that the method showed excellent repeatability in terms of the length of retardation time. The height of the peaks was seen to vary systematically with the position of the sample specimens in the apparatus, with those in the centre reaching a slightly higher peak temperature than those at the ends, where there was greater heat loss. This meant that the method was acceptable for the purpose for which it had been designed originally, the determination of the setting time or workable life of mortars.

Whilst the work had been proceeding it had become apparent that this original scope might well be capable of considerable expansion in a later phase, perhaps relating the peak height or the area under the curve to other aspects of the setting process. It was now clear that this would be complex, almost certainly requiring some sort of calibration or zeroing of the test for every piece of apparatus. This potential extension is certainly too complex to be considered in the current work programme and may well prove to be difficult to achieve with complete success. However, it could prove of great potential interest in a future programme of work to investigate in much greater detail more sophisticated aspects of the retardation and setting process.

Now that the equipment had been validated and improved to overcome initial minor problems and the repeatability had been shown to be good, work was instigated to confirm that the method worked satisfactorily with a wide range of differing mix constituents.

11.1 EFFECT OF OTHER PORTLAND CEMENTS

The work so far had all been carried out on the standard Portland cement and was now extended to include a wide variety of other Portland cement types. As well as the CAA standard cement, as wide a variety as possible of common occurring Portland cements was examined. Cement replacements were also tested. Other cement types included the specialist calcium sulphate cement used for the manufacture of proprietary flow applied floor screeds together with high alumina cement.

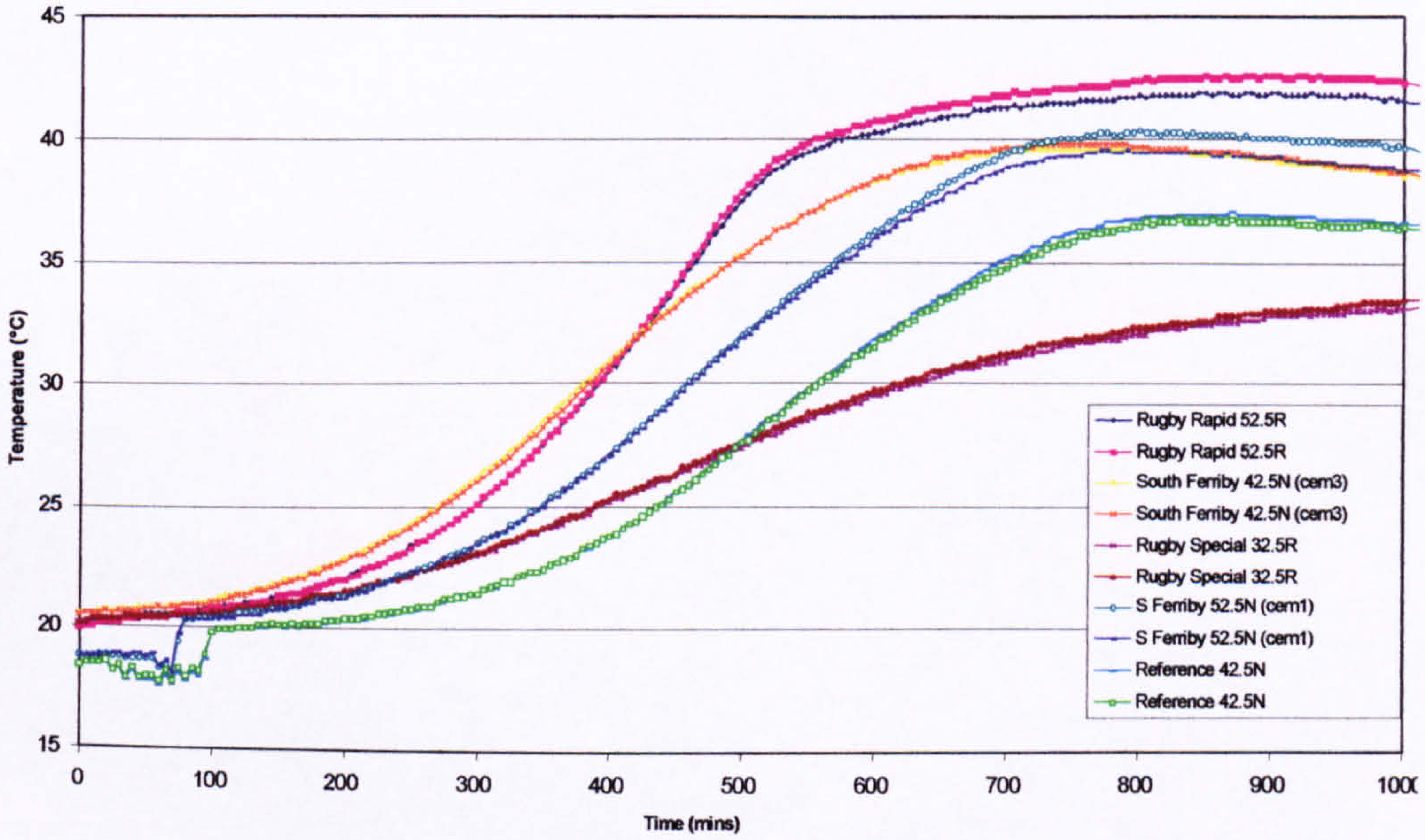
The first group of atypical Portland cements, results for which are shown in figure 11.15, comprised the following:

- Rugby rapid. CEM I 52.5N/mm^2. Finely ground, specific surface c. 495m^2/kg, with strength enhancer.
- Rugby South Ferriby works Cem II BV 42.5N/mm^2. 27% pfa, specific surface c. 425m^2/kg.
- Rugby special. Cem II BV 32.5N/mm^2. Specific surface c.350m^2/kg

- Rugby South Ferriby works CEM I 52.5N/mm^2. Finely ground, no additives, specific surface c.390m^2/kg.
- CAA reference cement

The results showed that the method discriminated between the setting behaviour and characteristics of finely ground and coarser cements, those with strength enhancers and those with pozzolanic components.

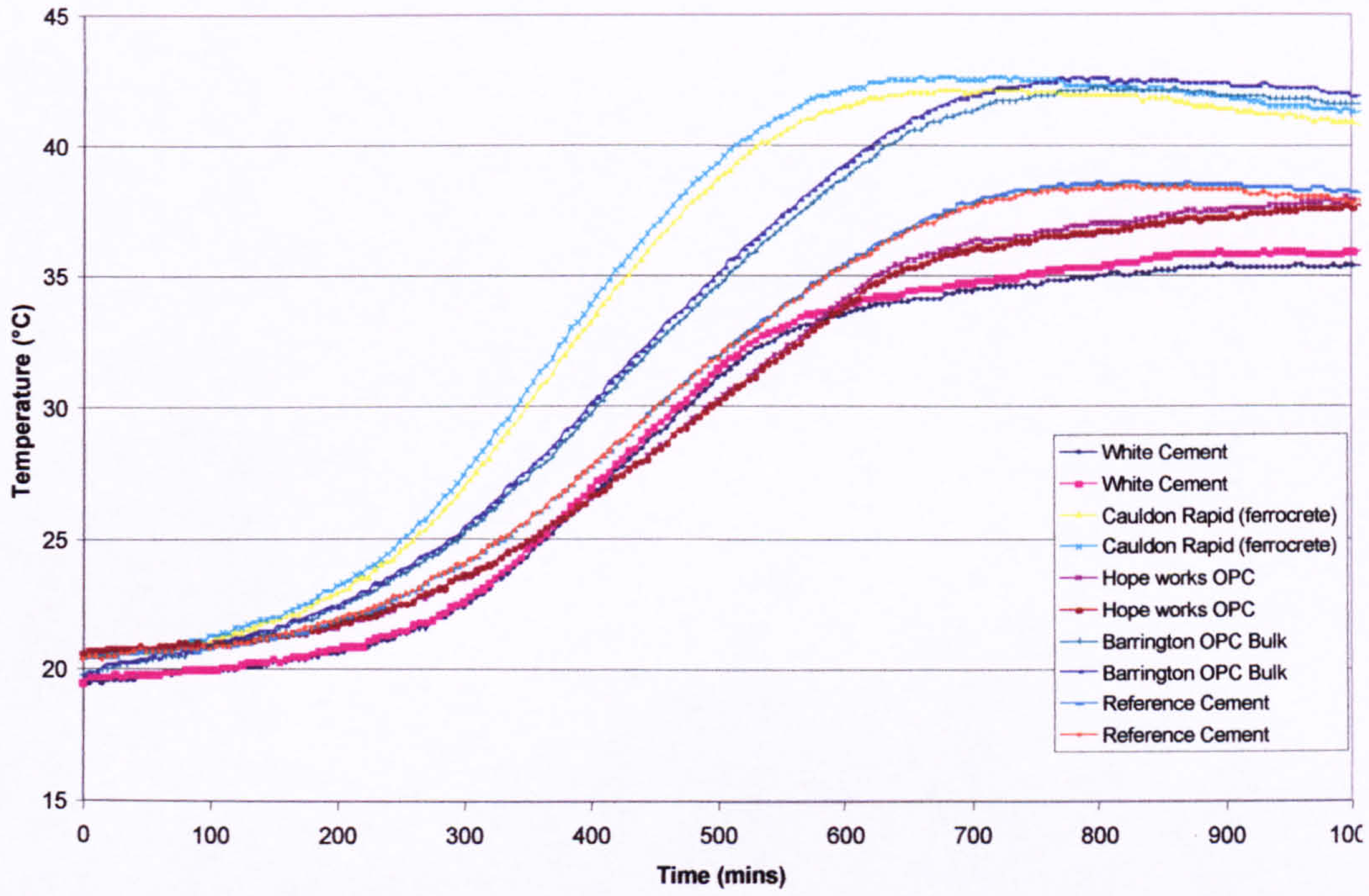
Figure 11.15 The effect of other Portland cements (1)



Further work was then carried out with additional atypical Portland cements, comprising the following, as shown in figure 11.16 below

- Aalborg white cement. Very low iron content
- Lafarge Cauldon rapid. 52.5N/mm^2. Finely ground
- Lafarge Hope. CEM I 42.5N/mm^2
- Rugby Barrington. CEM I 52.5N/mm^2
- CAA reference cement (specific surface 374mm^{2}kg), for comparison only.

Fig 11.16 The Effect of Different Portland Cements (II)



This work showed the quicker set of the rapid hardening materials and also shows how the much changed chemical composition of the white cement is carefully modified in order that the setting behaviour is substantially normal.

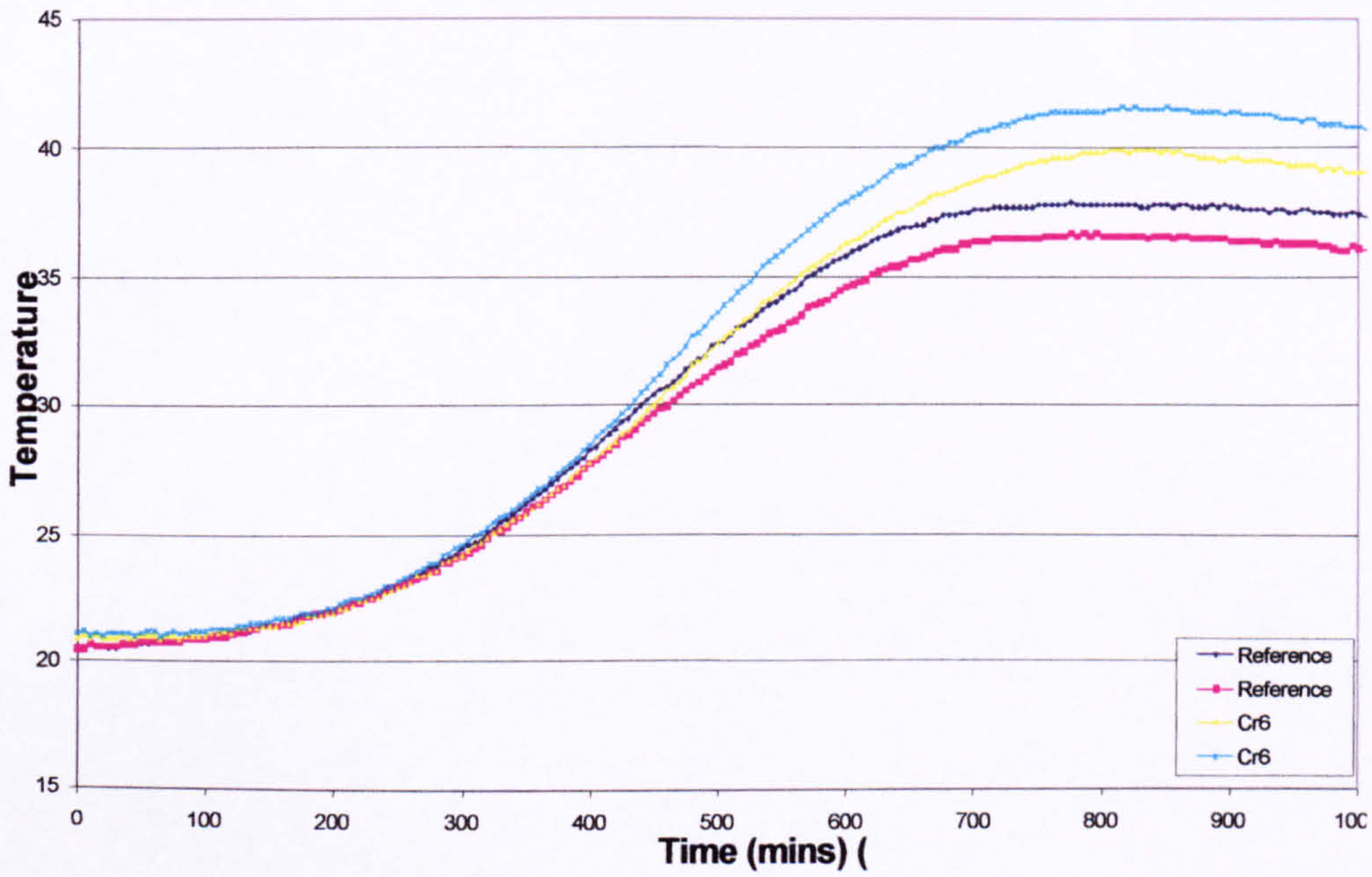
The final work on different cement types and sources, unfortunately not retained as data, confirmed the applicability of the procedure to a further selection of cement sources and types as follows:

- Castle Padeswood. CEM I 42.5N/mm/^{2}.
- Rugby South Ferriby rapid. CEM I 52.5N/mm/^{2}. c. 460M^2/kg
- Lafarge Northfleet. CEM I 42.5N/mm/^{2}.
- Lafarge Westbury. CEM I 42.5N/mm/^{2}.

All of these results showed that the method discriminated between different Portland cements, even when the differences were minor.

At the time this work was being carried out there was a proposal made within CEN that ferrous sulphate be added to Portland cements as an aid to health and safety, in that it would reduce the hexavalent chromium ion to the trivalent form which is not implicated in contact dermatitis, the so-called cement allergy. Accordingly, a trial was carried out into the effect of this material on the test procedure. The results, shown in figure 11.17 below, indicated that the inclusion of this material made no difference to test values.

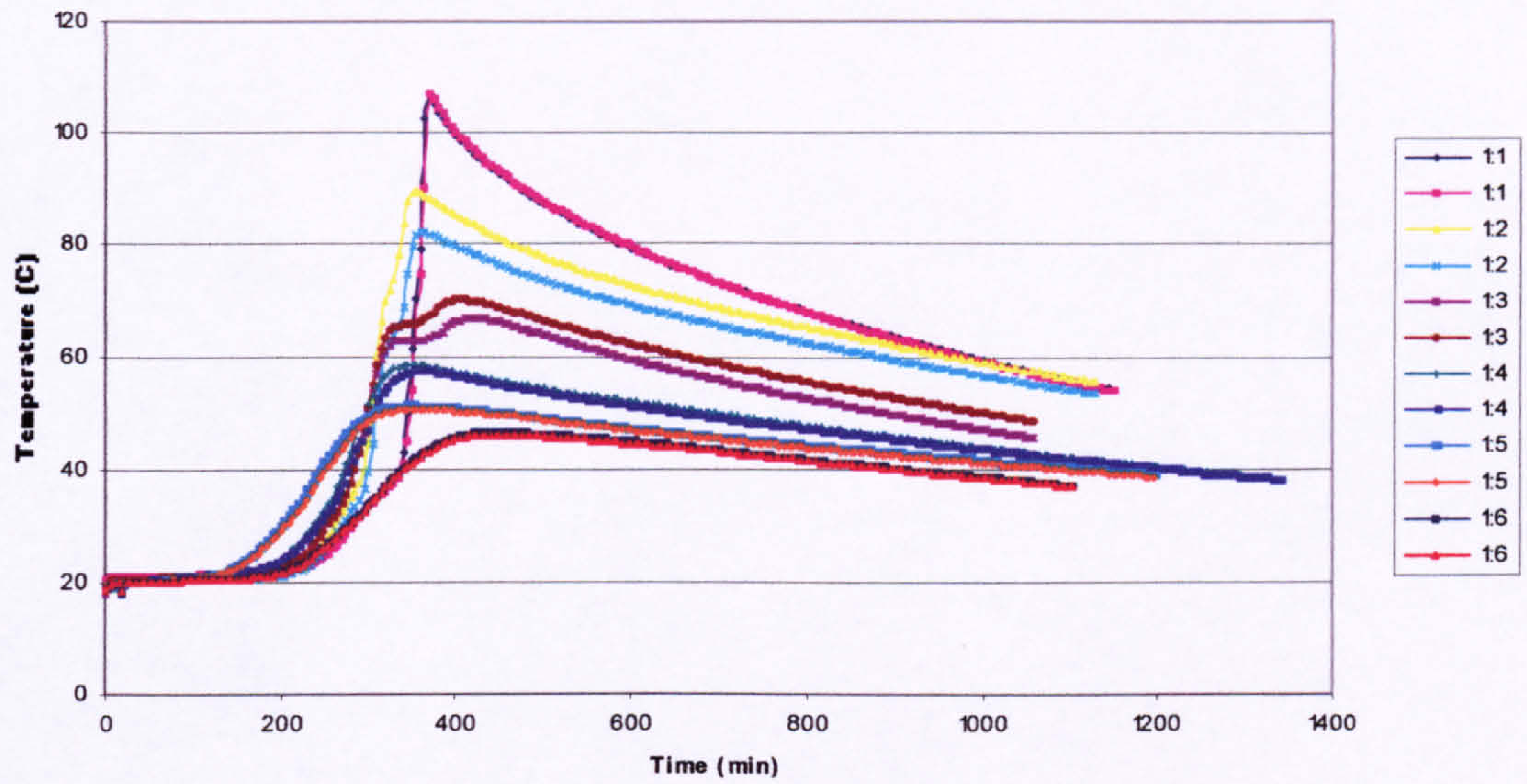
Fig 11.17 The effect of Ferrous sulfate



Non-Portland cements were then considered and trials were carried out using a high alumina, (sometimes now known as calcium aluminate) cement. The effect of this material, for duplicate determinations with six different mix designs is shown in figure 11.18. It is seen that the technique is still valid, although for the richer mixes the temperature rise was considerable. This probably accords with the known rapid early setting behaviour of this type of cement.

If further work was needed on this type of cement a smaller sample size or leaner mix would probably represent a worthwhile change, as Newtons Law of Cooling prescribes that there will be much greater heat losses with these higher temperatures.

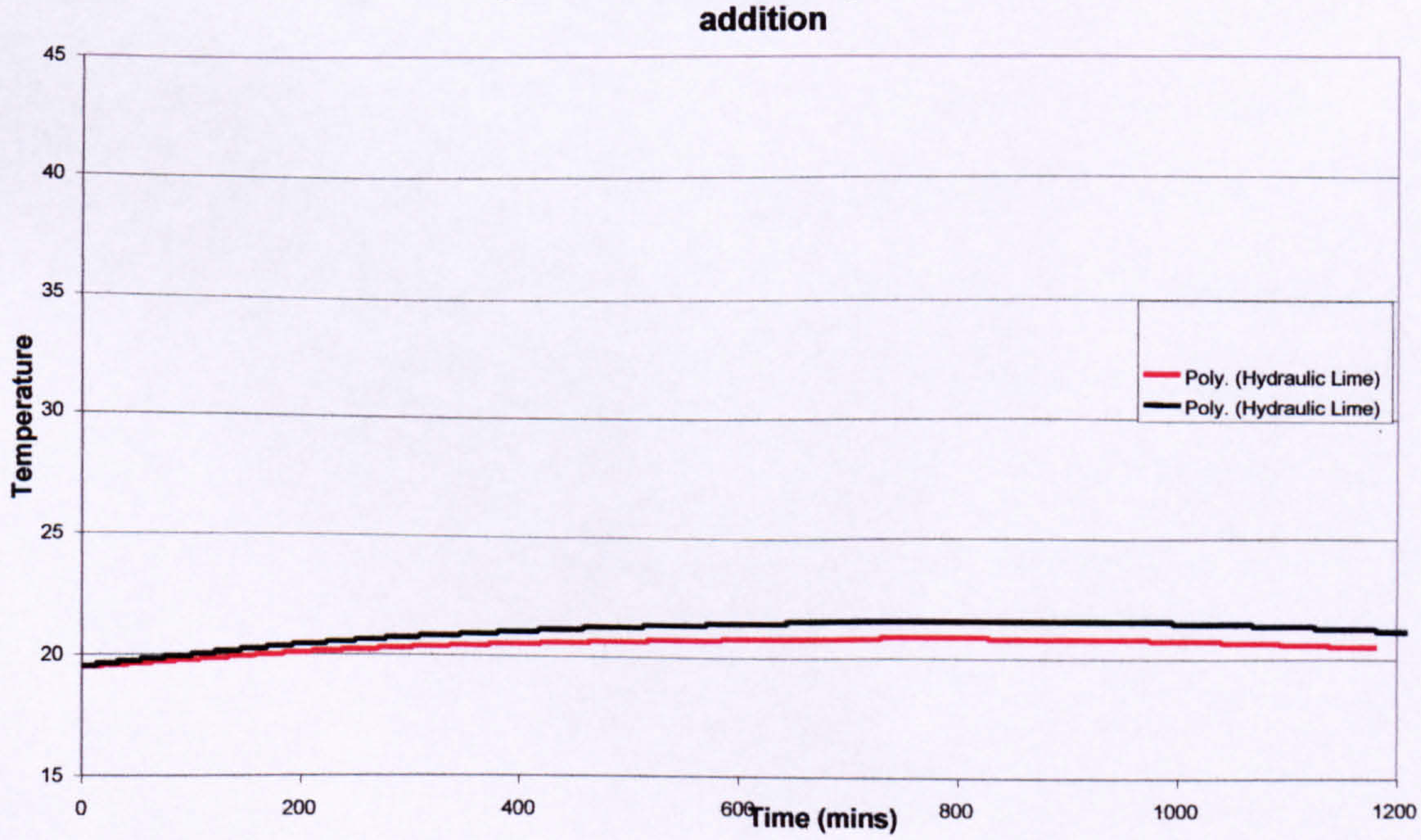
Fig 11.18 The effect of HAC on the heat of hydration



11.2 EFFECT OF SLOWER SETTING HYDRAULIC LIMES

The work so far had all been carried out on cements that hydrated reasonably rapidly, thus giving rise to the evolution of substantial amounts of heat. Further work was next carried out on a much slower setting hydraulic lime and the method was seen still to be quite usable, as shown in figure 11.19 below.

Fig 11.19 The effect of hydraulic Lime addition



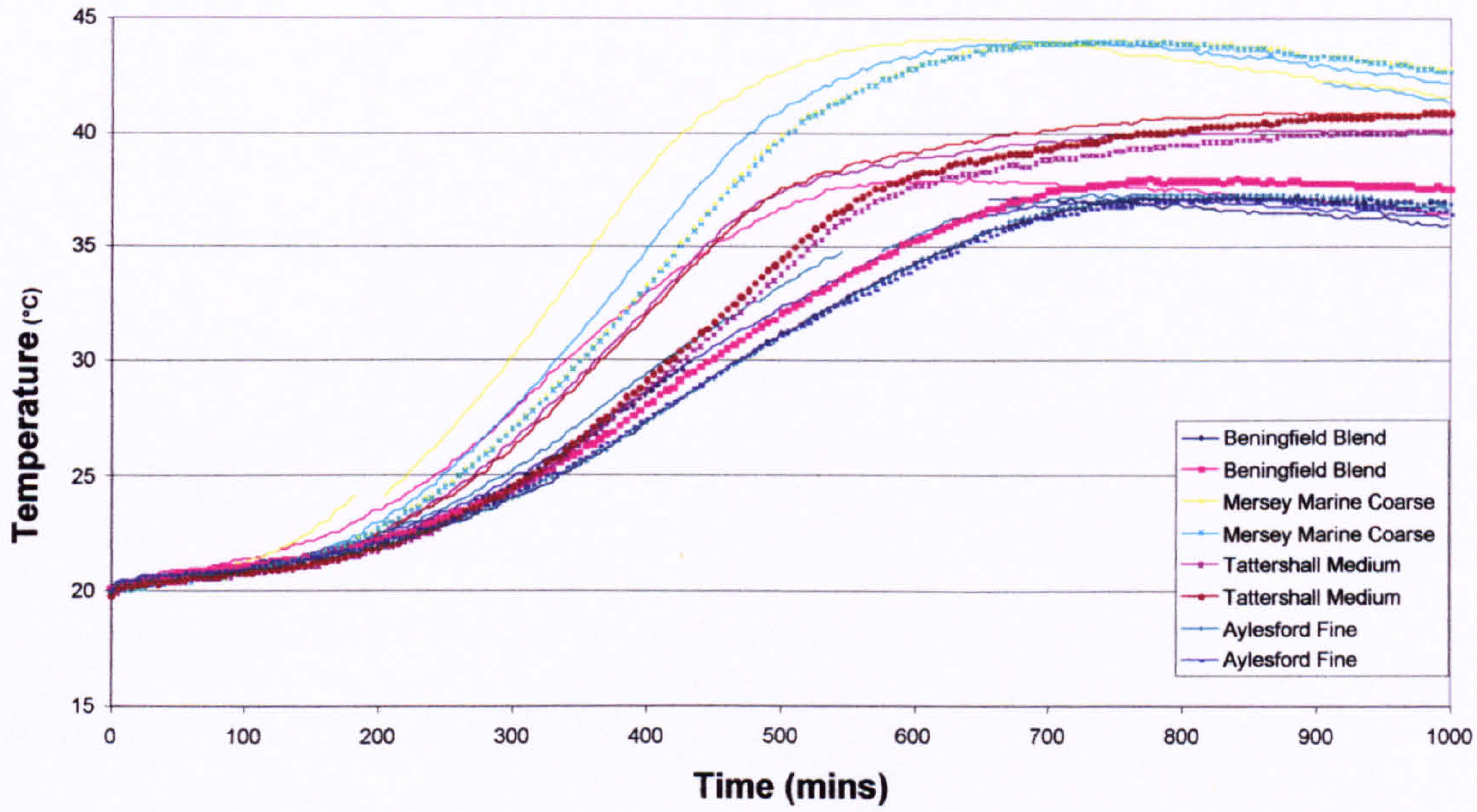
The results shown in this graph indicate the potential of the test method to examine in detail the setting mechanisms of other cementitious systems in addition to those based on simple Portland cement types. Unfortunately, however, the heat evolved by the much slower setting hydraulic lime was clearly far less than that with the Portland cements. In order to improve the accuracy with these materials it may be that larger specimen sizes or much greater insulation, to more nearly approach the true adiabatic state, would be required. This appeared to represent a considerable amount of further work and was unfortunately outside the scope of the current investigation. Nevertheless, the area shows promise.

11.3 EFFECT OF OTHER AGGREGATE TYPES

It was not expected that the aggregate type would greatly influence the result, but it was thought to be of interest to investigate any minor effects of aggregate type. Accordingly, fine, coarse, land based and sea dredged aggregates were tested.

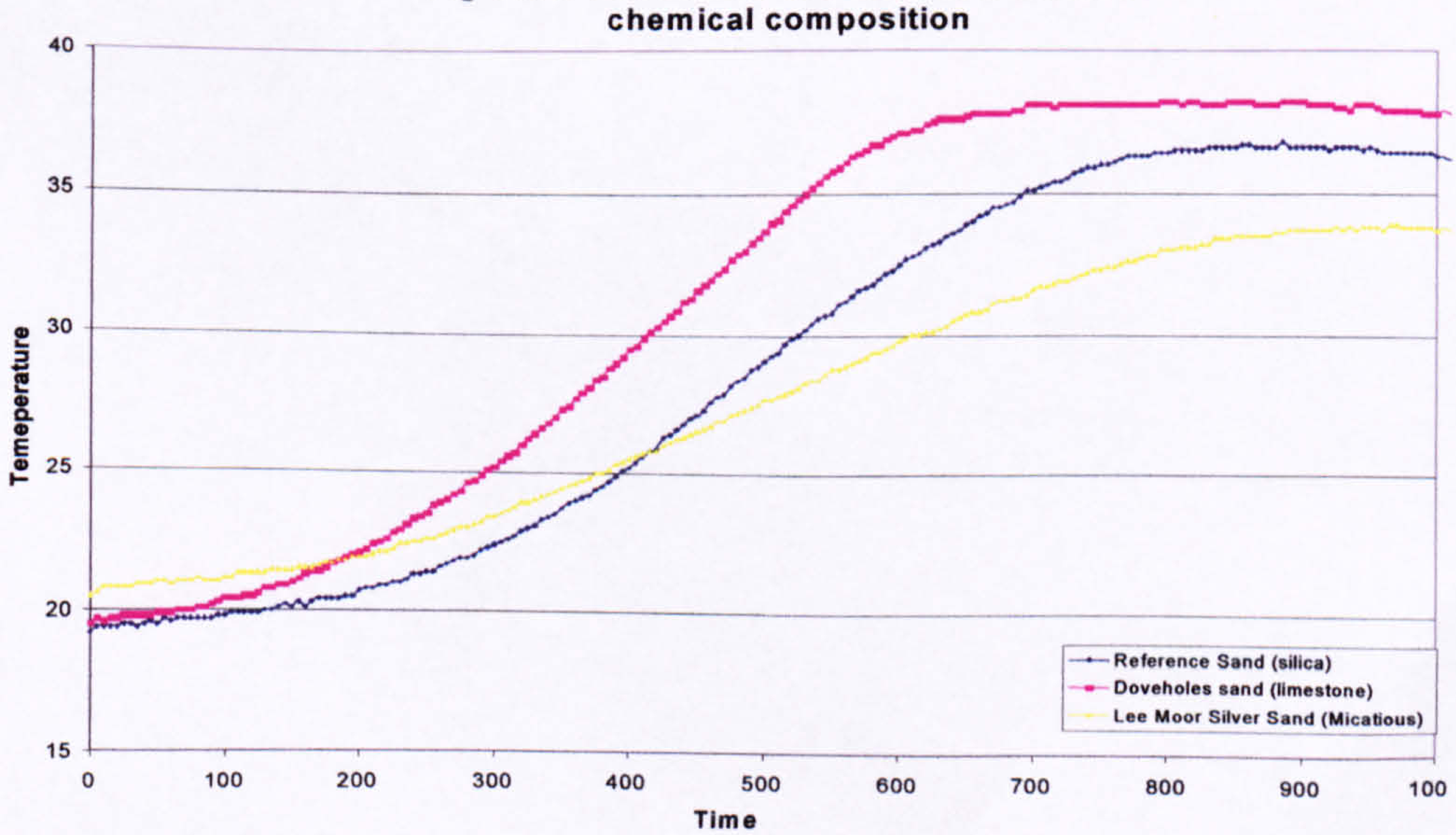
It was expected that the sea dredged material might set a little more rapidly, as a function of the chloride ion present and a minor effect was confirmed. In general, there was little effect although it was seen that the coarser sands also tended to set slightly quicker. This was also as expected and is as discussed earlier in the work, in that a lower water cement ratio is known to tend towards a slightly more rapid setting time. The results of this work are shown in figure 11.20 below which shows the results with four different sand types, the standard Beningfield blend, a coarse dredged marine sand, a fine land based sand from a quarry in Tattershall and a fine dredged land based sand from Aylesford.

Fig 11.20 The Effect of Different Sand Gradings



The final sand influence to be examined was that of chemical composition and in this trial a silicious sand, a limestone and a heavily micaceous sand derived from the production of china clay were examined. The results are shown in figure 11. 21 below and indicate that the chemical composition of the sand does not appear to exert an undue influence.

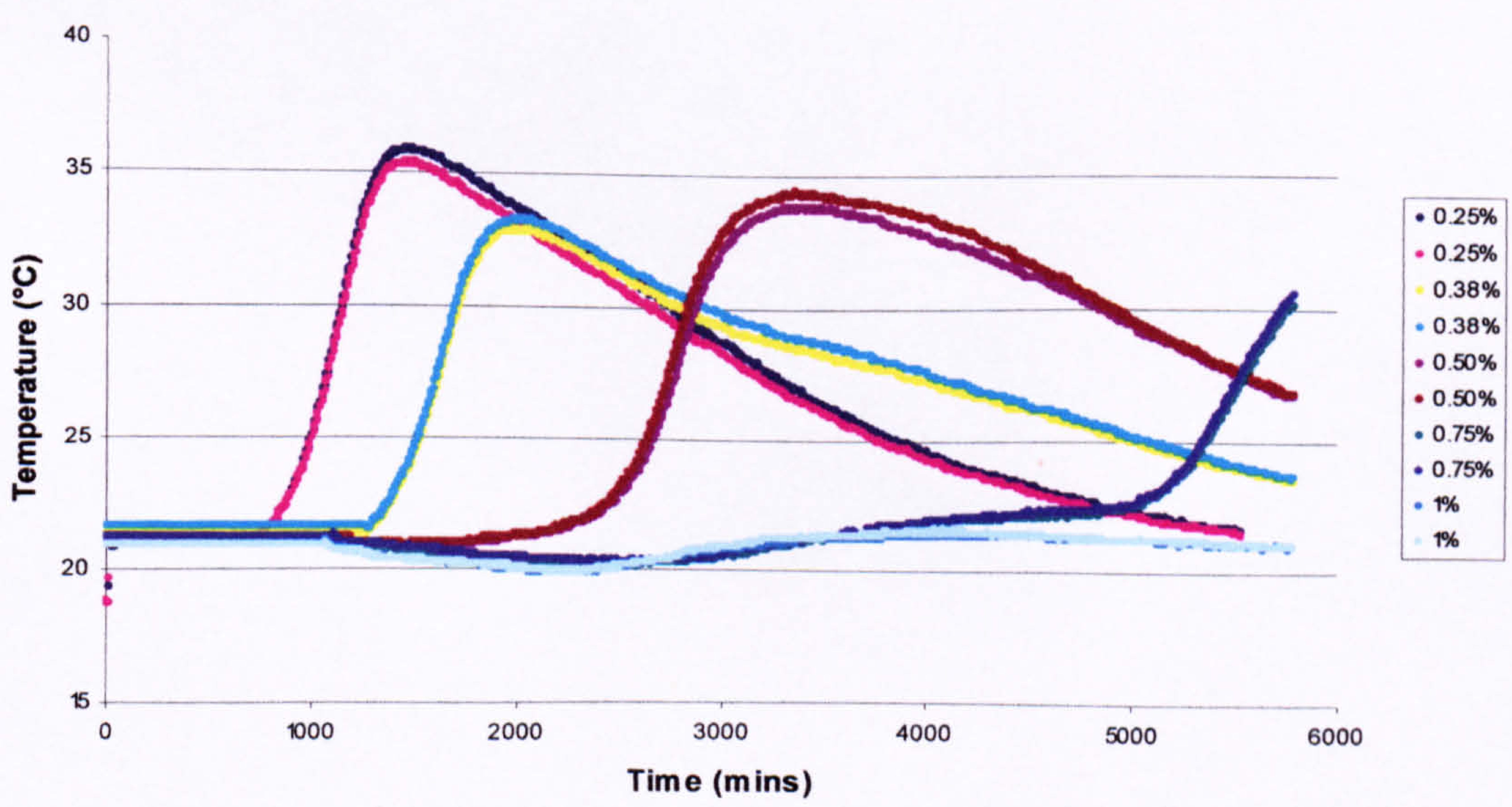
Fig 11.21 The effects of sands with different chemical composition



11.4 EFFECT OF RETARDED SYSTEMS

Although the research to date had been generally been carried out on non-retarded systems, albeit with some minor work on retarded systems, the real focus of the method in terms of its technical and commercial applications had always been planned to be with retarded systems. This was, as previously discussed, because existing manual methods were clearly not really practicable with the longer retardation times required by current construction practice. Now that the effect of major variables had been investigated in the simple non-retarded condition the final trial was to confirm the applicability to retarded mixes. Results for the first of this work are shown in figure 11.22 below, which further confirmed the earlier work on retarders shown in figure 11.14. It can again be seen that the duplicate determinations gave excellent agreement and the effect of increase in retarder addition was clearly seen.

Fig 11.22 The effect of retarder addition rate



This concluded the work on the proposed alternative method for stiffening/early life setting and the conclusions drawn from this part of the programme are discussed in section 12.3 below, immediately after the discussions concerning the research into the plastic properties.

12 CONCLUSIONS

This research programme comprised two investigations into the plastic mortar properties and behaviour, sometimes known as the fresh mortar properties.

The first investigation was into the very early life properties, when the mortar was still workable and the second into the stiffening and early setting properties, that is the behaviour of the mortar whilst it was becoming less workable and the set was commencing. The conclusions into each are outlined below.

As a preliminary, initial work was carried out to quantify the plastic properties from a practical point of view, and to relate those practical assessments to the existing test methods and the parameters that they measured. The objective of this was to confirm whether or not all of the practical criteria were adequately appraised by existing test methods.

This led to the view that there were areas that were not adequately covered, in particular a test was needed that related to ease of placement, deformabilty and thixotropy. In addition, the current procedures for the early life stiffening/setting behaviour, which takes place immediately after the period when the earlier plastic properties are dominant, were not really satisfactory. The programme that formed the major part of this work investigated and contrasted existing procedures, addressed shortcomings in these and suggested new or revised procedures if appropriate.

12.1 CONCLUSIONS FROM THE LITERATURE SURVEY

The literature survey gave rise to the following conclusions:

12.1.1 The plastic mortar properties are often overlooked and rarely specified although many will have a profound effect on the hardened properties, often in key areas such as bond.

12.1.2 Terminology in this field is loose and often misleading, with terms such as workability, plasticity, consistence, flow value and similar being used for essentially the same properties, often interchangeably.

12.1.3 More non-specific terms still such as spreadability, stickabilty and sand carrying capacity are used, sometimes even by engineers and researchers.

12.1.4 Where the plastic properties are specified or measured there is often a lack of clarity concerning what the resulting values actually mean.

12.1.5 The few crisp and accurate statements that have been published, for example, "consistence is a measure of workability, plasticity requires a more dynamic assessment", RILEM, 1969, appear to be largely ignored.

12.1.6 Whilst there has been a recent increase in the payment of masons on a piece work basis, which often seems to lead to corruption of the plastic properties, for example by addition of unauthorised admixtures in order to enhance productivity, there has been little concomitant additional concentration on the testing or control of these issues.

12.1.7 There are a great number of tests for the mortar plastic properties but the majority measure the same property or mix of properties. Notwithstanding this proliferation new tests continue to be devised, often being described as uniquely better or improved, whereas in reality they are all broadly similar, differing in mechanical detail but not in their basic scientific principles.

12.1.8 The tests in current usage are generally outdated and do not have regard to the basic rheological theory, or to recent developments in mortar production and technology, where considerations of the stability of entrained air and thixotropy are important.

12.1.9 Notwithstanding the large number of available tests, there is a surprising lack of comprehensive data to compare and contrast all of these, with the work that is available generally presenting relationships that appear to be over-simplified.

12.1.10 With regard to the test for water retentivity, the use of backgrounds that aim to produce a standard suction is ubiquitous, but some reported work on an alternative concept based on application of gas pressure appears to have a far more satisfactory theoretical basis, in that it is not reliant on the assumption of a constant suction being present from batch to batch of the test substrate.

12.1.11 The use of rheometry to characterise mortars and concretes in an objective way is well established in the research field, but it has not really been adopted in practice and is certainly not standardised or codified.

12.2 CONCLUSIONS FROM THE RESEARCH INTO PLASTIC PROPERTIES

A preliminary investigation first compared the properties of a mortar as appraised by practical considerations on the part of the user with the existing tests and the results obtained therefrom, in order to identify any shortcomings. The test programme then appraised the most used existing methods for the determination of consistence for sensitivity and then went on to compare and contrast them with each other.

Where existing test procedures did not cover a required property, or did so inadequately, suggestions were made as to test methods that could be adopted to correct these inadequacies. The proposed new test methods were then appraised.

The detailed findings of the test programmes are outlined below:

12.2.1 The preliminary work showed that there were mortar properties that were readily identifiable by the operative/user, and rated as important, for which there were no existing standardised tests.

12.2.2 The existing standardised test procedures failed to accurately measure the property usually known as plasticity.

12.2.3 They were inadequate where thixotropy was present

12.2.4 There was a relationship between the most common ways of determining consistence and water content but this was not of the straight line form often reported, but was of a polynomial form with a pronounced dip when shown as a graph.

12.2.5 There was a relationship between the different methods referred to above, but the methods differed in their ranges and sensitivities.

12.2.6 The ways of testing for consistence commonly used in the UK and Europe may be ranked in terms of the range of consistence that they are able to represent as shown below, with that with the greatest range shown first.

- Dropping ball
- DIN plunger
- Flow table

- Vicat

12.2.7 The methods may be ranked for sensitivity, with the most sensitive shown first

- DIN plunger
- Dropping ball
- Flow table

12.2.8 Of the two methods showing the better sensitivities, the dropping ball was the most operator dependant, so the DIN plunger appeared to be superior overall, although the apparatus is not quite optimised mechanically and would be enhanced by the adoption of electro mechanical release, eg as used in soils testing, BS 1377-2:1990.

As the preliminary work had shown that the existing tests were not adequate, and both theoretical considerations and the literature survey had indicated the possible potential of rheological techniques, the research investigated these, with the following findings:

12.2.9 The Tattersall rheometer gave results that were promising but too variable. The equipment was unsophisticated.

12.2.10 The Haake rheometer gave reasonable repeatability but, in the form available for use, was unable to handle particle sizes larger than about 150μm. It also lacked the features present in more complex machines, particularly in the differing modes of load application in which it could operate.

12.2.11 The Brookfield rheometer possessed neither of the disadvantages outlined in b) above and work with it produced the findings outlined below.

a) Increasing consistence increased both yield stress and viscosity.

b) Increasing air content increased yield stress and viscosity.

c) Increase in sand fines, ie a decrease in particle size, resulted in a decrease in yield stress and plastic viscosity.

d) The effect of lime was to increase both yield stress and plastic viscosity markedly.

e) Cellulose ethers, polysaccharides and acrylic copolymers all acted to increase viscosity markedly. They increased yield stress somewhat, with cellulose ether having the greatest effect, copolymer the smallest.

f) Rheometry appeared potentially to be a powerful technique for the study of thixotropic properties.

12.2.12 One of the most important findings was that the presence of air entraining agent and retarder substantially affected the plastic properties. This effect was not just in terms of the expected behaviour deriving from their basic effects, ie air entrainment and retardation, but was greatly in excess of that, presumably because of the residual effect of the chemical species.

12.2.13 It is suggested that usable mortars have yield values of between about 300 and 750. Below that value they will not support a unit well, above it they will resist initial movement with a trowel to a degree that may be judged unacceptable.

12.2.14 A viscosity range of between 500 and 4000 appears satisfactory, below that value segregation and bleed may occur, above it the material will appear “thick” and “heavy” to the operative.

12.3 CONCLUSIONS TO THE INVESTIGATIONS INTO THE STIFFENING AND EARLY AGE STRENGTH DEVELOPMENT

The investigations into this aspect of the plastic properties, at a time in the mortar maturity immediately following that encompassed by the work reported on in section 12.2 above, showed that in general the existing tests were based on mechanical methods that were not very satisfactory and that a non-mechanical instrumental technique could be used with advantage as a replacement procedure.

The literature survey showed that there were a number of standard tests for the workable life or stiffening rate but with the exception of one French method all of the standardised methods were based on mechanical procedures, generally those based on penetration of the sample by a rod, bar, needle or similar. There was a reasonable amount of literature concerning instrumental methods based on measurement of the exotherm arising from the hydration reactions and the research programme concentrated on the development of a simple, economic and robust procedure capable of handling multiple samples. The research produced the following findings :

12.3.1 The values for the workable life using traditional methods were affected by mix design.

12.3.2 They were also affected by a variety of mechanical and operator dependant issues.

12.3.3 An instrumental technique based on semi-adiabatic heat monitoring could be used instead of the current mechanical ones.

12.3.4 The technique worked well with different mix designs and with different cement and aggregate types. It worked with some non-Portland cements like high alumina cement and a calcium sulfate cement.

12.3.5 The technique was particularly suited to working with the retarded mortar systems that are in increasing usage today.

13 RECOMMENDATIONS FOR FUTURE WORK

The work on the initial plastic properties tests for consistence showed that of the existing methods the DIN plunger was marginally superior and it is recommended that this method is adopted where a standardised method is required. Of the non-standard methods, the small cone penetrometer showed great promise. Whilst it is felt that there are already an excessive number of methods available for the determination of consistence, and as a principle the proliferation of test methods in any field must be deprecated, it is felt that this method has the great advantages of simplicity, robustness and lack of need of a purpose made vessel in which to place the sample. It is therefore recommended that it be developed further. There is a requirement to confirm sensitivity, range, repeatability and in the longer term reproducibility.

The rheometry showed great promise and it is recommended that it be developed further. The accuracy and reproducibility of comparative work may also be further enhanced by additional refinements to the procedure, as discussed below.

Firstly, rather than using only temperature stabilised raw materials and laboratory environment, more sophisticated rheometers are available with the addition of temperature controlled water bath encapsulated specimen chambers so that extreme accuracy may be attained in this area, having regard to the fact that rheology is reasonably temperature dependant. Secondly, absolute rheological standards are available in the form of very stable, long chain calibration compounds. Use of these two enhancements will further improve numerical data in absolute terms and it is recommended that they are adopted for work where the best available accuracy is required. It is to be hoped that adoption of these enhancements will improve the repeatability of the attainment of absolute values, as although the current work showed good comparative power within a series, the shift in absolute values that is discussed in section 2, the review of literature, was disappointing.

Following on from refinement of the technique, it is recommended that it be used for further research into the effects of clays and of lime, as well as continuing to investigate those properties of "spreadability" and "finishability" that affect placement and deformability and were identified by users.

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APPENDICES

APPENDIX 1 A PROPOSED NOMENCLATURE FOR THE PLASTIC PROPERTIES OF MORTARS

As discussed in the literature survey on the requirements for the plastic mortar properties, terminology in this area is loose, inaccurate and misleading.

Many terms are used interchangeably, with confusion probably if an objective assessment is required. A number of terms are used in one context in the field of mortar, but a slightly different one with respect to concrete.

The proposed definitions below are suggested to ensure an accurate treatment of the topic.

ADHESION

Adherence to an external body or material.

COHESION

Internal adhesion.

CONSISTENCE

The degree of wetness.

Workability. A deprecated term.

Spreadability. A deprecated term.

Flowability. A deprecated term.

PLASTICITY

A complex sum of properties, including adhesion, cohesion, viscosity, yield, degree of segregation and consistence. A derived property.

Sand carrying capacity. A deprecated term.

Harsh. Deprecated in the context of mortar.

VISCOSITY

The resistance to motion offered by a material. A fundamental property.

WATER RETENTIVITY

A measure of the water retention of a mortar exposed to a background that produces suction. Exceptionally, may be derived from a consideration of applied pressure, rather than suction.

WORKABILITY

A measure of the energy required to move, deform or penetrate the mortar. A derived property term and one that is deprecated in the case of mortar.

YIELD, YIELD POINT

The point at which plastic deformation begins. A fundamental property.

APPENDIX 2 METHODOLOGY AND DESCRIPTION OF LEGENDS FOR FIG 7.1, SECTION 7.1

The description given in the text on page 119 concerning the test methodology and the labelling of the graphical legends is sparse and this appendix clarifies those issues.

The work addressed the repeatability and the ability to discriminate between different mix designs by evaluating five groups of materials.

Reading vertically down the list of legends shown in Fig 7.1 these were as follows:

- 3 identical mixes with cement content, (in the small laboratory mixer), of 80
- 2 identical mixes with cement content 75
- 2 identical mixes with cement content 75 but 25% pfa substitution
- 2 mixes with cement content 80 and 25% pfa
- 2 mixes with metakaolin, 25% and 50% respectively, with cement contents of 85 and 80

The graph show that the lines for identical mixes sometimes crossed and some appeared transposed. It was not possible generally to discriminate between different mix types that experience indicates to have had different working properties, as for example mixes of 100% ordinary Portland cement, (now referred to as CEM 1), and mixes with a considerable pfa content . For these reasons this work was not proceeded with.