

RESISTIVITY TESTING OF CONCRETE

BY

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R.MORELLI ,

TO MY PARENTS

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## NOTATION

A list of the symbols used in this work is given below.

A	: Aluminate ( $\text{Al}_2\text{O}_3$ ).
A	: Cross-sectional area.( $\text{m}^2$ )
a	: Coefficient of $\phi$ in Archie's FF equation.
A.C	: Alternating current.(Ampere)
$A_{ca}$	: Thirty minute absorption capacity of coarse aggregate.
$A_e$	: Average cross-sectional area of pores.( $\text{m}^2$ )
$a_e$	: amount of electrolyte contained in an average cross-section.
$\bar{a}_e$	: effective amount of electrolyte contained in an average cross-section.
a.r.	: Alumina ratio.
$A_s$	: Thirty minute absorption capacity of sand.
A/C	: Aggregate/cement ratio.
$A_1$	: Weight of fine aggregate.(kgs)
$A_2$	: Weight of coarse aggregate.(kgs)
C	: Lime (CaO).
C	: Capacitance.(Farad)
c	: Half depth of the beam.(m)
$C_a/C$	: Coarse aggregate/cement ratio.
C.F.	: Compacting factor.
c.f.	: Constriction factor.
$C_1$	: Length of pore.(m)
C-S-H	: Calcium Silicate Hydrate.
D	: Density.( $\text{kgs}/\text{m}^3$ )
d	: Diameter.(m)

D.C	: Direct current.(Ampere)
E	: Young's modulus.(N/m <sup>2</sup> )
E	: Energy.(Joule)
e	: Electrical charge.(Coulomb)
E.D.L.	: Electrical double layer.
F	: Iron oxide (Fe <sub>2</sub> O <sub>3</sub> ).
f	: Strength.(Pa) (Note 1Pa = 1N/m <sup>2</sup> )
F <sub>E</sub>	: Fracture energy.(Joule)
FF	: Formation factor.
I	: Electrical current.(Ampere)
J	: Moment of inertia of a cross-section.(m <sup>4</sup> )
k	: Numerical constant.
Kw	: Ionic products.
l	: Length of specimen.(m)
l <sub>e</sub>	: Average length of pores.(m)
l.s.f.	: lime saturation factor.
M	: Maximum bending moment.(Nm)
m	: Exponent of $\phi$ in Archie's FF equation.
m <sub>r</sub>	: Modulus of rupture.(N/m <sup>2</sup> )
n	: Average concentration of substance.
P	: Fractional pore volume.
P <sub>max</sub>	: Maximum applied load.(N)
R	: Resistance
r	: Pore radius.(m)
$\bar{r}$	: Equivalent pore radius.(m)
R <sub>a</sub>	: Electrical resistance of aggregate fraction.(ohm)
R <sub>e</sub>	: Resistance of electrolyte.(ohm)
R <sub>p</sub>	: Resistance of the cement paste

	: fraction or of a porous medium.(ohm)
$R_T$	: Total resistance.(ohm)
$r^2$	: Correlation coefficient.
$S$	: Silica ( $SiO_2$ ).
$s$	: Specific surface area.( $m^2$ )
$S_a$	: Non-wetting phase saturation.
s.r.	: Silica ratio.
$S_w$	: Fraction of the total pore volume which is filled with a conductive liquid.
S/C	: Sand/cement ratio.
T	: Temperature.(Degree Celcius)
t	: Time.
$T_r$	: Tortuosity.
$T_s$	: Splitting tensile stress.( $N/m^2$ )
V	: Voltage.(Volts)
v	: Volume.( $m^3$ )
W	: Weight of water.(kgs)
W/C	: Water/cement ratio.
$X_F$	: Volume fraction of conductive solids in the combination paste and solids.
Z	: Electrical impedance.(Ohm)
$\alpha$	: Temperature coefficient of resistivity.
$\alpha(r)$	: Distribution function of the pore radii.
$\beta$	: Regression Coefficient.
$\Delta(X)$	: Rate of change of X.
$\delta_F$	: Resistivity of conductive rocks as distributed.(Ohm.m)
$\delta_P$	: Resistivity of the paste as distributed.(Ohm.m)
$\phi$	: Fractional volume of cement paste.
$\epsilon$	: Dielectric constant.

$\lambda$	: Variable associated with patina aggregates.
$\mu$	: Permeability.(m/s)
$\rho$	: Resistivity.(Ohm.m)
$\rho_A$	: Resistivity of aggregate.(Ohm.m)
$\rho_c$	: Resistivity of concrete.(Ohm.m)
$\rho_e$	: Resistivity of an electrolyte.(Ohm.m)
$\rho_F$	: Bulk resistivity of the conductive solids.(Ohm.m)
$\rho_M$	: Resistivity of the mix.(Ohm.m)
$\rho_p$	: Resistivity of paste.(Ohm.m)
$\rho_S$	: Resistivity of conductive solids and electrolyte.(Ohm.m)
$\sigma$	: Electrical conductivity.(Siemen/m)
$\omega$	: Angular frequency.(Hertz)
$\psi$	: Electrical potential.
$\Psi$	: Sphericity.



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## ABSTRACT

This thesis is concerned with studies of the electrical resistivity characteristics of concrete and the possibility of applying them in assessing mix properties and quality.

The various existing concrete testing techniques are reviewed and the need for a novel approach justified.

The mechanics of electrical conduction through cement pastes and concrete is investigated in relation to the physio-chemical changes occurring during hydration.

A theory is put forward for the conductive process, based on the pore size distribution in the hydrating cement paste matrix. The theory is verified by experimental results.

A detailed description of the development of an automated system for the resistivity testing of concrete is given. The system, involving a hoist/tank layout, allows continuous monitoring of up to 15 separate experiments.

A number of cement pastes, mortars and concrete mixes are tested and their electrical resistivity characteristics investigated. From the output data empirical relationships are obtained which relate resistivity measurements to concrete mix proportions and compressive strengths.

**CHAPTER ONE**

**INTRODUCTION**



## INTRODUCTION

Growing dissatisfaction with the cube test, combined with an increasing awareness of the need to provide early and effective quality control information have resulted in a new impetus towards the developments of novel tests for concrete. The main disadvantage of presently used methods for testing concrete is that only after the concrete has been placed and cured, will test results be available. Other significant disadvantages include the non representative quality of the results obtained, as the cube is not from the structure; concrete quality may suffer during transportation, placing, vibration and curing.

Concrete, as a material, is very susceptible to strength variations and, even under laboratory controlled conditions, noticeable differences in quality are evident. On site, where supervision and control are less stringent, these strength variations can be two to four times as great. It is very difficult to quantify the benefits incurred in early detection of faulty concrete as very little data is available on the costs of remedial works which are required subsequent to placing.

In the majority of cases, however, large safety margins are employed at the design stage to allow for the high quality variability. Reducing the variation of quality about the mean would enhance reliability, increase the confidence of designers and ultimately result in a more economic design.

The basic lack of fundamental knowledge regarding the developing of mechanical characteristics of concrete at an early stage has lead to the widespread employment of empirical tests to monitor freshly mixed materials. These include tests such as Slump, Vebe, compacting factor and innumerable others. The degree of employment of each test varying not only from country to country, but from site to site.

These empirical tests suffer from the following disadvantages:-

1. They are not based on theory and as such should be used only as guides.

2. They are employed under a wide range of environmental conditions with no regard to the fact that fresh concrete is a very unstable material reacting rapidly to changes in temperature and humidity.
3. Whenever new circumstances arise (i.e. new information is required about the concrete) a totally novel test has to be developed.

Given that the main concern of Engineers is that the properties of the hardened concrete should be as close as possible to those specified in the design, it seems that an unduly large amount of interest has been shown in improving batching techniques. The quality control and analysis of the batched mix has been placed in a role of secondary importance.

Batching plants are now being produced which are almost totally automated and would, if employed correctly, almost nullify proportioning errors and variations in the mixing process. For the system to work however, the constituent materials should be constantly monitored for quality variations. The truth is that only very large works can afford such a high degree of sophistication.

A large amount of research has been directed towards the study of setting in freshly mixed cement based compounds. Most of this work has been carried out on a microscopic level and has advanced some way in relating the chemical reactions of the pastes with the mechanical properties of the hardened material<sup>1</sup>. The sheer intricacy and complexity of the chemistry involved in hydrating cement has, however, eluded the complete understanding of scientists for decades. In any case, it is generally believed that it is physically impossible to analyse the chemical state of a batch of concrete and as such, microscopic tests are of no direct use to the concrete user.

An approach to the problem is to bridge the gap between analytical and phenomenological approaches by means of an indirect method of monitoring the chemical changes within the material. Electrical characteristics of concrete vary in response to physico-chemical changes during hydration, and therefore offer the means of obtaining an 'overhead' picture of the evolution of

hardened concrete.

A figure of 100 million tons of cement based products a year has been put forward<sup>2</sup> as an estimate of the manufactured output of the U.K. alone, only 1% of the cost is channelled into the testing field. It would not be competent to suggest that the introduction of a new testing technique would affect this value. Results obtained, would however, add confidence in the designer's approach to design and therefore yield a more efficient use of materials and time. Recent surveys carried out by the SIRA Institute<sup>3</sup>, have brought to notice the intense interest of the majority of organisations in the development of a rapid analysis for fresh concrete.

It is on the whole, agreed that a new test would not replace the cube test as a way of confirming the structural quality of the material but would provide a much needed tool for quality control and potential performance investigations.

**CHAPTER TWO**

**CONCRETE AS A CIVIL ENGINEERING MATERIAL**

## 2.1 HISTORICAL PERSPECTIVE

Hydraulic cements, and cementitious compounds in general, have been classical building materials for many centuries. Their use as bonding agents was preceded by gravity type construction where stability of structures was achieved by the regular placing of large quantities of stone.

The use of mortars for masonry has been found in the remains of past great civilizations such as Egyptian, Greek and Roman<sup>4</sup>. The process through which it was made, however simple, was maintained with few alterations, up to the mid 18th century. It was around this period that Portland Cement was first used. Its origin is uncertain but it is most likely that various people were involved in the discovery and development of the manufacturing process. This, consists of grinding lime, or chalk, with an argillaceous material such as clay, mixing in preset proportions and burning them in a rotary kiln at high temperatures. The resulting clinker is then cooled and ground to a fine powder.

The use of concrete, a mix of coarse and fine aggregate with lime or cement is also of ancient origin. The Romans appear to have been the first people to employ it as a constructional material, some remains dating back as far as 300 years B.C. One of the finest concrete structures ever built is of Roman origin, the Pantheon in Rome. Its dome, 43.3 metres in diameter, is still the largest ever built and a wonder of Structural Engineering. Though it was built almost 2000 years ago, it shows considerable expertise in the use of concrete as a structural material. The thickness of the structure diminishes towards the top and so does the density of the aggregate employed in the mix<sup>5</sup>.

The weight of certain structures was lessened by casting hollow containers in the concrete as in the tomb of Empress Helena of Rome. Metal bars were also employed for reinforcement as in, for example, the roof of the Baths of Caracalla<sup>6</sup>.

It was also discovered that by adding pozzolana to the lime, a stronger and more durable material was produced. After its initial increase in popularity, though, the use and quality of concrete

declined right through the middle ages.

Some remains have been found in Norman castles, whose masons had found that quality improved with the addition of ground tiles and bricks. It was only with the development, in 1824, of Portland cement that concrete production gained momentum.

In these early stages, it was only in France that concrete construction was employed to any significant degree. There, existing knowledge on formwork, including slipforming, previously employed in mud construction was transferred to in-situ concrete construction. The discovery of composite steel/concrete construction helped to establish concrete as a major Civil Engineering material and led to the continuous improvement of cement and mix design.

## 2.2 THE NATURE AND PROPERTIES OF CONCRETE

The term concrete is used to define a constructional material consisting of Portland cement, water, air and mineral aggregate. More specifically it can be said to be divided into a binding agent and relatively inert mineral filler. The binding agent is made up by the cement and water fractions which, as hydration ensues, goes from being plastic in consistency to acquiring the properties of a stone like material. In commonly employed mixes, inert mineral fillers make up approximately 75% by volume of the total. Evidence shows that aggregate particles are, after batching, dispersed in a near uniform manner in the matrix composed of cement paste and air bubbles<sup>7,8</sup>.

Concrete is employed in a wide variety of situations and in a diversity of conditions. Engineers are therefore continuously faced with the problem of adapting the material to meet particular requirements. Different properties therefore assume varying degrees of importance with respect to the final product. All characteristics of concrete are affected by the type and proportioning of the individual constituents in the mix. Having established the required properties, the development of a suitable concrete is defined as mix design.

Since concrete is a material changing from a plastic to a brittle state, it is best to describe its properties in these two stages separately even though these are obviously interconnected.

### 2.2.1 Properties of Fresh or Plastic Concrete

Concrete, in a fresh state, is a relatively easy material to work with. If left for a period of time however, it will set and harden according to the external environment. The important properties at this stage of the concrete's life are workability and resistance to segregation and bleeding.

WORKABILITY - The workability of a mix can be defined as the ease with which the constituent materials can be mixed to produce concrete which can subsequently be handled, transported and placed with a minimum loss of homogeneity<sup>9</sup>. Another definition of workability is the ease with which air can be removed from the concrete to produce a fully compacted material<sup>10</sup>.

Plasticity and uniformity are essential as far as the finished product is concerned. The degree of workability required is dependent on the dimensions of the structure and on the type and quantity of reinforcement. Due to the difficulty in measuring the property directly, several empirical tests have been developed and are employed in the construction industry. None of these are able to evaluate all of the factors involved but may serve as indicators. The most important of these is the well known and widely employed slump test.

Factors affecting workability:-

- (i) Grading, shape, angularity and surface texture of aggregates.
- (ii) Water content (per cubic metre of concrete).
- (iii) The bulk densities and relative proportions of coarse and fine aggregates.
- (iv) Maximum size of aggregate.
- (v) Water absorption of aggregate.

SEGREGATION - Segregation is the loss of homogeneity of concrete caused by the constituent materials separating out and is primarily

due to size and density differences in the aggregate fractions. The cohesive bonds that hold concrete into a uniform state can be overcome by bad handling and placing. Over vibration is also a major cause of segregation in wet mixes, since large particles sink thereby displacing the mortar upwards. Segregation may also occur in dry mixes, especially during handling when larger aggregate particles tend to roll down the sides and concentrate at the bottom.

BLEEDING - Bleeding is a form of segregation occurring in the wet mixes where settlement of aggregate and fine mortar displaces water or water-cement paste upwards. This results in a weak surface liable to honeycombing, and a poor non durable porous finish. If superimposed concrete is applied unsatisfactory bonding will occur. The bleeding process continues until the mix has stiffened sufficiently to prevent sedimentation.

### 2.2.2 Properties of Hardened Concrete

Whilst the properties of fresh concrete are important at the construction stage, hardened characteristics determine its behaviour throughout its working life. As the fresh and hardened properties of concrete are interrelated, it is often the case that a compromise between the two has to be reached if a satisfactory material is to be produced. For example, it is generally known that concrete increases in strength with a reduction in water/cement ratio, this however might lead to a harsh, unworkable mix which could be difficult to place and compact.

The major properties of hardened concrete are strength, durability, permeability, shrinkage, elasticity and creep. Other properties worth noting although too specialised to be mentioned in detail include, thermal and acoustic characteristics, resistance to frost, abrasion, cavitation and chemical attack.

STRENGTH - Strength of concrete may be defined as the resistance of the material to rupture. It can be further classified as compressive, tensile, sheared and flexural according to the testing method employed.



Concrete is a brittle material whose compressive strength is approximately ten times greater than its tensile strength. It is therefore under compressive loading that concrete is generally employed.

Strength is widely recognised as being a main criterion for concrete quality for two reasons:-

- (i) In most cases it is the single most important property requirement.
- (ii) Other desirable properties, such as durability, density, permeability and so on are very difficult or impossible to measure, strength being indicative of the quality of the above parameters.

There are several contributing factors to the strength gaining characteristics of concrete. The following paragraphs describe the more significant ones.

WATER CONTENT - Once sufficient water has been provided to allow for full cement hydration, further increase will result in pore formation and a weak cement paste matrix. Low water contents, whilst increasing the strength of the paste, have the disadvantage of reducing the workability of the mix. This results in poor compactability and gives rise to entrapped air voids which also weaken the material.

AGGREGATES - Although of secondary importance compared to water content, the ratio of aggregate to cement is a major parameter affecting strength. The higher this value is, for a constant water/cement ratio and workability, the higher the strength. Other aggregate properties involved are grading, maximum particle size, angularity, surface texture, water content and absorption characteristics.

MIXING, PLACING, COMPACTING & CURING - The adequacy of all these processes is of key importance to the final strength of the material.

- (i) The concrete should be sufficiently well mixed to ensure an even distribution of aggregate and paste.

- (ii) Concrete should be placed carefully so as not to encourage segregation.
- (iii) The presence of air voids weakens the material which should therefore be properly compacted.
- (iv) Curing is essential in providing sufficient moisture and heat to adequately hydrate the cement.

PERMEABILITY - This is defined as that property of a material which allows fluid to enter its inner structure. Concrete due to its physical structure, is an inherently porous and therefore permeable material. The development of suitable mixes and production techniques has overcome this problem and concrete is now used in many situations where low permeability is of greater significance than strength.

Basic requirements for a low permeability mix are, as for strength, good quality materials in the correct proportions, effective mixing, compaction and curing. Water proofing agents have also been developed for use as concrete admixtures.

SHRINKAGE - Shrinkage is the reduction in volume of a mass of concrete caused by the processes of drying and carbonation.

Drying shrinkage refers to the reduction in water content within the cement gel causing the contraction (partly irreversible) of the calcium silicate phase (see chapter 3). As water loss occurs at the surface of a concrete specimen, a moisture gradient is established. Non uniform drying results in differential shrinkage which, in severe cases, may cause the build up of internal stresses.

Carbonation shrinkage, a process which has only recently been recognised, is caused by the chemical reaction of Carbon Dioxide with the cement constituents. It may, under certain favourable conditions, exhibit the same order of magnitude as drying shrinkage. Concrete is more susceptible to carbonation if porous and if cured at a relative humidity of around 50 per cent.

ELASTICITY - A body which returns to its original shape when the applied load has been removed is defined as elastic. Concrete, as a material, cannot be said to behave in a completely elastic manner. Creep occurs when loading is sustained and the physical structure is permanently deformed. It is nevertheless convenient in structural analysis to treat the material as elastic, defined by the relationship between applied stress and correspondingly induced strains.

CREEP - Creep in concrete is defined as that mechanism which allows a steady increase in strain without a corresponding increase in stress. It can be divided into permanent and recoverable creep according to the behaviour of the material upon removal of the applied load.

A principal view is that creep and shrinkage are interrelated properties in that both are affected by the movement of gel water. For creep, however, the gel water movement results from changes in applied pressure as opposed to varying hygrometric conditions<sup>11</sup>.

### 2.3 THE PROBLEM OF CONCRETE QUALITY

The characteristics of concrete should not be considered in an absolute sense but should be viewed in relation to the behavioural requirements for the finished product. Prior to the development of reliable tests, the engineer or contractor depended entirely on his own experience in judging the quality of materials. Subjective assessment will always be essential as it is impossible to quantify all the variables involved but calibrated tests are preferable. Different materials, new materials, and new uses of old materials all encourage the need for reliable, impartial tests to point the way to their correct use. This is especially true in the field of concrete construction where we see the development of bold and sophisticated structures that were not considered possible a few years ago.

The problem of concrete quality is one that cannot be overstated. Civil Engineering literature is full of examples of spectacular structural failures caused by inferior materials.

However, it is the less spectacular but more numerous small failures which have the biggest economic impact on the construction industry. Concrete is presently the only structural material to be manufactured on site, its main disadvantage being that the use of inferior materials, poor workmanship and disregard for manufacturing detail can all contribute to the production of a poor constructional material. Figure 2.1 exemplifies the problems that concrete manufacturers are faced with in trying to produce a good quality mix.

Defective concrete is, by definition, a material that fails to comply with engineering requirements and specifications. The major causes of defective concrete manufacture may be divided under the following headings:-

- (1) Mix proportioning at batching stage.
- (2) Variations in the quality of constituent materials.
- (3) Variations and/or erroneous mixing procedure.
- (4) Negligent Transportation/Placing.
- (5) Under or over compaction.
- (6) Curing Conditions.

Compressive tests on concrete cubes will only yield information concerning the first three of the above, and might not therefore be fully representative of the concrete within the structure. All six variables may only be considered to have been taken into account if the member were to be tested to failure. Indications of the in-situ concrete quality may however be obtained through the use of non-destructive and partially destructive testing techniques and by coring<sup>12,58</sup>.

#### **2.4 A GENERAL REVIEW OF CONCRETE TESTING**

The purpose of concrete testing is to ensure the production, at minimum expense, of structures whose concrete quality is uniform and of a sufficiently high standard to give satisfactory constructional behaviour. Such an objective can only be achieved through conscientious and systematic control of all operations, from the choice and production of materials to the finishing stages. Such control is evinced by the use of competent technicians working

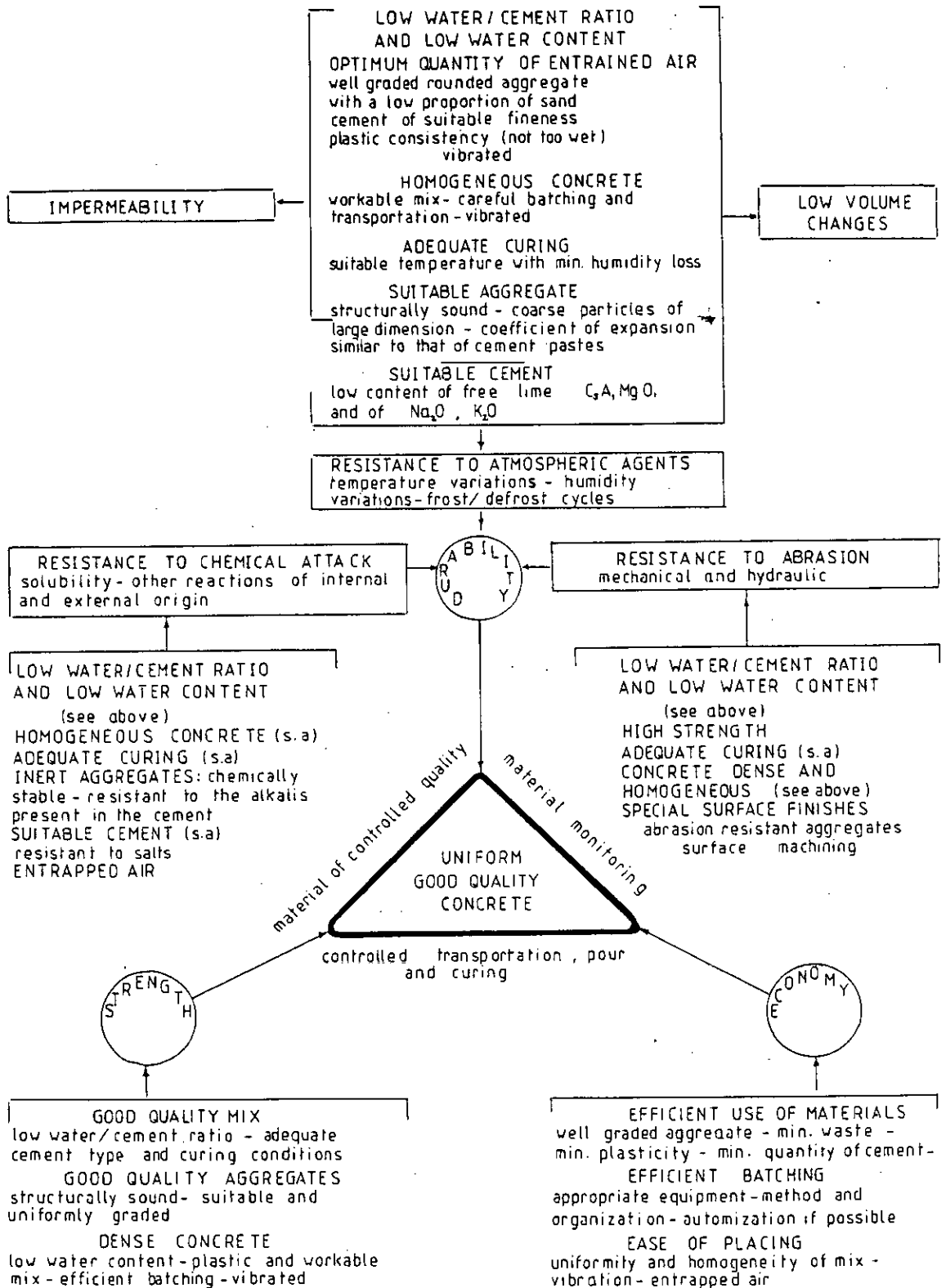


FIGURE 2.1 : Factors affecting concrete quality

within an organized schedule and under the direct supervision of suitably qualified engineers.

The testing of concrete may be divided into three basic categories<sup>12</sup>:-

- (a) Control Testing:- involves monitoring uniformity to ensure constant production of concrete with the required characteristics.
- (b) Compliance Testing:- carried out by the Engineer, it seeks to ascertain the compliance of the material with specifications.
- (c) Secondary Testing:- performed on in situ hardened concrete when (a) and (b) are either unavailable or unreliable.

Details of the most widely employed and recognized testing methods for fresh and hardened concrete are given in subsequent sections. Principal tests for both fresh and hardened concrete are listed in tables 2.1, 2.2 and 2.3 shown at the end of the chapter.

#### **2.4.1 Concrete Variability and Test Reliability**

It has long been accepted that concrete strength and durability will vary due to differences in curing, compaction and the physio-chemical characteristics of constituent materials. Knowledge and appreciation of these variations is essential to the planning of a test programme and the interpretation of results.

The nature and causes of concrete quality variations have been analysed previously whilst the degree of accuracy offered by different tests is discussed in detail in later sections. It is important that such factors are taken into account when considering the significance of test results.

Properties of concrete in a structure will inevitably be different from those of a sample which has been well compacted and cured for 28 days at optimum conditions. Given the limitations and restrictions imposed on concrete testing techniques, a number of questions arise:-

- (i) How many samples must be taken in order to obtain a valid indication of the quality of the overall work?
- (ii) How many tests are necessary to evaluate with desired accuracy a certain characteristic such as, for example, density?

(iii) How much importance should be given to a single result or to a given number of results which do not comply with pre-set limits and what provisions should be made?

None of the above questions has a straightforward answer, and in the majority of cases, it is left to the Engineer in charge, to assess the situation and act accordingly.

Concrete strength is the most common criterion for compliance testing and it is therefore usual practice, when specifying a concrete mix, to stipulate a minimum strength for the material. The mix will then be designed so that no failures, or only a limited number, fall short of requirements.

#### **2.4.2 Verifying the Quality of Fresh Concrete**

The need for accurate quality control of concrete, while still in a fresh state is indisputable; the economics of concrete structure manufacturing dictate the need for early detection of faulty material. Whilst it is generally accepted that early age analysis would not replace cube tests in so far as design requirements are concerned, it is agreed that it provides engineers with the means of obtaining, early, quality indicators.

Table 2.1 lists and comments on fresh concrete testing techniques which are either well established, new or still in the experimental stage.

##### **2.4.2.1 Workability Tests**

Many tests have been developed since the turn of the century to monitor the workability of fresh concrete. The difficulty in measuring such a complicated parameter has given rise to the employment of a whole range of tests, none of which has proved to be completely satisfactory although most are useful in providing relevant quality indicators.

The most important workability tests can be categorized under the following headings:

- |                              |  |
|------------------------------|--|
| (a) Compacting <sup>13</sup> | B.S.1881:Part2:1970 (103)                  |
| (b) Slump <sup>14</sup>      | B.S.1881:Part 2:1970 (102)<br>ASTM C143-78 |

- |                                |                           |
|--------------------------------|---------------------------|
| (c) Deforming <sup>15,16</sup> | B.S.1881:Part2:1970 (104) |
| (d) Impact <sup>17</sup>       |                           |
| (e) Flow <sup>17</sup>         | ASTM C124-39 (1966)       |
| (f) Penetration <sup>18</sup>  | ASTM C360-63 (1975)       |

Of all the methods that have been developed, none has been employed to the extent of the slump test. Although it is simple in conception and easy to operate, it is by no means a direct test for workability providing, however, useful information regarding homogeneity, consistency and cohesiveness.

#### 2.4.2.2 Compositional Tests

A wide variety of methods have been recommended in the technical literature for the determination of the cement and water contents of a fresh concrete. This not only reflects the importance of the problem but also the fact that none of the existing methods are completely satisfactory to the engineers.

Mix proportion verification includes a whole range of tests whose purposes may be summarized by the following:-

- (i) To ensure that composition complies with specifications.
- (ii) To check uniformity of batched materials.
- (iii) To evaluate the strength potential of the concrete.

The tests should be carried out before the placing of the material and it would be desirable if they were performed within 15 minutes of batching.

Mechanical Techniques:- Among the variety of physical testing methods which have been developed over the past years, that which was originally studied by Griesenauer<sup>19</sup> and Dunagan<sup>20</sup> and more recently by Kirkham<sup>21</sup>, has been considered reliable enough to be included in various standards. A detailed description of the method, which involves the sieving out of the individual fractions, can be found in B.S.1881:Part 2:1970.

The Rapid Analysis Machine (RAM) first reported in 1974 by Forrester<sup>22</sup>, is the latest evolution in the mechanical unscrambling of fresh concrete. Fully automated, it allows for a quicker testing procedure and results have been shown to be more accurate



and repeatable than those achieved through the Standard technique 23,24.

Use has also been made of ultrasonic pulse velocity and Cheeseman<sup>25</sup> has shown fresh concrete to be acoustically conductive as soon as it is cast. He was able to obtain strength predicting relationships of acceptable accuracy from readings taken as early as 5 hours after casting. Given, however, that fresh cement paste has little, if any, shear strength, it is likely that the ultrasonic pulses will be carried by the high density aggregate fraction alone. This would therefore make the test insensitive to water/cement ratio fluctuations, the most significant and common contributor to concrete quality variability.

Popovics<sup>25</sup> consistency tests for establishing the water content (W) of a fresh concrete mix may be summarized as follows:- A slump cone is made with the original mix and its displacement ( $y_1$ ), measured. A known quantity of water (W2) is then added and the test repeated. The original water content can then be calculated.

$$W_1 = \frac{W_2}{(y_2/y_1)^{0.1} - 1}$$

The test however suffers from low accuracy levels and poor repeatability.

Electrical:- An electrical conductivity test to determine the cement content of a freshly mixed concrete was first proposed by Chadda<sup>27</sup>. The test involves measuring the conductivity of water to which a known quantity of unset sand-cement mixture has been added. A pre-determined conductivity versus cement concentration relationship allows the extrapolation of the cement content of the sample. Sherwood and Fawcett's<sup>28</sup> investigation of the test's potential gave positive results. The study, also highlighted the necessity to control environmental conditions and other factors which would otherwise affect the results (i.e. variations in cement properties and dilution water employed; also, the test is specified for materials less than 1 hour old after which the hydrating process would start to interfere with the results).

Popovics<sup>26</sup> describes how use can be made of the dielectric constant to extrapolate the water content. The magnitude of this electrical parameter increasing with the amount of water present.

Nuclear:- The use of nuclear devices to determine water and cement contents of fresh concrete, although of recent origin<sup>29</sup>, has produced encouraging results. The water content may be determined by measuring the neutron moderation of a sample of the material, while tests are presently under way to investigate the feasibility of adding radioactive tracers to cement during the manufacturing stage. Cement content could then be established by radio-tracing techniques<sup>13</sup>.

Other nuclear techniques to measure cement content include gamma-ray back-scatter and absorption<sup>30</sup>, and neutron activation analysis<sup>31</sup>.

The main limiting factor in the application of nuclear techniques to the testing of concrete must lie in the level of expertise required in both the carrying out of the tests and in the interpretation of the results.

### Other Tests

Electromagnetic Radiation - in this test a specimen is irradiated with x-rays and the secondary x-rays which it emits are analysed and quantified. Each constituting element emits its own particular band which allows the operator to determine the cement content. Analysis requires approximately 5 minutes to carry out<sup>32</sup>.

Absorption Method - this technique is based on the different absorption characteristics of sand and cement. The latter, due to its greater surface area and chemical reactivity, is a better absorbent than sand, a relatively inert material. Two known quantities of sand-cement mixture and potassium permanganate are mixed for approximately half an hour and then filtered. The absorbed amount is found by back titration with a standard oxalic acid solution. The calculated value can then be related to the concentration of cement in the mixture<sup>27</sup>.

Chemical Techniques - The water content can be established by measuring the quantity of acetylene produced when a fixed amount of calcium carbide is added to fresh concrete<sup>26</sup>. A different approach by Kelly and Vail<sup>33</sup> involves the measurement of the dilution of chloride ions when a known quantity of sodium chloride is added to the fresh concrete. Subsequent studies by Howdysshell<sup>34</sup> found the results achieved with this technique representative of water content and in particular the free water in the mix.

Flame photometry has been employed in cement content verification<sup>35</sup>, by preparing a diluted solution and measuring the calcium concentration, cement content is determined by reference to a previously constructed calibration curve.

Microwave Absorption - Measurement of the absorption of electromagnetic radiation by the fresh concrete is made by determining the attenuation of the radiation as it passes from a transmitting horn antennae through the specimen to the receiving horn antennae. The attenuation is a function of the water content of the mix<sup>26</sup>.

The need to employ specialist subcontractors in the field, often requiring predetermined calibration curves, means that the above techniques are really only applicable in a limited number of situations and are of little relevancy in general practice.

#### **2.4.2.3 Setting Time Tests**

Setting time is an important parameter in concrete technology for a variety of reasons:-

- (a) As a means of checking the suitability of a mix with regard to structural considerations e.g. avoiding the cracking of the concrete deck when steel bridge girders deflect during placement of the deck.
- (b) To establish transit times. This is particularly important to ready-mix producers who have to transport the material sometimes for long distances. If the concrete is not allowed to set undisturbed, it will not reach its design strength.
- (c) As a monitor for effectiveness of set-controlling admixtures.
- (d) In preparing an accurate schedule for finishing operations such as formwork removal or slip form advancement.

The problems associated with trying to establish suitable methods for determining the setting time of concrete have been summarized by Scripture<sup>36</sup> who concluded that:-

- The setting time of cement paste does not adequately define that of concrete and misleading conclusions would result from such an assumption.
- None of the existing criteria for determining the setting time for concrete, such as workability loss, are satisfactory and will necessarily need to be defined by a particular test method or apparatus.

Setting time of cement pastes is, as defined by BS.12:1978, the achievement of a required stiffness, or rigidity, on a paste of standard consistency. The test, carried out using Vicat's equipment and procedure, basically involves the regular monitoring of the penetration of standard implements into the fresh paste until a required firmness has been achieved. When applied to concrete, however, the test suffers from a high level of inaccuracy caused by the physical presence of aggregates.

ASTM Test C403-77, lays down the procedure for determining the setting time of concrete. This is carried out by sieving out the larger particles from the concrete and then employing a Proctor penetration needle to test the remaining mortar fraction.

The difficulty in obtaining good results from mechanical tests has given rise to the development of several indirect methods of which those based on the electrical properties have been found to be the most promising<sup>37-48</sup>. The most common test is based on a marked increase in electrical resistivity at the time of final set. It should be noted, however, that electrical resistance testing is not a direct monitor of either physical or mechanical changes in the material. It is primarily dependent on the chemical composition of the aqueous phase and on the nature of the porous structure within which it is being held. Unawareness of the manner in which these variables might affect the electrical resistance of a setting cement paste could lead to misinterpretation of test results.

A more detailed description of the test, the factors affecting it and the present level of research is given in section 4.2.

Ultrasonic techniques have also received a certain amount of attention but the methods have only met with limited success due to the difficulty in passing sound waves through a liquid or semi-liquid material<sup>25,49,50</sup>.

Of the variety of other minor tests for determining the setting time of concrete, which have been listed in Table 2.2, a succinct description is given Kelly<sup>51</sup>.

#### 2.4.2.4 Air Content and Unit Weight Tests

Tests for air content and unit weight are carried out often, both on site and in laboratories. The determination of unit weight of concrete is an important control check which, combined with the knowledge of the unit weight of the constituents, provides a convenient method of approximating actual cement contents. It is also common to employ unit weight tests to determine the volume of concrete which corresponds to a certain weight of material in order to estimate the air content.

Air content has a marked effect on the properties of hardened concrete, its significance in this respect having been discussed in detail by Helms<sup>52</sup>. An increase in air content of 1%, for example, is likely to cause a reduction in strength of up to 6%<sup>53</sup>.

The following are the three most widely employed tests<sup>6</sup> of those which are currently available.

Gravimetric: B.S.1991:Part 6:1983; ASTM C138-77

Pressure: B.S.1881:Part 2:1970 (106); ASTM C231-78

Volumetric: ASTM C173-78

A review of the tests by Britton<sup>54</sup>, has shown that if the tests are carefully conducted, gravimetric, pressure and volumetric procedures give comparable results. It must be noted however, that all three tests are unable to distinguish between entrapped and entrained air and therefore provide a measurement of the total air content.

### 2.4.3 Loading Tests on Batch Samples

It is normal to estimate the strength of concrete in a structure on the basis of tests carried out on samples taken from the fresh material as it is being placed. Concrete strength, i.e. its resistance to rupture, is one basis of acceptance for concrete. Its primary requirement is obviously that of not falling below design value although it is often put to use in monitoring variations of properties which are difficult if not impossible to test directly, for example durability. Given that batch samples involve the use of a limited amount of material and that only a proportion of pours are usually sampled, loading tests should only be considered as 'indicators' of the general quality of the concrete in the structure. Also, because of the variability and non-homogeneity of the material, higher average strengths, than those required by design calculations are necessary for statistical compliance.

The fabrication of the specimen is described in detail in B.S.1881 together with compaction and curing conditions. The susceptibility of concrete strength to environmental and loading conditions is a factor which is often underrated.

It can be influenced by:

- size, shape and orientation of casting
- degree of compaction
- temperature of hardening
- moisture conditions during curing
- conditions of restraint
- disturbances during early life
- moisture conditions during testing, and
- rate of load application.

Of the above, the one which usually receives least attention, but is arguably the most important, is curing. The others being either strictly governed by Standards (i.e. size and shape of mould, compaction, loading conditions) or outwith the control of the operator (i.e. hardening time).

By curing of concrete is meant keeping it moist and, if necessary, controlling temperature so that the rate of hydration of

the cement is unaffected by environmental conditions. It can be divided into three categories:- standard, field and accelerated. Standard curing of batch samples provides potential strength values for structural concrete. It involves undisturbed storing for 24 hours at a temperature of 18 to 22°C and humidity higher than 90% followed by demoulding and curing under water at a temperature of 19 to 21°C.

Curing under field conditions implies the exposure of the specimens to the same environmental conditions experienced by the material in the structure. Loading test results for samples cured under site conditions can be deceiving because they are usually inferior to those of the material in the structure. This is due to the high surface area/volume ratio which prevails in the specimens. As loss of moisture to the environment occurs away from the surface of the specimens, sample cubes are most susceptible to drying out than, say, mass concrete pours thereby affecting the progress of hydration.

Accelerated curing comprises a wide range of procedures whose aim is to increase the rate of hydration of the cement so that potential strength values can be achieved sooner and the material be assessed quicker. Included are hot and boiling water methods, autogeneous and oven techniques. Of these, the autogeneous method has been reported as being the most exact although, if the techniques are properly carried out, all are able to predict 28-day strengths with equal accuracy. The variation in strengths detected have been shown to be the same as those obtained through 28-day or 1-year testing<sup>55</sup>.

It is usual to test non-accelerated samples at appointed ages, normally 28 days although 7-day testing is also common. Short term tests are useful during the initial stages of operation as they provide information on the suitability of different mixes. Testing samples at 7 days is excellent for this purpose although, in some cases, even younger specimens have been used with some success.

Load is generally applied hydraulically by specialised presses; care should be taken in applying the load evenly, without shocks and at a given rate (B.S.1881:Part 4:1970).

The mode of loading is dependent on the information required from the test, compression and indirect tension being the most common objectives. In structural applications where information on torsion, shear or impact strengths are required it is common to employ compressive strength as a form of indirect measurement. (More detailed information is provided in B.S.1881.) A listing of the more widely employed tests on batch samples is given in Table 2.2 at the end of the chapter.

#### 2.4.3.1 Compression Tests

Although compressive strength is widely regarded as one of the most direct property measurements in Civil Engineering, this is not so. The system of stresses imposed on the specimen is of such complexity that it defies quantitative assessment.

Platen effects are the main contributor to the deviation from uniaxial compression conditions.

Compressive strength tests can be divided into four categories according to the geometric shape of the specimen, the more popular ones being the cubes (B.S.1881) and the cylinder (ASTM C39-72 (1979)). The advantage of the cube lies mainly in the ease and accuracy of fabrication, and the fact that it can be tested between faces cast in the mould. The loaded faces of the cylinder, however, require capping, which is usually carried out with a water-cement paste.

The disadvantage of cube testing is the greater influence that platen/specimen frictional forces have on the failure characteristics of the specimen. The greater height/diameter ratio makes cylinders less susceptible to this effect and has been put forward<sup>56</sup> as a reason for the lower degree of scatter in test results.

A second reason put forward to explain the more erratic nature of cube results, is that specimens are tested in a direction normal to that of casting and tamping.

Although both methods strive to measure the same parameter, no direct link has yet been established between results obtained with



the two techniques.

B.S.1881 states that reasonable approximations are achieved if the following empirical relationship is employed:-

$$\begin{aligned} &5/4 \times (\text{C.S. of cylinder 12 in. ht. x 6 in. diam.}) \\ &= (\text{C.S. of 6 in. cube}) \end{aligned}$$

#### 2.4.3.2 Tests for Tensile Strength

Tensile strength of concrete is a parameter which is difficult to measure directly. It has so far proved impossible to produce suitably shaped samples and clamping conditions which do not induce unwanted secondary stresses. It is, however, an important property of the material determining as it does resistance to cracking. This is a particularly important consideration in underwater structural applications. In order to measure tensile strength, therefore, Engineers have had to resort to indirect techniques like flexure and splitting.

Splitting is done by loading a cylindrical specimen along its length thereby producing a tensile failure across the diameter. The test can also be carried out on cubes, either by providing semi-cylindrical loading platens, or by applying the load through opposite edges of the sample.

For cylinders, the splitting tensile strength can be calculated from

$$T_s = \frac{2P_{\max}}{ld} \quad \text{where } T_s = \text{splitting tensile strength}$$

$P_{\max}$  = maximum applied load  
 $l$  = length, and  
 $d$  = diameter.

The flexural test involves subjecting a simply supported standard beam to 1/3 point or mid-point loading until collapse ensues. Simple beam theory is then applied to obtain maximum tensile force.

$$m_r = \frac{Mc}{J} \quad \text{where } m_r = \text{modulus of rupture}$$

M = maximum bending moment

c = ½ depth of the beam

J = moment of inertia of the cross-section

Because it assumes stresses to vary linearly across the section of the beam the modulus of rupture overestimates tensile strength. Splitting tests yield more uniform results which are closer to the actual tensile strength of the material.

#### 2.4.4 Testing of Hardened Concrete in Structures

The aim of all concrete strength tests is to monitor the consistency and estimate the strength of the material in the structure. Batch samples have increasingly become the source of argument as to whether they are an effective method of compliance testing. Their unrepresentative nature, combined with the fact that no information can be deduced from them with regard to deterioration subsequent to placing, is the main cause of dissatisfaction.

A substantial amount of research and investment has therefore been recently directed towards new and more reliable testing procedures.

It is beyond the scope of this section to describe in detail the multitude of testing methods and procedures which are currently in use, this has been done in various publications e.g. Bungey<sup>12</sup> and Malhotra<sup>59</sup>. These reviews will also direct the reader towards more detailed sources of information on individual tests<sup>58,60</sup>. A comparison and evaluation of the existing techniques for assessing the strength and quality of in-situ hardened concrete is given in Table 2.3.

The tests can be classified in a variety of ways, depending for example on the type of information required, although considerations of damage caused, cost and reliability can be the overriding factors in certain cases. In general, however, the tests for hardened structural concrete can be divided into three simple categories<sup>58</sup>:-

- Destructive
- Direct strength measurement of a structural member, carried out by loading to destruction.

- Partially Destructive - Although a part of the structure is affected, damage is minimal and should not weaken the member, e.g. Core extraction, pull-off tests etc.
- Non Destructive                      - Indirect strength measurement, carried out by testing for a strength related parameter. i.e. ultrasonic pulse echo, magnetic etc.

The need for these tests arises when:-

- (a) Doubt is cast on the validity of test cube results, or measurements aside from that of compressive strength, are required.
- (b) It is necessary to assess a structure which has been exposed to fire, blast, unwarranted submerged conditions etc.
- (c) A change in the use of the structure is anticipated.
- (d) Acceptance of a structure by a purchaser or insurance company is desired.
- (e) Acceptance of a structure or structural member is required and it is known that it contains faulty material or that a design error has been made.
- (f) Monitoring the strength gained, so that formwork/slipform can be removed and prestressing or super-imposed loading applied.

The process of selecting the correct testing procedure should follow a logical progression so that, each further test, adds more reliable information whilst increasing the cost and the amount of damage caused.

## 2.5 DISCUSSION

This chapter has outlined the problems which Engineers have faced over the ages with both the production and the control of good quality concrete.

The many properties and characteristics of the material in both the fresh and hardened states have been described together with the multitude of tests in existence for assessing them.

The state of concrete testing has best been summarized by Mather<sup>160</sup>:-

....A great deal of attention is being given to perfecting methods for discovering more quickly and more quantitatively just how bad the concrete is in a structure and much less attention seems to be given to perfecting methods for assuring that the concrete in the structure will not be bad ....

Concrete is a complex, very sensitive structural material and one which cannot and should not be treated either as homogeneous or environmentally stable. Each mix, each site and each application demands particular attention.

Industry demands tests for the material. Engineers want to know whether the material is going to perform as expected once hardened and in a structure. Indirectly, however, they are asking a number of questions:-

- Does the batched mix correspond to the designed one?
- When will it be safe to remove the formwork?
- Once hardened, will it be sufficiently strong?
- Will it be permeable- etc. etc. ...

So many and varied are the questions that it would be impossible to develop a single test for all. Engineers in the past have therefore opted for simple, phenomenological approaches to their individual problems resulting in tests such as the Slump test for workability and the Vicat's test for setting time.

Accelerated curing techniques are increasingly being employed to provide the Engineer with ~~reliable~~ information regarding the quality of the material which he is using. Providing quality assessment after only the first day of curing is often suggested as an alternative although some argue that this is already too late and the only really advantageous time for testing fresh concrete is prior to placing. Whatever the views, all agree that the earlier the detection of faulty concrete is, the more economic remedial actions would be.

While still in a fresh state, hydration has not yet proceeded sufficiently for the minute physical changes to provide reliable, directly measureable, forecasts regarding the mechanical characteristics of the material.

This difficulty in determining quality control at such an early stage has been bypassed by employing either phenomenological methods or techniques which involve the separation of the material into its various constituents.

There is a need, therefore, for a test with an underlying theory, linking the hydration process to the mechanical properties. As this cannot be achieved directly, a secondary measureable parameter has to be employed.

Electrical resistivity testing, related as it is to both the physical and the chemical characteristics of a hardening concrete mix, provides a suitable means to this end. A further advantage of this technique is that, since it is non-destructive, it allows for the continuous monitoring of the hydration process.

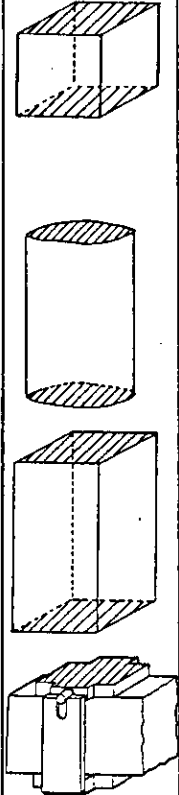
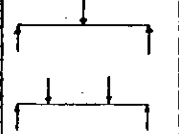
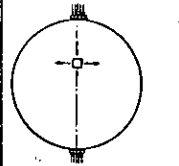
Although concrete, as a heterogeneous material is dependent on all of its constituent fractions, cement is generally regarded as the single most important one of them all. It provides the binding, strength yielding material which makes concrete such an excellent structural medium. When combined with water it also provides the electrolyte through which the largest proportion of an applied current is carried. For a thorough understanding of the hardening characteristics, the development of mechanical properties and the conductive mechanism through the material it is necessary to study the chemical composition of cements and the physio-chemical changes which occur during the hydration process; this is the topic of the next chapter.

INFORMATION REQUIRED	METHODS AVAILABLE	
WORKABILITY (Consistency- Plasticity)	Slump	Extensively employed throughout the world - good for monitoring uniformity.
	Flow	Good in assessing consistency and tendency to segregation.
	Remoulding	More sensitive to changes in air content hence the slump test.
	Compacting Factor	Good for medium consistency concretes but performs poorly in the testing of very dry mixes.
	Vebe Apparatus	A development of the remoulding test. It is particularly good in dealing with dry mixes.
	Ball Penetration	Employed mainly in the control of mix consistency.
	Thoulow's con- crete tester	Is considered to be best employed in measuring consistency of very dry mixes.
	Wigmore Consistometer	Considered better than the slump test as it comes closer to field conditions.
	Nesser's K-probe	The most recent of specialised workability tests, it is claimed to be affected by cohesive, adhesive and friction forces within the mix. Relies on Bingham's model to describe the flow of concrete.
	Tatersall's two- point tests	Sensitive to variations in water content.
SETTING TIME	Penetration Resistance	Although coarse aggregates are removed by sieving, sand in the remaining material has a deleterious effect on the results.
	Ultrasonic Measurements	Good, but equipment cost limits its widespread application.
	Electrical Measurements	Very promising early work but insufficient data on the setting of concrete mixes.
	Heat of Hydration	Too sensitive to chemical rather than physical changes in the material.
	Troweling	Very much "troweler" dependant. Satisfactory mainly because the test performed on the concrete rather than the mortar fraction.
	Bond pull-out pin method	Modifications and refinements are needed.
	Bleeding Characterisations	Because of variability in the bleeding characteristics of different concrete mixes, this method is not considered promising.
	Volume Changes	Suffers from the same drawback as the heat of hydration method.
	Strength Determination	Showed some promise but only limited results are available.
	Deformatibility	Only applicable to air entrained concrete.

Table 2.1 - Testing methods for plastic concrete

INFORMATION REQUIRED	METHODS AVAILABLE	
COMPOSITION		
Water Content	Dunagen	Although promising, inaccuracies occur in determining immersed weights and in correcting cement content to the presence of sand.
	Oven Drying	Inaccurate but simple to perform and inexpensive.
Cement Content	Chloride Titration Calcium Titration	Restrictions on their use may be imposed by some aggregate types, and extensive variations in the calcium content of the cements used.
	Flame Photometry	Accuracies of $\pm 8\%$ have been reported. Disadvantages are twofold, cost of equipment and skilled operator requirements.
	Rapid Analysis Machine (RAM)	Although arguments have been put forward on the rationality of results, this is the most promising of compositional tests available.
	Gamma Ray Back- Scatter and Absorption	Expensive, calibration is difficult and time consuming to perform.
	Neutron Activation	Measures water content.
	X-ray Spectrometer	A quick test (approx. 5 mins.) which relies on the measurement of calcium levels in the cement paste phase. Its accuracy is dependent on the calcium content of the aggregates.
	Electrical Conductivity	Test must be performed within an hour of mixing, and is sensitive to temperature variations.
	Centrifuge	Insensitive to fine aggregates with specific densities similar to that of cements.
AIR CONTENT	Gravimetric	Good in laboratory conditions but suffers serious limitations as a field test. Its accuracy is largely dependent on accurate knowledge of batch proportions, specific gravities and moisture contents.
	Pressure	Relies on the fact that air is the only compressible ingredient of a concrete mix. Sole disadvantage is that air in porous aggregates affects results.
	Volumetric	Best of the three methods. Only disadvantage lies in the amount of work needed to remove air from the mix.
UNIT WEIGHT	Gravimetric	See above.

Table 2.1 - Testing methods for plastic concrete (Continued)

TEST (PARAMETER MEASURED)	NATURE OF SAMPLE	COUNTRY OF USE	LOADING MODE	SECONDARY CHARACTERISTICS INFERRED FROM TEST
<p>COMPRESSION (COMPRESSIVE STRENGTH)</p>	<p><b>CUBE</b> 6 M. OF MAX. AGGREGATE SIZE 1½ IN. 8, 10 M. MAY BE USED IF MAX. AGG. SIZE 1/ IN. 4 M. MAY BE USED IF MAX. AGG. SIZE ¾ IN.</p> <p><b>CYLINDER</b> 12 IN. HIGH X 6 IN. DIA. OF MAX. AGG. SIZE 2 IN. OTHERWISE CYLINDERS OF UP TO 36 M. DIA. MAY BE EMPLOYED</p> <p><b>PRISM</b> HEIGHT TO CROSS-SECTION RATIO SIMILAR TO CYLINDERS, CAST WITH LARGER SIDES HORIZONTAL AND TESTED IN A POSITION NORMAL TO THIS.</p> <p><b>EQUIVALENT CUBE</b> USING PARTS OF A FLEXURAL SPECIMEN WHICH HAVE BEEN LEFT INTACT AFTER TESTING.</p>	<p>U.K. GERMANY AND MANY OTHER COUNTRIES</p> <p>U.S.A. FRANCE AUSTRALIA NEW ZEALAND</p>		<ul style="list-style-type: none"> <li>- QUALITY OF CONCRETE MIX</li> <li>- LOAD CARRYING CAPACITY OF STRUCTURE</li> <li>- TENSILE STRENGTH</li> <li>- FLEXURAL STRENGTH</li> <li>- MODULUS OF ELASTICITY</li> <li>- IMPACT STRENGTH</li> <li>- DENSITY</li> <li>- DURABILITY</li> <li>- RESISTANCE TO DETERIORATION (BY FIRE, WEAR ETC.)</li> <li>- PERMEABILITY</li> <li>- INDIRECT EFFECTS OF ADMIXTURES</li> <li>- EFFICIENCY OF CONCRETE MIXERS</li> </ul>
<p>FLEXURE (TENSILE STRENGTH IN BENDING)</p>	<p><b>PRISM</b> 28 IN. X 6 IN. X 6 IN. IF MAX. AGG. SIZE ¾ IN. 20 IN. X 4 IN. X 4 IN. IF MAX. AGG. SIZE ¾ IN.</p>			<ul style="list-style-type: none"> <li>- LOADS AT WHICH CRACKING OCCURS (WIDELY EMPLOYED IN PAVEMENT CONSTRUCTION)</li> </ul>
<p>SPLITTING (TENSILE STRENGTH IN SPLITTING)</p>	<p><b>CYLINDER</b> SPECIMEN SIZES AS FOR THE COMPRESSION TEST ABOVE</p>			<ul style="list-style-type: none"> <li>- BOND SPLITTING RESISTANCE HAS ALSO BEEN USED TO MEASURE OTHER SECONDARY CHARACTERISTICS</li> </ul>

TITLE:- LOADING TESTS ON BATCH SAMPLES

TABLE 2.2



TEST METHOD	COST	DAMAGE CAUSED	SPEED	QUALITY	INFORMATION PROVIDED
Absorption flow tests	Low	Cores required	Moderate	Comparative and difficult on site	Permeability Durability
Acoustic emission and holography	High	None		still at laboratory stage	Cracking
Break-off and Pull-off		Moderate surface damage	Moderate	Good but still at development stage Localised	Strength
Chemical Analysis	High	Moderate (cores required)	Slow	Moderate because of difficulty of tests	Cement content mix properties and constituents. Denotation
Cores	High	Moderate to high	Slow	Good	Strength Honeycombing & Compaction Mix properties and constituents
Electrical Resistivity	Moderate	Minor	Moderate	Good but applications dependent on development of cheap portable equipment	Moisture content Reinforcement corrosion Pavement thickness
Infrared thermography	High			Still at development stage	Abrasion resistance and soundness
Load test with deflection and strain measurement	High	Variable	Slow	Good	Member behaviour and strength
Load test to collapse	Very High	Total	Slow	Good	Member behaviour and strength
Magnetic	Low	None	Fast	Unreliable if reinforcement congested	Reinforcement detection
Micrometric	Low	Cores required	Slow	Moderate	Mix properties and constituents Deterioration
Microwave absorption	Low	None	Fast	As low as +30% further developments required	Moisture content
Nuclear	High	None	Fast	Still at development stage	Moisture content Cement content
Penetration Resistance (Windsor probe)	Moderate	Minor	Fast	Good +20% accuracy claimed given calibration charts	Strength
Pull-out and internal fracture	Moderate	Minor	Fast	Good but localized to surface	Strength
Pulse echo techniques	Low	None	Fast	Moderate	Honeycombing and compaction. Integrity of members, i.e. piles
Rebound hammer	Very low	Unlikely	Fast	Poor and localized to surface. Max. accuracy $\pm 25\%$	Strength Abrasion resistance and soundness
Thermoluminescence	Moderate	Minimum	Moderate	Under development Potential	Deterioration by fire
Ultrasonic pulse Velocity	Low Low Low Low Low	None None None None None	Fast Fast Fast Fast Fast	Moderate +28% Good + 15% Good - Moderate Good $\pm 5\%$	Strength Cracking Location Honeycombing & Compaction Deterioration Pavement thickness
Wear tests	High	Total	Dependent on test	Good but not widely employed	Abrasion resistance and soundness
X- and Gamma radiography	High	None	Moderate	Good but requires stringent safety precautions	Reinforcement and crack detection
Gamma Radiometry	High	None	Fast	Good	Honeycombing & compaction Density

Table 2.3: Appraisal of in-situ concrete tests

**CHAPTER THREE**

**CHEMICAL CHARACTERISTICS OF ORDINARY PORTLAND CEMENT**

**BEFORE AND AFTER HYDRATION**

### 3.1 INTRODUCTION

The performance of concrete is dependent on both chemical and physical characteristics of the binding fraction of hydrated cement, commonly referred to as cement gel. Cement, being the main ion releasing agent in the cement paste, is also the single most important contributing factor to the conductivity of pastes, mortars and concretes. The chapter has been divided into two main sections:-

**SECTION 3.2:** "Chemical and mineralogical composition of unhydrated O.P.C."

**SECTION 3.3:** "Mechanism of hydration and hardening of cement pastes".

**NOTE:-** This study is only concerned with the properties of good quality Ordinary Portland cement.

### 3.2 CHEMICAL AND MINERALOGICAL COMPOSITION OF ORDINARY PORTLAND CEMENT

Although cement is often treated and considered as a singular material, it is not so, its composition is a complex combination of ingredients all of which affect the physical characteristics of the hydrated product.

The hydraulic properties of cement depend largely on its mineralogical composition, and this in turn depends on the cement production process; knowledge of the mineralogical composition can therefore provide valuable information on quality and potential of the material. Two types of analysis can be carried out on a cement specimen to determine its composition:- chemical and mineralogical.

A chemical analysis of a typical O.P.C. would yield the following<sup>10</sup>:-

CaO	MgO	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Na <sub>2</sub> O + K <sub>2</sub> O	SO <sub>3</sub>	Ins. Resid.	loss on Ignit.
% 63.0	1.5	6.0	3.0	20.0	1.0	2.0	0.5	2

A mineralogical analysis on the same specimen would yield the following compounds:-

	$C_4AF$	$C_3A$	$C_3S$	$C_2S$
%	9.1	10.8	54.1	16.6

NOTE:- It is customary to express cement minerals in shorthand notation:-

- Lime (CaO) = C
- Silica ( $SiO_2$ ) = S
- Aluminate ( $Al_2O_3$ ) = A
- Iron Oxide ( $Fe_2O_3$ ) = F

In the U.K. the chemical composition of a cement is usually defined by three ratios<sup>62</sup>:-

$$\text{Lime saturation factor (l.s.f.)} = \frac{(CaO - 0.7(SO_3))}{2.8(SiO_2) + 1.2(Al_2O_3) + 0.65(Fe_2O_3)}$$

$$\text{Silica ratio (s.r.)} = \frac{SiO_2}{Al_2O_3 + Fe_2O_3}$$

$$\text{Alumina ratio (a.r.)} = \frac{Al_2O_3}{Fe_2O_3}$$

From these, the potential hydrated strength and the effectiveness of the combination of the different phases can be deduced.

Of particular importance is the lime saturation factor representing the ratio of the quantity of lime present in the cement to that required by the oxides to form  $C_3A$ ,  $C_3S$  and  $C_4AF$ . If the value exceeds unity, the preset maximum lime content has been exceeded and free lime appears leading to unsound cement. Conversely, a deficiency in lime corresponds to a loss of strength as lime is required to produce Tricalcium Silicate, a major strength yielding compound.

### 3.2.1 Cement Compounds and their Properties

Four compounds are usually regarded as the major constituents of cement, they are:-

Tricalcium silicate (alite) .....  $3CaO.SiO_2$

Dicalcium silicate (belite) .....  $2\text{CaO} \cdot \text{SiO}_2$   
 Tricalcium aluminate (celite) .....  $3\text{CaO} \cdot \text{Al}_2\text{O}_3$   
 Tetra calcium aluminoferrite (felite) ...  $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$

**Tricalcium silicate:-** ( $\text{C}_3\text{S}$ ) constitutes the main phase of cement clinker and is often employed singularly as the model system by experimentalists studying cement chemistry. It has been found in seven polymorphic forms so that alite composition in a cement is far from being a unique parameter. Over the past hundred years the level of  $\text{C}_3\text{S}$  in O.P.C. has risen from 14 to 45-64%, this has been accompanied by a corresponding decrease in the amount of  $\text{C}_2\text{S}$ <sup>63</sup>.

**Dicalcium silicate:-** ( $\text{C}_2\text{S}$ ) is known to have four polymorphic forms:-  $\alpha$ ,  $\alpha'$ ,  $\beta$  and  $\gamma$ . The usual form of  $\text{C}_2\text{S}$  found in Portland cement clinker is, however,  $\beta\text{C}_2\text{S}$ , commonly known as belite.

**Tricalcium aluminate ( $\text{C}_3\text{A}$ ) and Tetracalcium Aluminoferrite ( $\text{C}_4\text{AF}$ ):-** Known as the celite and the felite compounds respectively, these are sometimes referred to as interstitial phases. Although they do not contribute significantly to cement strength, they play a major part in the setting process of cement pastes. The rapid hydration rate of the celite is controlled by the addition of  $\text{SO}_3$ , in the form of gypsum, to the clinker.

The main hydration characteristics of the above compounds have been summarized in Table 3.4 while their rate of gain of strengths are shown graphically by Bogue<sup>64</sup>, see figure 3.1. Bogue's graph has, however, been obtained by testing individual cement compounds, the interaction which occurs between them during the hydration of a cement paste remains the source of intense research<sup>65</sup>.

It can be seen from figure 3.1 that, in the hydration of Ordinary Portland Cement, Tricalcium Silicate is the main contributor to early strength while Dicalcium Silicate shows a linear increase in strength between 28 days and 6 months.

**Remaining Constituents:-** The minor elements in cement composition and their respective proportion of the total as percentages are as follows:-

$\text{MgO}$	$\text{Na}_2\text{O}$	$\text{K}_2\text{O}$	$\text{Mn}_2\text{O}_3$	$\text{TiO}_2$	$\text{P}_2\text{O}_5$
1.5	0.2—1.3		0.1	0.1-0.4	0.2

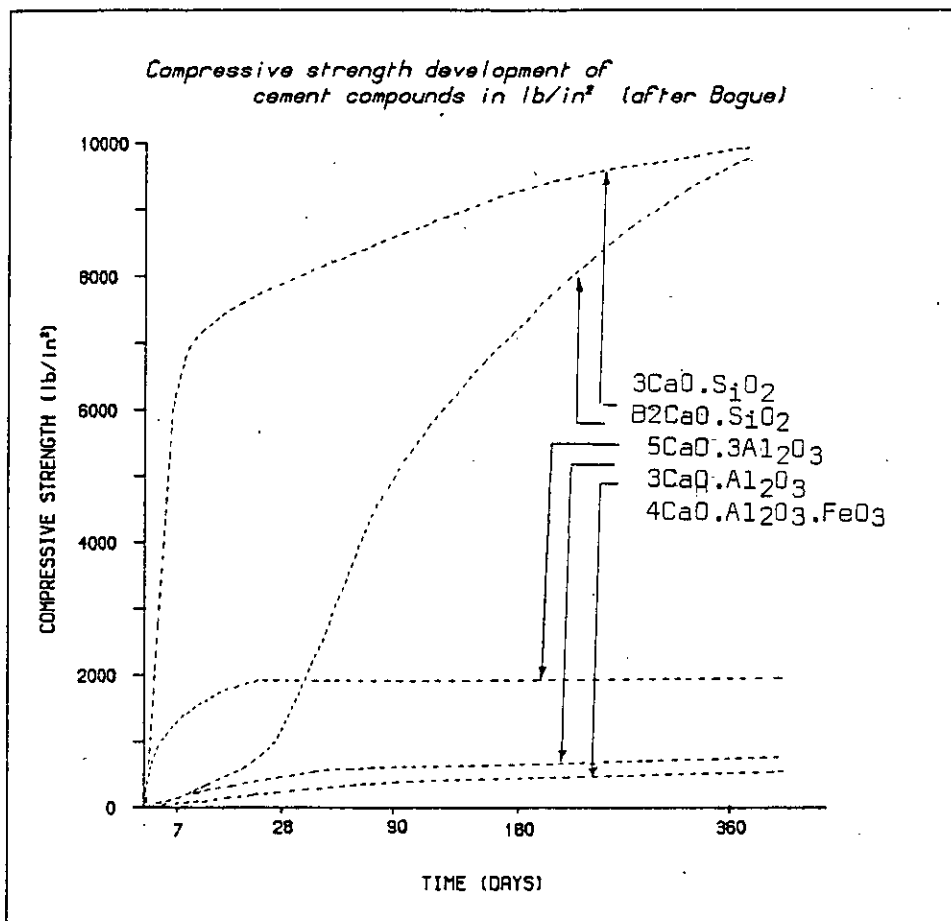


Figure 3.1 (After Bogue<sup>64</sup>)

### 3.2.2 Cement Composition Calculations

The identification and quantitative analysis of a cement by techniques such as Bogue's potential compound composition, optical point counting and x-ray diffraction have become routine procedures, each associated with a particular precision. More direct methods of separating the constituent compounds by chemical or physical means have met with only partial success due to the small size, inhomogeneity and range of properties of the crystals involved<sup>68</sup>. There is, therefore, no procedure available at present that will produce an absolute quantitative analysis of the phases present.

Of the techniques available, Bogue's potential compound composition, because of its relative accuracy and more widespread use, was adopted for the purpose of this research. This method relies on the chemical analysis of a cement sample to determine the relative

proportions of the mineral compounds.

$$C_3S = 4.07 \text{ CaO} - (7.60\text{SiO}_2 + 6.72\text{Al}_2\text{O}_3 + 1.43\text{Fe}_2\text{O}_3 + 2.85\text{SO}_3)$$

$$C_2S = 2.87 \text{ SiO}_2 - 0.754(3\text{CaO} \cdot \text{SiO}_2)$$

$$C_3A = 2.65 \text{ Al}_2\text{O}_3 - 1.69 \text{ Fe}_2\text{O}_3$$

$$C_4AF = 3.04 \text{ Fe}_2\text{O}_3$$

The table below shows how sensitive the mineralogical composition of cement is to changes in its chemical make-up:-

OXIDES	1	2	3
SiO <sub>2</sub>	20.0	22.0	20.0
Al <sub>2</sub> O <sub>3</sub>	1.0	7.7	5.5
Fe <sub>2</sub> O <sub>3</sub>	30.0	3.3	4.5
CaO	66.0	63.0	66.0
Balance	4.0	4.0	5.0

MINERALS	1	2	3
C <sub>3</sub> S	65.0	33.0	73.0
C <sub>2</sub> S	8.0	38.0	2.0
C <sub>3</sub> A	14.0	15.0	7.0
C <sub>4</sub> AF	9.0	10.0	14.0

"Table 3.1 Susceptibility of cement compounds to changes in the chemical make up" (after Czernin<sup>69</sup>).

The sensitivity to variations in the lime content are particularly noticeable. In column 2, a decrease in lime of 3% has caused a 50% drop in Tricalcium Silicate content and a fourfold increase in Dicalcium Silicate. Column 3 shows how, although not directly responsible for strength themselves, the Aluminates and Ferrites can, by the amount in which they are present, affect the cementitious qualities of the hydrated product.

### 3.2.3 Experimental Investigation of the Variability of Cements Employed

Due to the lack of information regarding the compositional

variations and uniformity of the cements used in the experimental programme, it was decided to analyse their chemical make up.

X-ray fluorescence spectroscopy<sup>68</sup> was chosen using a Phillips 1540 Automatic x-ray Spectrometer. This relies on the secondary (fluorescent) rays which are emitted by each constituent once the sample has been irradiated with a primary, high-energy x-ray beam.

This method enabled the characterization of 97% of the total composition of the cements. The only element not identifiable was  $\text{SO}_3$ , found in the gypsum and added to cement for the control of setting time, which was dispersed during the firing process. This obstacle was overcome by employing the value of 2.6% as provided by the cement laboratories for a typical O.P.C. from the Dunbar mill.

As a means of establishing the limits within which the industry maintains the compound composition of their Ordinary Portland Cements, Blue Circle Laboratories were approached for typical analyses from various works<sup>70</sup>. Table 3.2 has been drawn up from the data obtained and the following observations were drawn:-

- Substantial differences were noted in the percentage compound composition. Especially noticeable was the difference between the proportion of Tricalcium Silicate from the cement produced at Hope (63.3%) and that from the Oxford works (51.7%).
- The variation in cement composition was reflected in both the 7 and the 28 day compressive strengths of the cement pastes ( $W/C=0.6$ ) with the maximum difference again occurring between the cements from the Hope ( $46.6 \text{ MN/m}^2$ ) and the Oxford ( $41.4 \text{ MN/m}^2$ ) works.

Because of the variability in the composition of cements from different kilns, it was decided to employ material from a single source for research purposes. Also, aside from the characterization of the chemical elements present, x-ray testing was employed to monitor cement variability during the experimental programme.

Table 3.3 shows results of the analysis carried out on both the chemical and the mineralogical composition of cement samples collected during the two year testing period.



	CEMENT SOURCE							
	Cauldon	Dunbar	Hope	Masons	Norman	Oxford	Tunstead	Weardale
Type of Analysis								
Chemical: %S <sub>i</sub> O <sub>2</sub>	20.1	18.6	19.7	20.7	20.6	19.5	20.0	19.9
Al <sub>2</sub> O <sub>3</sub>	5.3	6.1	5.5	5.0	5.4	5.6	5.6	5.1
Fe <sub>2</sub> O <sub>3</sub>	3.4	3.5	2.2	2.3	1.9	3.3	2.6	2.7
C <sub>a</sub> O	63.9	63.3	65.9	64.5	65.3	63.4	64.5	63.8
M <sub>g</sub> O	1.4	2.7	1.0	1.0	1.0	1.5	1.5	2.6
SO <sub>3</sub>	2.9	2.9	2.8	2.8	2.4	2.9	2.8	2.9
L.S.F.:%								
Mineralogical: %C <sub>3</sub> S	52.3	52.4	63.3	52.8	55.8	51.7	53.6	52.4
C <sub>2</sub> S	18.2	13.8	8.7	19.5	17.0	16.9	16.9	17.5
C <sub>3</sub> A	8.3	10.2	10.8	9.3	11.1	9.2	10.4	8.9
C <sub>4</sub> AF	10.3	10.6	6.7	7.0	5.8	10.0	7.9	8.2
Compressive Strength (MN/m <sup>2</sup> )								
7 days	33.2	31.6	38.1	34.8	33.9	32.8	34.8	35.5
28 days	45.2	43.3	46.6	45.2	45.2	41.1	46.6	47.2

ANALYSIS CARRIED OUT BY BLUE CIRCLE CEMENT LABORATORIES (JULY-DEC. 1980)

ON ORDINARY PORTLAND CEMENT OF THEIR PRODUCTION FROM NORTHERN AREA HILLS

- (INFORMATION COURTESY OF B.C. INDUSTRIES)

Cement	Chemical Composition				Mineralogical Composition				
	$S_{i_2}O_2$	$Al_2O_3$	$Fe_2O_3$	$C_aO$	$C_3S$	$C_2S$	$C_3A$	$C_4AF$	L.S.F.
Hope	20.48	5.69	2.70	66.34	63.993	10.527	10.515	8.208	0.975
Wearsdale	21.08	5.78	2.49	64.98	57.154	17.405	11.109	7.569	0.931
Dunbar 1*	19.91	5.97	3.40	64.44	57.709	13.629	10.074	10.336	0.958
2	20.08	5.96	3.26	64.35	56.318	15.166	10.285	9.9104	0.952
3	18.656	5.906	3.803	63.700	64.082	5.225	9.224	11.5661	0.998
4	18.759	5.928	3.819	64.111	64.801	4.978	9.255	11.610	0.999
5	18.457	5.877	3.628	63.686	65.982	3.221	9.443	11.029	1.009
6	19.026	6.058	3.698	64.597	64.049	6.312	9.804	11.242	0.994
7	18.624	5.951	3.640	63.788	64.614	4.732	9.618	11.066	1.002
8	19.033	5.877	3.394	63.318	62.884	7.210	9.838	10.318	0.989
9	19.166	5.954	3.707	64.332	62.593	7.811	9.513	11.269	0.985
10	18.952	6.149	3.483	64.406	63.530	6.491	10.408	10.588	0.995
11	18.615	6.054	3.423	63.781	64.272	4.964	10.258	10.406	1.002
12	19.087	6.143	3.487	64.432	62.645	7.545	10.386	10.600	0.992

*\*Cement samples taken at regular intervals during the course of the experimental programme*

CHEMICAL ANALYSIS OF CEMENT SAMPLES INCLUDING MINERAGOLICAL COMPOSITION AS OBTAINED USING BOGUE'S EQUATIONS

Considering the fact that all but two of the cements employed originated from one source, variations in the compound composition were significantly greater than expected. With reference to the results obtained from the cement companies themselves and those attained experimentally, it is justified to assume that the cements employed did not possess the same strength achieving potential. The effect that the observed variations in the cement percentage compound composition had on the 28-day strength of concrete mixes could not, however, be quantitatively assessed.

Nevertheless, tests were carried out on concrete mixes of equal proportions but containing cements of differing composition (batches 5 and 8 in table 3.3). Although cement pastes might have showed up discrepancies in the mechanical characteristics, this was not evident in the concrete mixes. Too many factors can effect the hardened strength of concrete, two samples can never be prepared in the same manner and, the heterogeneous nature of the material itself, ensures a difference in the number, size and position of flaws in their structure.

Slight changes in the structure of the paste can therefore be said to be important but their significance should be assessed in the light of the heterogeneity of concrete as a material.

Concluding, the investigation points to the need for better control of the uniformity of cement production overall but, especially, within individual sources.

Anhydrous compound	Rate of Hydration	Heat Evolved	Strength Contribution
C <sub>3</sub> S	Moderately fast	Considerable over a long period	1-28 days
C <sub>2</sub> S	Slow	Little	14-28 days
C <sub>3</sub> A	Very rapid	Very considerable	Up to 1 day
C <sub>4</sub> AF	Slow	Little	No cementitious properties

Table 3.4 "Hydration characteristics of cement compounds"

### 3.3 MECHANISM OF HYDRATION AND HARDENING OF CEMENT PASTES

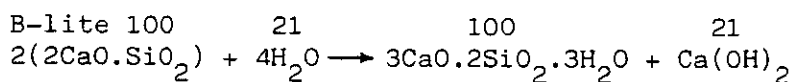
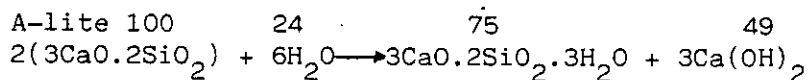
The processes of hydration and hardening of cements, although at times treated as separate events<sup>71</sup>, are really one and the same thing, the separation made when chemical or physical-mechanical changes are being analysed independently.

#### 3.3.1 Chemistry of Hydration

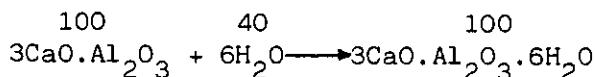
All the compounds which constitute Portland cements are anhydrous in nature and react with water at various rates to produce stable hydrates. The system does not achieve equilibrium until all the compounds have fully hydrated.

In order to simplify analysis and reduce the number of secondary reactions, researchers have resorted to studying cement constituents individually<sup>72</sup>. The Calcium Silicates in particular, being the most important strength yielding compounds, have been the source of a number of studies<sup>73,74,75</sup>.

Both C<sub>3</sub>S and C<sub>2</sub>S react with water to produce Tetracalcium Disilicate Hydrate and Calcium Hydrate as follows<sup>76</sup>:-



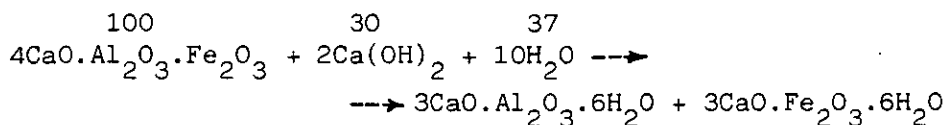
Slower in the rate of reaction, the Belite hydrates combine with Alite products to form what is generally known as C-S-H gel. Although dominated by the Calcium Silicates, the characteristics of hydrated cements are also affected by the remaining constituents. Tricalcium Aluminate reacts with water and tends to the following reaction<sup>76</sup>:-



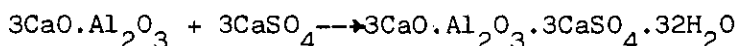
The reaction is however slightly simplified by the above equation as Tricalcium Aluminate forms unstable compounds before completing the reaction shown.

Equal doubts occur about the manner in which Tetracalcium

Aluminoferrite combines with lime and water. If, however, the hydration end products are considered, the following is a good approximation to the hydration process<sup>76</sup>.



The Calcium Hydrate used in the reaction being taken up from Silicate hydration. The above mentioned reaction of the Aluminates only gets under way once the gypsum has completed its reactions. Gypsum, approximately 3-4% is added to control the setting of the cement, and is normally exhausted within the first 24 hours of reaction<sup>77</sup>.



**precipitates as an insoluble water-rich compound  
: Calcium Sulphoaluminate (ettringite)**

It is possible that ettringite is then transformed into a sulphate poor double salt<sup>76</sup>:-  $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaSO}_4 \cdot 14\text{H}_2\text{O}$ .

The hydrolysis of  $\text{C}_3\text{S}$  causes a rapid saturation of the water with Calcium Hydroxide, other compounds dissolved rapidly being Calcium Aluminate and Alkali hydroxides. It is thought that both flash set and retardation of set result from the formation of Calcium Aluminate Hydrates<sup>78</sup>. As shown by the reaction above, the addition of gypsum controls this process by reacting with Calcium Aluminate to form either of two practically insoluble compounds<sup>78</sup>:-  
Calcium Aluminate Trisulphate Hydrate  $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$   
Calcium Aluminate Monosulphate Hydrate  $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaSO}_4 \cdot 14\text{H}_2\text{O}$

As shown by the coefficients involved in the individual hydration of the various cement compounds and by the table below, the water-binding characteristics of separate minerals differ significantly. The Silicates are however similar to each other in hydrating water requirement and so are the Aluminates.

Cement Constituent	Water bound chemically by weight
$\text{C}_3\text{S}$	24
$\text{C}_2\text{S}$	21
$\text{C}_3\text{A}$	40
$\text{C}_4\text{AF}$	37

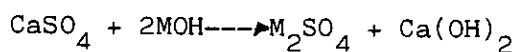
Although the figures above are not completely accurate, as the knowledge of cement stoichiometry is still limited, large changes would have to be made to alter the overall water-binding requirements which, in the majority of cases, is in the region of 28% by weight of cement<sup>79</sup>.

To summarize, the hydrogel developed in a Portland cement mix of ordinary composition is, in the main, a combination of the following phases:-

- **C-S-H (Calcium Silicate Hydrate)** is the predominant phase.
- **C-H (Hydrated Lime)** The second major constituent, is dissolved and precipitated in the process of hydration of the calcium silicates.
- **C-A-H (Calcium Aluminate Hydrate)** This is a complex phase made up of compounds formed as products of hydration of the aluminate bearing and ferrite bearing clinker constituents in a solution containing excess of calcium and sulphate ions, and also reactive silica complexes.

### 3.3.2 The Aqueous Phase

Once water is added to cement, a high alkaline solution (approximately 13 pH<sup>20</sup>) is formed by hydrolysis containing hydroxides and sulphates of Calcium, Sodium and Potassium; these tend towards the following equilibrium:-



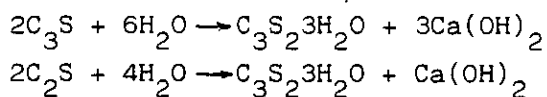
where M is either K or Na or both<sup>80</sup>.

A mere 1% of the cement is used up in this initial reaction.

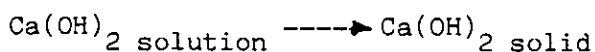
The reaction which releases the above ions is very rapid and slows down within four or five minutes of water having been added to the cement. Greenberg and Mehra<sup>81</sup> established that the aqueous phase can be regarded as being composed essentially of a supersaturated Calcium Hydroxide solution saturated with Calcium Sulphate Hydrate. Sodium and Potassium ions are also present but at concentrations well below equilibrium levels and these remain relatively constant through the early hydration stage. The aqueous phase also contains Fe<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> but, because of the extreme insolubility of their hydration products, these are present at concentrations which are low and can be disregarded.

Double<sup>82</sup> analysed solutions filtered from Portland cement paste, figure 3.2, showing rising concentrations of Calcium and Hydroxyl ions to a supersaturated peak. It was also observed that silica levels remained low throughout.

The reason for the supersaturation with hydrated lime being that, following the hydration of the two silicates:-



lime is released into the solution. The rate at which this reaction proceeds is faster than the rate at which lime can be removed from the solution by crystallization<sup>83</sup>.



Research by Lawrence<sup>84</sup> showed that, approximately five hours after the mixing with water, relatively large Hydroxide crystals were beginning to form and it is therefore probable that the fall in the degree of super saturation arises simply because of the nucleation and growth of Calcium Hydroxide crystals from the aqueous phase. The concentration of Calcium Hydroxide in the aqueous phase in contact with hydrating cement compounds can therefore be considered as an indicator to the rate at which hydration proceeds.

Another important constituent of the aqueous phase over the early, i.e. less than 24 hours, period is Sulphate which results from the dissolution of the gypsum. Double's graph shows how rapidly the concentration of Sulphate decreases as it combines with the fast hydrating Aluminates to precipitate in the form of Sulphoaluminate Hydrates. After approximately 24 hours, the Sulphate has almost completely been removed from the solution.

After the initial 24 hours of hydration the liquid phase in cement paste becomes a NaOH and/or KOH solution which is saturated, or perhaps supersaturated with respect to Calcium Hydroxide and, in general, the lime concentration continues to decrease gradually with slow further release of Alkali from the cement.

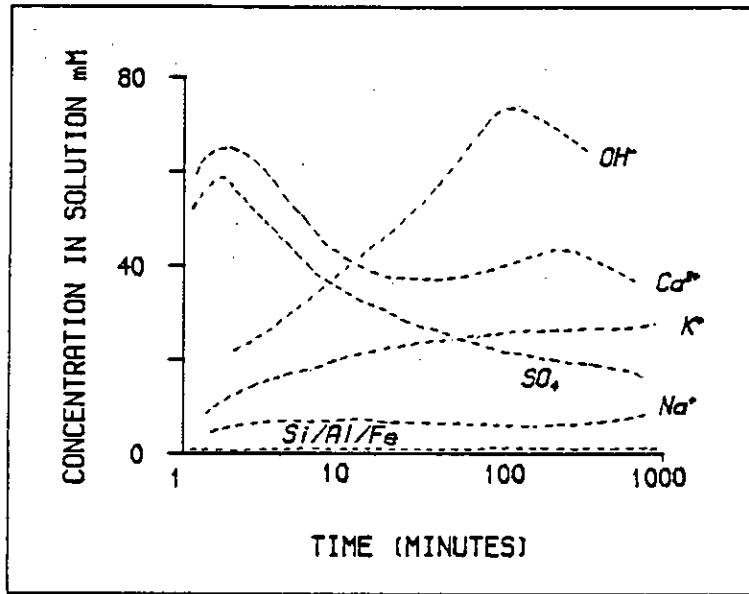


Figure 3.2 "Concentration against time curves obtained by chemical analyses of a solution filtered from a Portland cement paste (w/c ratio=2) (after Double<sup>82</sup>)"

### 3.3.3 The Development of the Microstructure

The hardening process is generally considered as a consolidating operation of a mineral skeleton, taking place by the gradual filling up of the water filled spaces with C-S-H gel and  $\text{Ca}(\text{OH})_2$  crystals. One may therefore consider cement paste to be made up of water filled pores in a matrix of continuously evolving hydrated solids.

#### 3.3.3.1 Evolution of Hydrates

Figure 3.3<sup>82</sup> shows the roles played by the individual cement compounds during hydration and the products resulting from the reaction of each of them. As can be seen from the right hand side of the figure, fully hydrated Portland cement can be divided into three main categories:- The Calcium Silicate Hydrates, Calcium Hydroxide and the Aluminate Hydrate phases.

- The Calcium Silicate Hydrates, generally known as the gel phase of the hydrated paste, are sometimes compared to the mineral Tobermorite because of similarities in their structural makeup. The gel has also been termed colloidal because of the very



small size ( $>50\text{nm}$ ) of the particles involved which are held very strongly together partly by Van der Waal forces and partly by chemical bonds<sup>86</sup>.

- Calcium Hydroxide, the solid form, is sometimes referred to as Portlandite, a naturally occurring form of the mineral. This phase has been observed to occur in different forms:- monocrystalline, macro-crystalline, microcrystalline, slightly crystallized or even amorphous. At one end of the scale it is possible to find anhedral crystals of up to 100 to 200 microns in diameter while at the other it can exist in a completely amorphous state. The latter case has been found to occur when crystallization has been disturbed by atmospheric Carbon Dioxide<sup>87</sup>.
- The Aluminate Hydrates take up approximately 7% of the overall volume of hydrated products and occurs predominantly as Ettringite, a crystal formation of Calcium Sulphoaluminate Hydrate although ferrite intrusions of the lattice have also been observed<sup>88</sup>.

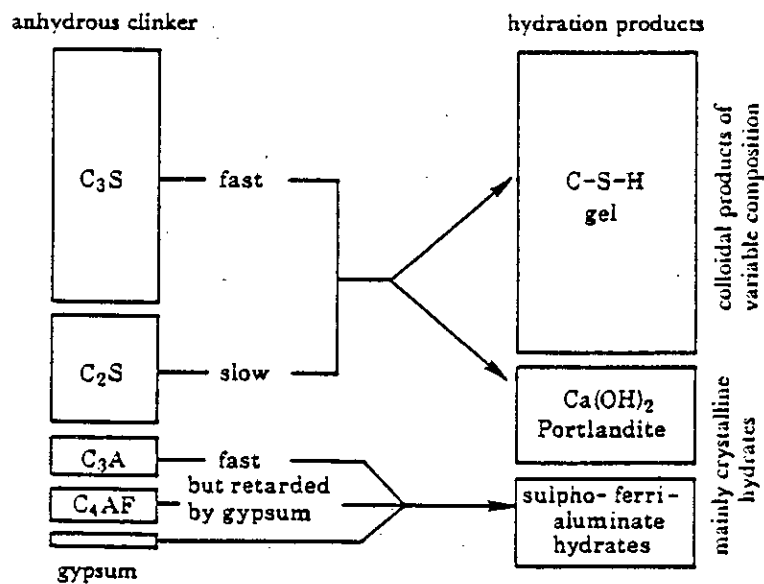
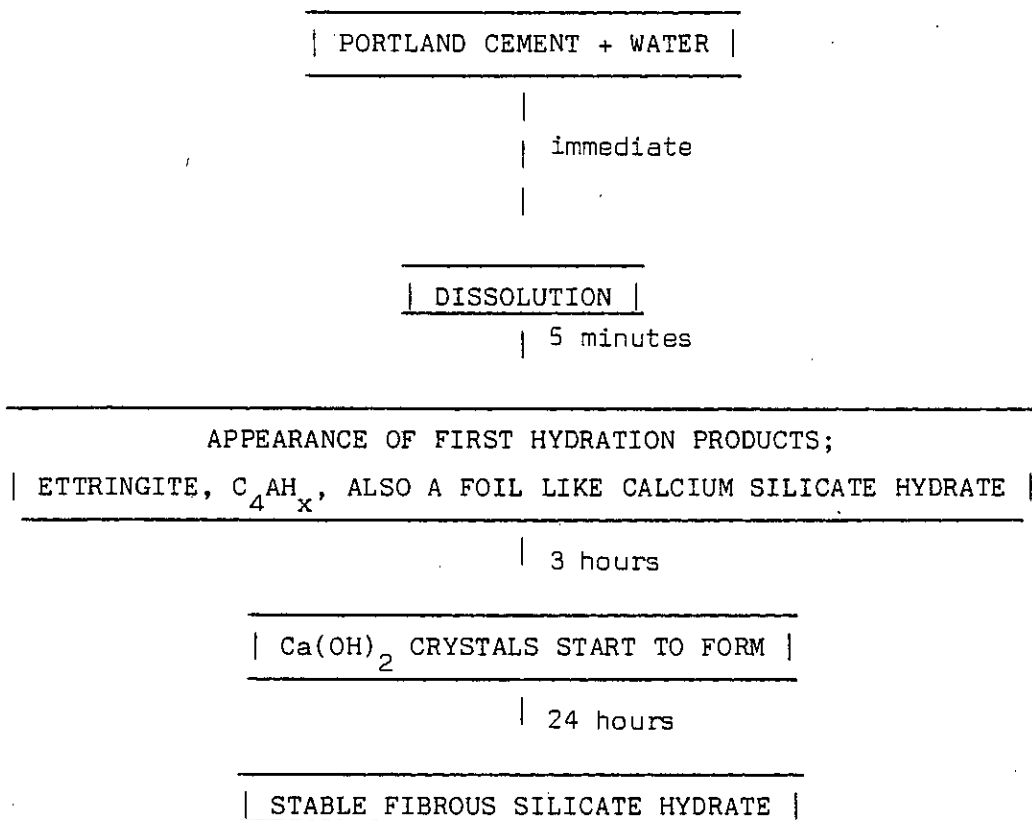


Figure 3.3 "Schematic representation of the anhydrous constituents in Portland cement clinker and the products formed during hydration. The areas of the 'boxes' give the approximate volume proportions of the phases. (after Double<sup>82</sup>)"

The early age development of the microstructure has been summarized by the flow chart below:-



No physical structure can form until the mixing is finished and the paste has been left undisturbed however, the hydrolysis of cement compounds in water begins as soon as the two are combined. Almost immediately after the rapid dissolution stage, the outer surface of each cement particle forms a film of hydrate which, by reducing the contact area, slows down the progress of hydration<sup>62</sup>. This dormant stage is defined as the induction period the duration of which is largely dependent on the cement type. Although not as rapid as the initial reaction was, the induction period involves a slow, gradual build up of colloidal hydrates forming a very weak, permeable solid on the surface of the cement particle.

The dormant stage is followed by a period of relatively high chemical activity caused by the breaking up of the hydrated surface layer. The rupture of this coating was suggested by Powers<sup>89</sup> to be the direct result of osmotic pressures on either side of the film.

This next stage of the hydration process involves the gradual loss of plasticity of the paste and lasts in the region of three hours, within which lie the arbitrarily decided 'initial' and 'final' set defining chosen degrees of firmness of the material.

Micro-graphs<sup>90</sup> taken of early age hydrated cement and hydrated clinker components show the individual particles clearly discernable with a substantial amount of space between them. As hydration progresses C-S-H gel radiates out in the form of needles crumpled or rolled foils and tubes. Rods of Ettringite and thin Calcium Hydroxide crystals are also visible, the latter as intrusions to the C-S-H gel clusters.

In hardened cement pastes, the clusters of gel which radiate from the individual cement particles join up and intertwine to such an extent that the material assumes a very compact and rigid formation with very little unhydrated constituents remaining.

### 3.3.3.2 Cement Paste Porosity

**DEFINITION:-** Porosity - Volume of voids/volume of paste

Most of the important properties of hardened concretes are related to the quantity and characteristics of the pores in the cement phase. Strength, for example, is dictated by the pore volume whilst permeability is influenced by their volume, relative size and continuity. Other properties such as durability, shrinkage and frost resistance can also be related to the pore structure<sup>91</sup>.

The following section aims to describe the way the pores originate, their nature, the factors that affect them and the limits within which they occur.

**Nature and Development of Pore Structure:-** The space not occupied by cement particles in a freshly mixed paste could be termed as the initial porosity of the system. At water/cement ratios higher than approximately 0.35 by weight, the hydrated cement compounds are unable to fill up all the volume originally occupied by water and capillary pores are formed. The gel itself is also porous, a porosity which is intrinsic of the hydration process and therefore independent of the water/cement ratio. The gel pores are

much smaller than the capillaries, only one order of magnitude larger than the size of a molecule of water, and tend to occupy a fixed volume percentage of the total gel, approximately 28%<sup>92</sup>, see figure 3.4.

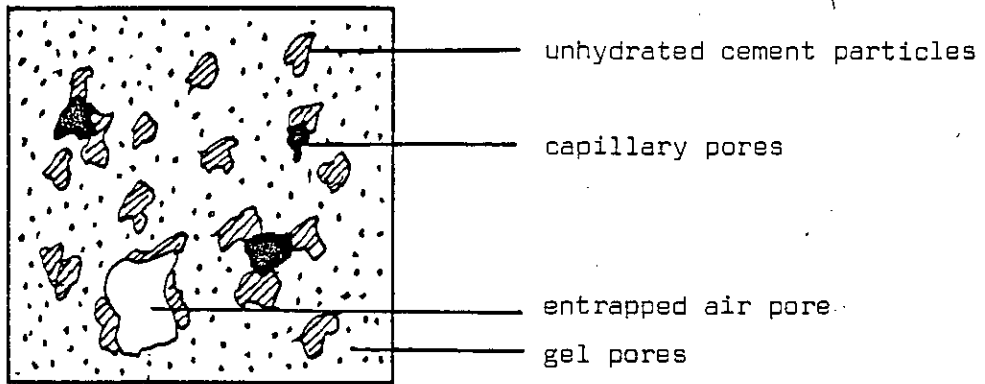


Figure 3.4

The volume of gel pores increases as hydration progresses whilst capillaries decrease in size, number and continuity. The relative volume changes in a simple Tricalcium silicate cement as a function of hydration are shown in figure 3.5.

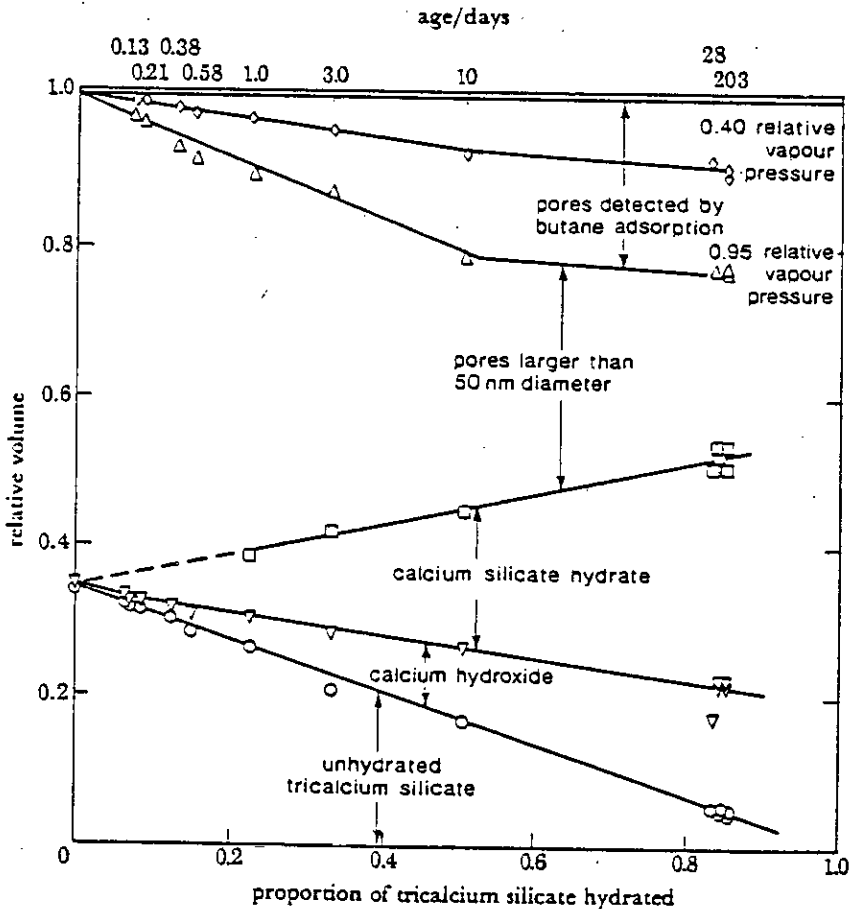


Figure 3.5 "Phases present in a simple  $C_3S$  cement as a function of hydration (W/C ratio 0.59 by mass)". (After Parrott<sup>98</sup>)

The more water is added to the paste, the larger and more numerous the capillary pores become. The system could therefore be visualized as a network of interconnected voids distributed at random through the hydrated cement paste matrix.

Capillary pores constitute 0-40% of the paste by volume, depending on the water/cement ratio and the stage of hydration, and may vary between  $8 \times 10^{-6}$  and  $1.3 \times 10^{-2}$  mm in diameter<sup>91</sup>. Pores smaller than the arbitrarily set limit of  $8 \times 10^{-6}$  mm are generally considered as gel pores whilst larger pores, up to a few millimetres in size, are attributed to poor particle packing and occluded air.

**Pore Size Distribution:-** Although, as will be shown later, it is the larger pores that determine the strength of the hydrated material, it is the more numerous smaller capillaries that occupy the largest volume. Their size range defies quantitative analysis by optical microscopy and the presence of water is an obstacle to the straightforward application of electron-microscopy.

Various indirect methods for analysing pore size and pore size distribution, such as mercury intrusion<sup>100</sup> and water vapour absorption<sup>101</sup>, are in existence but controversy surrounds measurement interpretation.

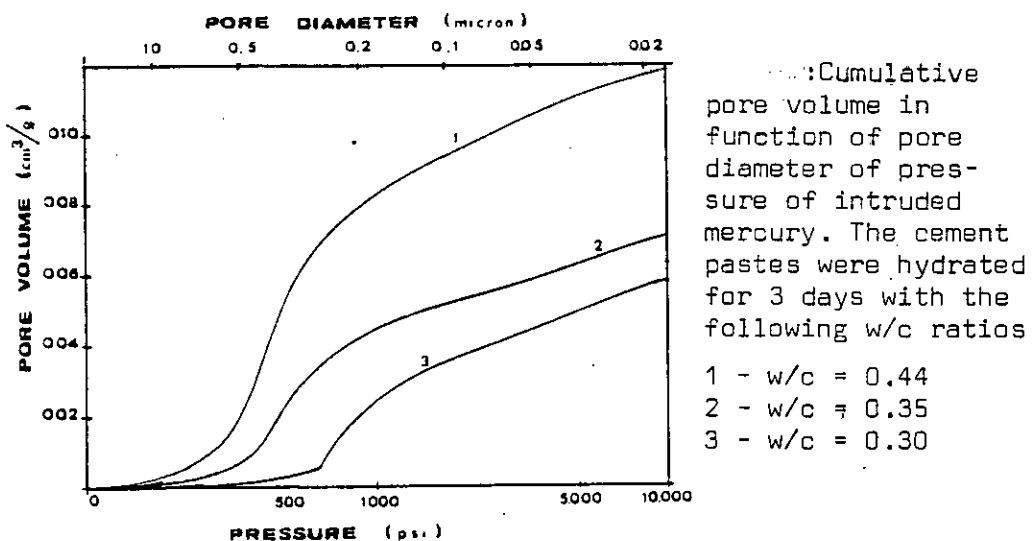
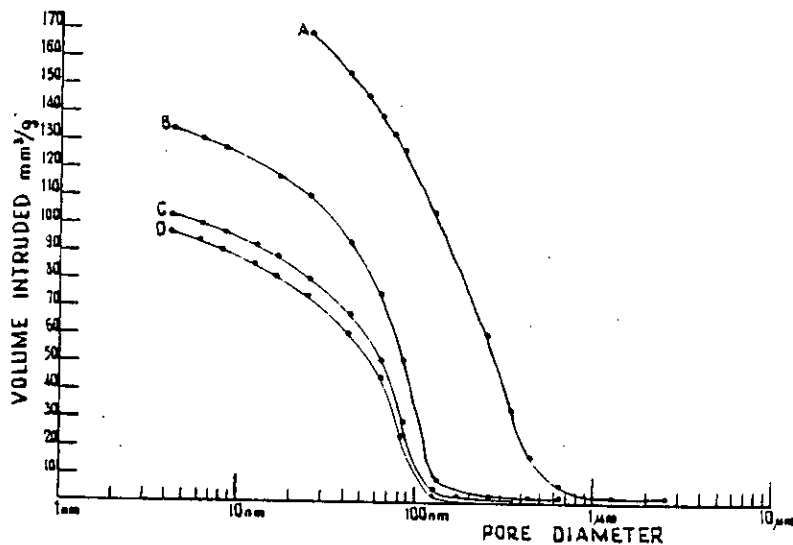


Figure 3.6 (After Collepardi & Massida<sup>99</sup>)



The effect that varying the water/cement ratio has on the pore characteristics of a cement paste is shown in figure 3.6 where the increase in bulk density, which accompanies a reduction in water content, results in a decrease in overall porosity as well as average micropore radius.

A very similar effect on porosity results from the progress of hydration of a cement paste from one to twenty-eight days, figure 3.7. It is also of interest to note the decrease in the rate of change of pore volume as hydration progresses from A to D.



- Pore size distribution curves of normally cured specimens, w/C = 0.3.

- A : 1 Day
- B : 3 Days
- C : 7 Days
- D : 28 Days

Figure 3.7 (After Kayyall & Page<sup>99</sup>)

**Porosity and Strength:** The notion that strength is not determined by the total porosity but by the large diameter pores was first expressed by Griffiths<sup>105</sup>, who investigated the failure in tension, by bending, of porous materials.

$$\sigma = \frac{ER}{\pi C_L}$$

where  $\sigma$  = strength,

E = Young's modulus

R = Fracture energy, and

$C_L$  = Length of pore.

It can be inferred from the above that strength decreases as flow size rises whilst total porosity is of secondary importance, determining as it does, the statistical probability of a crack of length  $C_L$  occurring within the stressed region<sup>97</sup>.

A significant conclusion to be drawn from Griffiths theory is that, in the presence of large pores, the microstructure and morphology of the hydrated phases can have but a secondary influence on the failure characteristics of hardened cement paste.

In order to reduce the size and proportion of large capillary pores, decreasing the water/cement ratio is often counter productive as it significantly affects workability thereby allowing for the formation of occluded air cavities. Various techniques have been developed to optimise pore size distribution such as:- high energy mixing, gap graded cement and chemical additives<sup>94</sup>. Admixtures, such as dispersing agents and the addition of fine silica have resulted in concretes of strength in excess of 250 MPa<sup>95</sup>. Water soluble organic polymers<sup>96</sup> have also been recently employed to reduce water requirements and improve particle lubrication with excellent results (total porosities less than 1%)<sup>93</sup>. The processed material is, however, very expensive as a substantial proportion of the cement is effectively employed as micro-aggregate and does not hydrate.

**Porosity and Permeability:** Fluid flow through concrete is similar to that through any porous body in that it is not a simple function of porosity but is related to pore size, distribution and continuity. From the practical point of view, cement paste permeability may be considered to depend on capillary porosity only, as the coefficient of permeability of gel is exceedingly low ( $7 \times 10^{-16}$  m/s as opposed to  $1 \times 10^{-13}$  m/s)<sup>92</sup>.

In concrete, all the permeating water must pass through the cement paste phase and, if the latter is of low permeability so is the concrete.

Water flow through cement pastes is, in the main, a function of two factors:- water/cement ratio and curing time.

Given that permeability increases with voids ratio, reducing the water/cement ratio will directly lessen permeability, in fact a decrease from 0.8 to 0.4 would reduce permeability by approximately a thousandfold<sup>91</sup>. If, however, the water content is too low for complete compaction, the matrix will be riddled with macropores and, the high proportion of cement, will produce excessive hydrating contraction and result in shrinkage cracks<sup>7</sup>. The manner in which water/cement ratio affects permeability for a fully compacted mix is shown in figure 3.8<sup>104</sup>.

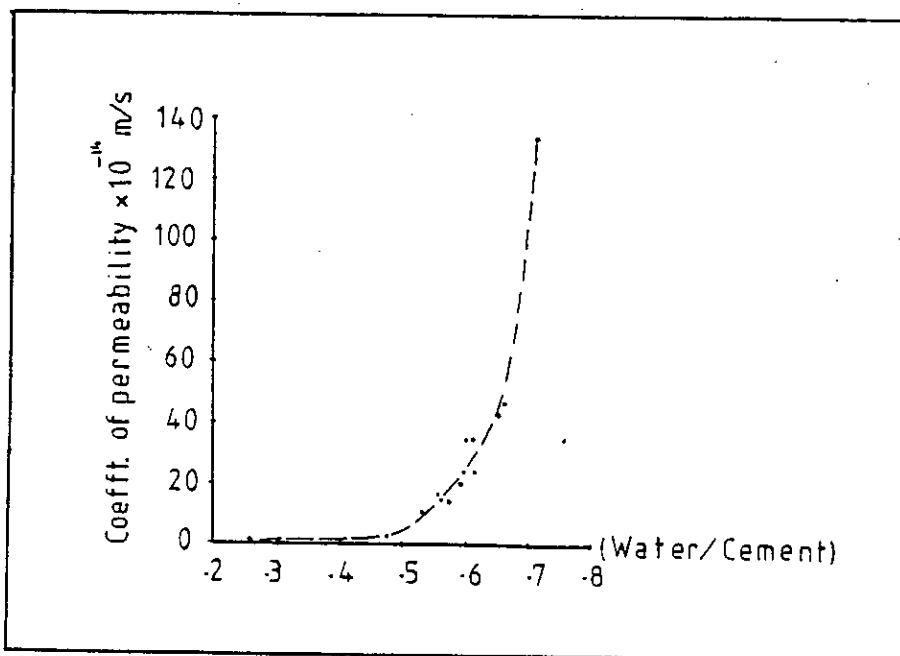


Figure 3.8 "Permeability response to changes in water/cement ratio of hardened cement pastes" (After Woods<sup>104</sup>)

As hydration progresses, permeability can drop dramatically; a paste with a water/cement ratio of 0.8 will in fact become a thousand times less permeable between the seventh day and the first year of curing<sup>91</sup>. This decrease in the coefficient of permeability with time is due to the capillary ducts becoming blocked with hydration products and no longer forming a continuous system.



Experimental and theoretical analysis by Powers<sup>102</sup> has yielded the following times at which capillaries become segmented for different water/cement ratios:-

Water/Cement Ratio	Time
0.40	3 days
0.45	7 days
0.50	14 days
0.60	6 months
0.70	1 year
over 0.70	Impossible

There is evidence, however, that even when the capillary system becomes 'discontinuous' (as measured by water permeability) there still remains some continuity in the large pore system<sup>91</sup>. This would account for the spontaneous inward movement of freezing in mature cement pastes. The number of continuous paths is seemingly too low to significantly influence permeability measurements. Figure 3.9, drawn using data obtained by Powers<sup>103</sup>, shows clearly the decrease in both porosity and permeability as hydration progresses for a paste with a water/cement ratio of 0.645.

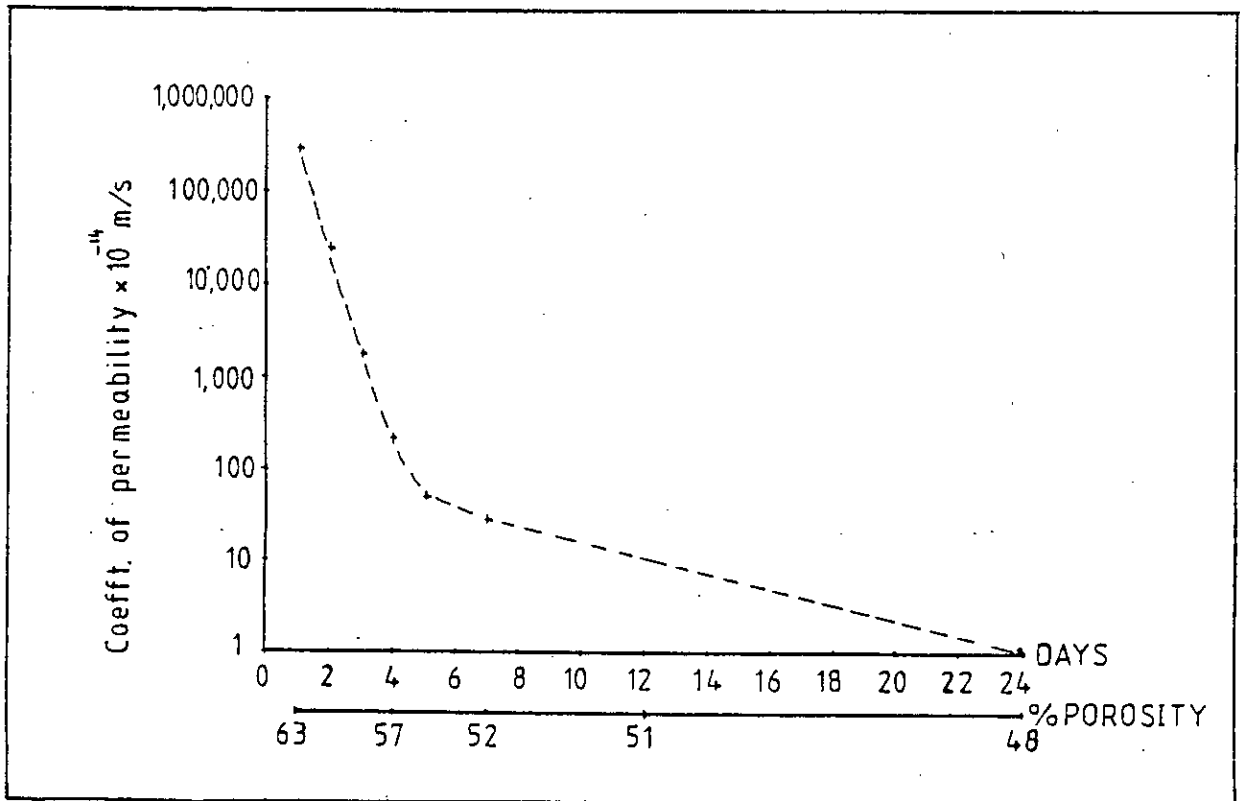


Figure 3.9 "Effect of hydration on porosity and permeability" (w/c ratio = 0.645) (After Powers<sup>103</sup>)

### 3.4 SUMMARY AND CONCLUSIONS

The aims of chapter three have been to:-

- (a) Study the composition and variability of Ordinary Portland cement,
- (b) Investigate the hydration process,
- (c) Establish which of the cement compounds are responsible for the gain in strength and how the latter is dependent on variations in percentage compound composition, and
- (d) Relate hydration to the formation of the solid structure and to subsequent physical properties, i.e. strength and permeability.

It has been shown that, in order to produce a cement of uniform strength gaining characteristics and potential, mineralogical composition must be maintained within strict limits. It has also been established that knowledge of the cement compound composition can yield information regarding the quality and strength of the hydrated cement.

An important conclusion drawn from the study of the physical characteristics of a hydrated cement paste was that, in the presence of structural flaws such as pores, the morphological properties and inherent strength of the cement paste matrix are of secondary importance with regard to the failure of the system under stress.

Workability requirements, set by the vast majority of structural engineering works, dictate the use of water/cement ratios which are much higher than needed for the hydration of the cement. The net result is a concrete whose binding matrix is riddled with pores of various sizes.

Like strength, the resistance offered by concrete to the flow of an electrical current has also been shown<sup>118,122</sup> to be largely dependent on the presence of a porous structure in the cement paste matrix. It is on this common dependence on the pore system of the cement paste that the relationship between physical properties and electrical resistivity of concrete is based.

Having established links between the chemical composition of O.P.C., the hydration process and the physical structure of the

hydrated paste, the aim now must therefore be to investigate the conduction mechanism through concrete and the different factors which can affect it.

This, together with a review of the research carried out to date on the subject, is the topic of chapter four.

**CHAPTER FOUR**

**ELECTRICAL PROPERTIES AND TESTING OF CONCRETE**

## 4.1 ELECTRICAL CONDUCTION THROUGH CONCRETE

### 4.1.1 Introduction

The resistance of a conductor is dependent on:-

- (1) The temperature,
- (2) The material of which the conductor is made,
- (3) The length of material through which the current is made to pass,
- (4) The cross-section.

At a given temperature the resistance,  $R$ , is directly proportional to the length,  $l$ , traversed by the current, and is inversely proportional to the area of the cross-section,  $A$  at right angles to the direction of the current.

Hence:-

$$R \propto \frac{l}{A} \text{ or, } R = \rho \frac{l}{A}$$

The proportionality constant,  $\rho$ , is only constant for the particular substance of which the conductor is made and is known as resistivity.

The conductivity of a material is defined as the reciprocal of its resistivity, i.e.

$$\sigma = \frac{1}{\rho} \text{ siemens.m}^{-1}$$

Conduction of electricity through a heterogeneous, time-dependent material such as concrete is a function of two factors: Ohmic resistivity and Ionic conductivity. Ohmic resistivity is dependent on:

- (a) The fractional volume of aggregate, and
- (b) The cross-sectional area left available for conduction by the non-conductive cement paste compounds.

Concrete may be visualized as consisting of aggregate particles embedded in a cement paste matrix. The latter may also be sub-divided into a solid phase of hydrated and unhydrated cement compounds and a liquid phase consisting of ions in an aqueous solution, see Fig. 4.1.

The ability of the liquid phase to transport electrical charges is dependent on the type, concentration and speed of the ions available in the solution. To simplify the analysis, electrical conduction through concrete is divided into three levels:-

- (i) **MACROSCOPIC** - Aggregate particles embedded in a cement paste matrix.
- (ii) **MICROSCOPIC** - Physical structure of the cement paste phase.
- (iii) **SUB-MICROSCOPIC** - Electrolytic characteristics of the aqueous phase.

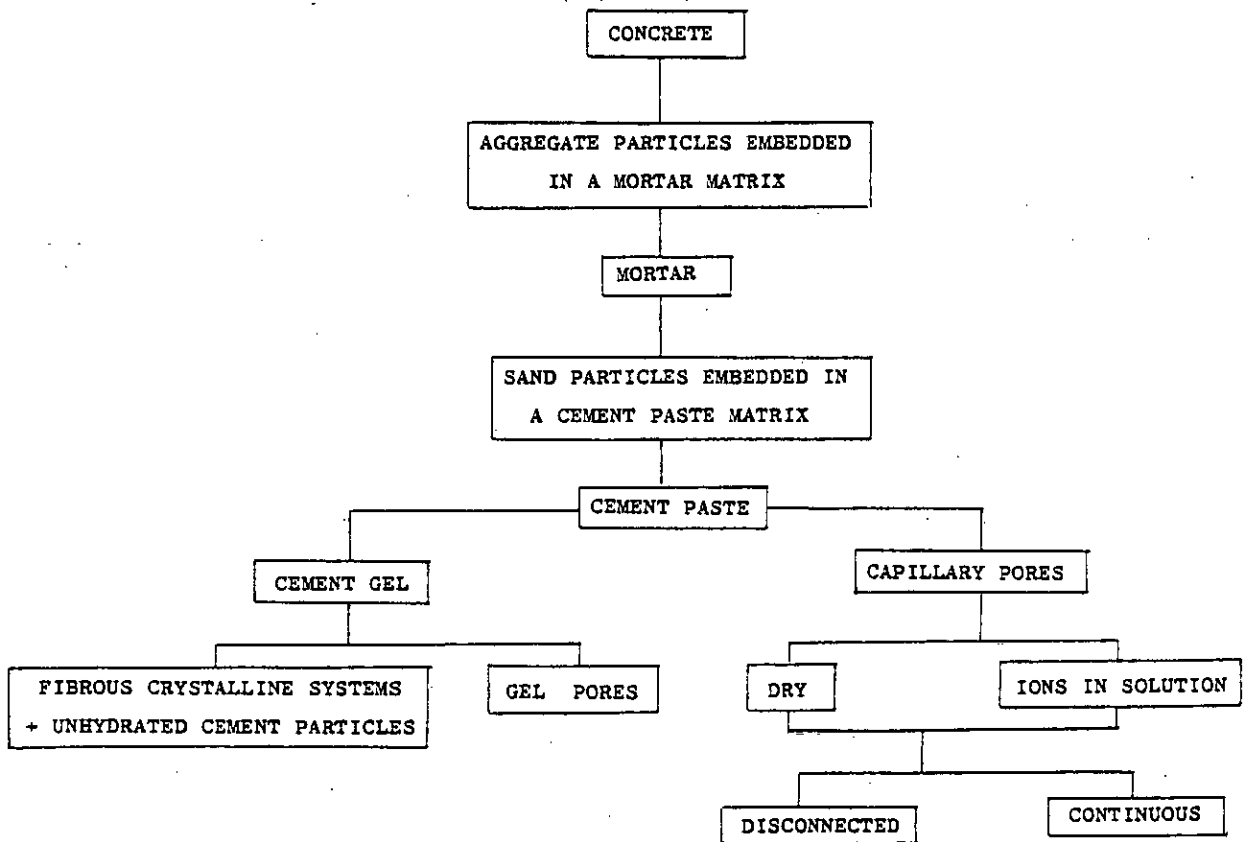


Figure 4.1 Schematic breakdown of concrete composition.

#### 4.1.2 Macroscopic Level

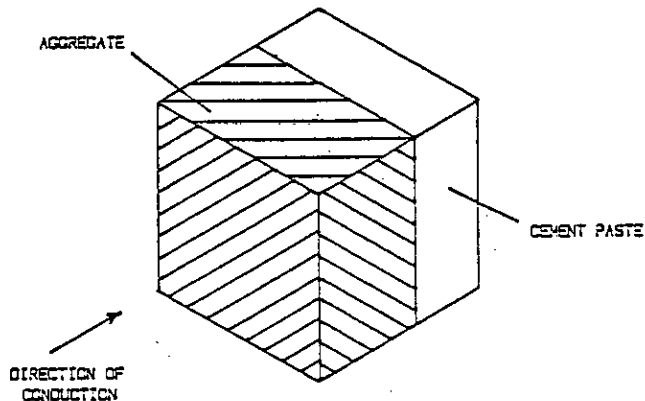
##### "The Effect of Aggregate on the Conductive Properties of Concrete"

Aside from the particular cases where specially developed conductive aggregates are employed, rock aggregates are, in general, very poor conductors and their direct contribution to the

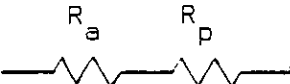
electrical conductivity of concrete is therefore very limited. Because of the much lower resistance offered to electrical flow by the cement paste compared to normal aggregates (approx. 100 times lower)<sup>106</sup>, the latter are considered as simple obstacles to the flow of electricity which is, on the main, carried by the aqueous phase in the cement paste fraction.

In order to model this insulating effect electrically, two approaches are possible<sup>107</sup>:

- (a) Consider the aggregate as lying in a single block across the path of electrical flow:-



The length of the block (l) being related to the volumetric fraction of aggregate in the mix.

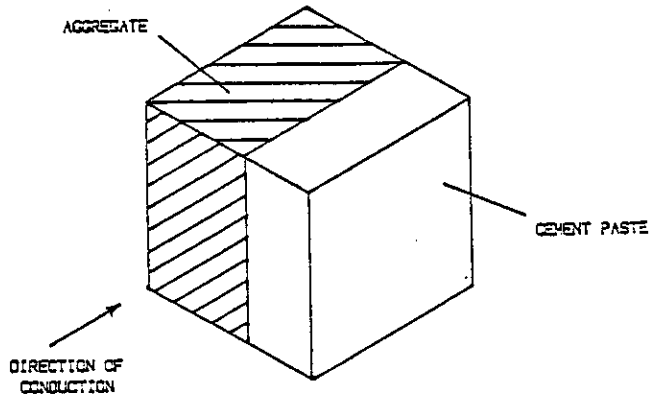
Equivalent electrical representation = 

Where  $R_a \gg R_p$

Total Resistance  $R_T = R_a + R_p$

Because of the high resistance offered by the aggregate, the conductivity of this model will be very low.

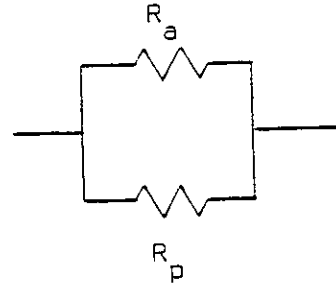
- (b) A second model could be visualized as having the aggregate still in a block form but this time in parallel with the electric field:-



In this case, it is the cross-sectional area of the block which is related to the volumetric fraction of aggregate.

Equivalent electrical representation =

$$\frac{1}{R_T} = \frac{1}{R_a} + \frac{1}{R_p}$$



As electricity flows along the path of least resistance, the greatest proportion of the current passes through the cement paste phase and only a small fraction through the aggregate (see sect. 4.1.5). Model (b) would therefore yield a much lower resistivity value than model (a).

For practical purposes however, both models suffer from inaccuracies:-

Model (a) 'forces' the flow through the aggregate and assumes that the aggregate offers a continuous conductive path while, in reality, each particle is coated with a cement paste layer. Model (b) is much nearer the real situation whereby the cement pastes provides a continuous route through the concrete, but it is also an over simplification as it neglects the physical aggregate-paste interaction.

Various researchers have adopted model (b)<sup>108,106</sup> as the basis



of empirical relationships for the conductivity of concrete. These have, on the main, been concerned with either fresh or hardened concrete, the transitional period between the two having received no attention from a modelling aspect.

#### 4.1.3 Microscopic Level

##### "Conductive Process through the Cement Paste Phase"

In conjunction with the aggregates, further ohmic resistance is caused by the physical restriction to the passage of current through the formation of the hydrated micro-structure. Electrical conductivity tests on oven dried cement paste samples have shown them to be 10,000 times less conductive than their damp counterparts thereby classifying the solid phase of the cement paste in the insulator category<sup>108</sup>. At a microscopic level, cement paste consists of a system of capillary pores, water filled or dry, through the cement gel. At a smaller level, the gel itself is made up by a network of crystalline formations and unhydrated cement particles making up a less continuous system of minute water-filled voids, the gel pores.

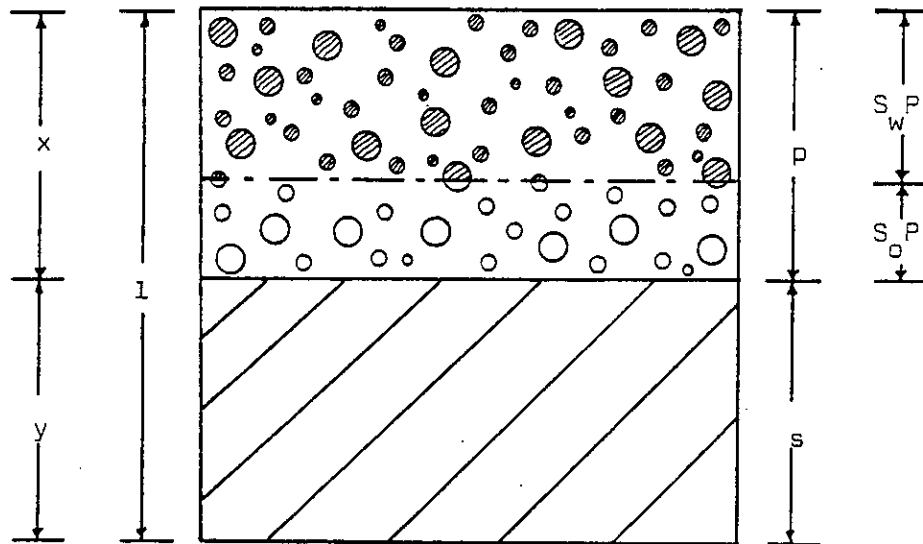
Given the complicated layout of crystal formations, it is impossible to adopt a strict geometrical approach to the solid to void ratio and, electrical measurements, are therefore confined to monitoring reductions in the effective cross-sectional area available for conduction.

##### 4.1.3.1 Relating Resistivity to Pore Characteristics

The specific resistance of hardening cement stone and concretes of a given composition is dependent on the density of the mix and on the quantity of free water. Increases in resistivity are caused by both the increment of the solid phase (increase in density) and by the dehydration of the pores as a result of evaporation or autodehydration by contraction. The objective of the following section is to relate the effective cross-sectional area to the effective pore radius, the pore saturation and the probability of pore continuity.

The following is an adaptation of Kozeny-Carman's equation for fluid flow in a multiphase porous material<sup>109</sup>.

Consider concretes, mortars and pastes to be divided into two distinct fractions, solids and pores. Furthermore, assume that not all pores are filled with a conductive liquid but only a fraction,  $S_w$ , of the total pore volume.



/// = Solids (i.e. coarse and fine aggregates, unhydrated cement, hydrates)

○ = Pores

⊙ = Fluid filled pores

$S_w$  = Actual-wetting phase saturation

$S_o$  = Non-wetting phase saturation

**Figure 4.2: Idealized capillary model of concrete**

Pores are idealized to be circular in cross-section and to have radii varying between  $r_o$  and  $r_{max}$ , according to a distribution function  $\alpha(r)$ .

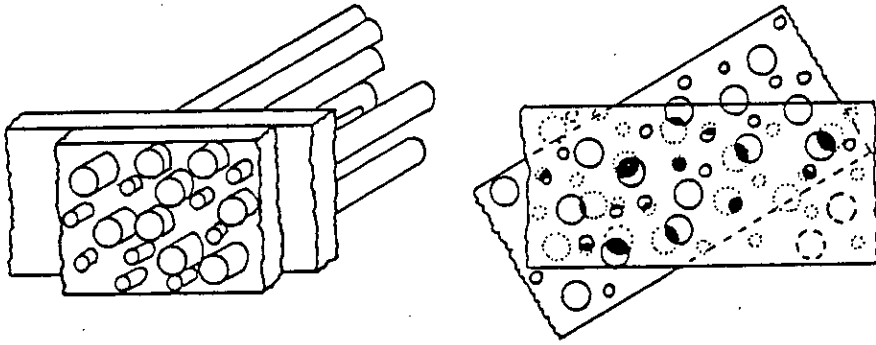


Figure 4.2(a)

The bundle of capillaries can then be pictured to be divided into thin slices as in figure 4.2(a) above. Each slice contains an equal amount of electrolyte:-

$$a = A.P.S_w \quad \begin{array}{l} A = \text{Total cross-sectional area of the slice} \\ P = \text{Fractional pore volume} \\ S_w = \text{Actual wetting phase saturation.} \end{array}$$

The probability that a random point on the surface of a slice lies within the wetted fraction is  $(PS_w)$ . The probability that it lies in the area occupied by liquid in a succession of  $n$  number of slices is  $(PS_w)^n$ . The factor  $PS_w$  could be termed as the constricting coefficient; a pore, of cross-sectional area  $\pi r^2$  would be constricted to a fraction  $PS_w$  of its area.

The equivalent radius could then be expressed as:-

$$\bar{r} = \frac{(PS_w)^{1/2} r}{k^{1/2}} \quad \text{For each of the slices}$$

where  $r$  is proportional to  $(PS_w)^{1/2}$  since the cross-sectional area is taken as  $(PS_w) \cdot A$  and  $k$  is a numerical constant reflecting the manner in which the available interconnecting pores are divided, ( $k \geq 1$ ).<sup>109</sup>

$$a_e = \pi \bar{r}^2 = \pi \left( \frac{(PS_w)^{1/2} r}{k^{1/2}} \right)^2 = \frac{\pi PS_w r^2}{k}$$

where

$a_e$  = effective cross-sectional area  
summing for all the pore sizes,

$$\bar{a}_e = \frac{\pi(PS_w)}{k} \int_{r_0}^{r_{\max}} r^2 \cdot \alpha(r) dr$$

The above equation shows the following:-

- $\bar{a}_e$  increases with the number and size of pores present in the paste matrix.
- $\bar{a}_e$  is constricted by a factor  $(PS_w)$ , the fractional cross-sectional area of fluid filled pores.

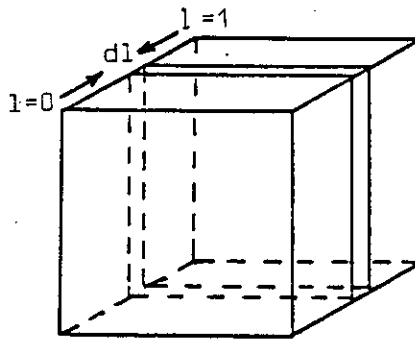


Figure 4.2(b)

V = Effective volume of liquid filled pores

$$V = \bar{a}_e \int_{l=0}^{l=1} dl = \bar{a}_e$$

$$= \frac{\pi PS_w}{k} \int_{r_0}^{r_{\max}} r^2 \cdot \alpha(r) dr$$

#### 4.1.3.2 Electrochemical Structure of Cement Paste

The electrochemical characteristics of a cement paste are time dependent and, in order to simplify the analysis, the material is best divided into three broad categories:- unhydrated cement compounds, solid by-products of hydration and the aqueous phase.

Although semi-conductive properties have been reported for certain hydrated cementitious compounds<sup>85</sup>, both the unhydrated cement and the solid hydrates are in general regarded as insulators due to their low ability to carry electrical charges.

The conductance of ordinary concrete in a damp state is therefore due to the presence of an electrolyte in the form of the aqueous phase containing ionic compounds. That the latter is responsible for the large proportion of the conductive behaviour is reflected by the following:

- (a) Concrete exhibits a negative temperature coefficient of resistivity an effect which is particular to electrolytic behaviour.
- (b) Oven dried concrete, prepared from cement and conventional sand and rock particles, has a high resistance ( $2.6 \times 10^3$  ohm.m)<sup>111</sup> and is generally classified as an insulator.

Electrical conduction through a damp cement paste is by means of ions in the evaporable water. The aqueous phase is predominantly saturated or, in the initial stages, supersaturated with Calcium and Hydroxyl ions which are the main contributors to the conductive process. Other ions such as Sodium, Potassium, Alumina, Silicates and other oxides occur at well below equilibrium level and are only found at very low concentrations. During the initial twenty four hours Sulphates are also present, but these gradually precipitate in the form of Sulphoaluminate Hydrate.

Because of their low solubility, Calcium Silicate Hydrates, Ferrites, Silicates and Aluminates are only present in the aqueous phase at low concentration values.

The exact ionic composition of the aqueous phase is dependent on the type of cement, time elapsed since the addition of water and temperature. Cement type and hydration time do not hinder the use of conductivity measurements, where relative changes are being monitored at fixed times and given cement type. Changes in temperature, however, have to be corrected for by means of a temperature coefficient of resistivity.

#### 4.1.4 Sub-Microscopic Level

##### "Ionic Conductivity"

##### 4.1.4.1. Chemical Analysis of a Water-Cement Solution

The following test was carried out to assess the concentration level of Calcium and Hydroxide ions in an electrolytically saturated water-cement solution.

A solution was prepared with a water/cement ratio of 1.0 and allowed to rest for a period of 15 minutes. The sample was then centrifuged to remove unhydrated cement particles and HCl added to stabilize ions and prevent precipitation.

Chemical analysis of the clear solution showed only Calcium to be present at high levels (7.2 mg/ml) all other compounds such as ferrites, aluminates and other oxides were only detected at very low concentrations (Ranging from  $10^{-3}$  to  $10^{-5}$  mg/ml). The alkalinity of the solution was tested prior to acidification and a pH value of 13.2 measured at a temperature of 20°C.

1 M = 1 mole of solute per litre of solution

(Where a mole of any chemical compound corresponds to a mass equal to the relative molecular mass in grams.)

1 mole of CaO = 56.08 gms (Calcium is present in the solution in the form of Calcium Oxide.)

$$\frac{7.2 \times 10^3}{56.08} = 128 \text{ mM of CaO was established as the concentration level of Calcium ions (Ca}^{2+}\text{) in the solution.}$$

The equation of equilibrium between the concentration of Hydrogen and Hydroxide ions in an aqueous solution is expressed by:

$$[H^+] \times [OH^-] = K_w$$

where  $K_w$  is known as the ionic product<sup>110</sup>.

In logarithmic form:

$$-\log[H^+] - \log[OH^-] = -\log K_w$$

hence:

$$pH + pOH = pK_w$$

where  $pK_w = 14.1669$  at  $20^\circ\text{C}$

$$\begin{aligned} \text{OH} &= \log^{-1} - (pK_w - \text{pH}) \\ &= \log^{-1}(-14.1669 + 13.2) \\ &\approx 107.9\text{mM} \end{aligned}$$

#### 4.1.4.2 Electrolytic Conductance

The passage of an electric current through a liquid causes chemical changes, the process is called electrolysis. Conduction is possible only in electrolytes, liquids which are, at least, partly dissociated into oppositely charged ions. The electrodes through which the current enters and leaves the electrolyte are known as the cathode and the anode. The positive ions, cations, flow to the negative electrode, cathode. The negative ions, anions, flow to the positive electrode, anode.

At the surface of the electrodes where the current passes between electrode and electrolyte, the mechanism of conduction must change. This can happen in two ways:

- (1) The discharge of an ion, causing the substance concerned to come out of the solution and be liberated either as a deposit on the electrode or as bubbles of gas.
- (2) Fresh ions are formed from the material of the electrode and passed into the water.

The reaction, taking place at the interface between an electrolyte solution and inert electrodes, interfere with the measurement of the true conductance of the solution. For this reasons alternating current is used for electrical resistivity measurement of electrolytes. A.C. at about 1000 Hz is normally employed so that the small amount of electrolysis taking place is completely reversed every half-cycle, see Section 4.1.4.2 (iv).

##### (i) **Ionic Mobility and Ionic Conductance**

Conductance of an electrolyte is a measure of the current it can carry. The magnitude of a current carried by ions in solution, at a given temperature, is dependent upon the number and speed of the ions, and also upon the charges carried by each of them 110,112,113

Thus, conductance is dependent upon three main variables:

(a) **Charges carried by each ion:** Faraday's second law states that there is a definite unit charge of electricity, positive or negative, and that each univalent ion carries one such charge, each bivalent two and so on.

(b) **Ionic concentration:** When an ionic compound dissolves, it spontaneously dissociates into ions, the more dilute an electrolyte is the more complete the ionic dissociation. As the solution becomes more concentrated less ions dissociate from the compound being added, although overall there will be more free ions in solution. The more ions present, the greater the rate at which charge may be transferred.

(c) **Ionic speed:** The faster an ion travels the greater the rate of transfer of charge, however the speed of an ion is, itself, dependent upon four factors.

(1) **Electric field intensity:** Current flows through an electrolyte due to charge being transferred between the electrodes. A difference of potential across the electrodes gives rise to an electric field in the solution. A charged particle in an electric field experiences a force which is proportional to the electric field intensity.

(2) **The viscosity of the solvent:** The viscous drag on the ions by the solvent, increases with velocity and opposes the accelerating force until, after a short period of time, the ion reaches its terminal velocity.

(3) **The asymmetry effect:** A positively charged ion will attract, towards itself, negatively charged ions and tend to repel other positively charged ions. Every positive ion will therefore be surrounded by an ion cloud containing more negative than positive ions. The reverse will be true for a negative ion.

Consider one particle, a positive ion. When a current is applied, the positive ion will move towards the cathode and its ion cloud will move in the opposite direction. The original ion cloud will break up and a new one will form. In practice, a short time is



required for the completion of this operation, which is known as the time of relaxation. Before the original ion cloud has decayed there will be a net backward attraction and the speed of the positive ion is correspondingly reduced.

**(4) Electrophoretic effect:** Positively and negatively charged ions are moving in opposite directions through the solvent and in doing so transfer momentum to the solvent molecules; but, as each ion is surrounded by an excess of those going in the opposite direction, it will be moving against the flow and will be opposed by a greater frictional force than the solvent would otherwise exert.

#### **(ii) Temperature Dependence**

Electrolytes are effected by temperature in two ways:

- (1) With a rise in temperature solvents become less viscous as each solute particle executes a more lively random motion. As a result the mobility of each species of ion increases and so in turn does the conductivity of the electrolyte.
- (2) In an electrolyte with a high temperature more ions will dissociate than in an electrolyte of the same concentration at a low temperature. Thus the ionic concentration increases with temperature.

It follows from the above that electrolytes have a negative temperature coefficient of resistivity. i.e. conductivity increases with temperature.

The conductivity of an aqueous suspension of cementitious material is also dependent on the hydration rate of the cement which results in the release of more ions in the solution. Temperature affects the rate at which the hydration reactions take place, the rate at which it does so is, however, dependent on the composition of the cement and would be difficult, if not impossible to predict.

The increase in ionic mobility with increase in temperature can, however, be predicted through the use of temperature coefficients which allow resistivity measurements at any temperature to be converted to those at a datum one.

### (iii) The Electric Double Layer

The electric double layer (E.D.L.) is a term used to describe the distribution of ions on the surface of a solid which has been placed in an electrolyte. As all solids have an electrostatic imbalance at their surface, in order to establish a condition of electroneutrality a balancing of charges at the solid/electrolyte interface has to occur. Ions of opposite charge to that of the surface are attracted to it whilst ions of equal charge are repelled. This electrostatic model, initially put forward by Helmholtz, has been developed by Grahame<sup>114</sup> who explained it as being made up of two layers, the first consisting of rigidly fixed ions, the second of more mobile ones.

Debye and Huckel<sup>115</sup> calculated the charge density from Boltzmann law of energy distribution, according to which if  $n$  is the average concentration of a substance, its concentration  $n^1$  at a situation where it possesses extra energy  $E$  per molecule will be given.

$$n^1 = n \cdot e^{-E/kT}$$

where  $k$  is Boltzmann's constant. The work required to bring a unit positive charge to  $dV$  is  $e\psi$ , so the number of cations and anions in the element of volume is:

$$dn^+ = n \cdot e^{-e\psi/kT} dV \text{ and } dn^- = n \cdot e^{+e\psi/kT} dV$$

where

$dn$  = Number of particles in a volume  $dV$ ,

$T$  = Temperature,

$e$  = Electrical charge, and

$\psi$  = Electrical potential.

### Electrode (Metal) - Electrolyte Interface

Examination of the cathode in an electrolytic solution shows that it is surrounded by a layer of cations. (The negative charge on the electrode has attracted an excess of positively charged ions.) The two layers of charge on either side of the interface constitute an electric double layer (Helmholtz layer). Beyond the layer of cations is the diffuse layer. Ions are constantly being

transferred to and from this layer between the bulk of the solution and the Helmholtz layer. The diffuse layer has a predominant amount of anions which are attracted there by the cations in the Helmholtz layer. The bulk solution is much larger than any of the layers and has a random distribution of ions in it.

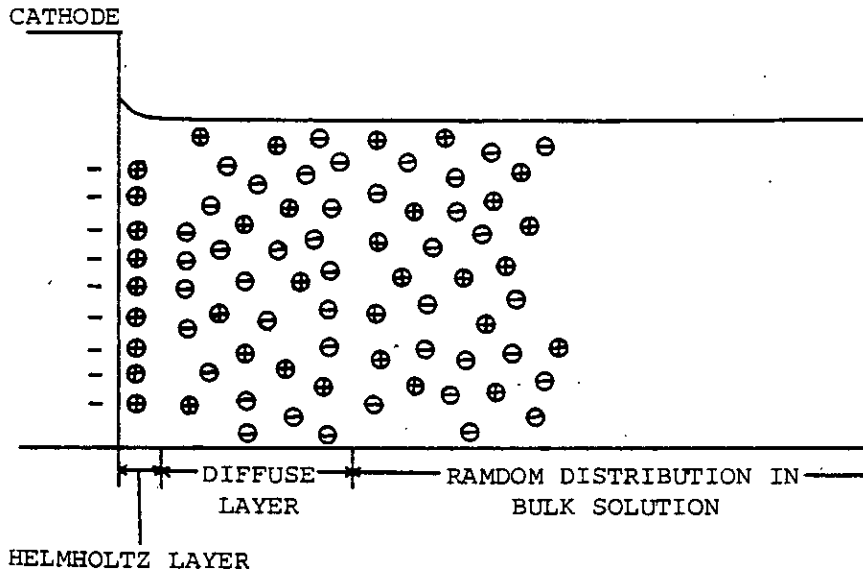


Figure 4.3 Ionic distribution at the cathode-electrolyte interface

This effect also happens at the anode only in reverse as the anode has a positive charge.

#### Semiconductor - Electrolyte Interface

The concentration of charge carriers in a semi-conductor is very low and the dielectric constant has a finite value. Under these conditions not only does the electrolyte form a diffuse double layer at the interface, as in the metal-electrolyte interface, but the semiconductor also forms one<sup>113</sup>.

#### (iv) Polarization

Polarization is defined as the change in the electrochemical structure of a dielectric as a result of sustaining a steady electric field<sup>116</sup>. In an electrolyte subjected to the flow of electricity this change can be characterized according to whether

it occurs in the vicinity of the electrodes or in the bulk electrolyte.

**Polarization of electrodes:-** Electrode polarization is the result of the slow process by which charged particles are transferred from the solution to the electrodes. Charged particles delaying the conduction process cause an increase in resistance and a corresponding rise in potential difference. The anode becomes more positive and the cathode more negative.

The discharge of ions at an electrode involves three main stages<sup>110</sup>:-

- (1) Transport of ions to the electrode surface,
- (2) Discharge of the ions to form atoms,
- (3) Conversion of the atoms to the normal stable form.

The overpotential caused by the transportation process (1) is known as 'concentration polarization' while that due to the actual charge transfer mechanism, (2) and (3), is termed 'activation polarization'.

The overpotential  $\eta$  at each electrode is given by:-

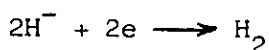
$$\eta = E^1 - E$$

where

$E^1$  = actual electrode working potential, and

$E$  = equilibrium potential.

The greatest hindrance to the application of D.C. to a fresh cement paste is the activation polarization caused by the formation and liberation of Hydrogen gas at the electrodes effectively obstructing the flow of electrical charges to the electrode's surface.



**Polarization of bulk electrolyte:-** Aside from the electrode processes, a secondary polarization effect occurs away from the electrode/electrolyte interface. This phenomenon involves the alignment of dipoles and other charged particles in the direction of current flow. If D.C. is applied, this re-alignment results in a more efficient conductive medium while, if subjected to A.C., the continuous change in polarity causes a proportion of the charge to

be stored in between cycle reversals.

The magnitude of the stored charge is measured by means of the dielectric constant:-

$$\epsilon = \frac{C}{C_0} \quad \text{where } \epsilon = \text{dielectric constant}$$

$C = \text{capacitance of the specimen}$   
 $C_0 = \text{capacitance of equivalent sample size}$   
occupied by a vacuum.

The dielectric constant, like resistivity, is independent of specimen dimensions and is particular to any given material.

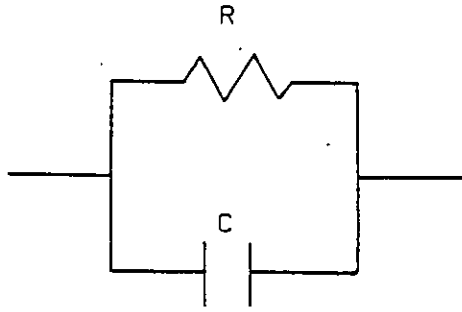
Bulk electrolyte polarization effects which are likely to occur at a low frequency (1000 Hz) input pulse are<sup>117</sup>:-

- (a) Molecular and dipole rotation aligning with applied electric field.
- (b) Charges build up on the surface of hydrated and unhydrated compounds where they are bound to the crystal lattice.
- (c) Ion atmosphere distortion - where intermolecular atomic distances are increased by the opposite attractions of the constituent atoms.
- (d) Displacement of the electrical double layers along the applied electric field.

Ohm's law states that, in a metallic conductor, the passage of current is inversely proportional to the applied voltage:

$$I = \frac{V}{R}$$

In an electrolyte, polarization effects at the electrodes interface, known as back e.m.f., result in reduced conductance. This phenomenon can be minimized through the use of alternating current although the net result is the introduction of a capacitive element in parallel with the resistance of the system. Concrete, in an A.C. field, may be considered as a network of capacitors and resistors, this model may be simplified to a single capacitor and resistor in parallel:



for the above,  $I = \frac{V}{Z}$

where

$$Z, \text{ the impedance} = \frac{R}{(1 + \omega^2 C^2 R^2)^{1/2}}$$

$Z$  = impedance ( $\Omega$ )

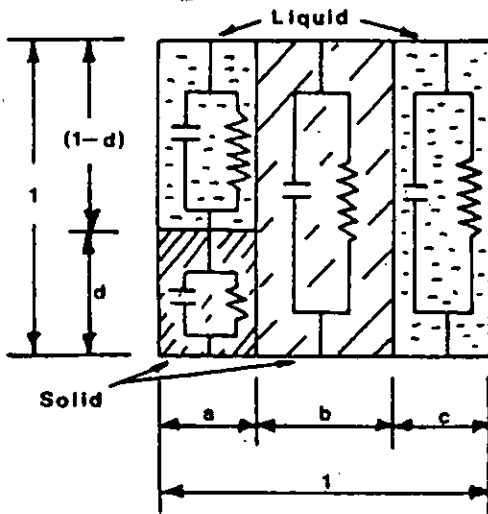
$R$  = resistance ( $\Omega$ )

$\omega$  = angular frequency =  $2\pi f$

$f$  = frequency in cycles per second (Hz)

$C$  = capacitance

#### 4.1.5 Electrical Modelling



a, b, c = fractional cross-sections of paths 1, 2, 3, respectively

1. Through solution and conducting particles in series.

2. Through particles in contact with each other.

3. Through solution.

Figure 4.4 "Electrical analogue model of concrete" (After Taylor)

The above method although allowing for polarization of bulk electrolyte does not take into account electrode surface polarization.

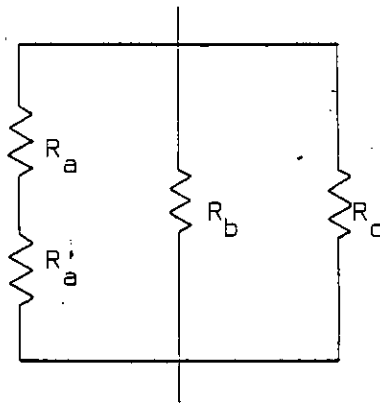


Figure 4.4(a) Simplified Model

$$\frac{1}{R_{TOTAL}} = \frac{1}{R_a + R'_a} + \frac{1}{R_b} + \frac{1}{R_c} \quad \dots\dots(4.1)$$

Because of high contact resistance,  $1/R_b$  is extremely small and can therefore be removed from the equation<sup>106</sup>.

Equation (4.1), can be further expanded:

$$\frac{1}{R_T} = \left( \frac{\rho_a \times (d)}{a \times 1} + \frac{\rho_p \times (1-d)}{a \times 1} \right)^{-1} + \left( \frac{\rho_p \times 1}{c \times 1} \right)^{-1} \quad \dots\dots(4.2)$$

where  $\rho_a$  and  $\rho_p$  are the resistivities of aggregate and paste respectively.

$$\text{also, } (1 - d)a + c \times 1 = \phi \quad \dots\dots(4.3)$$

$$\text{and } c + a = 1.0 \quad \dots\dots(4.4)$$

where  $\phi$  = Fractional volume of cement paste

In order to resolve equation (4.2) in terms of c, equations (4.3) and (4.4) are substituted:

$$1/R_T = \left( \frac{\rho_a(1.0 - \phi) + \rho_p(\phi - c)}{(1.0 - c)^2} \right)^{-1} + \left( \frac{\rho_p}{c} \right)^{-1} \quad \dots\dots(4.5)$$

Let us consider a concrete mix 1:1.5:3 with a water/cement ratio of 0.5 and a cement paste of equal water/cement ratio. The resistivities of the two in a fresh state are 11.4 ohm.m and 1.63 ohm.m respectively. The fractional volume of cement paste in the concrete mix is 0.33050 and  $\rho_a$ , the resistivity of the aggregate employed (granite), is 5000 ohm.m.

Substituting the values into equation (4.5) yields a value for  $c$  of 0.1426

$$\text{therefore, } R_c = \frac{1.63 \times 1}{0.143 \times 1} = 11.43 \text{ ohm.m}$$

As the resistance of the whole block was 11.40, the above calculations show that ~~the~~ bulk of the conduction is through  $R_c$ , the cement paste phase alone.

The above example involved resistivity measurements taken after approximately 1.5 hour of the setting stage.

## 4.2 TESTING OF CONCRETE BY ELECTRICAL MEANS

A substantial amount of research has been carried out on the electrical testing of cement pastes, mortars and concretes. For the sake of clarity the following literature review has been divided into two main sections. One is concerned with the practical applications of the method, such as determining the setting time and forecasting mechanical properties, the other is theoretical, involving relationships with mix parameters and ambient conditions.

### 4.2.1. Practical Applications

#### 4.2.1.1 Setting Time

The earliest work involving the electrical resistivity measurement of cementitious materials falls under the practical applications category; the aim, to determine the setting time of cement paste.

The Vicat apparatus, an empirical test for determining setting times, (see section 2.4.2.3) has the disadvantage of not providing reliable information when applied to mortar and concrete. Thus a new method was required.

In 1928, Yosomatsu Shimizu<sup>37</sup>, working with mixed cements containing blast furnace slag, determined that, at the point of final set, there was a characteristic change in the electrical resistance. The sudden change is expressed graphically in figure 4.5. The author concluded that the method was better adapted and more accurate than the mechanical and thermal methods used at that time for determining final set.



Electrical Conductivity ( $K_{18^{\circ}C} \times 10^4$ )

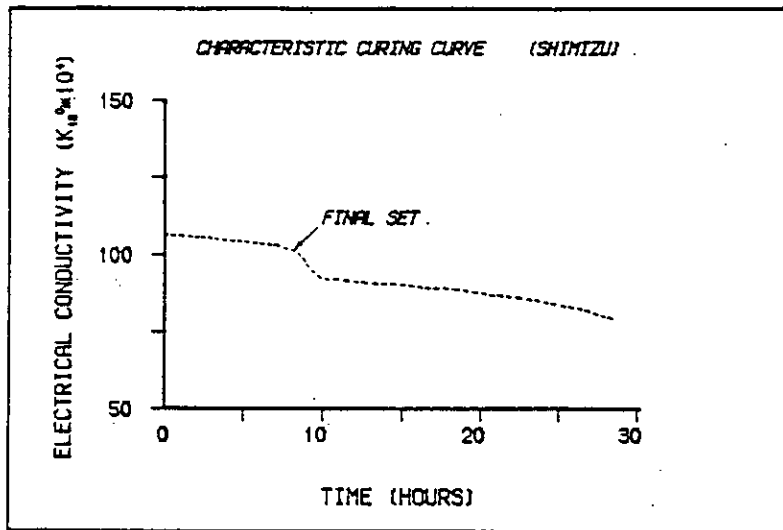


Figure 4.5 Characteristic curing curve (after Shimizu<sup>37</sup>)

Petin et al<sup>38</sup> and Baire<sup>39</sup>, in 1932, confirmed Shimizu's findings but also added that there were several other factors, such as water content and temperature, which greatly affected the electrical conductivity of a cement paste.

In the following years, Michelsen<sup>118</sup>, Dorsch<sup>119</sup> and Boast<sup>120</sup> all carried out various tests involving the measurement of the electrical conductivity of pastes during the setting period and the effect of controlling factors such as the composition of the cement, temperature and the type of electrodes employed. Michelsen made use of the galvanic current induced by metals placed in a fresh mix to study the early stages of hydration, while Dorsh established that the resistivity of different cements was inversely related to their lime content. Figure 4.6 shows, graphically, Dorsh's results;

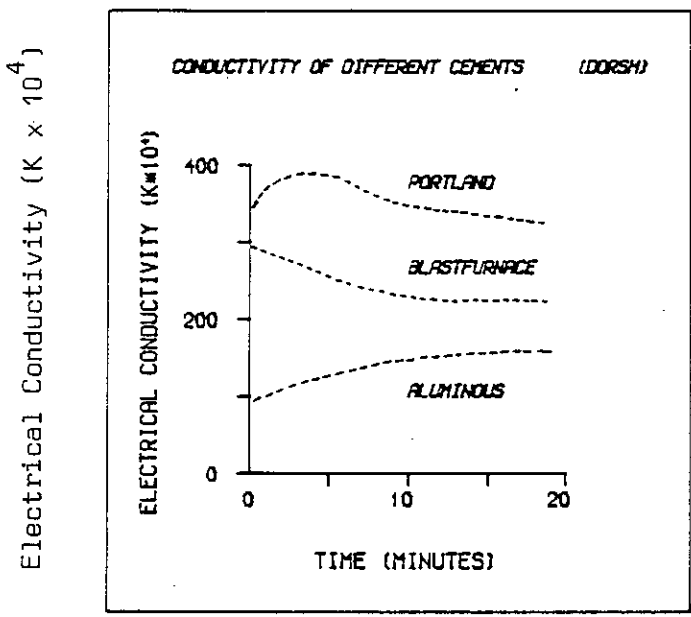


Figure 4.6 Conductivity of different cements (after Dorsh<sup>119</sup>)

(Note that Portland, Blast furnace and Aluminous cements have, in that order, an increasing proportion of lime). Dorsh repeated his experiments several times and, as the results were satisfactorily reproduced, concluded that there were definite relationships between the composition of cement and the electrical conductivity of pastes. Boast attempted to relate, unsuccessfully, electrical measurements to 28 day compressive strengths. The reason for this failing being the attempt to relate the resistivity of both fully compacted and mixes with entrapped air, to compressive strength. For a fully compacted mix, a decrease in water/cement ratio is accompanied by corresponding increases in resistivity and compressive strength. In an uncompact mix, however, decreasing the water content increases the resistivity but adversely affects the compressive strength. What the author succeeded in showing, however, was the ability of the method to detect the effect of water content on the workability and conductivity of a fresh concrete mix and the way in which this could be related to compressive strength, figure 4.7.

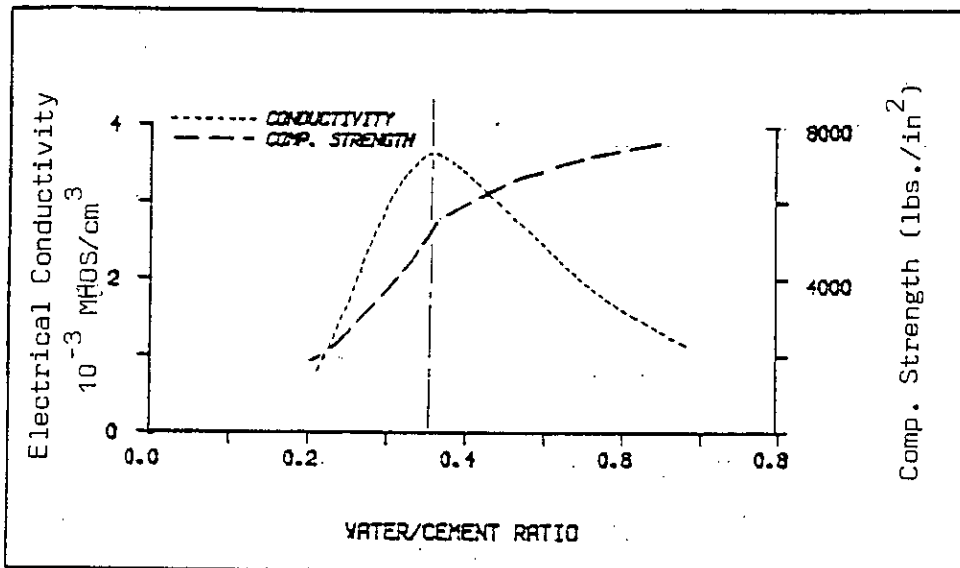


Figure 4.7 Electrical conductivity of fresh concrete vs. its compressive strength at 28 days as a function of water/cement ratio. (after Boast<sup>120</sup>).

Kind et al<sup>40</sup>, in 1937, investigated the relationship between electrical conductivity of fresh cement pastes and their subsequent mechanical durability characteristics. The authors concluded that a relation with durability exists but, given the limited amount of data available, were unable to establish a clear cut relationship.

Having examined the dielectric properties during the setting of four Spanish cements, Tobio<sup>41,46</sup> concluded that the changes in admittance (reciprocal of impedance) which occurred during that period were characteristic of each of the cements tested. However, although the author maintains good replicability of results, data published appears to be very erratic (see figure 4.8).

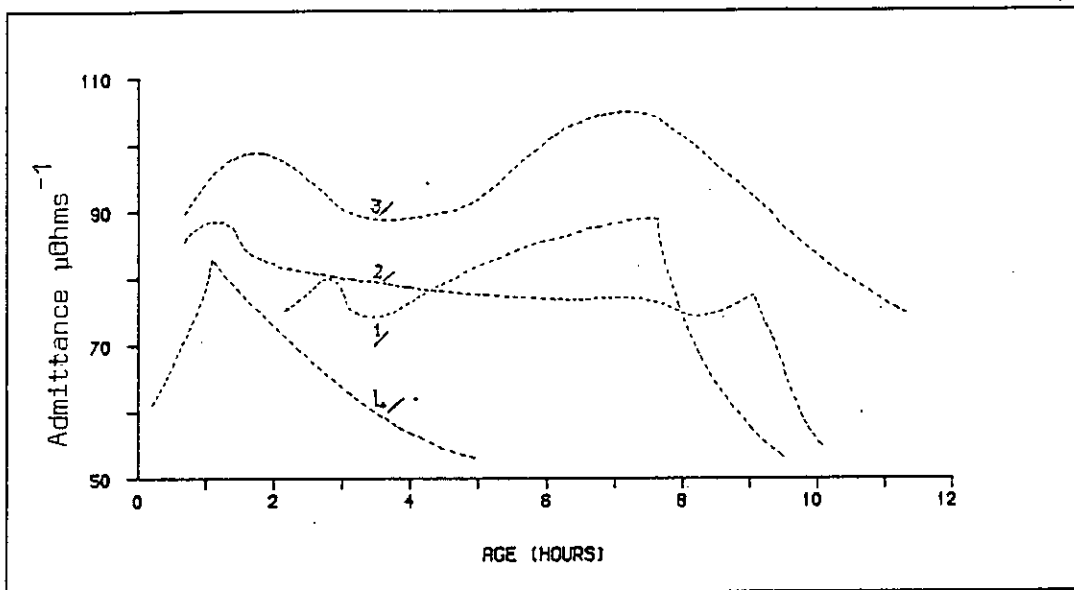


Figure 4.8 Conductivity of four Spanish cements (after Tobio<sup>46</sup>)

In 1957, Naray-Szabo et al<sup>121</sup> carried out an extensive series of tests monitoring the setting process by electrical, temperature, Vicat's and pH measurements. Results confirmed Dorsh's<sup>119</sup> findings relating cement composition to the electrical response of hardening cement pastes.

Nikkanen<sup>122</sup> and Ashan<sup>47</sup> continued Michelsen's<sup>118</sup> work on the use of galvanic currents to monitor the setting process. Nikkanen, in particular, produced an extensive study of the conductivity, electrolytic and dielectric properties of pastes and concretes. The influence of accelerators and retarders (figure 4.10) and water/cement ratio (figure 4.9) upon the voltage generated by copper and lead electrodes show quite clear trends.

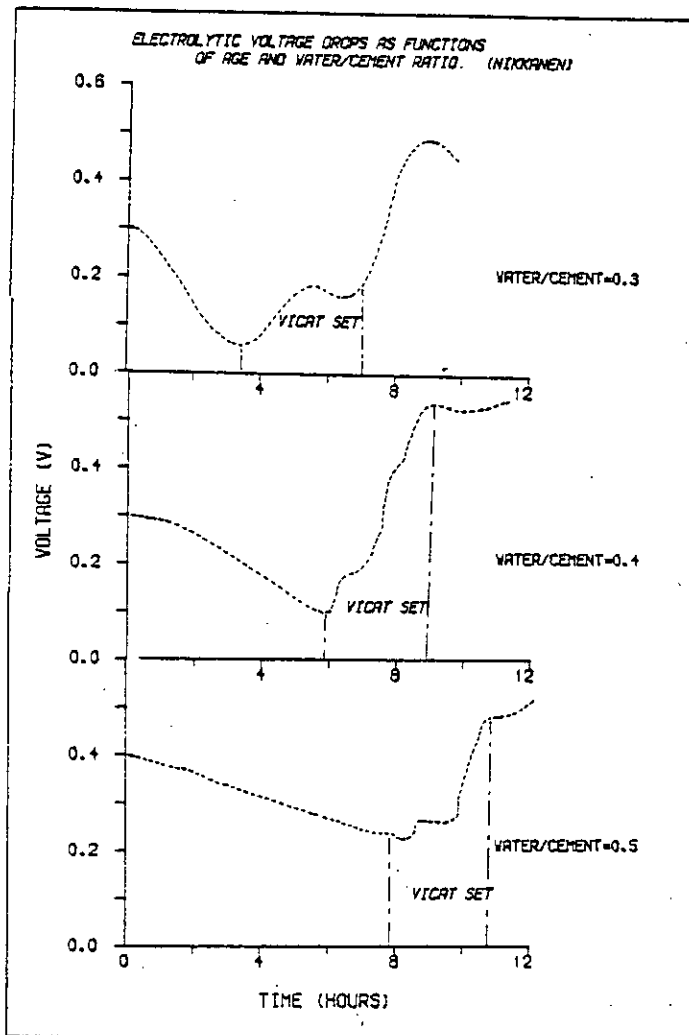


Figure 4.9 Electrolytic voltage drops of cement pastes as functions of age and water/cement ratio (after Nikkanen<sup>122</sup>)

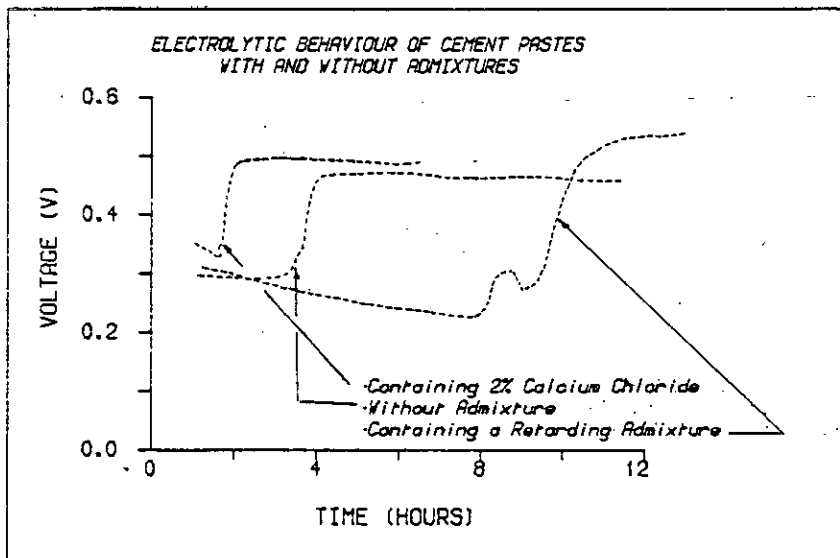


Figure 4.10 Electrolytic behaviour of cement pastes with and without admixtures (after Nikkanen<sup>122</sup>)

Ashan researched into the effects of curing temperature upon the electrical characteristics during setting and his results have been summarised in figure 4.11.

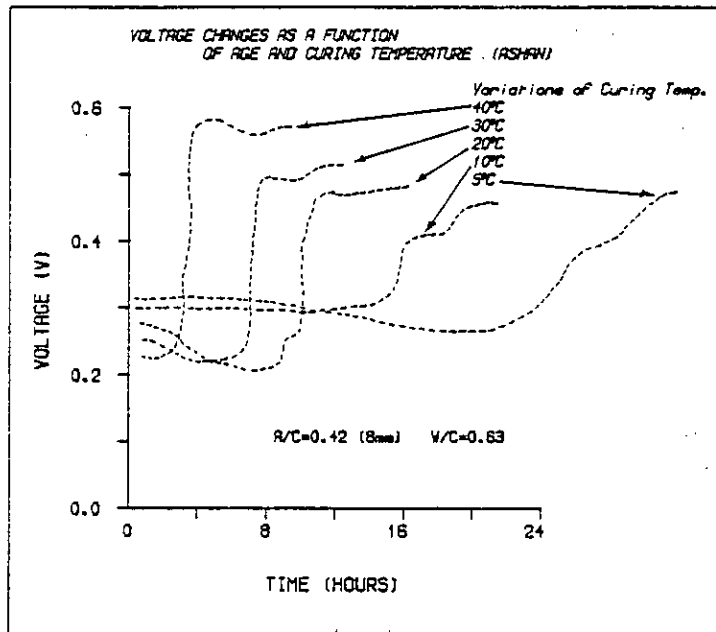


Figure 4.11 Electrolytic voltage changes as a function of age and curing temperature (after Ashan<sup>47</sup>)

Bars et al<sup>123</sup>, in 1982 derived a method for determining the conductivity of solid state electrolytes by drawing complex impedance diagrams, which were employed to monitor the evolution of the conductivity of cement pastes during setting. The advantage of this system being that polarization effects could be easily accounted for through the use of a wide frequency range.

A three dimensional probe electrode recently developed by Swamy et al<sup>48</sup>, is the latest in conductivity measuring devices. The system relies on the electrodes being embedded in the mix immediately after batching and left in position through the hardening stage. The size of the probe unit, which contains 24 sets of electrodes could however form a physical obstruction to moisture movements in its vicinity.

#### 4.2.1.2 Other Applications

Aside from the measurement of setting times, electrical conductivity/resistivity testing has been applied to a variety of problems.

Slack<sup>124</sup>, in 1933 devised an electrical conductivity test to determine the moisture content of pavement concretes which had been cured by different methods. A definite pattern was observed in the results.

Slack's work was continued in 1937 by Spencer<sup>125</sup> who looked more closely at the relationship between electrical resistance and moisture content in mass concrete. He found that several factors affected the relation, such as temperature type of cement, age of concrete, sealing of concrete and presence of salts. After extensive testing, however, the author concluded that, if suitable precautions were taken, direct resistance measurements of hardened concrete could offer an inexpensive and simple means of determining moisture content.

A number of researchers<sup>108,126,127,128,111</sup> have been investigating the problem of how to increase or decrease the resistivity of a concrete mix. A decrease in insulation resistance is necessary in cases where the build up of static charges may be the cause of equipment breakdown. An increase in resistivity, on the other hand, is required for insulating concrete components as those employed, for instance, by electric railways. Conductivity testing is presently regarded as being the only satisfactory method of monitoring the efficiency of the mixes developed.

A new method of determining the cement content in fresh concrete and mortar mixes was developed by Chadda<sup>27</sup> in 1955. The test based its theory on Dorsh's<sup>119</sup> conclusions that conductivity of a water/cement solution is related to the lime content of the cement used. The method was further improved by Sherwood and Fawcett<sup>28</sup> in 1963 who concluded that results compared favourably with those achieved by chemical and mechanical tests in use at the time.

Yamada<sup>129</sup> and Ohama<sup>130</sup> also published data on the resistivity of concrete; the results are however of little relevance as they were obtained through the application of large currents employed for the electrical heating of concrete for curing purposes.

Gjorv et al<sup>135</sup> in 1977, examined the ability of the conductivity test to monitor the contamination of concrete with salt water concluding that, amounts of up to 1%  $\text{CaCl}_2$ , based on the cement weight, slightly increased the resistivity, while larger amounts of up to about 8%  $\text{CaCl}_2$ , reduced resistivity to approximately 50%, see figure 4.12.

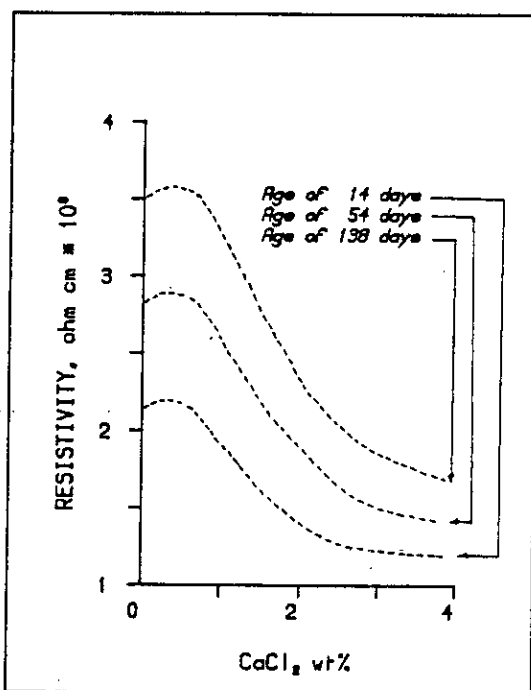


Figure 4.12 Effect of Chlorides on the resistivity of mortars (after Gjorv<sup>135</sup>)

The test therefore proved to be very sensitive to the level of salt water infiltrating hardened concrete.



### 4.3. Theoretical Investigations

Investigations which come within this category involve research concentrating mainly on the study of those factors which affect the electrical properties of cement based materials. Obviously, a large amount of the work carried out to date covers both the practical and the theoretical aspects of conductivity testing. Thus, only those tests which have not been previously mentioned will be cited in this section.

In 1952, Calleja's<sup>42</sup> adiabatically controlled experiments on cement pastes, showed that temperatures and electrical resistance were definitely related. Previous researchers had been unable to determine whether variations in resistivity were due to the hydration process or to temperature changes induced by the exothermic chemical reactions involved. In a later paper, Calleja<sup>43</sup> also examined the effect that variations in the frequency of measuring current had on resistivity values (see figure 4.13). He concluded that, although resistance values were altered, this did not affect results obtained for setting times.

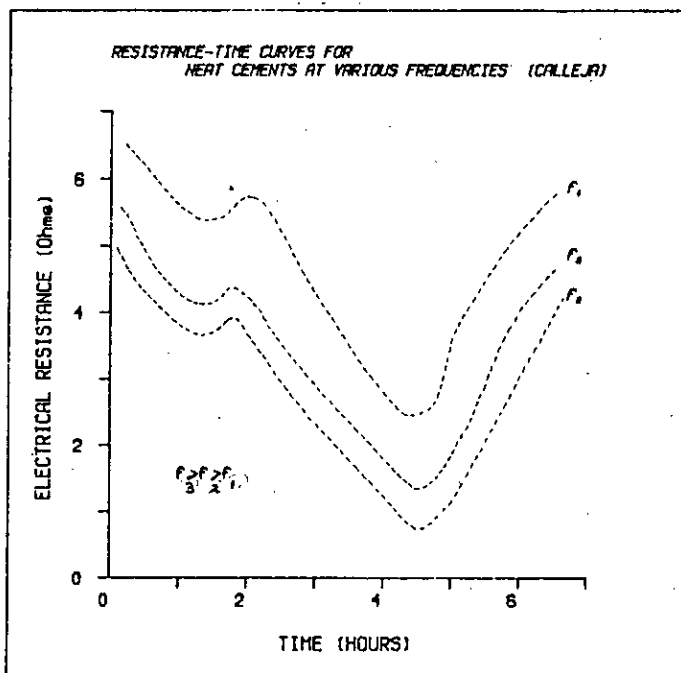


Figure 4.13 Resistance-time curves for neat cements at various frequencies after Calleja<sup>43</sup>)

Several factors which affect the electrical properties of concrete were investigated by Hammond and Robson<sup>45</sup> in 1955. These

included:- size and shape of specimen, mix proportions, aggregate type and water/cement ratio. Figure 4.14 shows graphically some of their results.

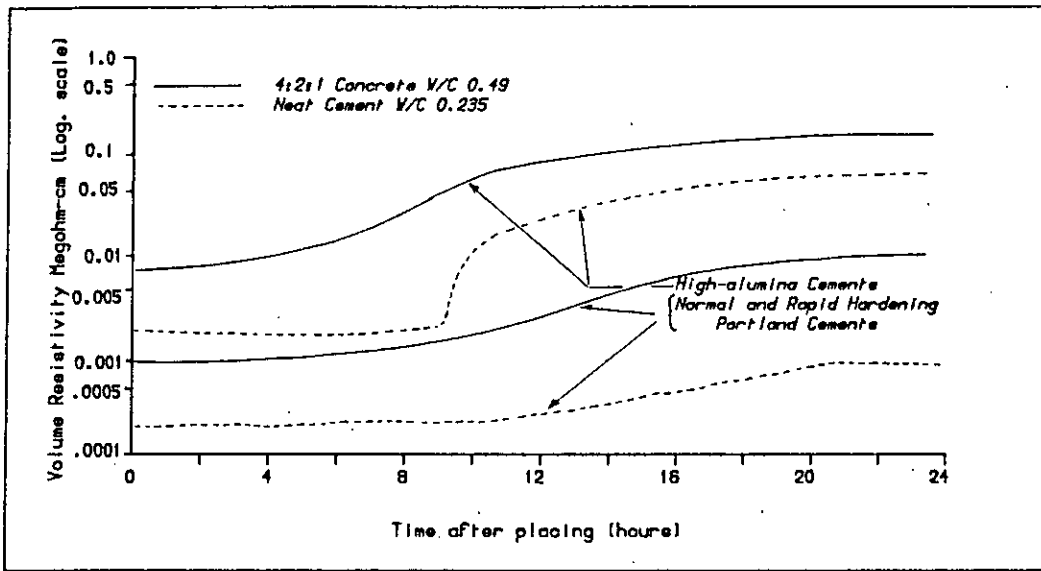


Figure 4.14 Development of the volume resistivities of 4:2:1 concretes and neat cements during the first 24 hours of the curing period. (after Hammond and Robson<sup>45</sup>)

The problem found with the data they published being, however, that mix proportions, water/cement ratio and cement type were varied concurrently thereby making the individual study of the effect of one or the other impossible. The authors also put forward the well known Hinrichson-Rash law to quantify resistivity variations with temperature. This law has recently been employed by McCarter et al<sup>131</sup> to establish a temperature coefficient for cement based mixes of 0.022.

Ganin<sup>132</sup> studied the relationship between the electrical resistance of concrete and its density concluding that, in doing so, account must be taken of the phenomenon of contraction through dehydration, evaporation and the water binding characteristics of the cement gel.

Employing a high frequency (10 KHz) input current, Nikkanen<sup>122</sup> monitored, simultaneously, the capacitance and the resistance of fresh cement paste (figure 4.15) showing how both displayed time-dependent characteristics during the setting time period.

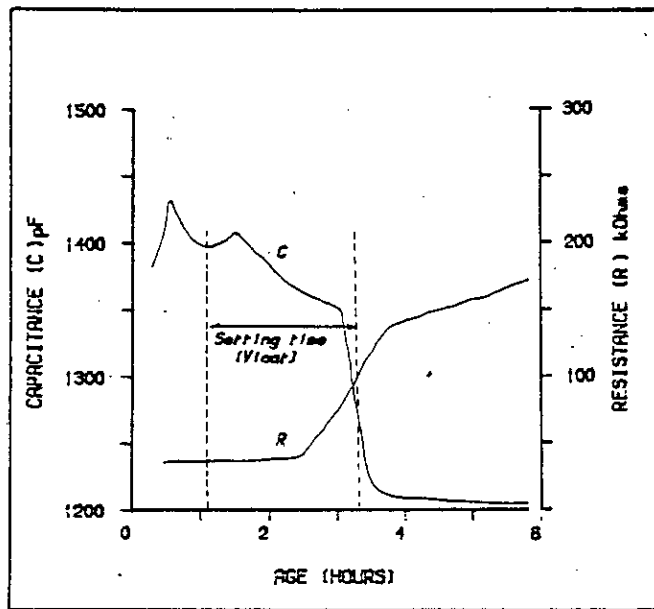


Figure 4.15 Changes of capacitance (C) and resistance (R) in a capacitor with fresh paste used as a dielectric (after Nikkanen<sup>122</sup>)

Nikkanen's work was followed in 1974 by Taylor et al<sup>1</sup> who investigated the relationship between physical and electrical properties of cement pastes through the study of dispersion curves figure 4.16. The authors looked at the dielectric behaviour of pastes of different water/cement ratio over the early hardening stage by employing input currents ranging from 1 to 100 MHz. Radio frequency dispersion measurements, they concluded, can provide useful knowledge on the developing microstructure of fresh and hardened pastes.

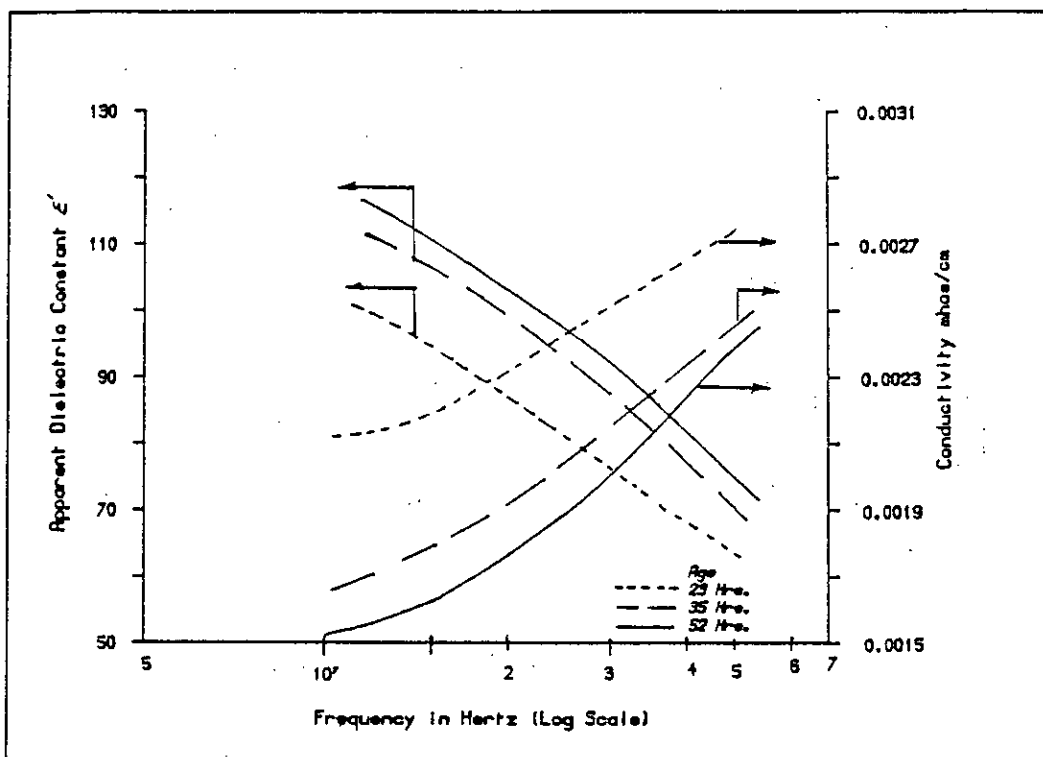


Figure 4.16 Dielectric dispersion of cement paste (Water/Cement ratio of 0.35) as a function of age (after Taylor and Arulandan<sup>1</sup>)

As part of his research on the electrical resistivity testing of a piled foundation - soil system, McCarter<sup>133</sup> carried out an investigative study of the conductive properties of concrete. Typical mixes were tested in both controlled and field conditions. The results of a controlled experiment are shown in figures 4.17 and 4.18.

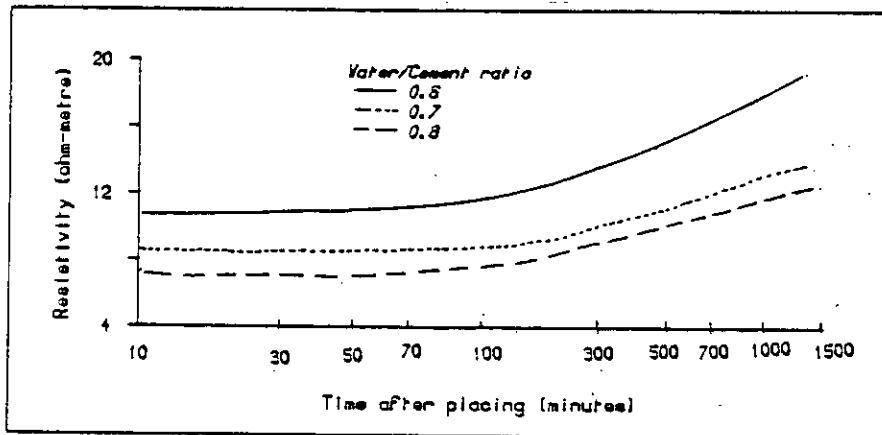


Figure 4.17 Development of resistivity during first 24 hours after placing for 1:2:4 mix specimens (after McCarter<sup>134</sup>)

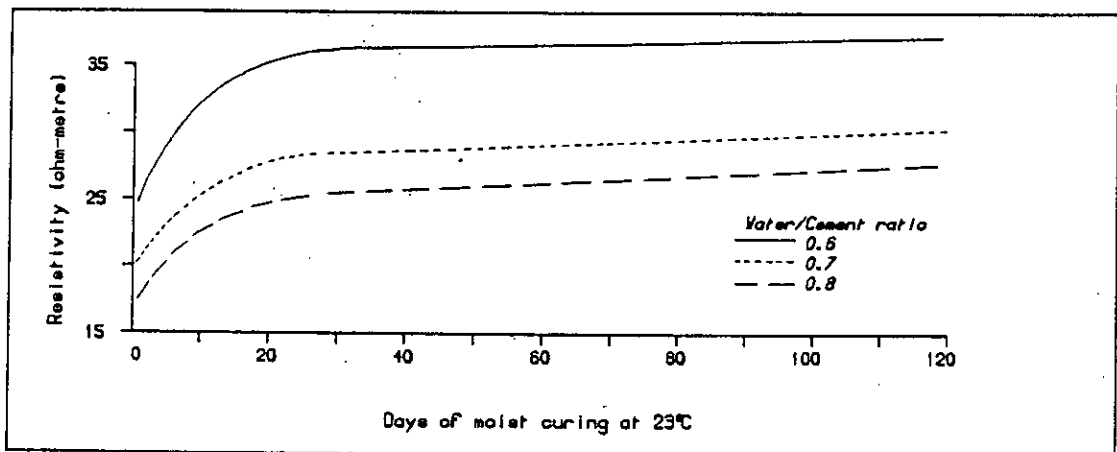


Figure 4.18 Change in resistivity with time for 1:2:4 mix specimens (after McCarter<sup>134</sup>)

The results and conclusions achieved by McCarter have instigated the development of new experimental techniques and provided the background on which this research has been based.

#### 4.4 DISCUSSION

The objectives of chapter four have been to investigate the conduction mechanism through concrete and to review research carried out to date in the field of electrical testing.

To facilitate the analysis of the conduction process, the

material has been viewed at three levels:- macro, micro and sub-microscopic and classified as an electrolyte whose conduction is to be attributed to the presence of ions in the aqueous phase of the cement paste matrix. By substituting experimentally obtained values into an electrical model for concrete, aggregate was confirmed as making a negligible contribution to the conduction of the material.

A theoretical model developed to study the effective cross-sectional area has shown it to increase with the number and size of pores present and to be constricted by a factor  $(PS_w)$ , the fractional cross-sectional area of fluid filled pores.

The electrochemical structure of the cement paste has been studied and the various factors affecting ionic conductivity examined in detail. A chemical analysis carried out on a water-cement solution showed Calcium and Hydroxide to be the ions with, by far, the highest concentration level.

A review of existing literature has shown that the electrical properties of concrete have, as yet, not been extensively investigated although a considerable amount of research has been directed towards the study of fresh cement pastes and, in particular, to developing and improving techniques for establishing setting times.

Research into the electrical properties of concrete, most probably because of the greater amount of experimental effort which is required, has been carried out in a very limited and sporadic manner. The lack of standardized experimental procedures has meant new published material contributing only in part toward a better understanding of the conductive mechanism through concrete.

Conductivity or resistivity measurement have been the most widely employed of all electrical techniques although some tests have been carried out on the basis of galvanic or capacitance measurements which, due to the difficulty in their accurate measurement have had a narrower field of application.

It has become apparent that, for a satisfactory relationship to be made between the electrical and mechanical characteristics of

the material, a fundamental knowledge of the chemical and physical processes involved in cement hydration is required. The effect of aggregate addition on the evolution of the porous structure in the cement paste matrix and the corresponding changes in the conductive properties of the material are the topic of chapter five.

**CHAPTER FIVE**

**FORMATION FACTOR THEORIES**



## 5.1 INTRODUCTION

The objective of the following chapter is to develop theories, or empirical relationships, describing the conductive behaviour of concrete mixes. The study takes the form of an investigation of the formation factor - porosity relationships (formation factor being the ratio of the resistivity of a porous medium to the resistivity of the pore fluid):-

$$FF = \frac{\rho_m}{\rho_p} \quad \dots\dots(5.1)$$

where  $\rho_m$  = Resistivity of the mix, and

$\rho_p$  = Resistivity of the cement paste.

Given that, in normal circumstances, rocks are practically non-conductive, transport of electrical charges is, in the main, of an electrolytic type through the cement paste phase. Concretes and mortars can therefore be considered as made up by electrically inert aggregates embedded in a conductive cement paste matrix. The assumption made being that the cement paste is to be treated as an electrolyte rather than a porous material in its own right.

Justifications for this assumption are as follows:-

- (a) The formation factor technique is, in this case, being employed to study the conductive changes in concrete as a conglomerate material.
- (b) It would be difficult and approximate to attempt to quantify the porosity of the cement paste phase. In any case, the effect of aggregate on the pores of the mix is unknown and is one of the areas which is hopefully to be investigated.
- (c) Although as an electrolyte cement paste is not stable with time, it is hardening at the same rate in the mix as it is by itself. As it hardens the paste becomes less conductive and, as an electrolyte, less effective. The formation factor, by its own formulation, is independent of the strength of the electrolyte.

Hence, from here on, the mix porosity ( $\Phi$ ) shall be taken to signify the fractional volume of cement paste:-

$$V_{\text{paste}} = \Phi V_{\text{mix}} = \Phi A l \quad \dots\dots(5.2)$$

where A = cross-sectional area

l = length of specimen

## 5.2 FORMATION FACTOR OF AN ELECTROLYTE CONTAINING DIELECTRIC PARTICLES

The problem of theoretically describing the flow of electricity in a solid/electrolyte system is a classical one in physics. The first eminent scientist to turn his attention to it was Maxwell<sup>136</sup> in the 19th century. His theory was concerned with the conductivity of a disperse system (i.e. large interparticle distance) made up of spherical solids, of given conductivity, and electrolyte. Letting the conductivity of the spheres equal zero, the following equation is obtained:-

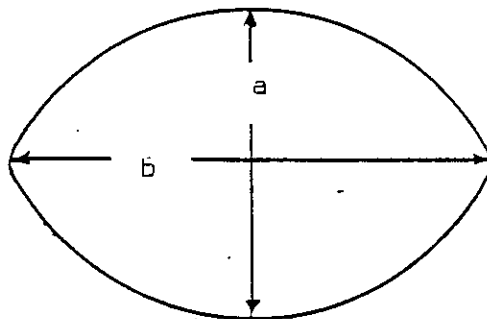
$$FF = \frac{3 - \phi}{2\phi} \quad \dots\dots(5.3)$$

Although Lord Rayleigh<sup>137</sup> claimed the above yielded erroneous results, the theory which he himself developed was too complicated for practical use and, in any case, defied experimental verification.

A development on Maxwell's theory was made by Fricke<sup>138</sup> who generalized equation (5.3) to include a shape factor. Rather than considering spheres, he analysed ellipsoids and formulated the following:-

$$FF = \frac{(x + 1) - \phi}{x\phi} \quad \dots\dots(5.4)$$

where x is a variable dependent on the axial ratio of the ellipsoid.



$$X = f(a/b)$$

For a particular sand employed during his tests, Fricke calculated  $x$  to be = 1.4

therefore,

$$FF = \frac{(2.4 - \phi)}{1.4 \phi} \quad \dots\dots(5.5)$$

In the mid 1920's Slawinski<sup>139</sup> developed semi-empirical equations for non-conductive spheres in an electrolyte. The spheres were treated solely as obstacles to the flow of electricity and two particular cases were investigated.

- (a) Spheres dispersed uniformly in an electrolyte, and
- (b) Spheres in perfect contact with one another.

For the latter case, which more closely resembles the packing nature of aggregates in a cement paste matrix, the following equation was put forward:-

$$FF = (1.3219 - 0.3219\phi)^2 / \phi \quad \dots\dots(5.6)$$

#### 5.6 THEORY OF FORMATION FACTORS AS APPLIED TO SATURATED ROCK SYSTEMS

Electrical resistivity measurements are being increasingly employed, in various fields, to assess other physical properties of porous formations. Such measurements offer a convenient method of characterizing the pore structure of rocks and have been extensively employed to appraise the oil bearing capacity of underground deposits<sup>140</sup>.

Considering the fact that resistance of a sample of given resistivity is proportional to its length in the direction of current flow and inversely proportional to its cross-sectional area, it is evident that a relationship must exist between porosity and the formation factor.

Archie's<sup>140</sup> equation of 1942 was the first approximation to this relationship:-

$$FF = \phi^{-m} \quad \dots\dots(5.7)$$

Research carried out by Helander and Campbell<sup>141</sup> on synthetic

cores cited the following as having an effect on the magnitude of Archie's exponent,  $m$ .

1. Degree of cementation,
2. Shape, sorting and packing of the particle system,
3. Type of pore system (intergranular, inter-crystalline, vugular),
4. Tortuosity of the pore system,
5. Constrictions existing in porous system,
6. Presence of conductive solids,
7. Compacting due to overburden pressure, and
8. Thermal expansion.

Because of the large number of variables which precluded a generalised theoretical approach, investigators were required to establish empirical relationships from a large number of laboratory studies.

Humble's coefficients, leading to Humble's relation, were established after extensive laboratory measurements on a variety of samples<sup>142</sup>. These samples were obtained from cores of different sites in the United States and covered a wide range of physical characteristics such as carbonates and clay content, porosity, tortuosity, permeability, formation factor and various others. Regression techniques were applied to the variables in an attempt to establish meaningful relationships between them and the best fitting correlation was found to be between the formation factor and the porosity.

$$\text{Humble's equation } FF = 0.62\phi^{-2.15} \quad \dots\dots(5.8)$$

The above equation is of an empirical and general nature but, nevertheless, tentatively applicable to the problem of conductivity of concretes. By comparing it to experimentally obtained results it will at least be possible to examine the relevance of this empirical format to the problem under consideration.

#### 5.4 THE EFFECT OF PORE TORTUOSITY OF THE FORMATION FACTOR - POROSITY RELATIONSHIP

Tortuosity is defined as the mean amount of deviation that

current path is subjected to when travelling through a porous media<sup>143,144</sup>.

$$FF = (T_r, \phi)$$

where FF = Formation Factor,

$\phi$  = Porosity, and

$T_r$  = Tortuosity.

with reference to figures 5.1(a) and (b), porosity could be expressed as:-

$$\phi = \frac{A_e l_e}{A l} \quad \dots\dots(5.9)$$

where  $A_e$  = average cross-sectional area of pores

$l_e$  = average length of pores

A = area of porous solid

l = length of porous medium

Formation factor being the ratio of the resistance of equal volumes of porous medium and electrolyte:-

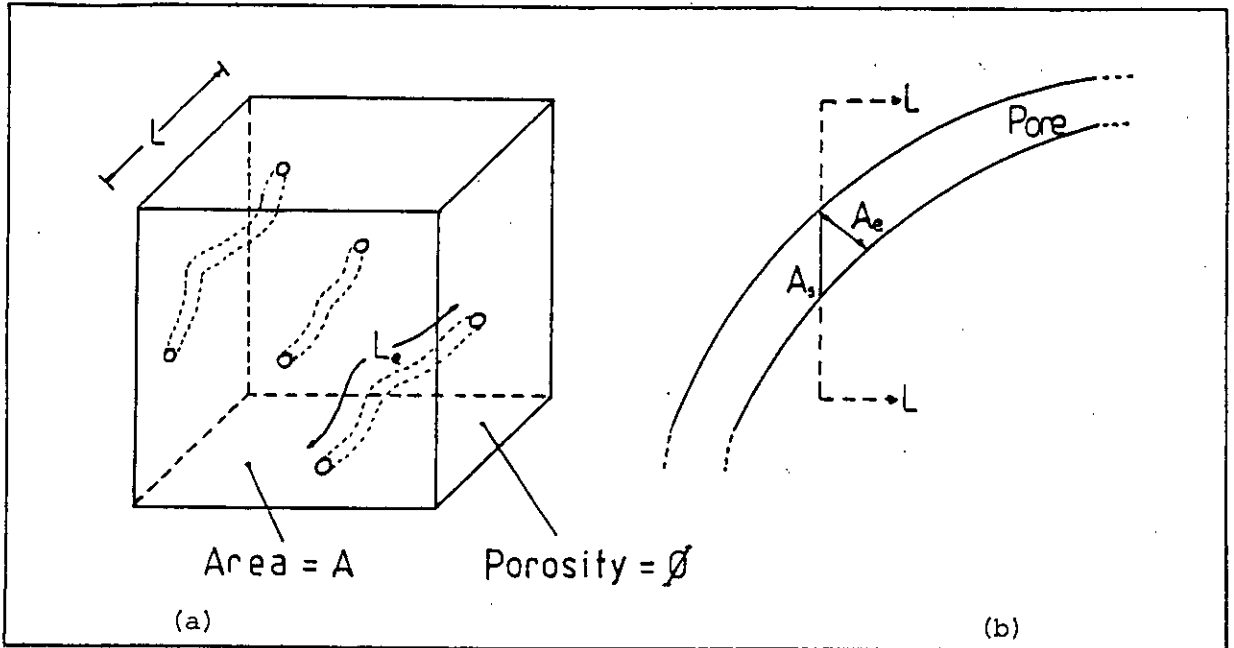


Figure 5.1(a) Diagram of porous medium illustrating external area  $A$ , Length  $L$ , and actual length of pores  $L_e$ .

Figure 5.1(b) Magnified section of a pore in rock:  $A_e$  is the cross-sectional area of the pore and  $A_s$  is the average area of the pore exposed by cross-section cut normal to the macroscopic flow direction  $L$ .

$$FF = \frac{R_p}{R_e}$$

$$R_e = \frac{\rho_e l}{A}$$

$$*R_p = \frac{\rho_e l_e}{A_e}$$

where  $R_p$  = Resistance of porous medium

$R_e$  = Resistance of electrolyte

$\rho_e$  = Resistivity of electrolyte.

\*It is assumed that the aggregate does not conduct and that the pores are interconnected.

$$FF = \frac{l_e/l}{A_e/A} \quad \dots\dots(5.10)$$

From (5.9) and (5.10)

$$FF = \frac{(l_e/l)^2}{\phi} = \frac{T_r}{\phi} \quad \dots\dots(5.11) \quad ?$$

i.e. Tortuosity =  $T_r = \left(\frac{l_e}{l}\right)^2$

From the above definition of tortuosity, it follows that the formation factor is a function of the shape of the electrically inert obstacles and of their degree of dispersion.

Research carried out by Agrawal and Bhandari<sup>145</sup> on sands, spheres and triangular prisms, has shown the formation factor increasing with loss of sphericity, see figure 5.2. The following relation was put forward:-

$$FF = \left\{1 + \frac{0.3219}{\psi} (1 - \phi)\right\}^2 / \phi \quad \dots\dots(5.12)$$

where  $\psi$  = Sphericity = Ratio of the surface area of a sphere having a volume equal to that of the particle to the surface area of the particle itself.

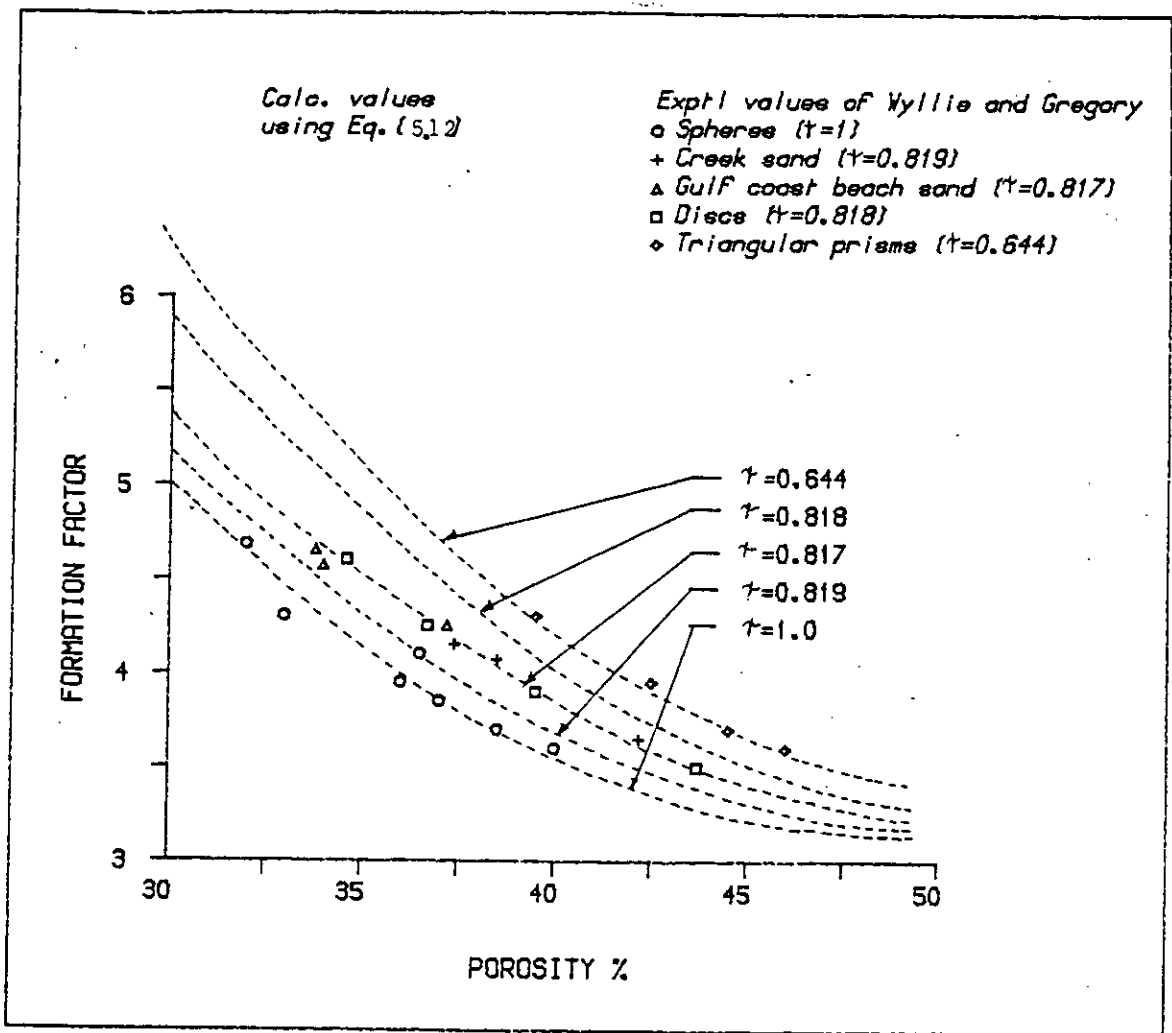


Figure 5.2<sup>145</sup>

It can be seen that, by substituting  $\psi = 1$  into the above equation, it reverts to Slawinski's formula for the conductivity of an electrolyte containing non-conductive spheres.

### 5.5 THE GEOMETRICAL CONSTRICTION FACTOR

Any explanation of high Formation Factors which depends solely on the tortuosity of uniform pore paths, necessarily ignores the effect that variations in the cross-sectional area of the conductive paths have on the resistivity of the porous body.

It is known that for two equal volumes of different sized particles, the one with the smaller particles will have the greater surface area. There will also be an increase in the number of contacts between particles and in the relative change of pore sizes along their length. It can therefore be deduced that a decrease in



particle size, accompanied by an increase in surface area, causes a rise in the constriction factor of the pore structure.

The constriction factor (c.f.) is therefore an indicator of the variation of the cross-sectional area of the pore network which, although easy to visualize, is difficult to define and impossible to quantify in practice. Owen carried out a series of tests on synthetic models containing voids interconnected by tubes and succeeded in expressing the c.f. as the ratio of pore-neck size to the pore size. His results showed that, for equal porosity and tortuosity, an increase in c.f. caused corresponding increases in formation factor values.

It is interesting to consider the effect that hydration has upon the pore network when certain assumptions are made regarding initial conditions and the manner in which hydration takes place.

Consider a number of samples identical to each other but for the relative stage of hydration. Given that a hydration effect is to progressively reduce the pore diameter available for conduction, the samples in a later hydrating stage will yield higher formation factors than the others. The rise in resistivity is, however, not only to be attributed to the amount of pores which have been completely obstructed but also to the amount and number of constrictions that the pores have experienced.

## 5.6 THE ELECTRIC DOUBLE LAYER

### 5.6.1 The Influence of the E.D.L. on the Electrical Characteristics of Cement Pastes

Electrochemical phenomenon which are inherent in water disperse systems, such as the formation of the electrical double layer, are not in general considered responsible for the binding characteristics of cement pastes although they do play a part in the hardening process.

Studies<sup>85</sup> of the electrostatic characteristics of cements before and during hydration have shown that, although clinker minerals have different electrochemical characteristics,

(Alites and Belites : -ve surface charge

Aluminates and Alumino Ferrites : +ve surface charge),

the hydrated gel is positively charged and is therefore surrounded by a -ve surface charge. The inner layer of the E.D.L. is made up of groups of anions  $\text{OH}^-$ , whilst in the diffusion region cations,  $\text{Ca}^{2+}$ , predominate.

As hydration progresses, particles bind together and a network of capillary pores is formed whose electrostatic cross-section can be divided into two regions : the E.D.L. and the disperse electrolyte.

Biryukov and Plugin<sup>147</sup> have shown that on an average grain size of cement,  $3.2 \times 10^{-6}$  m, the thickness of the E.D.L. can range from  $1 \times 10^{-9}$  to  $1 \times 10^{-8}$  m. Given the fact that capillary pores can have a diameter of as little as  $8 \times 10^{-9}$  m<sup>147</sup>, the effect of the E.D.L. is really recognizable.

Because ions in the E.D.L. are rigidly bound to the perimeter of the pore, electrical conduction in strong electrolytes is, in the main, carried out by the free ions close to the centre of the pore. In weak electrolytes, however, the higher ionic concentration in the E.D.L. offsets the lack of mobility of the ions and becomes a major contributor to the conductive characteristics.

It is therefore evident that the influence of the E.D.L. on the conductive characteristics of a pore is dependent on the pore's diameter and on the number of constrictions along its length. In micro-capillaries, the effect can be an overlapping of the diffusion area of the E.D.L. thereby causing a reduction in the effective cross-sectional area available for conduction.

Because of the relatively high electrolytic strength of the pore fluid in cement pastes, it is safe to assume the resistivity of the E.D.L. to be much higher than that at the pore core. Given that the thickness of the E.D.L. is independent of pore size and considering a porous medium, such as cement paste, with a considerable spread in pore sizes, it is evident that the resistivity of the small diameter pores will be higher due to the greater influence of the E.D.L.

Effectively, the existence of an electrical constriction

factor can be put forward, analogous to the geometric one in the manner in which pore size and pore size distribution affect the resistivity of the conductive matrix.

### **5.6.2 The Occurrence of E.D.L. in Silty Aggregates**

Clay or silt is a common impurity found in many sands and gravel aggregates, apart from fine coatings on the aggregate particles themselves. Although limitations exist concerning the allowable quantities of clay-type materials in concrete making aggregates, see the table below, it is of interest to note the implications of silt content with respect to the electrical resistivity of mortar and concrete mixes.

- Coarse aggregate 1% by weight
- Natural sand or crushed gravel sand 3% by weight
- Crushed stone sand 15% by weight

#### **Clay content limitations on naturally occurring aggregates (B.S.882: 1973)**

It can be readily shown that the conductivity of clay, and argillaceous material in general, is negligible when in a dry state<sup>148</sup>. When wetted however, they readily absorb water and, if exposed to electrolytic solutions, tend to adsorb more negative than positive ions. The result is a negatively charged clay surface attracting positive ions to its vicinity and the consequent formation of an E.D.L.<sup>149</sup> Figure 5.3 shows the ionic distribution in the vicinity of silty sand particles. As previously stated, with regard to cement paste, the effect of the E.D.L. on the overall conductivity of the system is very much dependent on pore size, pore size distribution and ionic concentration of the electrolyte.

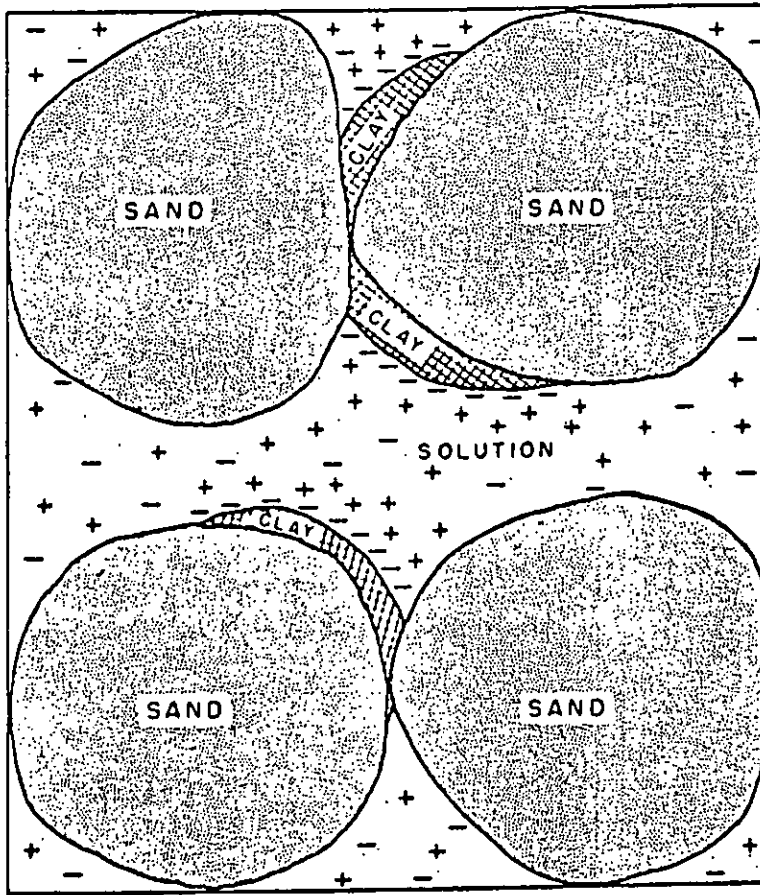


Figure 5.3<sup>149</sup>

Research by Patnode and Wyllie<sup>149</sup> has shown the resistivity of a sand/weak electrolyte system to decrease with increases in the proportion of argillaceous material present, the decrease being accentuated for mixes with high resistivity solutions.

Given the high conductivity of the cement paste's pore fluid, it is doubtful that the E.D.L. on the surface of clay intrusions would be chosen as a preferential conductive path. It is much more likely to perform as a secondary contributor to the electrical constriction factor as previously defined (Section 5.6.1).

#### 5.7 TIME DEPENDENCY OF THE FORMATION FACTOR

Unlike rock/solution systems, concretes, and cementitious materials in general, are not stable with time, their physical, rheological and electrical properties are continuously changing and

never as quickly as they are in the early hydration period. These changes, due to the setting and hardening of the cement paste, occur at the same rate in pastes, mortars and concretes, as ordinary aggregates are not involved in the hydration reactions.

The following section aims to describe how the aggregate, although not directly involved in the hydration process, affects the pore structure, and consequently the electrical characteristics, of a mix during the setting and hardening period. Use is made of the formation factor to examine the relative changes in resistivity with respect to time of concrete and pastes.

$$FF = \frac{\rho_c}{\rho_p}$$

$$\frac{dFF}{dt} < 0 \quad \text{Resistivity of the paste increasing faster than the concrete}$$

$$\frac{dFF}{dt} = 0 \quad \text{Resistivity of paste and concrete increasing at the same rate,}$$

$$\frac{dFF}{dt} > 0 \quad \text{Resistivity of concrete increasing faster than the paste.}$$

Considering an idealized fresh cement paste with uniformly distributed conductive paths ranging from  $r_0$  to  $r_{max}$ , see figure 5.4(a). As hydration progresses, and the cement paste begins to lose its plasticity, C-S-H gel is formed on the surface of the individual cement particles which, as hardening begins, joins up to that of surrounding particles to form a continuous structure. From here on, the conduction mechanism is taken up by the capillary pores within the gel.

During the setting stage, the cement particles are dispersed through the aqueous phase and the very thin crystal formations which have been produced do not affect the conductive properties of the paste to any significant extent.

As hydration continues past the start of the hardening process, the capillaries are progressively blocked off, starting

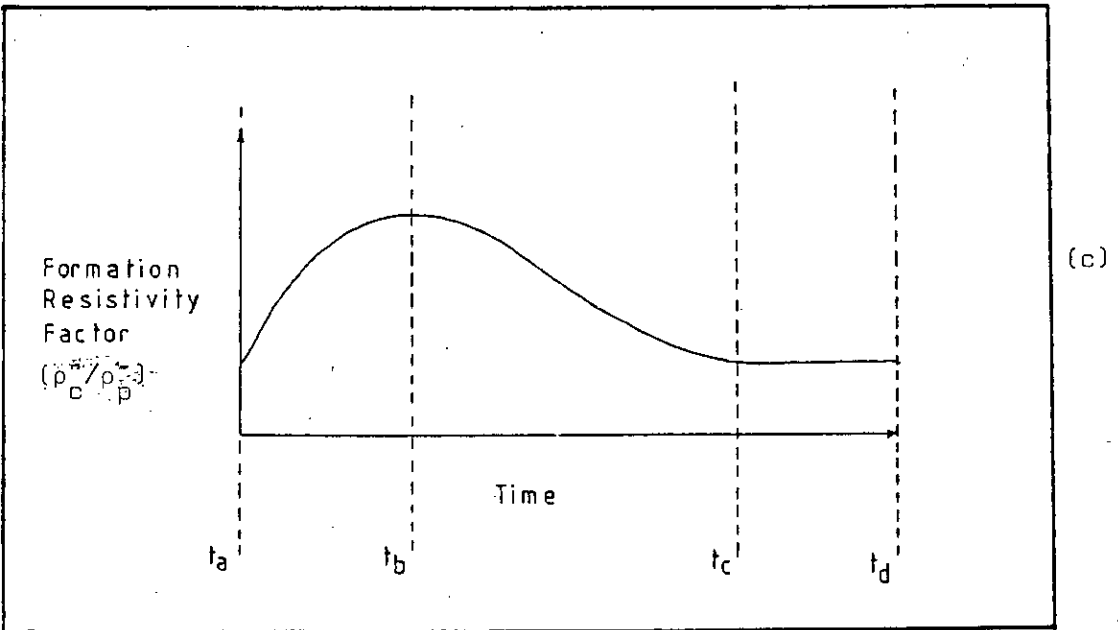
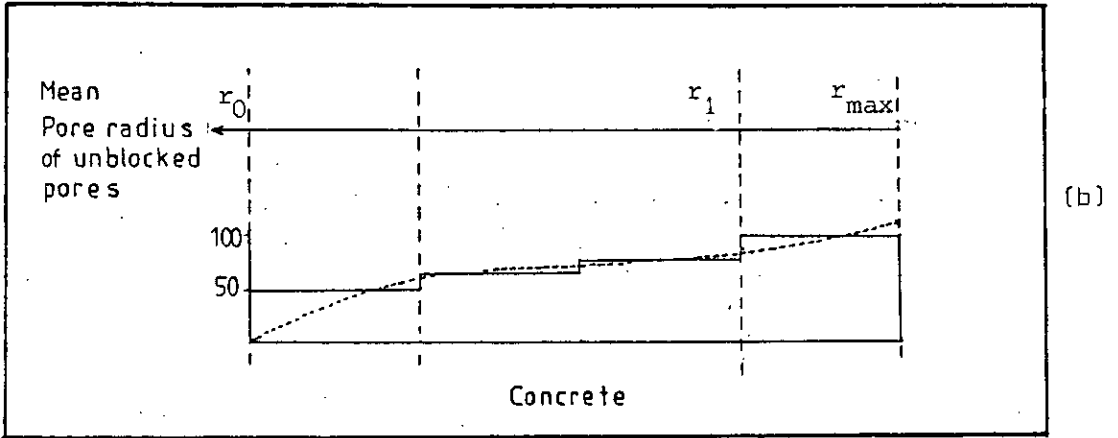
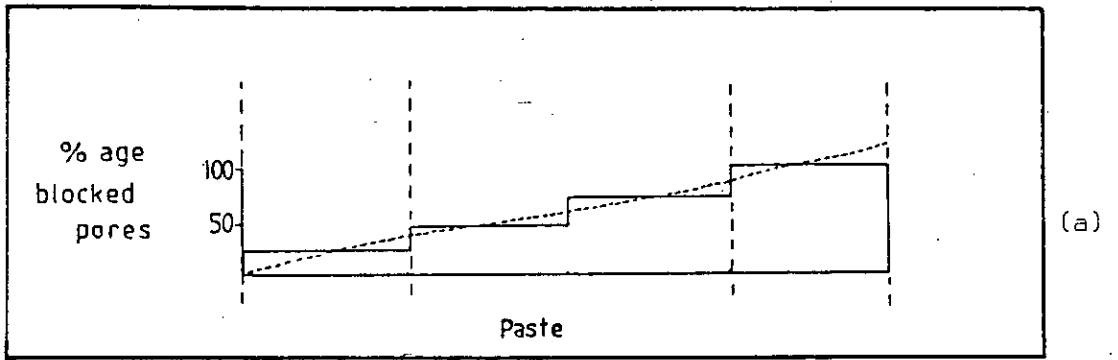


Figure 5.4 Effect of aggregate on pore size distribution and on the Formation Factor - time relationship

with the smaller diameter pores, until all have been segmented and no longer form a continuous system.

This might never occur in pastes with a high water-cement ratio where the total amount of hydrates produced is insufficient to complete the segmentation.

Considering now the effect of introducing aggregate into the system. Provided that the particles are not in contact with each other and are well spaced within the paste, the sizes of the conductive paths, and eventually that of the capillary pores, remain unaffected; the only conductive parameters which have been influenced are:

- (a) The cross-sectional area available for conduction; and
- (b) The effective path-pore length.

As further amounts of aggregates are added and the particles compact together, a third effect is introduced, the larger conductive paths are constricted to smaller diameters and a consequent shift in pore size distribution takes place. This is shown schematically in figure 5.4(b) where a large percentage of the conductive paths can now be found in the small diameter range. The effect of this shift in pore size distribution is twofold. Because of the fact that cement particles have, in some cases, been pushed very close together, they are much more readily formed into clusters by early hydration products. The presence of the E.D.L. on the particles surface makes a larger contribution to the conductive characteristics during the setting stage.

The net effect on the Formation Factor-Time relationship is evident in figure 5.4(c). Because of the increased number of low diameter conductive paths, concrete has a greater initial rise in conductivity, however, the lack in the middle to high pore size range causes the concrete's rise in resistivity to be first equalled,  $t_b$ , and then surpassed,  $t_b - t_c$ , by that of the cement paste.

## 5.8 DISCUSSION

The effect of adding aggregates to cement paste on the rate of change of resistivity with hydration has been theoretically investigated in this chapter. The variables which determine the electrical resistivity of concrete have been examined on the basis of the formation factor theory.

The theoretical work on the conduction through concrete has yielded several important conclusions:-

- (a) The aggregate phase within the concrete, although having been shown to conduct a negligible amount of electricity, has, nevertheless, an important influence on the overall resistivity of the material.
- (b) The effect of tortuosity and constriction of the capillary pores caused by the presence of aggregate has been shown to affect the relative changes in resistivity between a concrete and a paste of equal water/cement ratio.
- (c) Aside from the geometrical lengthening and constricting of the capillary system, the notion has been introduced of an electrical constriction factor caused by the presence of the Electrical Double Layer.
- (d) The formation factor theory could be a good theoretical method for investigating the conduction of electricity through concrete as a heterogeneous material, provided the results obtained above are in agreement with those achieved experimentally.



**CHAPTER SIX**

**APPARATUS AND EXPERIMENTAL PROCEDURE**

The Author was responsible for the design and construction of the curing tank, hoist system and moulding units. The development of the electronic automated measuring unit was carried out by J. Wilson, a research student in the Electrical Engineering Department at the University of Edinburgh.

## 6.1 INTRODUCTION

The large amount of experimental effort required in the testing of concrete has been the main reason for the limited research on the electrical resistivity characteristics of different mixes. The heterogeneous nature of concrete as a material makes it imperative to test more than one sample per mix for statistical significance. The testing, if executed manually, is tedious and extremely laborious to carry out. A review of methods which have previously been employed to measure resistivity has shown marked differences in both equipment and procedures.

The system described in this chapter allows automatic measurement of a large number of samples over an extended period of time and has been developed on the basis of a critical analysis of previously employed methods.

An overall view of the apparatus is shown in plate (6.1).

## 6.2 EXPERIMENTAL OBJECTIVES

The main aim of the testing programme is to develop relationships between electrical resistivity and physical parameters such as mix proportions and compressive strength. In the course of the investigation however, it is hoped to achieve a better understanding of both the hydration process and the conduction mechanism through the concrete as a composite material.

Planned objectives were set initially which could be classified under the following categories:

- (1) To develop suitable apparatus and experimental techniques.
- (2) To relate electrical resistivity characteristics of mixes during a 28-day period.
- (3) To relate electrical resistivity changes to the physical - chemical processes occurring during hydration.
- (4) To investigate the possibility of relating resistivity parameters to mix proportions such as percentage aggregate content and water/cement ratio.
- (5) To establish relationships between resistivity and strength.

These objectives were however constrained by a number of factors which are discussed in the next section.

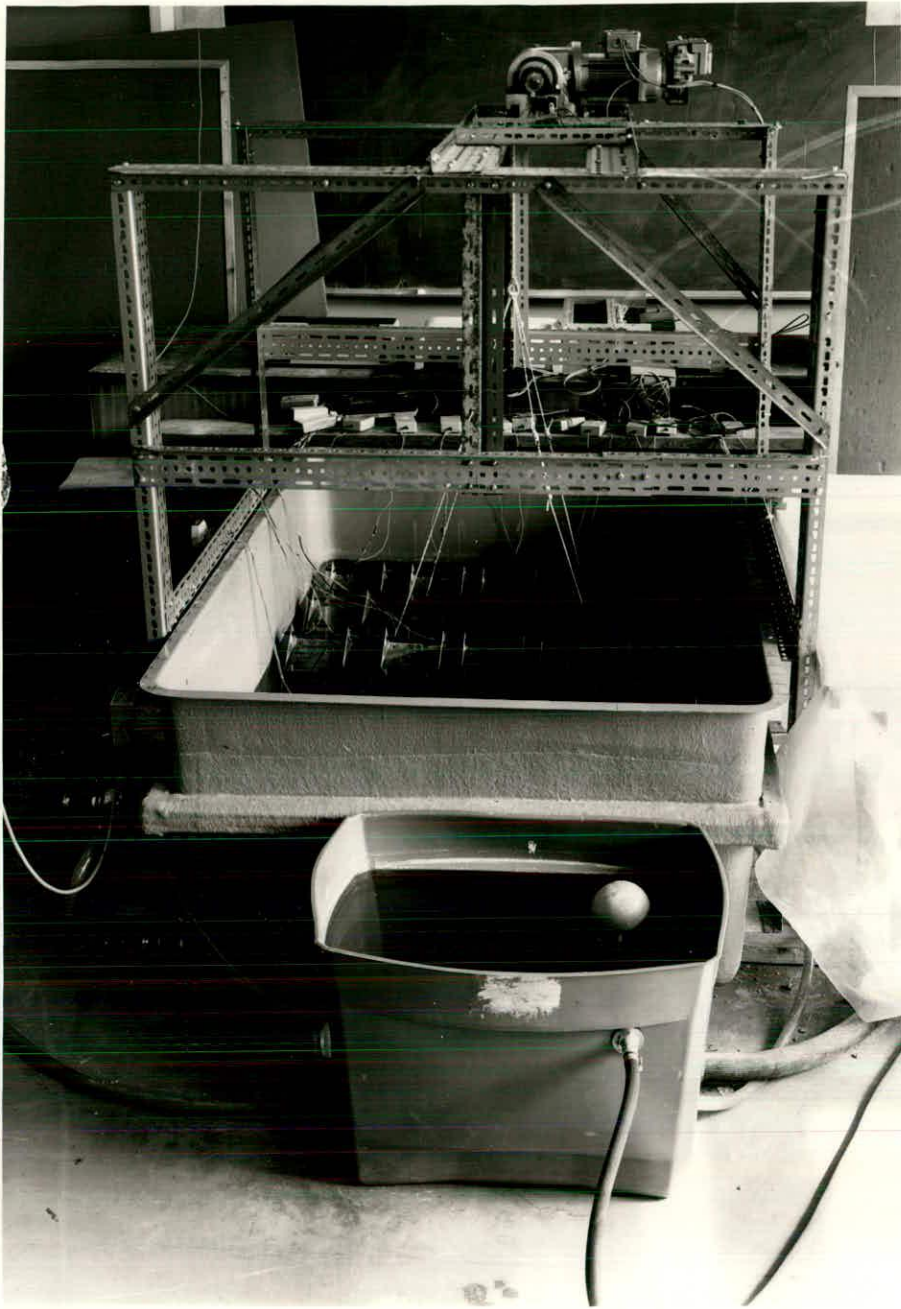


Plate 6.1 :Overall view of apparatus

## 6.3 EXPERIMENTAL CONFINES

### 6.3.1 Physical Restrictions

British Standards governed the size and shape of the specimens, the way they were prepared, the environmental conditions to which they were exposed during the length of the curing period and the manner in which they were tested (see appendix 1).

B.S.1881: Part 3: 1970 "Methods of making and curing test specimens"

B.S.1881: Part 4: 1970 "Methods of testing concrete for strength"

Because of the susceptibility of concrete to changes in the properties and composition of the constituent materials, these were, wherever possible, maintained constant for the duration of the programme. Analysis of the materials employed is presented in detail in section 6.5.1.

Given the infinite number of possible concrete mixes, this study was restricted, in the main, to fully compactable nominal mixes of common use although rich mixes (high in cement content) were also investigated. See Table 7.1.

### 6.3.2 Electrical Requirements

The restrictions placed on the electrical properties of the equipment can be categorized into the following:

- (a) Voltage size.
- (b) Current type.
- (c) Frequency of the measuring pulse.
- (d) Measurements during the water curing stage.

(a) The voltage employed for the resistivity measurement must (i) be low enough to prevent it from interfering with the chemical reactions in the mix; and (ii) be sufficiently high to override the electrokinetic potential of the pastes which, in certain cases, has been measured at  $0.24^{147}$  Volts. Four Volts was therefore selected as the measuring voltage.

(b) As shown in section 4.1.4.2 (iv), direct current causes formation of gas at the electrodes and has the effect of making it progressively more difficult for the ions to reach the electrodes thereby increasing resistivity. To counteract this phenomenon, alternating current was employed but, as electrode processes are minimized, the polarization of the electrolyte with each change in direction of electrical flow becomes the most significant adverse factor in the direct measurement of true resistivity. The effect of polarization is to introduce a large equivalent electrical capacitance in series with the resistance of the concrete. Allowances for this capacitance will have to be made in the a.c. measurement of resistivity, see measurement technique section 6.4.5.1.

(c) Research by McCarter<sup>150</sup> shows a large drop in resistivity when switching from direct to alternating current which, although it initially continues with pulse frequency increases, becomes minimal at frequency values of over 1 KHz (e.g. raising the frequency from 1 KHz to a MHz only decreases resistivity by approx.  $10^{-2}$  ohm-m). The use of even higher frequencies, in the Gegahertz region would result in what is known as the 'skin effect' whereby conduction tends to be confined to a thin surface layer and not through the bulk specimen. A current frequency of 2000 Hz was therefore adopted for measurement purposes.

(d) Although the conductivity of water is low, it is nevertheless significant when compared to the values measured during the investigation and it is therefore impossible to monitor resistivity while the cubes and electrodes are submerged. It is also necessary to have the concrete in a surface dry condition so that no conduction can take place along the surfaces of the cubes.

An automatic hoisting mechanism was therefore developed to lift the specimens above the surface of the water at the measuring stage.

## 6.4 EXPERIMENTAL DESIGN

### 6.4.1 Electrode Geometry

Because of the need to employ samples which could eventually be tested for compressive strength in accordance with B.S.1881, external plate electrodes were adopted for experimental purposes.

The electrodes employed consisted of thin, 14 s.w.g., stainless steel plates positioned on opposite faces of the 150 mm specimen cubes. Each plate was designed to cover one whole surface of the cube while extending far enough above it to keep the electrical connections dry when the samples were cured under water. See plate (6.2).

Each electrode was provided with a socket connection to the measuring device while one, out of each pair, was also fitted with a temperature transducer thereby allowing the resistivity readings to be corrected to 20°C.

The electrode configuration, in combination with the specially developed moulding units, see section 6.4.2, allowed the electrodes to be included in the testing process from the casting stage. Aside from enabling measurements to be taken almost immediately after batching, this configuration offered two other main advantages:

- (a) It provided a well defined conductive area, difficult to achieve with embedded electrodes.
- (b) It allowed a good bond to be made between concrete and electrodes.

The interface between the electrodes and the concrete surface is very important. If the area of surface contact were to vary from cube to cube, resistivity readings could not be calculated correctly and experimental results would be meaningless.

As it was expected that electrodes would detach from the cube surfaces on demoulding, various forms of conductive glues and clamping devices were considered. It was found, however, that by casting the electrodes in the mould, with the cube, a significant natural adhesion developed between the two surfaces to produce an

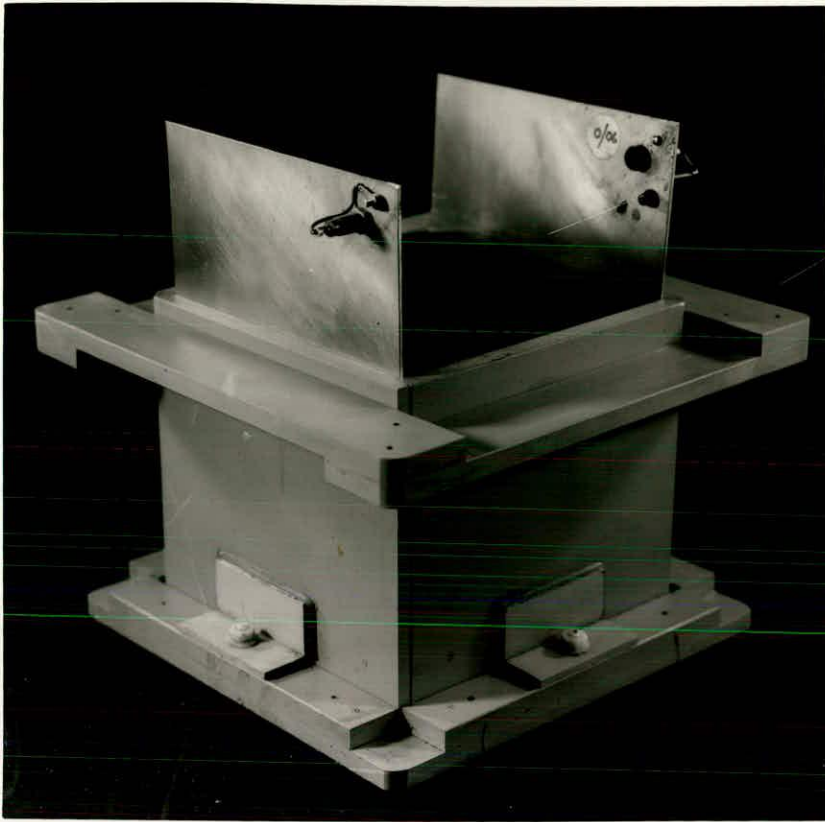


Plate 6.2 : Mould and electrodes



Plate 6.3 : Specimens during initial 24 hours of test



ideal interface. The bonding was sufficiently strong to maintain a sound connection throughout the duration of the testing period.

#### 6.4.2 Moulds

During the initial hydration period, concrete does not possess sufficient strength to maintain any required shape and has to be cast into suitable moulds. For the purpose of this study, it was decided to carry out tests on 150 mm cubes, a size which was considered sufficient to hold a representative quantity of material.

Conventional concrete moulds for producing standard cubes are made of cast iron (B.S.1881: Part 108: 1983) but their implementation was found to be problematical for the following reasons:

- (1) So that face electrodes could be cast with the concrete, shims had to be fitted to the mould in order to achieve the required dimensions.
- (2) Concrete and electrodes had to be electrically insulated from the cast iron surfaces to prevent short circuiting of the system. The moulds were therefore lined with thin polythene sheeting, a process which proved effective in many cases, although time consuming to prepare. The major difficulty was found to be in preventing the polythene sheeting from puncturing during vibration.

Given the number of cubes to be tested, it was decided to design and develop suitable moulds, (see appendix A2.1). Between fibreglass and P.V.C., both good electrical insulators, the latter material was chosen for its greater durability and ability to achieve close tolerances ( $\pm 0.25\text{mm}$ ). The moulds, including nylon nuts, bolts and washers, were therefore fabricated entirely of non-conductive materials, see plate (6.2).

(NOTE : The moulds were supplied by I.C.L. "Technical Plastics Limited : Registered Office Grovepark Mills, Hopehill Road, Glasgow, G20 7NF.)

### 6.4.3 Curing Tank and Tray System

After the initial 24 hour curing period under polythene covering, the concrete specimens acquire sufficient strength for the moulds to be removed and the cubes placed under water for the remainder of the curing period.

A wooden tray and a fibreglass tank were employed for this purpose as both fibreglass and wood offer the advantage of being non-corrosive and electrically inert. The curing tank contained one tray capable of holding up to 15 cubes.

The tray was fitted with inclined supports holding the cubes at an angle of  $40^\circ$  to the horizontal thereby allowing drainage of free water from the top surfaces as the specimens were lifted out of the water. Anchor points were provided to which the nylon lines, connecting the tray to the hoisting mechanism, were fixed. Tensioners were also provided to ensure that the tray would rise without swaying and remain parallel to the water surface. (Schematic diagram of tray: figure A2.3).

The temperature of the water in the tank was maintained at  $20^\circ\text{C}$ , ( $\pm 2$  degrees) as specified by B.S.1881: Part 111: 1983. In order to provide sufficient room for the inclusion of a second tray, an extra tank was added to the system to accommodate for the pumping and heating apparatus. Figure A2.2 gives a schematic representation of the tank system layout.

The water temperature in the curing tank was monitored by a TRAFAG thermostat connected to a relay mechanism which, as the temperature dropped below  $20^\circ\text{C}$ , triggered on both the pump and the immersion heater.

While the pump was therefore responsible for the flow of hot water into the curing tank, back flow was ensured by a large diameter pipe connecting the back of both tanks. A Technoterm-Digital instant action thermometer, (accurate to  $0.1^\circ\text{C}$ ), was employed to verify that temperature gradients between different areas of the tank were negligible.

In order to maintain a constant head of water, the heating tank was connected to the water mains via a ball-cock mechanism (plate 6.5).

#### **6.4.4 Hoisting Equipment**

To fully automate the experimental procedure, a hoisting system was developed to lift the cubes from the curing tank prior to each reading being taken.

A Dexion frame was designed and constructed to support the hoisting equipment and two fully loaded trays. The electric motor selected was a NECO DR D71 single phase geared motor with capacitor start and fitted with an electro-magnetic drum brake. A maximum torque output rating of 100 lbs.f.ft with a shaft rotational speed of 1.06 r.p.m. proved sufficient to lift a maximum design weight of 200 kgs clear of the water surface.

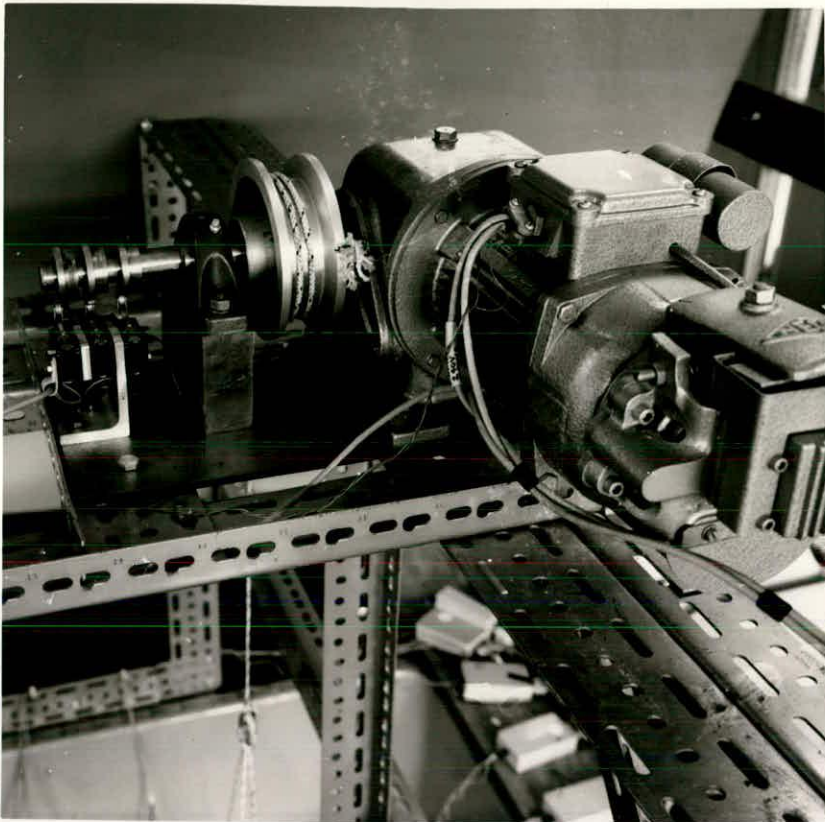
The motor shaft was supplied with cams which, when rotating, engaged or disengaged microswitches thereby controlling the end position of the lifted tray. As a safety precaution, an override microswitch was also provided to disconnect the mains if a fault in the system were to occur and the other switches bypassed. (Schematic diagram : figure A2.3, plate 6.4)

#### **6.4.5 Automatic Resistivity Measurement System**

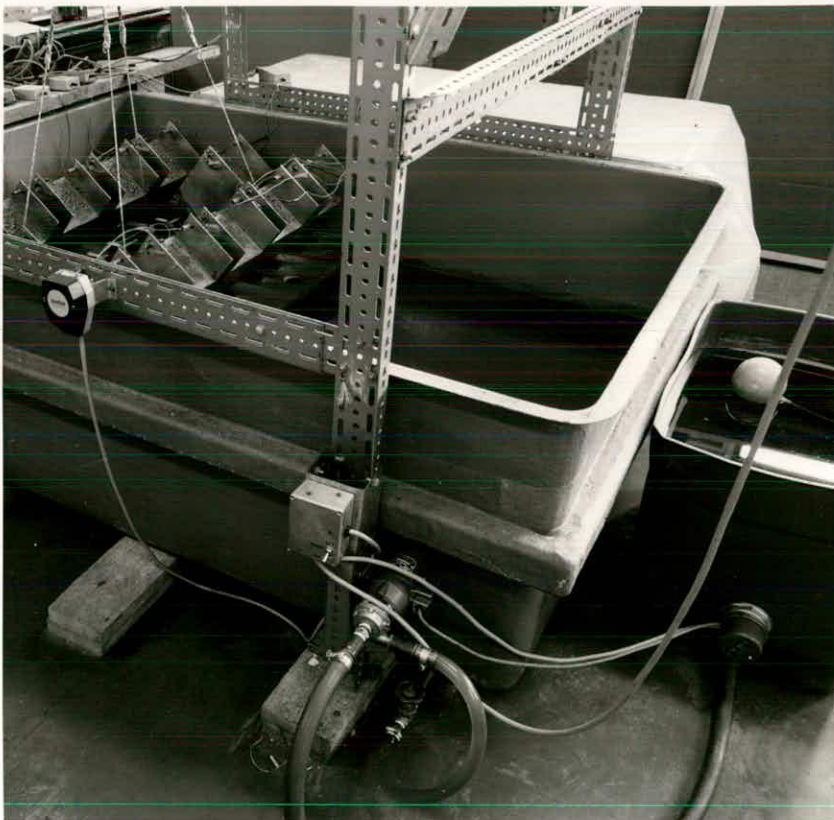
##### **6.4.5.1 Measurement Technique**

As described in section 4.1.4.2 (iv) applying alternating current to an electrolytic conductor such as fresh concrete causes the material to store a certain amount of electrical charge as the electrodes reverse polarity. The resulting effect is that of a capacitor in series with the concrete's resistance.

Previous investigators have, in certain cases, resorted to monitoring both resistance and capacitance of the sample in order to calculate the correct value of resistivity.



**Plate 6.4 : Hoisting mechanism**



**Plate 6-5 : Curing tank ; water temperature and level control system**

This procedure is laborious and time consuming to perform, and an alternative solution was therefore developed<sup>107</sup> which could be easily and effectively implemented by the automated electronic system.

A pulse frequency of 2000 Hz was employed to minimise polarization effects at the electrode - aqueous phase interface, and a square input waveform was adopted as opposed to the conventional sinusoidal one.

The layout and output pulses are shown diagrammatically in figure 6.1. The rise in voltage during each half cycle of output wave is caused by the polarization capacitance effect.

Analysing the output signal by means of Laplace transform, Wilson et al<sup>107</sup> have shown that the effect of the rise can be eliminated if measurements are taken at  $T/4$ . Knowing the value of  $V_o$ , the series resistance  $R_e$  and  $V_i$ , the input voltage, the resistance of a sample can be calculated by measuring the amplitude of the output waveform.

#### 6.4.5.2 System Operation

Using the 1 MHz clock signal produced by the computer, the timing circuit is able to generate all the wave forms necessary for the operating system. This includes a signal every 43.2 seconds which is equal to  $0.0005 * \text{Day}$  and is the basic timing interval in use. The timing circuit also produces 2.0 KHz and 7.2 KHz signals which activate the resistivity measurements and the temperature transducers respectively.

The resistivity is measured by (a) amplifying the 2.0 KHz square wave signal received from the timing circuit, passing it through the concrete cube via high accuracy resistors and (b) measuring the potential drop across the specimen.

The temperature of each concrete cube is measured by a platinum film temperature transducer which receives a signal that has been amplified from the timing circuit (7.2 KHz signal). Temperature increases within the sample will cause a corresponding

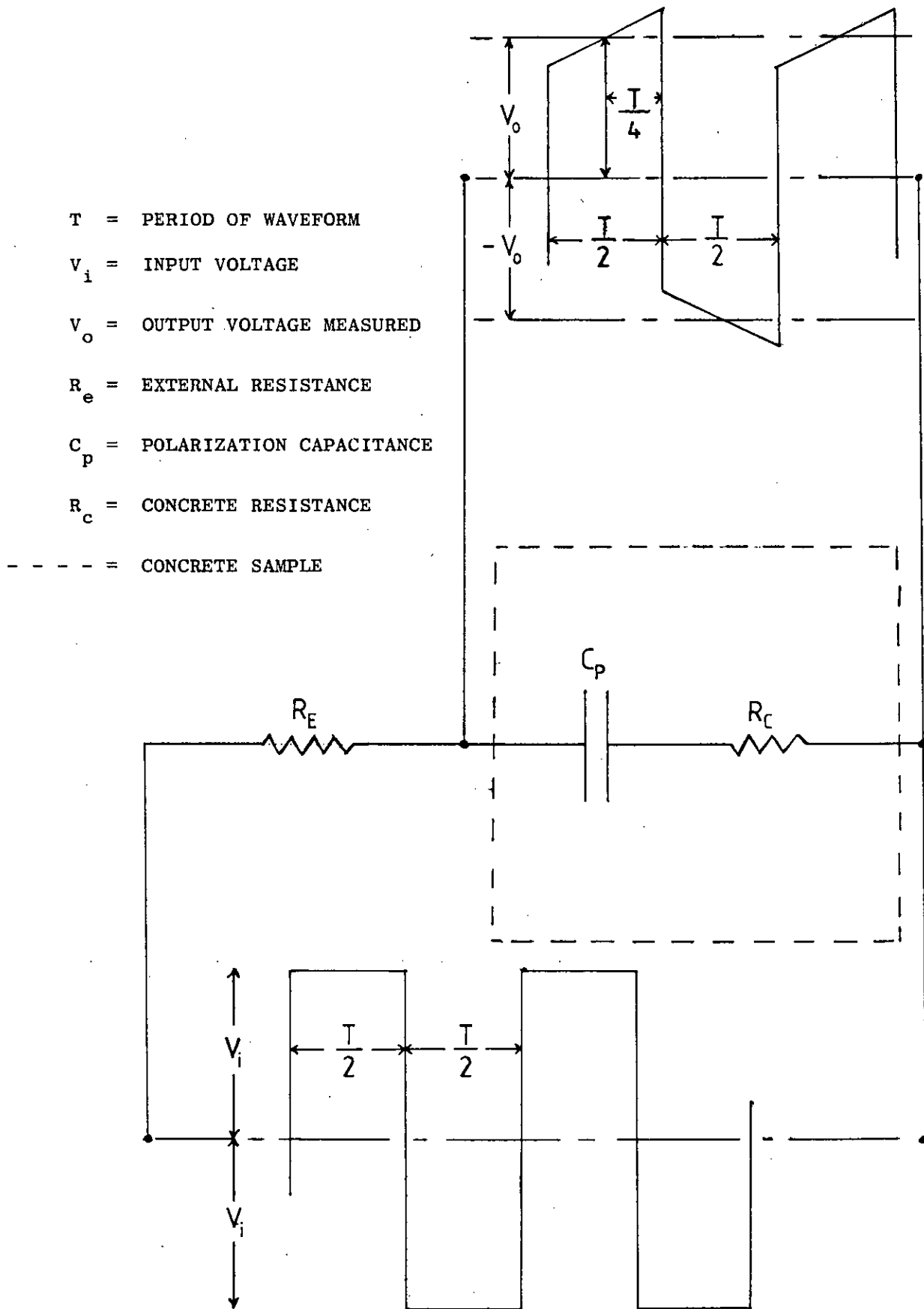


FIGURE 6.1 : Schematic representation of measurement technique.

drop in potential across the sensor which is then amplified and converted into digital form for the computer to employ.

At predetermined times the computer selects, in sequential order, the resistivity and temperature measurements of the various specimens via the respective multiplexing units. The function multiplexer then takes each reading one at a time and puts it through an A/D (analogue to digital) converter, hence to the computer. Once all the data has been gathered, the computer proceeds to calculate the resistivity, temperature and resistivity corrected to 20°C. A typical output from the dot matrix printer for a one sample experiment is as shown below:

```
010.0000          - Actual time (days)
UNIT TIME 00.0650 - Time that has elapsed since water
                  addition (days)
0/00             - Address label of cube
011.2  21.1  011.5 - Res (ohm.m), Temp(c), Corr. Res
                  (ohm.m)
```

The computer also controls the hoisting system, the upper and lower limits of movement of the tray being governed by limit microswitches.

The computer program controlling the overall process is stored in an Erasable Programmable Read Only Memory (EPROM) located in the electronics unit. (see plate 6.7 - figure 6.2)

#### 6.4.5.3 Accuracy Trial

Before testing commenced, and at regular intervals during the course of the experiment, the system was checked for accuracy.

Figure 6.3 shows the percentage error in the resistivity readings obtained from the system. Readings from both resistors and concrete mixes are compared to those accurately determined using a Wheatstone bridge.

The graph shows relatively constant accuracy limits ( $\pm 2\%$ ) for resistivities greater than 2 ohm.m, this being the lower limit of

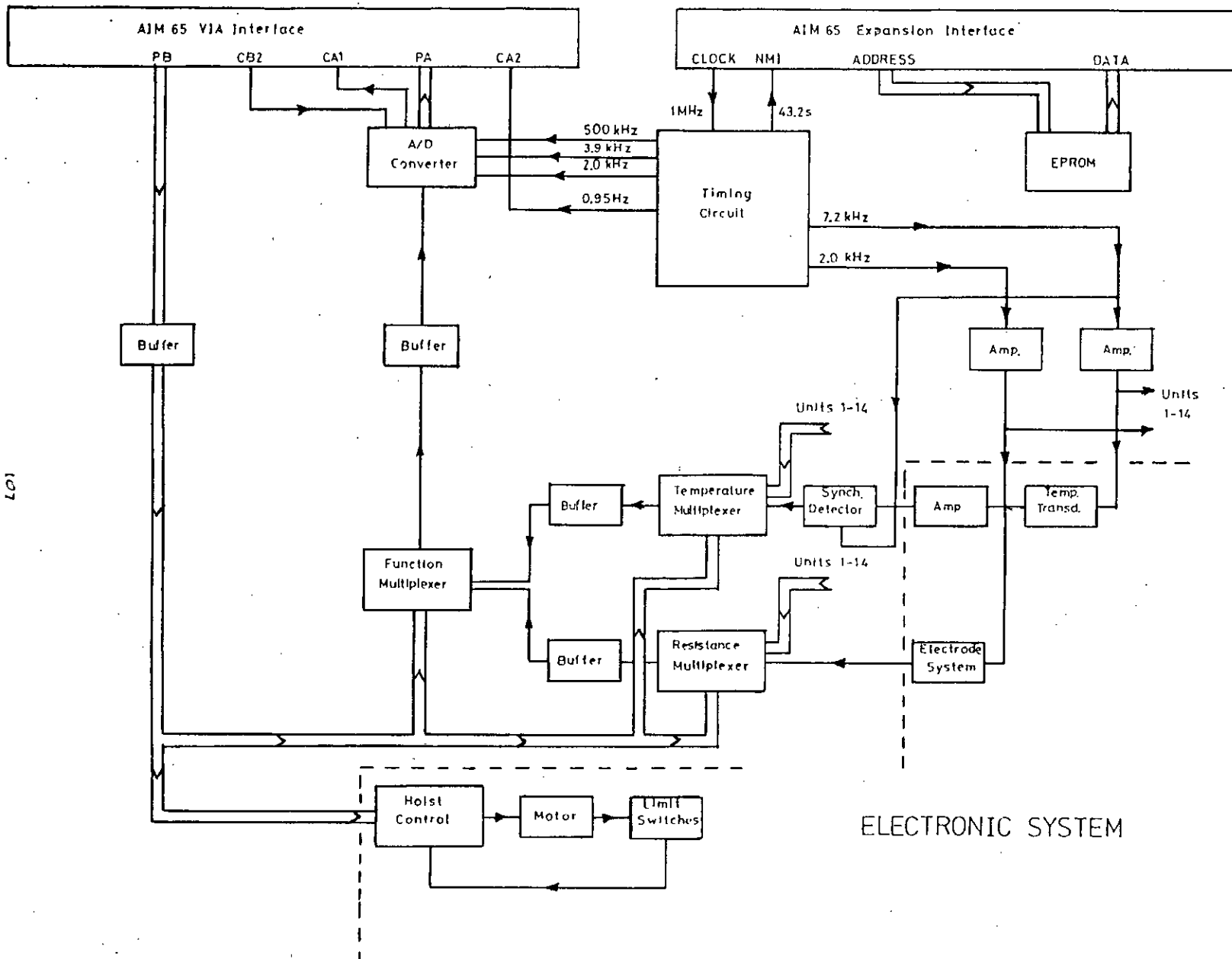


FIGURE 6.2 107





Plate 6.6 : Data output – storage and plotting system

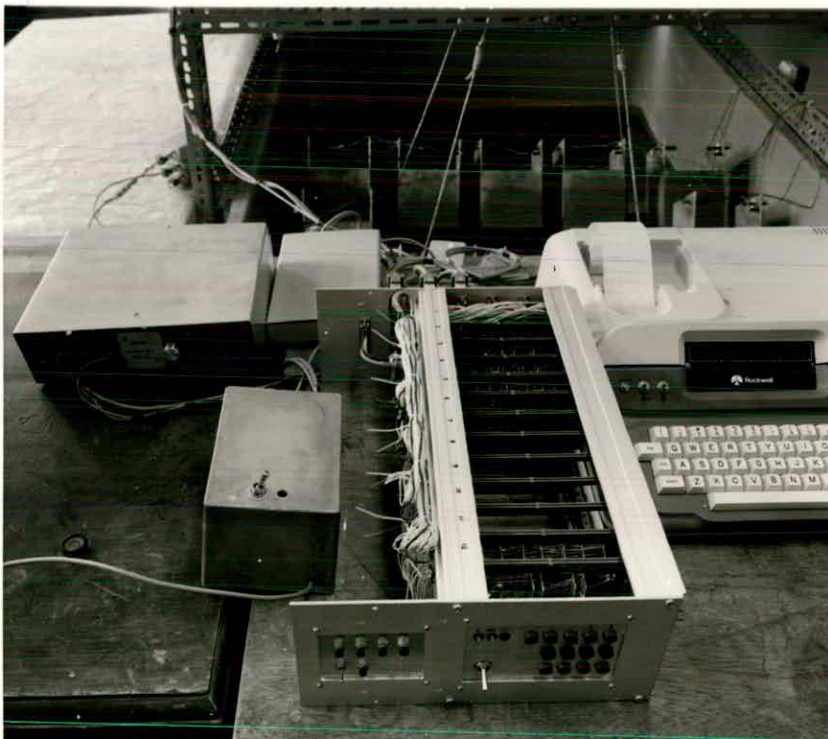
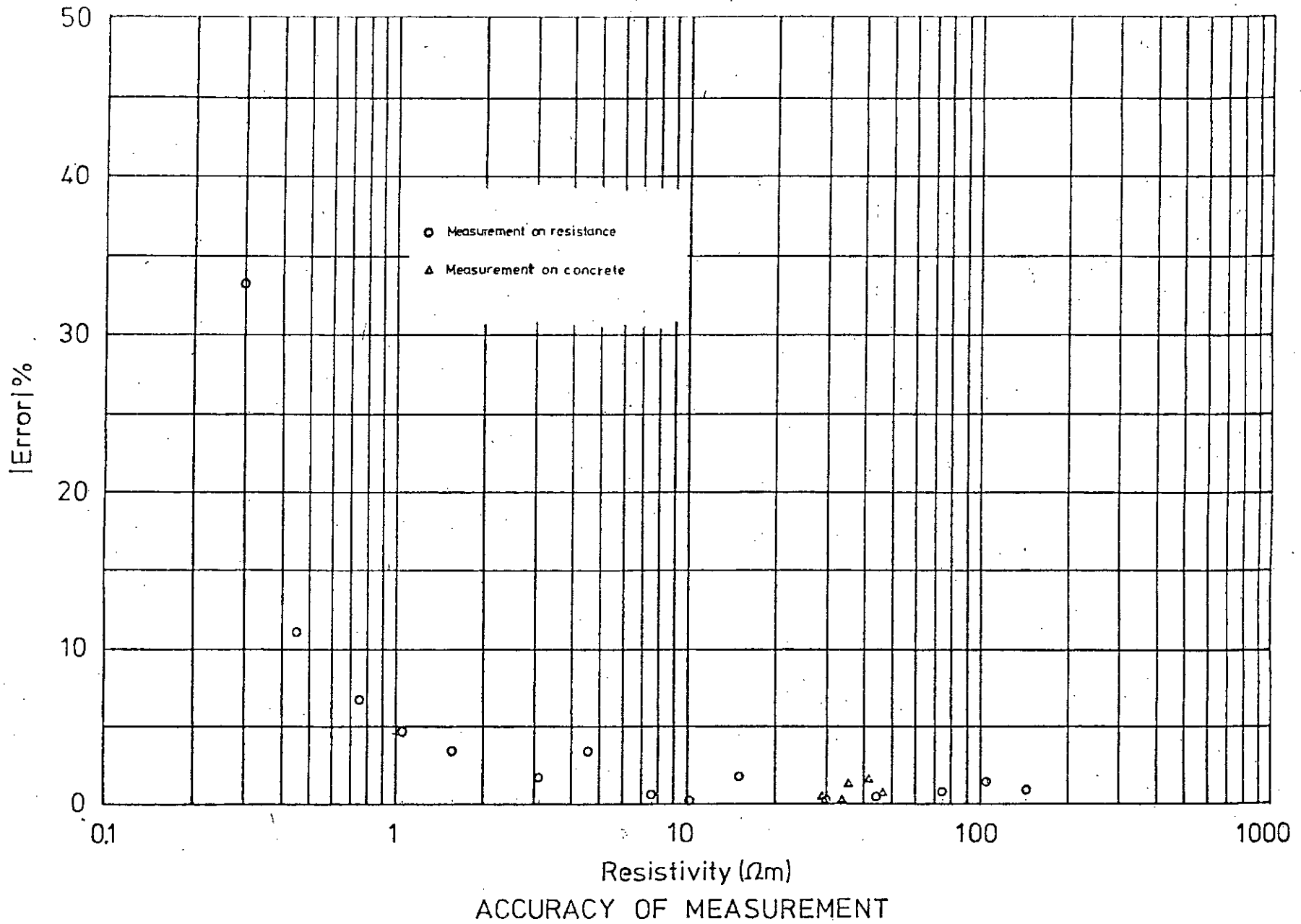


Plate 6.7 : Automatic resistivity measurement system

FIGURE 6.3



the resistivity range for nominal concrete mixes. Because of inaccuracies in the analogue to digital conversion of the small voltage drops induced by smaller resistivity readings, the accuracy of the measurement drops drastically when dealing with resistivity less than 1 ohm.m in magnitude.

## 6.5 EXPERIMENTAL PROCEDURE

### 6.5.1 Selection and Properties of Constituent Materials

**WATER:-** The main criterion employed in adopting tap water as mix water was its obvious conveniency, although resistivity tests showed it to be too poor a conductor to make a significant contribution to the conductive mechanism of a concrete mix. Tap water resistivity was measured at 230 ohm.m, negligible when compared to that of cement paste, 2 ohm.m.

**CEMENT:-** Ordinary Portland Cement was employed throughout the testing programme, and care was taken to ensure a continuous supply of fresh material.

Cement was stored in air tight containers when not in use to prevent humidity affecting its hardening characteristics. Typical chemical and mineralogical analysis of the cements employed during the testing period is given in chapter 3 section 3.2. The relative density of Ordinary Portland Cement was measured at 2.99 (see Appendix 4).

**AGGREGATE:-** One fine and two coarsely graded aggregates were employed for test purposes. These had to comply with the following criteria:

- (a) Be available locally in sufficiently high quantity to ensure a continuous supply during the experimental programme.
- (b) Comply with grading zones specified by B.S.812: Part 1: 1975.
- (c) Be representative of material commonly used in practice.
- (d) Be free from water soluble salts, clay and other organic impurities liable to interfere with the hydration process of the cement paste.
- (e) Have, as the majority of rocks do, a high electrical resistivity.

A petrological analysis of the aggregates which were selected yielded the following:

Sand : Concrete sand (Quartzite) of varied crystal size, angular particle shape. Relative Density = 2.608. Grading zone 2.

Gravel : Granite (Granulite), coarse grained crystalline structure, rounded particle shape, smooth surface texture.

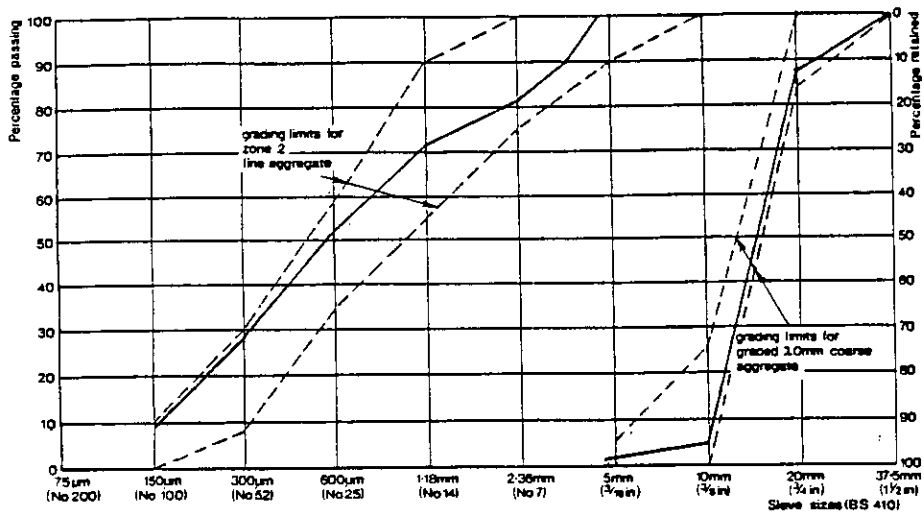
Relative Density = 2.62. Grading as shown in figure 6.4.

Whinstone : Basalt (Dark grey Olivine Tholeite), fine grained crystalline structure, angular particle shape with a granular surface texture. Relative Density = 2.69. Grading as shown in figure 6.5.

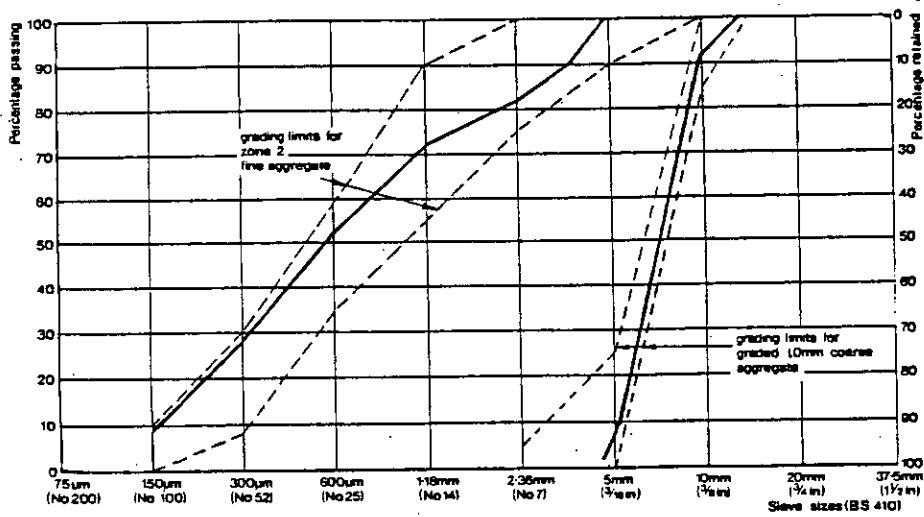
The materials selected satisfied the preset requisites as follows:

- (i) All aggregate employed was obtained directly from quarries around the Edinburgh area. Stocks were replenished at 6 month intervals.
- (ii) The grading characteristics were as shown in figures 6.4 and 6.5.
- (iii) Economics dictate the use of local aggregates in all but the extremely specialised Civil Engineering works. The aggregate employed, although particular to the geological conditions of the Edinburgh region, are widely employed locally.
- (iv) Aggregates employed are igneous in nature and therefore free from organic impurities. As a precautionary measure, however, the aggregates were washed prior to use to remove any fine particles of silt and dust.

The table below shows the result of chemical analysis carried out on samples of the three aggregates.



(a) Concrete sand and 20mm granite gravel



(b) Concrete sand and 10mm granite gravel

Figure 6.4: Grading characteristics of concrete, sand and gravel aggregate

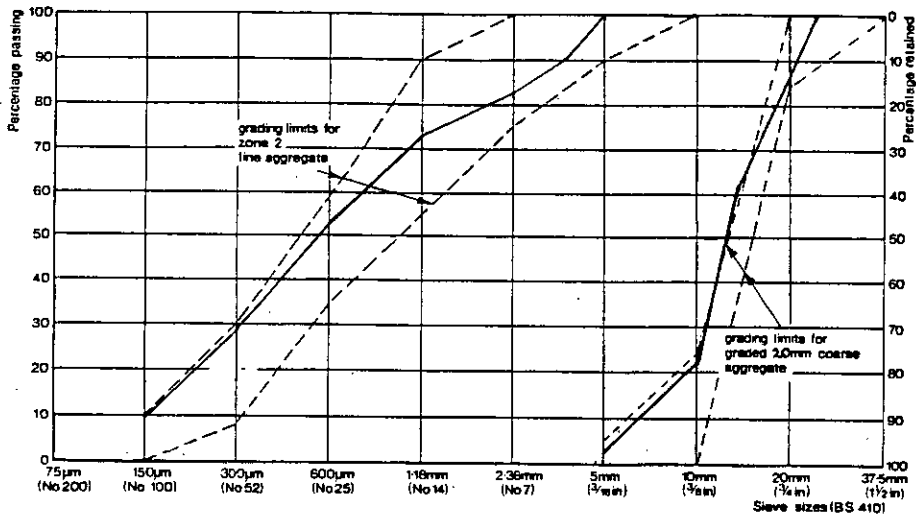


Figure 6.5 Grading characteristics of concrete sand and 20mm whinstone aggregate

Materials %Composition	SAND	GRANITE	BASALT
SiO <sub>2</sub>	80.72	73.82	54.00
Al <sub>2</sub> O <sub>3</sub>	5.95	10.78	16.00
Fe <sub>2</sub> O <sub>3</sub>	4.01	4.01	12.46
CaO	1.75	1.61	6.20
Na <sub>2</sub> O	1.00	2.16	4.10
TiO <sub>2</sub>	0.50	0.54	2.50
P <sub>2</sub> O <sub>5</sub>	0.08	0.12	2.2
MgO	1.1	1.47	2.4
K <sub>2</sub> O	0.96	1.88	-
MnO	0.07	0.08	-
TOTAL	96.15	96.47	99.86

(v) Electrical resistivity of the aggregates employed were<sup>151</sup>:

Sand (Quartz) =  $3.8 \times 10^4 - 1.2 \times 10^{12}$  ohm.m  
 Gravel (Granite) =  $5 \times 10^3 - 1 \times 10^6$  ohm.m  
 Whinstone (Basalt) =  $1 \times 10^5 - 1.4 \times 10^7$  ohm.m

### 6.5.2 Preliminary Operations

Prior to batching, all materials and apparatus were prepared to ensure constant test conditions throughout the length of the experimental programme. The following procedure was adopted:

The aggregate to be used was; (a) washed to remove any silt and dust and, (b) oven dried to minimise fluctuations in the water content from batch to batch.

Electrodes were cleaned of any solid residue, and were washed and dried to prevent oxidation of the wet surfaces. The accuracy of the temperature gauges was checked at regular intervals by immersing the electrodes in water at a temperature of 20°C. If necessary the units were then calibrated.

In assembling the moulds, electrically - chemically inert silicon grease was applied to the joints between the different components to prevent fluid loss during compaction.

Although the curing tank water was changed at regular intervals, it was nevertheless checked during the testing programme to ensure that its alkalinity was sufficiently low not to have any significant effect on hydration reactions. This was carried out by means of conductivity measurements, the minimum resistivity observed being 10 ohm.m.

The alkalinity of the water was measured at regular intervals and found to oscillate around a pH value of 7. The difference between the alkalinity of the curing water and that of the aqueous phase of the cement paste (pH=13) being sufficiently small to prevent any significant leaching out of  $\text{Ca}^{++}(\text{OH}^{2-})$  ions from the specimens.

Care was also taken to ensure that the constituent materials, electrodes and moulds were kept at a temperature of 20°C prior to batching.

### 6.5.3 Batching Procedure

To enable accurate mix proportioning all constituents of the concrete were batched by weight, and each batch was carried out in sufficient quantity to allow for the testing of a representative number of samples. When investigating the resistivity - strength relationship with time, large batches, involving 15 cubes were prepared so that cubes could be removed at regular intervals for crushing.

To ensure efficient batching, two different mixers were used. A tilting mixer was employed for batches involving five cubes or less while for greater numbers a pan mixer was used.

Compaction was carried out by means of a vibrating table.

Once properly compacted the cubes were immediately connected to the automated resistivity measuring system.



#### 6.5.4 Curing Conditions

The cubes were, for the first 24 hours, cured in a temperature controlled environment under polythene covering, at an ambient temperature of 20°C.

Subsequent to demoulding specimens were disconnected, placed on a tray in the curing tank and reconnected up to the measuring equipment.

As it is not possible for accurate resistivity readings to be taken while the cubes are submerged, the curing tray was lifted out by an automatically controlled hoisting mechanism. Prior to a resistivity measurement being taken, the hoist lifted out and held the cubes above the water surface for approximately 4 minutes, a sufficient interval to allow for the drainage of any free water from the cube's surface. Once the readings had been taken, the cubes were repositioned immediately in the curing tank (See plate 6.8).

The cubes were cured in water for up to 28 days.

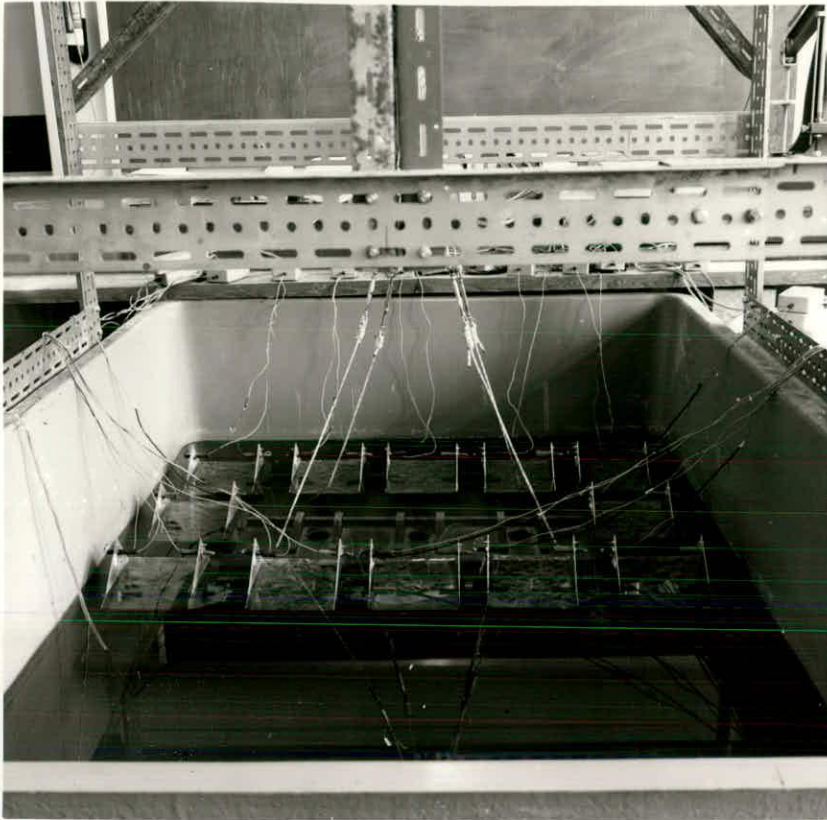
#### 6.5.5 Weight and Strength Measurement

The weight of the concrete samples was taken at day one and also prior to testing at the crushing stage. The aim of weight measurement was twofold:

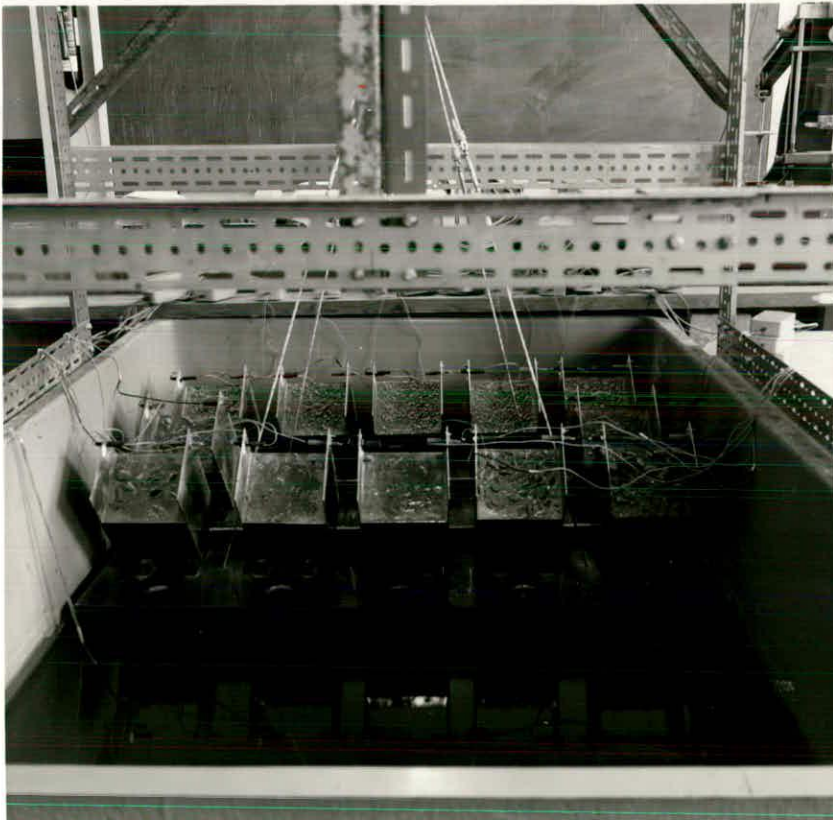
- (a) It allowed for the calculation of concrete density; and
- (b) Comparing the weights of the cubes before and after curing in water, enabled an assessment to be made regarding permeability of the mixes.

To measure compressive strength, a 2000 KN Avery-Dewson load testing machine (type 353) was employed. The machine was tested in accordance to B.S.1610: 1964 "Methods for the load verification of testing machines" (maximum error in accuracy of  $\pm 1\%$ ).

The specimens were tested at 28 days, unless compressive strength-time relationship was being investigated in which case testing was carried out at intervals during the 28-day period. As



a.



b.

Plate 6.8 : Positioning of cubes :

a. During curing

b. During resistivity measurement

for resistivity measurements, a minimum number of 3 cubes was employed for compressive strength testing.

A diagrammatical representation of the overall testing procedure is shown in figure 6.6.

#### **6.5.6 Data Handling Operations**

At the start of each test the mix characteristics were noted along with any other relevant information regarding the batch and the test given a numerical label. Upon completion of each experiment, the data output from the Rockwell Aim 65 was loaded onto a Hewlett Packard 85 microcomputer, tabulated and then stored on magnetic tape cartridges.

Subsequently, resistivity measurements corrected for temperature were plotted against a logarithmic time scale by means of a Hewlett Packard 7470A graphics plotter (see plate 6.6). If more than four samples were employed the average value was calculated and plotted, if less than four, individual readings were presented by identifying symbols.

A typical experiment output is given in appendix 3.

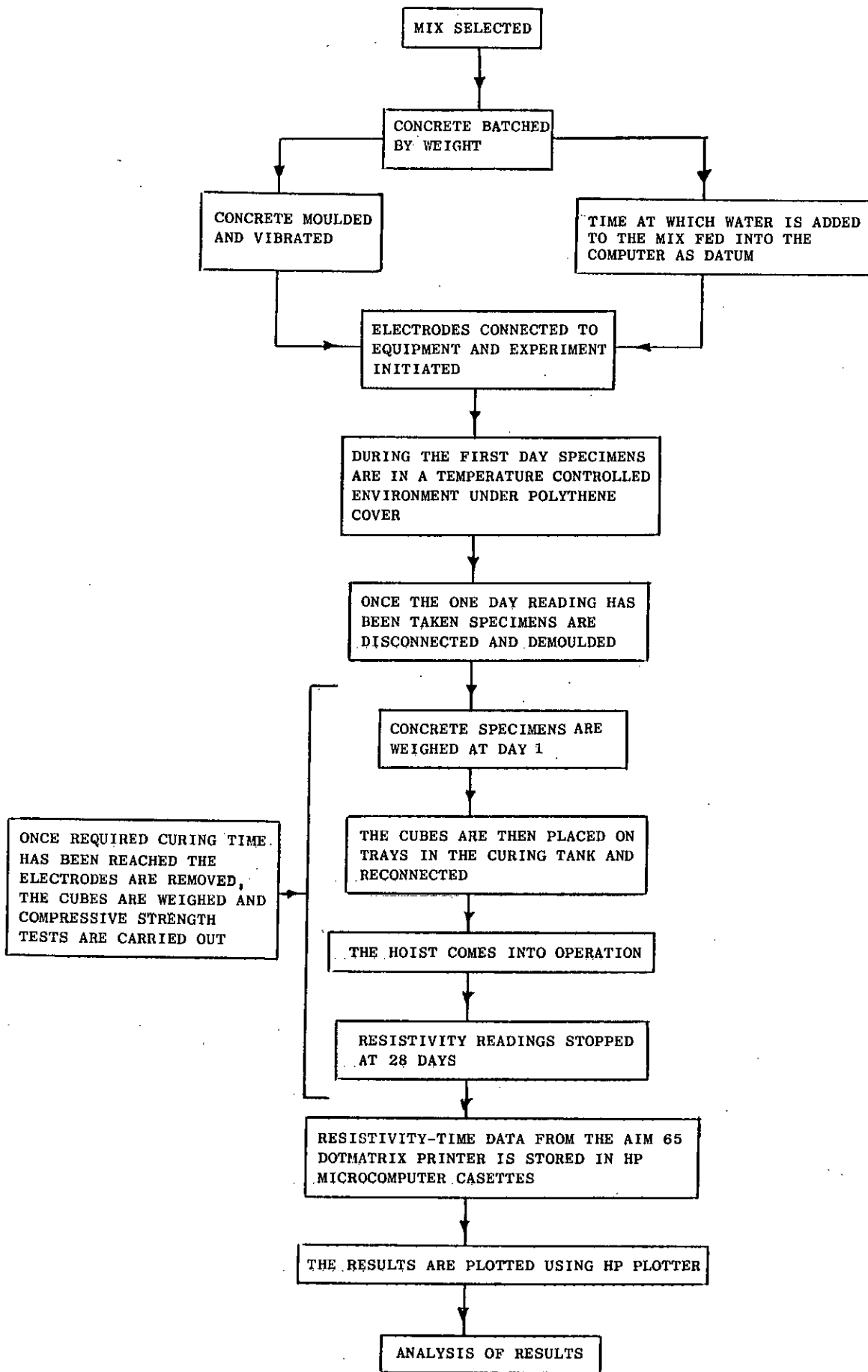


Figure 6.6) Flowchart of overall testing procedure

**CHAPTER SEVEN**

**FUNDAMENTAL EXAMINATION OF CONDUCTION**

**THROUGH CONCRETE**

## 7.1 INTRODUCTION

Earlier in Chapter 5, a theoretical approach that may be applicable in describing the conductive behaviour of concrete mixes was put forward. It is one of the aims of this chapter to test this model of postulated behaviour.

Before embarking upon the main experimental programme, material control tests were carried out to determine the absorption characteristics of the aggregates, the electrolytic saturation point and the temperature coefficient of resistivity for a water/cement solution. These tests are reported in sections 7.2 to 7.5 under the heading 'Preliminary Studies'. The main experiments are reported in the section headed 'Electrical Resistivity Characteristics of Cement Pastes, Mortars and Concretes' which also contains the discussions and conclusions to this chapter.

The objective of the investigation has been to provide a clearer understanding of the conduction mechanism through both fresh and hardened concrete, also forecasting parameters for various mix characteristics and properties have been singled out from the resistivity-time data.

### PRELIMINARY STUDIES

## 7.2 ABSORPTION TESTS ON AGGREGATES

The following concepts have been drawn from existing information on the moisture movement between aggregate and cement paste phases during the initial stages of hydration:-<sup>152,153,154,155</sup>

- (a) Permeability of granite =  $5.35 \times 10^{-11}$  m/s.
- (b) In general, crushed rock has a lower rate of absorption than gravel. The weathering factor, to which the latter has been subjected, making its surface more porous and absorbent.
- (c) Due to the effect of cement paste clotting on the surface of the aggregate, absorption by the latter occurs mainly during the initial 30 minutes from the time water is added.
- (d) Also, in the first 30 minutes after gauging with water, the aggregate will only retain water in small surface fissures and crevices.

- (e) Most of the water occupying the aggregate's surface will be available for hydration during the setting period.
- (f) During long term submerged curing, dry aggregates will, eventually, become saturated but the effective water/cement ratio of the paste will remain unaffected.

Tests were carried out to assess and quantify the influence of aggregate absorption on the water/cement ratio of the cement paste. The test described in BS.812: Part 2: 1975 was employed to determine the 30 minute absorption capacity for the sand and coarse aggregate employed.

Between the two coarse aggregates which were used during the test programme (granite gravel and crushed basalt) the granite was selected for absorption testing. Its weathered surface texture and slightly coarser crystalline structure making it more permeable to water than the basalt.

Granite gravel - 0.49% (by dry mass)  
 Sand - 0.21% (by dry mass)

The above absorption capacities are for aggregates going from a dry to a saturated, surface dry condition. The latter was a particularly difficult condition to achieve in practice but results were taken with a safe, i.e. wetter, margin. Newman<sup>153</sup> developed an equation by which the effective water/cement ratio could be calculated knowing the 30 minute absorption capacity of the coarse and fine aggregates.

This equation is not based on the direct measurement of water contents but on the strength of the material as a secondary monitor for water/cement ratio. However approximate, it nevertheless provides an indicator to the water content of the mix:-

Effective water/cement ratio for strength

$$= \text{Actual water/cement ratio} - (A_s \cdot S/C + \lambda \cdot A_{ca} \cdot C_a/C)$$

where:

$A_s$  = 30 min. absorption capacity of sand

$A_{ca}$  = 30 min. absorption capacity of coarse aggregate

$S/C$  = Sand/Cement ratio by weight

$C_a/C$  = Coarse-aggregate/Cement ratio by weight

$\lambda$  = Variable associated with patina aggregates. (This can be ignored when non-patina aggregates such as granite are employed.)

For a 1:1.5:3 mix with a water/cement ratio of 0.50,

$$\begin{aligned} \text{Effective water/cement ratio} \\ &= 0.5 - (0.0021 \times 1.5 + 0.0049 \times 3) \\ &= 0.482 \end{aligned}$$

Since this figure is assumed to be a low estimate (for reasons already stated), aggregate absorption effects do not appear to be of an appreciable size (-3.5% of original water/cement ratio).

Although during the testing programme high aggregate content mixes were not employed, it is of interest to note that a 1:4:6 mix would have an original water/cement ratio of 0.5 reduced to 0.462, a drop of approximately 8 per cent.

### 7.3 ELECTROLYTIC SATURATION OF A WATER-CEMENT SOLUTION

Electrolytic saturation and chemical saturation are two different physical states. A cement-water solution is said to achieve electrolytic saturation when a sufficient number of ions is present to transport all the electrical charges from one electrode to the other. Chemical saturation, however, takes place when the solution reaches a concentration level, with respect to a particular solute, above which precipitation or crystallisation occurs.

To establish the water/cement ratio at which a fresh cement-water solution becomes electrolytically saturated, conductivity measurements of solutions of increasing concentrations were taken. Measurements were taken with a portable conductivity set (model 5003 - E.I.L. Analytical Instruments) offering an accuracy of  $\pm 3\%$  over the tested range, and the test was carried out at a temperature of 20°C. An electrolyte was made with 37% cement content above which further increase in cement content did not increase the conductivity of the solution, see figure 7.1. The maximum conductivity



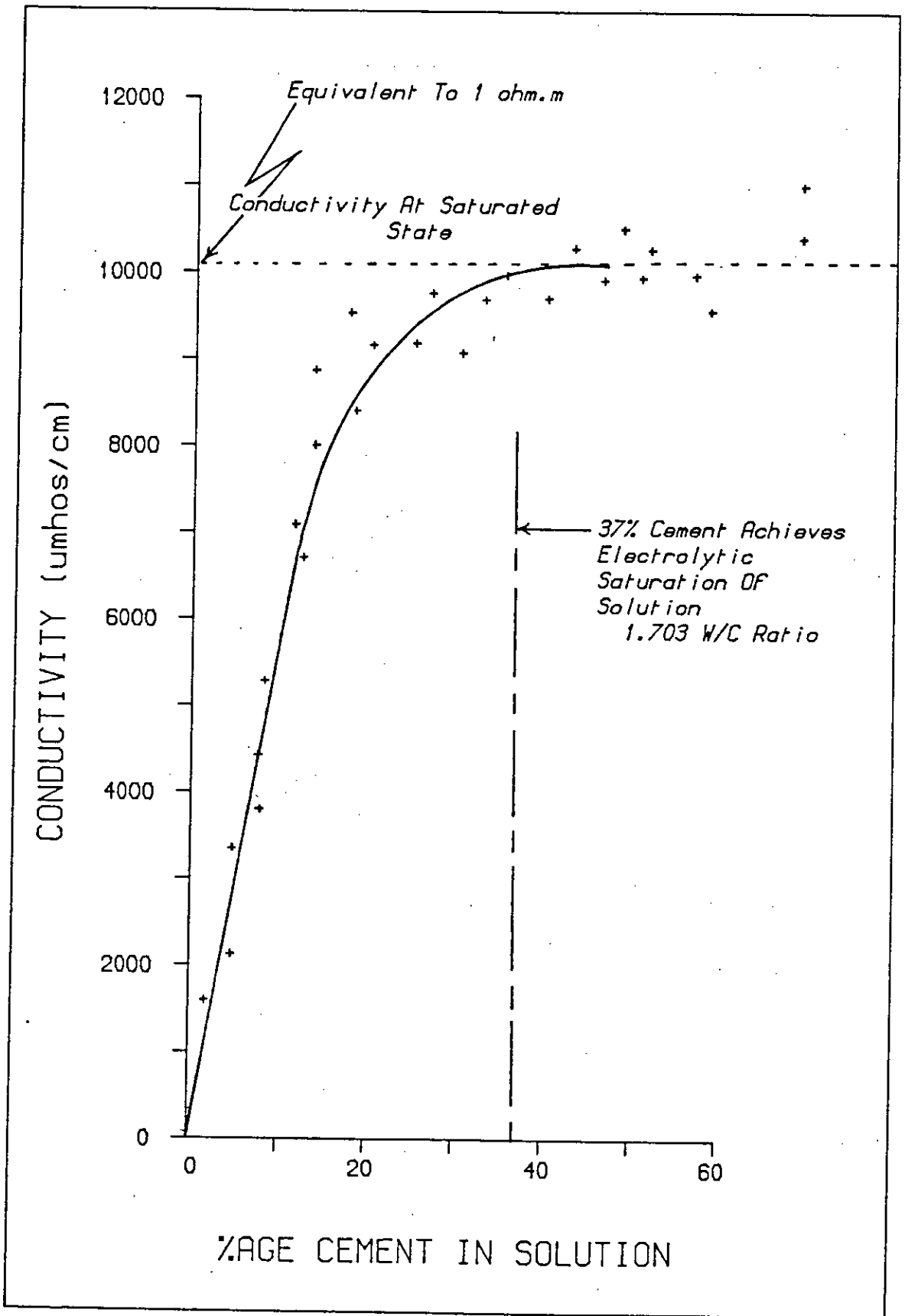


FIGURE 7.1 : Conductivity of a water/cement solution as a function of the percentage cement in solution.

achieved, approximately 1 Siemen/m, corresponded to that of all the fresh cement pastes tested. As the highest water/cement ratio employed in the experimental programme was 0.8, 56% cement in solution, variations in conductivity, in the initial hydration period, due to a partially saturated aqueous solution can be neglected.

Also, due to the continuing hydration of the Silicates (the reaction of the  $C_2S$  continuing even past the 28-day stage), the aqueous phase can be considered to remain saturated, or perhaps supersaturated, with Calcium Hydroxide (see section 3.3.2) thereby ensuring constancy in the level of electrolytic conduction.

#### 7.4 DETERMINING THE TEMPERATURE COEFFICIENT OF A WATER-CEMENT SOLUTION

Temperature variations affect the rate at which the different mineral constituents of cement hydrate. Although, for any given cement, it ought to be possible to correct for this variation, the discrepancies in cement compound composition shown in chapter 3 (Section 3.2.3) make any form of quantitative assessment impractical. All tests must therefore be carried out at a standard temperature. Minute changes in temperature, and corresponding variations in ionic mobility, can, however, be corrected for by means of a temperature coefficient of resistivity.

Raising the temperature of an electrolyte has the effect of decreasing its viscosity thereby allowing the ions to move more freely; a temperature rise therefore decreases the resistivity of an electrolyte. By simultaneously monitoring the resistivity and temperature of a material, and knowing the material's temperature coefficient of resistivity, a value corrected to a standard temperature can be obtained.

To determine the temperature coefficient of resistivity,  $\alpha$ ; experimentally, a series of tests <sup>W-C-E</sup> was carried out. The apparatus employed for the purpose consisted of an Electrolytic Conductivity Measuring Set (Kent Industrial Measurements, model MC-Mark 5), with a +1% accuracy over the tested range. The apparatus employed an alternating current measurement pulse with an operating frequency

of 1400Hz. Temperature monitoring was conducted by means of a Technoterm 5500 digital instant action thermometer ( $\pm 0.1^\circ\text{C}$  accuracy).

Deionized water was heated and poured in a conductivity cell, Ordinary Portland Cement was then added to it, the solution stirred and left for exactly 20 minutes.

To ensure standard conditions throughout, cement was taken from the same source and the water/cement ratio accurately maintained to 1.3, a sufficiently high proportion of cement to yield an electrolytically saturated solution, see section 7.3. The electrode configuration in the conductivity cell was such that the unhydrated cement (which settles during the 20 minutes prior to the reading being taken) would not interfere with the conductivity measurement of the solution.

The conductivity results were converted to resistivity:-

$$\text{Resistivity} = \frac{1}{\text{Conductivity}}$$

From the relationship  $\rho_0 = \rho_T(1 + \alpha \Delta T)$

let resistivity at temperature  $T_1$  be  $\rho_1$ ,

$$\rho_0 = \rho_1(1 + \alpha \Delta T_1) \quad \dots\dots(7.1)$$

also let resistivity at temperature  $T_2$  be  $\rho_2$ , then:

$$\rho_0 = \rho_2(1 + \alpha \Delta T_2) \quad \dots\dots(7.2)$$

dividing (7.1) by (7.2):

$$\frac{\rho_1}{\rho_2} = \frac{1 + \alpha \Delta T_1}{1 + \alpha \Delta T_2}$$

therefore,

$$\alpha = \frac{\rho_1 - \rho_2}{\rho_2 \Delta T_2 - \rho_1 \Delta T_1}$$

where  $\alpha$  is the temperature coefficient of resistivity.

The experimental standard was taken as resistivity equal to 1.148 ohm.m at a temperature of  $15.5^\circ\text{C}$  as a number of tests were carried out at this temperature, the ambient temperature at the

time.

A graph was then plotted of  $(\rho_1 - \rho_2)$  as a function of  $(\rho_2 \Delta T_2 - \rho_1 \Delta T_1)$ ;  $\alpha$  being the gradient of the best fit straight line through the points, see figure 7.2. Using regression techniques the temperature coefficient of resistivity was found to be equal to 0.0216. The correlation coefficient of the fitted line to the data was 0.76. This value compares favourably with that of 0.022 obtained by McCarter et al<sup>131</sup> using pastes in a solid rather than a liquid state, indicating that no change in  $\alpha$  takes place during the hardening of the material.

Returning to the original equation:

$$\rho_0 = \rho_T (1 + \alpha \Delta T)$$

where  $\rho_T$  is the resistivity reading and knowing that  $\alpha$  is approximately 0.022 then all the experimental results were able to be corrected to a temperature of 20°C.  $\rho_0$  being the corrected resistivity.

(Note on figure 7.2: Error bar in Y-axis =  $\pm 0.01$  Ohm.m

Error bar in X-axis =  $\pm 8\%$ )

## 7.5 THE RESISTIVITY TIME ( $\rho - t$ ) GRAPH

Experimental output takes the form of Resistivity-Time graphs, each of which is particular to the mix employed. It is by analysing, and comparing the graphs to one another, that relationships between resistivity and physical parameters are obtained together with a better understanding of the conductive process and the factors affecting it.

Although it was only at the end of the experimental programme that trends were noted, analysed and identifying parameters selected, it is thought helpful to provide, at this stage, the following description of the measurements taken and the general trends observed.

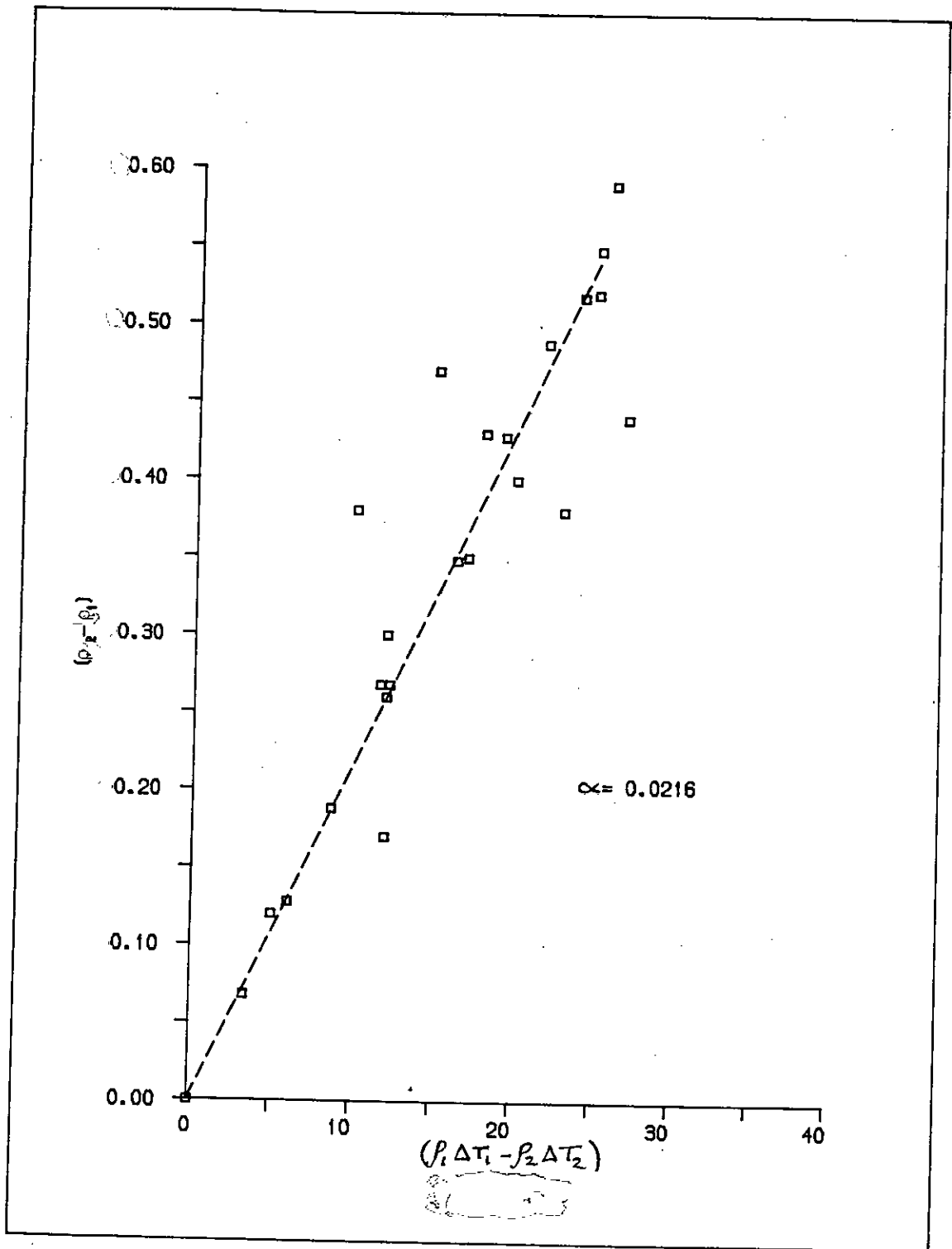


Figure 7.2) "Determination of  $\alpha$ , the temperature coefficient of resistivity"

### 7.5.1 General ( $\rho - t$ ) Trends

Figure 7.3 shows the typical format of a resistivity time graph for a concrete mix tested over a 28 day period. In general, three distinct regions are discernable in all of the graphs:- Setting period, unsaturated hardening and saturated hardening.

(i) **Setting period:** The stage during which the material ceases to behave as a liquid and gains in plasticity. Aside from a marked decrease in resistivity in the initial minutes, caused by a gradual dissolution of cement compounds in the mix water, the first few hours are only characterized by a gentle increase in resistivity. The end of setting and the beginning of the hardening proper is marked by a substantial rise in resistivity. The period at which this occurs is termed the "Knee point".

(ii) **Unsaturated hardening:** During this period the material rapidly gains in strength, water is gradually taken up by hydration and capillary pores are blocked off by crystals and gel formations. All these factors contribute to a rapid rise in resistivity which continues to the demoulding stage at the end of the first day of curing.

(iii) **Saturated hardening:** As the cubes are submerged for curing, water permeates the air-filled cavities causing resistivity readings to drop. The size of the day-one discontinuity is therefore related to the permeability and porosity of the specimen. From the reduced level, resistivity continues to rise as an increased proportion of the conductive area is blocked by the products of hydration. The rate of increase of resistivity is, however, not as rapid as during the initial air-curing period and tends to level at the 28-day period.

Also in figure 7.3 are shown the parameters which have been employed in the analysis of the different mixes, these were:-

- $\rho_{0.065}$  - Resistivity at 0.065 of a day
- The knee point - End of the setting period
- $\rho_{1.0}$  - Resistivity at day one, prior to demoulding
- $\rho_{1.5}$  - Resistivity at 1.5 days, after 12 hours submerged in water

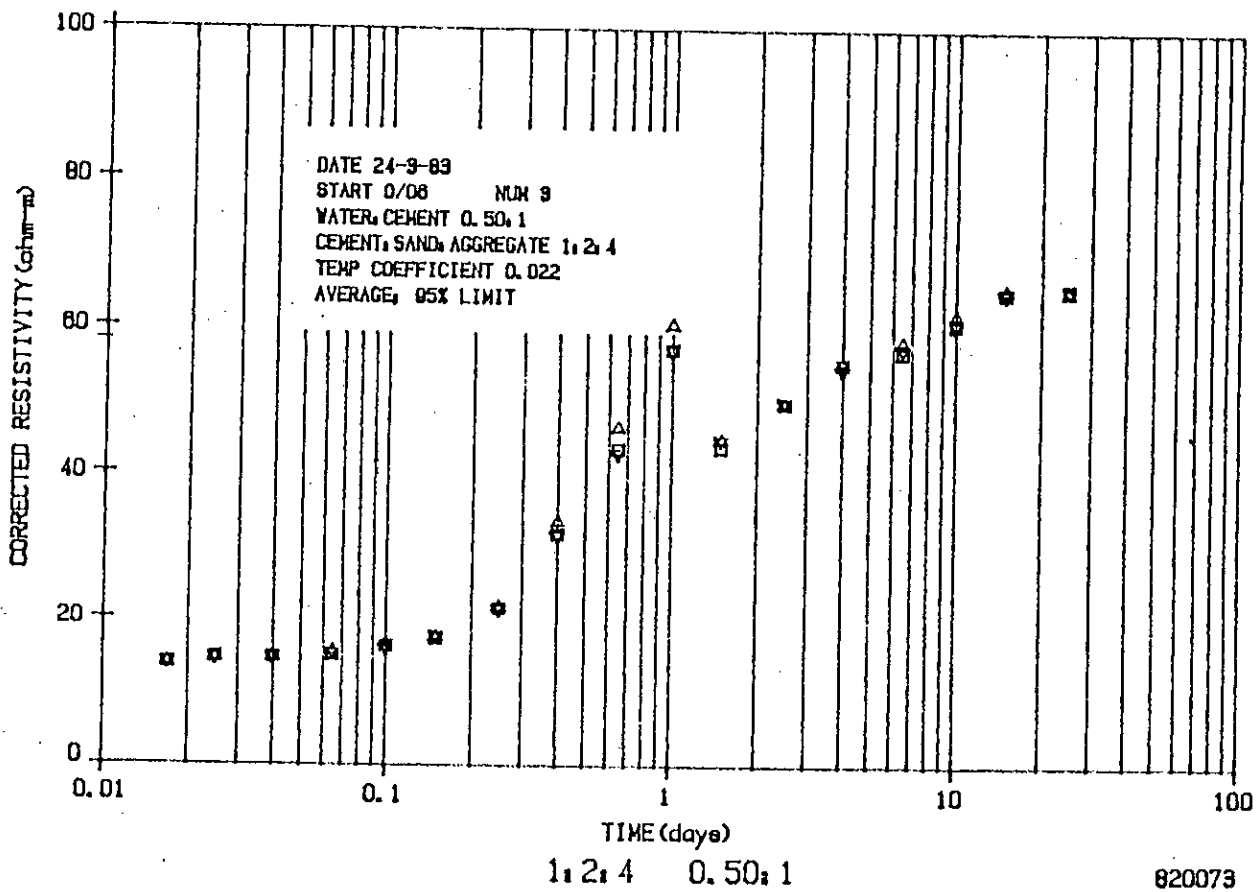


FIGURE 7.3

## ELECTRICAL RESISTIVITY CHARACTERISTICS OF CEMENT PASTES, MORTARS AND CONCRETES

### 7.6 THE SETTING CHARACTERISTICS OF CEMENT PASTES

A number of 1-day electrical resistivity tests were carried out on cement pastes with water/cement ratios ranging from 0.30 through to 0.60, Ordinary Portland Cement was used throughout and three cubes cast for each of the water/cement ratios considered, see figure 7.4.

The following observations were noted:-

- (i) The drop in resistivity caused by the dissolution of cement in the initial minutes was not registered. This is explained by the fact that the hydrolysis process, which releases Hydroxides and Sulphates of Calcium, Sodium and Potassium, is very rapid and slows down within four or five minutes of water having been added to the cement, see section 3.3.2. (The first reading being taken usually 15 minutes after the gauging of water).
- (ii) Although, as shown by Double<sup>82</sup>, figure 3.2, the concentration levels of the various ionic groups alters considerably during the initial hydration stages, this was not registered by the resistivity measurements. Whilst setting, all pastes maintained a constant resistivity level of approximately 1.5 ohm.m. The notion that, within the range of water/cement ratio tested, the pastes were equally as conductive, confirming the saturation test carried out with cement-water solutions.
- (iii) The sharp increase in hydration activity which characterizes the end of the setting period and the beginning of the hardening process is defined on a Resistivity-Time graph by a marked rise in resistivity.
- (iv) An increase in the water/cement ratio results in larger and more numerous capillary pores, thus the amount of hydrates



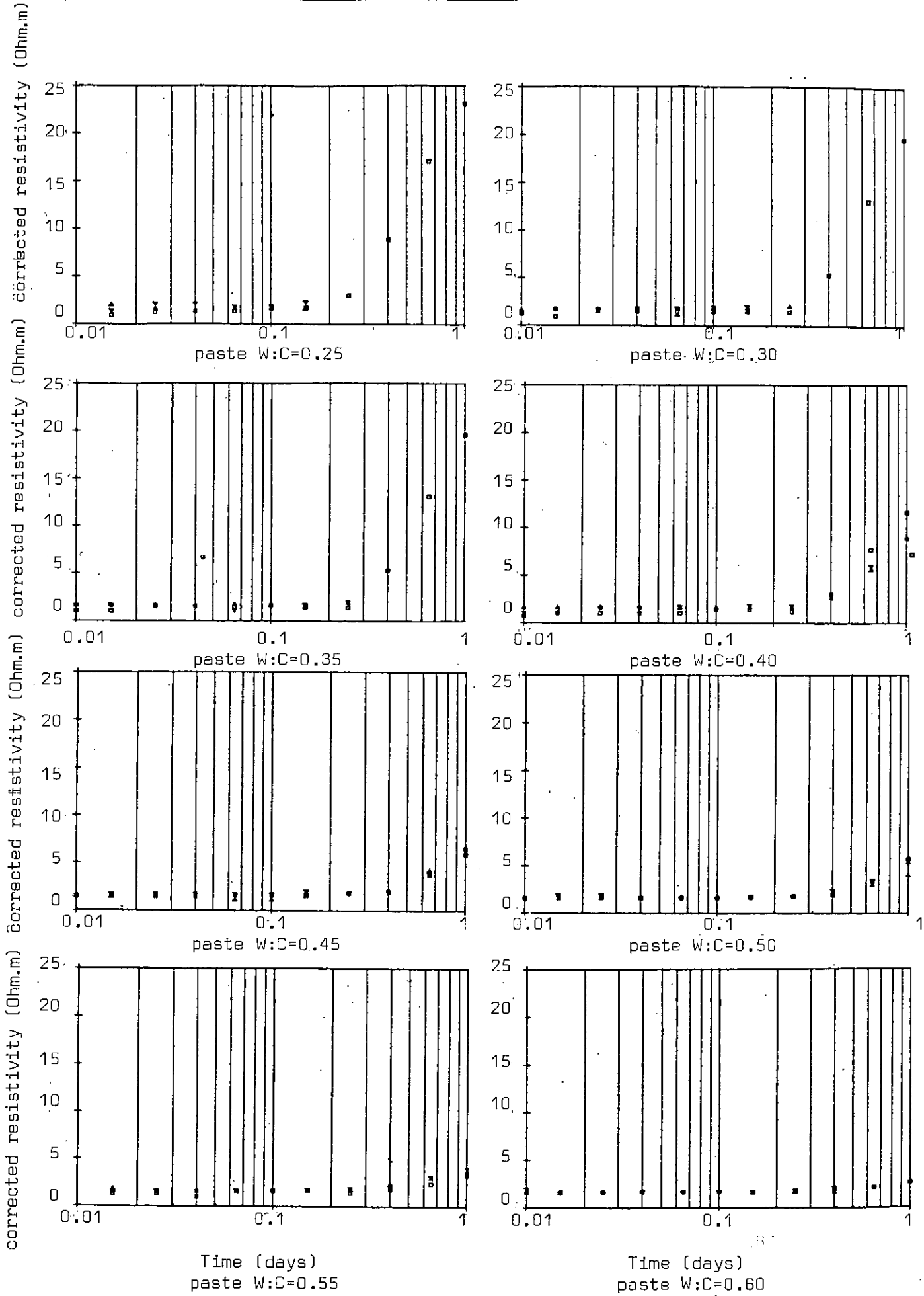


Figure 7.4 Day-one resistivity graphs for O.P.C. pastes of different water/cement ratio

Mix Proportions (by Weight) cement = 1			Fractional Volume of cement paste (on demoulding) $\phi$	Length of test Period (DAYS)	
Aggregate	Sand	Water			
PASTES	0	0.25	1.0	All one day	
		0.30	1.0		
		0.35	1.0		
		0.40	1.0		
		0.45	1.0		
		0.50	1.0		
		0.55	1.0		
		0.60	1.0		
MORTAR	0	1.5	0.40	0.5608	All one day
		0.45	0.5770		
		0.50	0.5920		
		0.55	0.6060		
		0.60	0.6190		
	0	2.0	0.40	0.4892	
		0.45	0.5057		
		0.50	0.5211		
		0.55	0.5336		
		0.60	0.5492		
	0	2.5	0.40	0.4338	
		0.45	0.4500		
		0.50	0.4654		
		0.55	0.4799		
		0.60	0.4936		
	0	3.0	0.40	0.3897	
		0.45	0.4054		
		0.50	0.4204		
		0.55	0.4347		
	0	3.5	0.50	0.3834	
		4.0	0.50	0.3523	
	0	4.5	0.50	0.3260	
	0	5.0	0.50	0.3032	
	0	5.5	0.50	0.2835	
0	6.0	0.50	0.2662		

TABLE 7.1

Summary of Mixes Studied

Mix Proportions (by Weight)			Fractional Volume of cement paste (on demoulding) $\phi$	Length of test Period (DAYS)
Aggregate	Sand	Water		
CONCRETE with 10mm granite				
3	1.5	0.40	0.2992	28 days
		0.45	0.3132	
		0.50	0.3266	
		0.55	0.3395	
		0.60	0.3520	
4	2.0	0.40	0.2425	28 days
		0.45	0.2548	
		0.50	0.2667	
		0.55	0.2783	
		0.60	0.2895	
6	4.0	0.50	0.1798	1 day
		0.60	0.1968	
		0.70	0.2129	
		0.80	0.2288	
CONCRETE with 20mm whin- stone				
1	1.0	0.40	0.493	28 days
		0.45	0.5095	
		0.50	0.5249	
		0.55	0.5393	
		0.60	0.5530	
2	1.0	0.40	0.3945	28 days
		0.45	0.4104	
		0.50	0.4254	
		0.55	0.4397	
		0.60	0.4533	
3	1.5	0.40	0.3020	28 days
		0.45	0.317	
		0.50	0.3305	
		0.55	0.3435	
		0.60	0.3560	
4	2.0	0.40	0.2458	28 days
		0.45	0.2582	
		0.50	0.2702	
		0.55	0.2818	
		0.60	0.2931	

TABLE 7.1 (cont.)

Summary of Mixes Studied

Mix Proportions (By Weight) (1 part of cement employed)			% Total Aggregate (By Volume)	Resistivity at 0.065 * day (Ohm-m)	Resistivity at 1.0 * day (Ohm-m)	$R_{1.0} - R_{0.065}$ (Ohm-m)
Coarse Aggregate	sand	water				
PASTES						
0	0	0.25	0	1.57	23.10	21.53
		0.30		1.47	19.60	18.13
		0.35		1.43	19.60	18.17
		0.40		1.57	9.80	8.23
		0.45		1.47	6.30	4.83
		0.50		1.63	4.97	3.34
		0.55		1.63	3.67	2.04
		0.60		1.67	2.47	0.80
MORTARS						
0	1.5	0.40	43.91	3.40	12.40	9.00
		0.45	42.30	3.10	11.50	7.40
		0.50	40.80	3.10	8.10	5.00
		0.55	39.40	2.60	6.30	3.70
		0.60	38.10	2.90	5.60	2.70
0	2.0	0.40	51.08	4.70	16.70	12.00
		0.45	49.43	4.30	13.90	9.60
		0.50	47.89	3.90	11.30	7.40
		0.55	46.53	3.30	9.30	6.00
		0.60	45.07	3.30	6.60	3.30
0	2.5	0.40	56.62	5.90	21.50	15.60
		0.45	54.99	5.20	18.50	13.30
		0.50	53.46	4.80	16.30	11.50
		0.55	52.01	4.50	13.10	8.60
		0.60	50.63	3.90	10.00	6.10
0	3.0	0.40	61.03	6.90	23.90	17.00
		0.45	59.45	6.50	21.60	15.10
		0.50	57.95	5.80	17.80	12.00
		0.55	56.53	5.10	14.50	9.40
		0.60	55.17	5.20	12.60	7.40
CONCRETES						
1	1	0.40	50.70	6.17	32.63	26.46
		0.45	49.05	5.03	23.60	18.57
		0.50	47.51	4.87	17.50	12.63
		0.55	46.07	4.83	15.50	10.67
		0.60	44.70	4.27	13.83	9.56
2	1	0.40	60.55	8.07	52.70	44.63
		0.45	58.95	7.40	41.97	34.57
		0.50	57.56	7.23	33.53	26.30
		0.55	56.03	6.27	25.40	19.13
		0.60	54.67	5.53	18.73	13.20
3	1.5	0.40	69.71	12.77	81.13	68.36
		0.45	68.30	12.57	70.03	57.46
		0.50	66.95	11.41	62.75	51.34
		0.55	65.65	10.00	43.00	33.00
		0.60	64.40	9.23	36.40	26.87
4	2	0.40	75.42	16.96	124.93	107.97
		0.45	74.18	16.90	95.87	78.97
		0.50	72.98	15.20	58.17	42.97
		0.55	71.82	13.82	52.13	38.31
		0.60	70.69	12.50	48.17	35.67

TABLE 7.2 DAY-ONE RESISTIVITY CHARACTERISTICS OF TESTED MIXES

required to obstruct them increases accordingly. Pastes with high water content will, therefore acquire a given resistivity value at a more advanced stage of hydration than pastes with less water, see figure 7.5.

(v) Researchers<sup>37,39,42,47,48</sup> have, in the past, related the position of the knee point to the final set.

The relationship between water/cement ratio and the position of the 'Knee point' for the different pastes is shown in figure 7.6 together with results obtained from Vicat's test. Direct comparison is not possible, as both measure parameters which cannot be related; it is of interest, however, to note that both show a high degree of linearity.

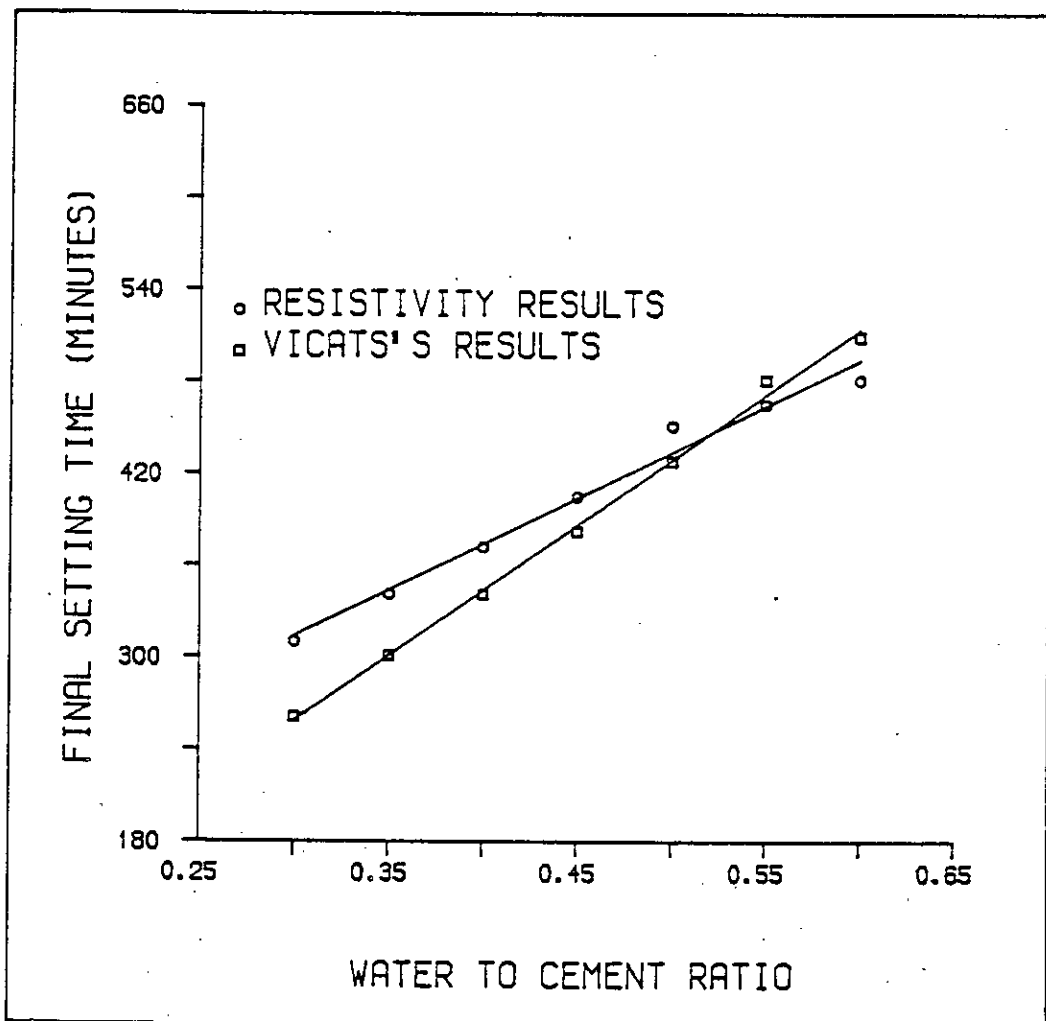
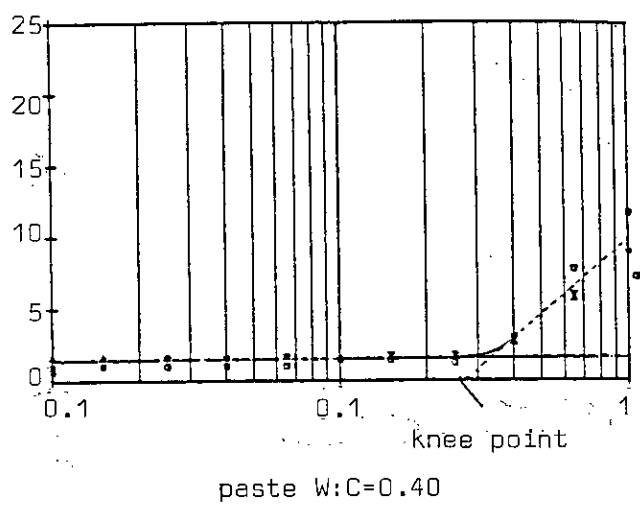
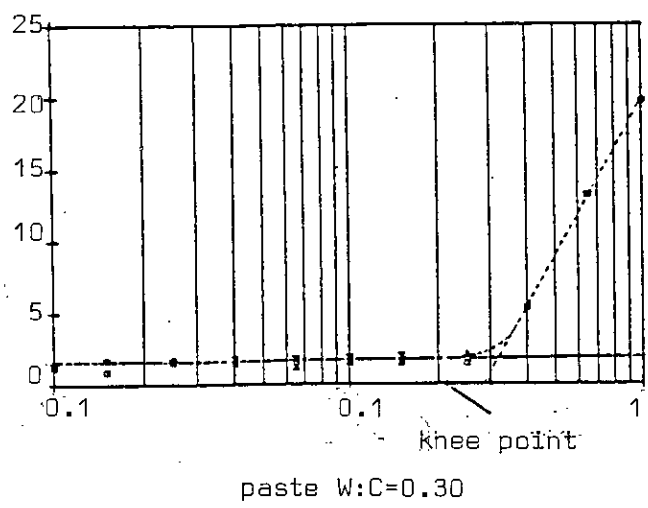


Figure 7.6 Final setting times as determined by means of Vicat's test and the Electrical Resistivity testing technique

corrected resistivity (Ohm.m)



corrected resistivity (Ohm.m)

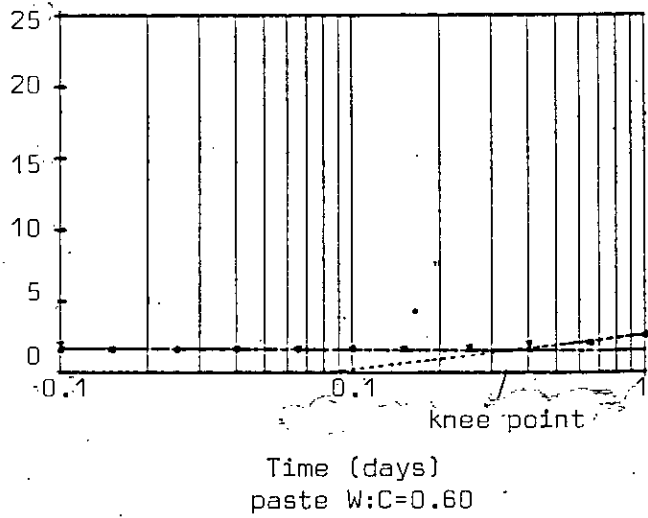
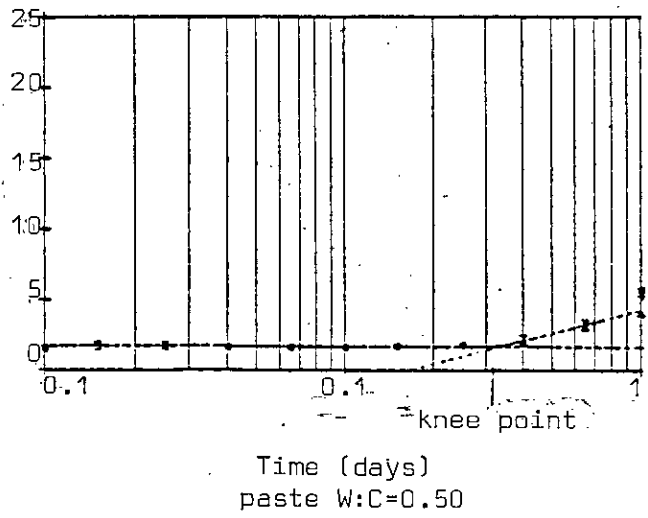


Figure 7.5. Effect of varying water/cement ratio on the position of the 'knee point' of different cement pastes.

## 7.7 THE EFFECT OF AGGREGATE ON THE SETTING CHARACTERISTICS OF CEMENT PASTES

The addition of aggregate to a fresh cement paste matrix has the following effects on the electrical resistivity characteristics of the material during the setting stage:-

- (a) Because of the very high resistivity of the aggregates employed, in relation to that of fresh pastes, the cross-sectional area available for conduction is reduced. The result is an increase in the resistivity datum level of the mix.
- (b) Whilst for the cement pastes no marked changes in resistivity level were observed during the setting period, the same was not true for mortars and concretes.

The gradient of the resistivity versus time line increased from zero, for pastes, in proportion to the quantity of aggregate added (see Figure 7.8).

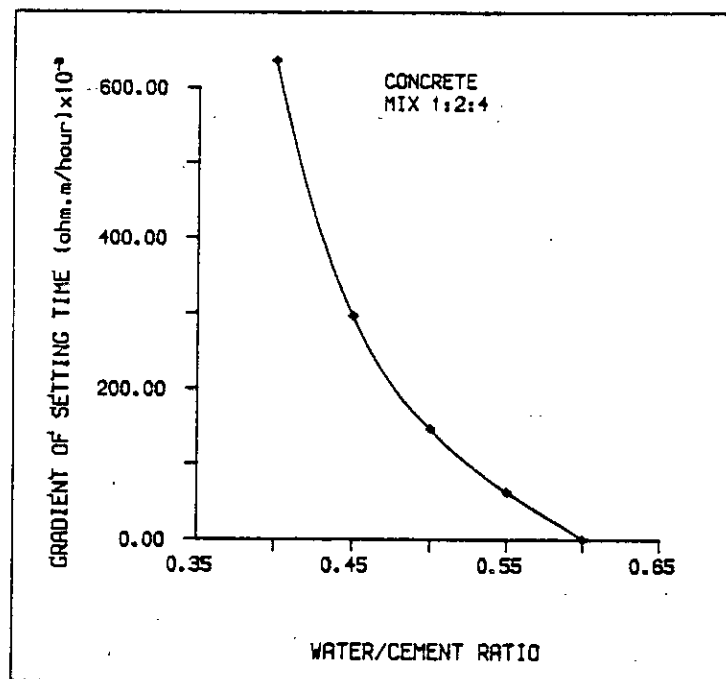


Figure 7.7: Gradient of setting time resistivity vs. water/cement ratio

The following explanation is put forward for this phenomenon:- The addition of aggregate to the paste has the effect of increasing the tortuosity of the network of continuous capillary within the matrix.

Although hardening proper does not commence until a later stage it is evident (see Figures 7.8, 7.9) that conductive paths are being obstructed by hydration products as early as 30 minutes after water gauging.

The presence of aggregate in the matrix has the effect of speeding up the process of reduction in the number of continuous capillary pores during hydration. The result is a shift in the knee point to an earlier stage than obtained with a cement paste alone. See figure 7.8, 7.9.

#### **7.8 INFLUENCE OF MIX DESIGN ON THE 1-DAY RESISTIVITY ANOMALY**

A major initial area of investigation was the study of an anomaly noted in published data<sup>134</sup> reporting resistivity-time graphs of equal mixes in a dry and subsequently saturated condition. Due to the mould/electrode assembly employed, McCarter et al<sup>134</sup> were forced to disconnect the cubes from the electrodes prior to submerging for curing. Further resistivity readings were obtained by clamping the electrodes firmly against the cube surfaces and the data plotted on a different graph (see figures 4.17 and 4.18).

Super positioning of the graphs however, showed that resistivity readings had dropped once the cubes had undergone submergence. A series of tests were therefore carried out to establish the cause of this discontinuity.

After the first day of curing under polythene sheeting, the specimens were demoulded and immersed in the curing tank for the remainder of the test period. Since the conductivity of water is greater than that of air, the rate of increase of resistivity subsequently dropped. This was found to continue until (a), the samples had become fully saturated or, (b) the gain in resistivity



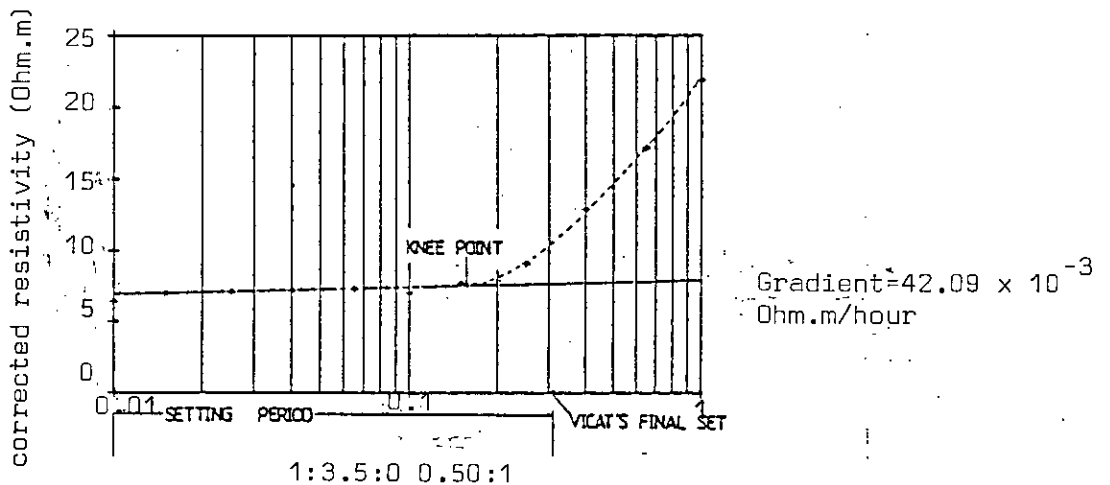
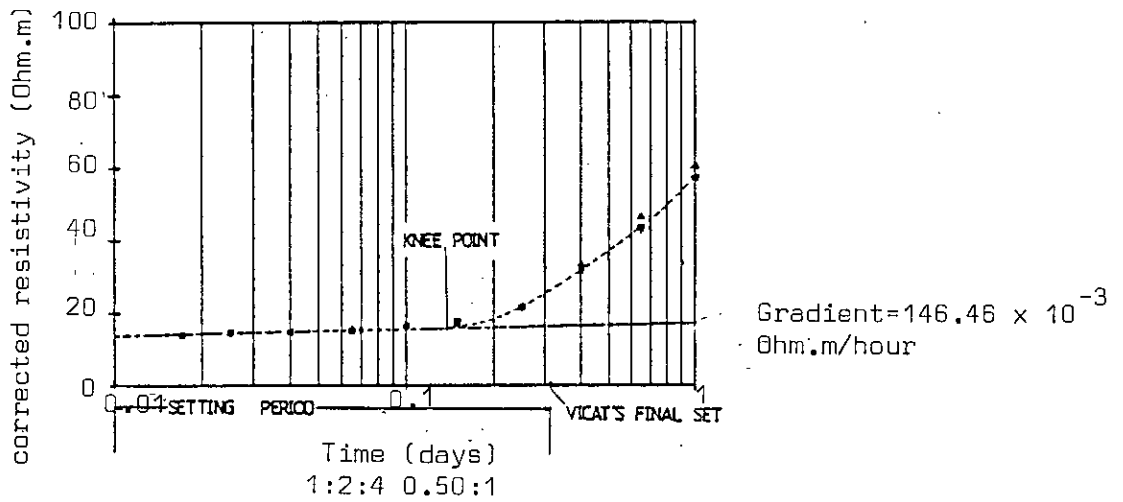
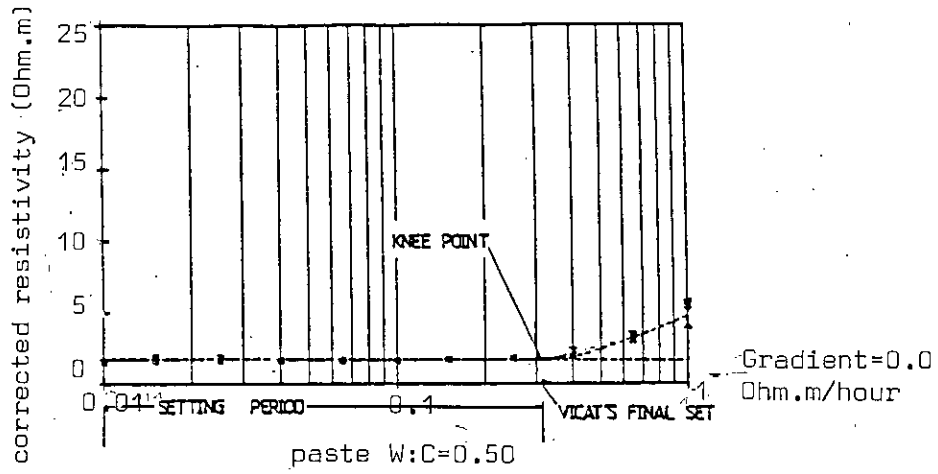


Figure 7.8. Effect of aggregate on the position of the resistivity 'knee point' for two concretes and also a paste of equal water/cement ratio

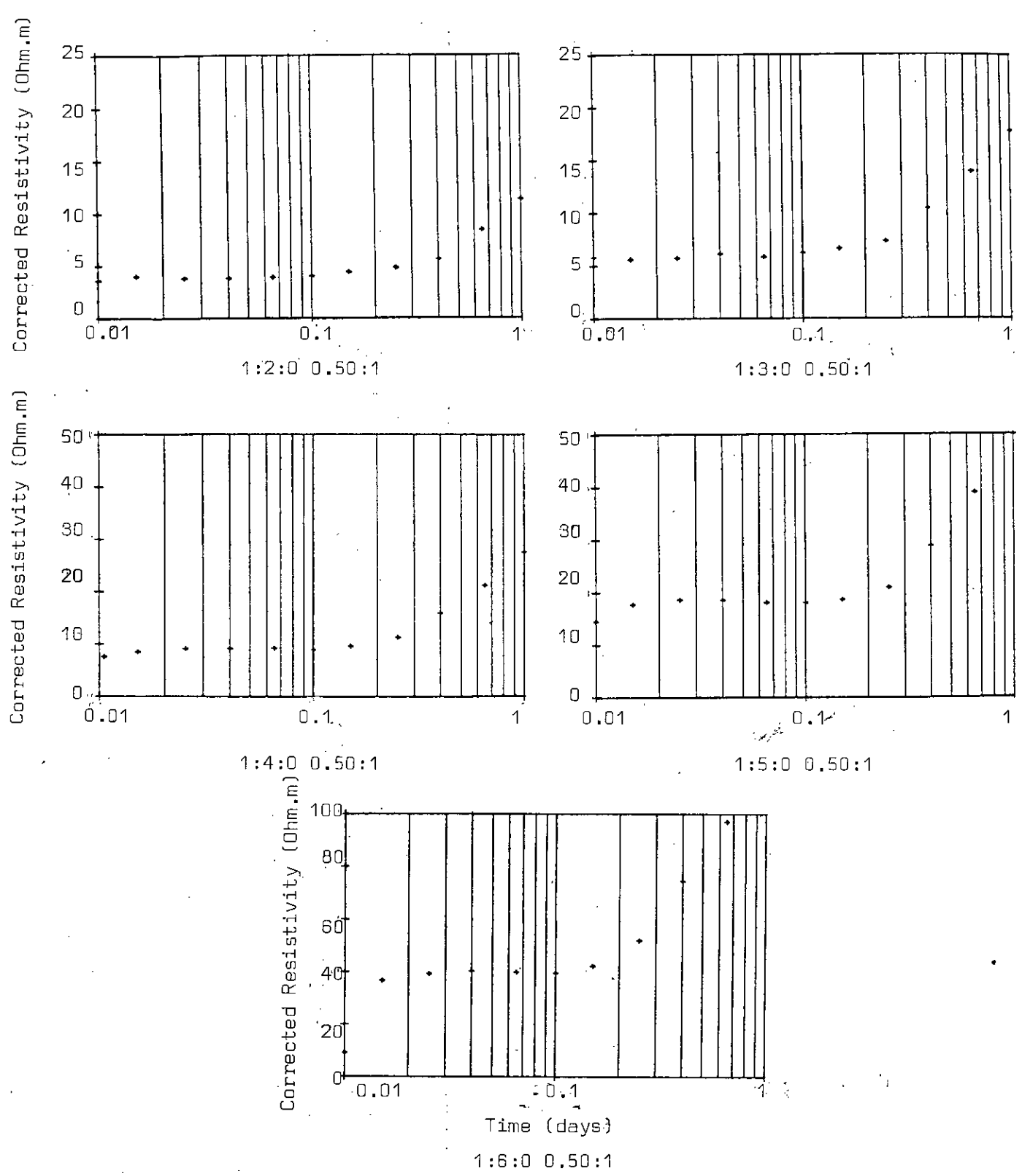


Figure 7.9 Effect of sand/cement ratio on the day one resistivity characteristics of O.P.C. mortar

due to the continuing hardening had risen above it. The stage, in the  $\rho$ -t curve, where resistivity drops due to the ingress of water was termed the 'Discontinuity', see figure 7.3.

Although the magnitude of the discontinuity was found to be largely dependent on the degree of compaction of the specimens (see figure 7.10), differences were noted between equally well compacted mixes of different water/cement ratio. This is to be attributed to the increase in both porosity and permeability of the cement paste phase with water/cement ratio.

As the intensity of the hydration process decreased, so did the rate of increase in resistivity and, by the 28 day stage, the resistivity of the saturated samples had, in the majority of cases levelled off to finite values.

By subtracting the one-day mass of the samples from the 28 day saturated value, the percentage absorbed water by volume was calculated.

$$\% \text{ VOLUME OF VOIDS} = \frac{(\text{MASS}_{28} - \text{MASS}_{1.0})}{1000} \times \frac{100}{\text{VOLUME OF CUBE}}$$

Figures 7.11(a) and (b) show the relationship between the above and discontinuity as a function of water/cement ratio and mix proportioning respectively.

Figure 7.11(a) shows an exponential increase in discontinuity with a decrease in water/cement ratio. Progressive loss in workability was the main cause for this behaviour.

Figure 7.11(b), shows the marked increase in both the percentage absorbed water and discontinuity size with decrease in the compactability of the mix (results from 1:1.5:3 and 1:2:4 mixes).

#### 7.9 EFFECT OF MACROVOIDS ON THE CONDUCTIVITY CHARACTERISTICS OF A CONCRETE MIX

The high resistivity of the aggregate, when compared to that of the cement pastes, indicated a need for tests on the ability of the apparatus to distinguish between non-conductive solids and air

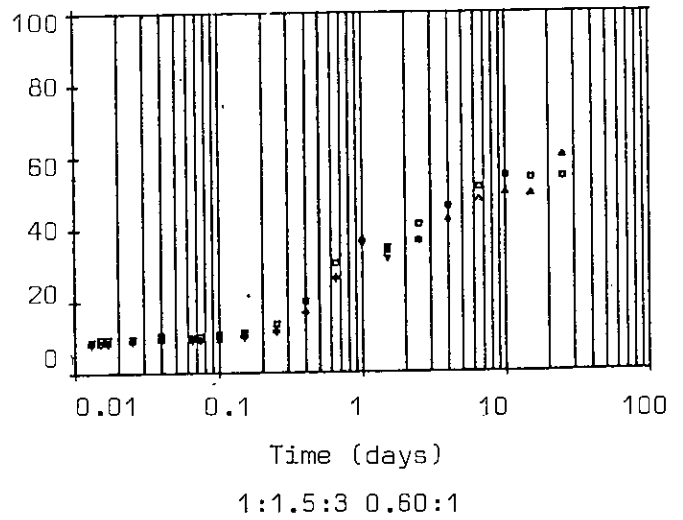
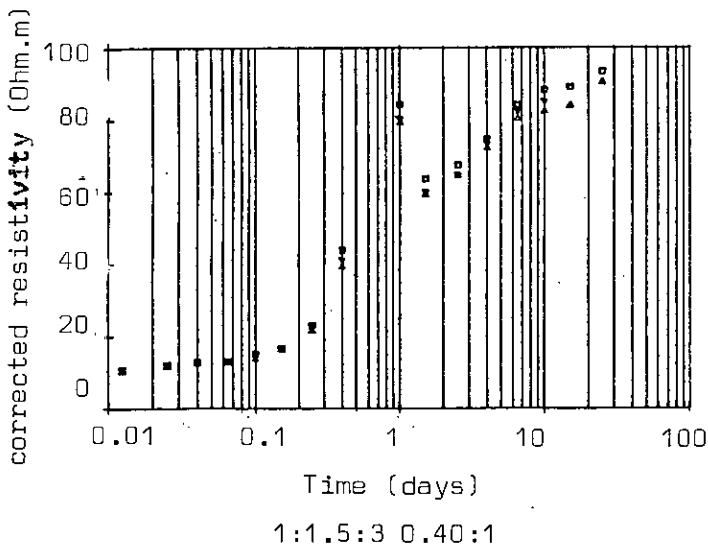
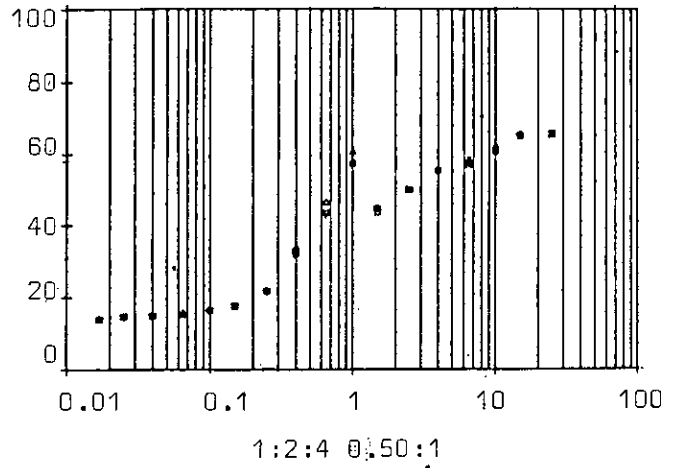
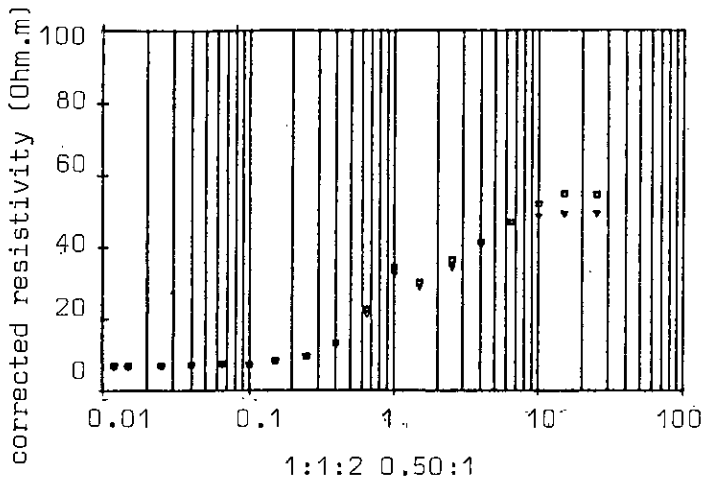
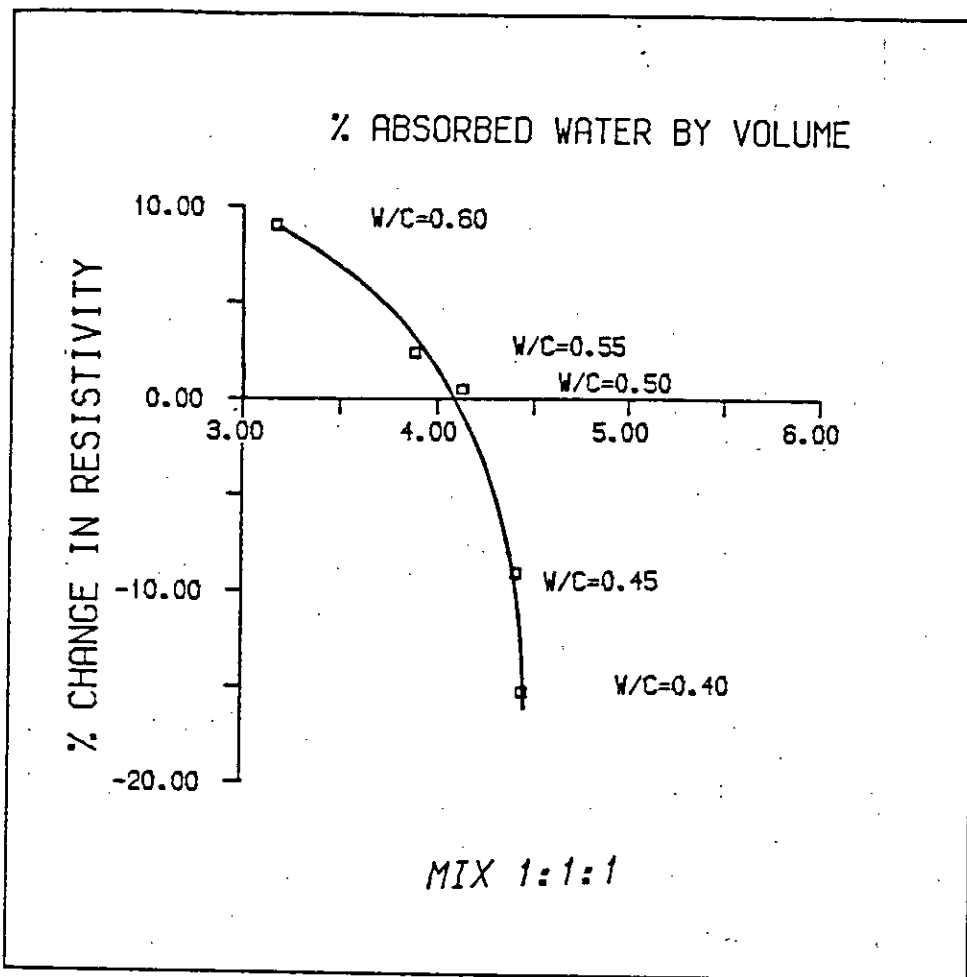
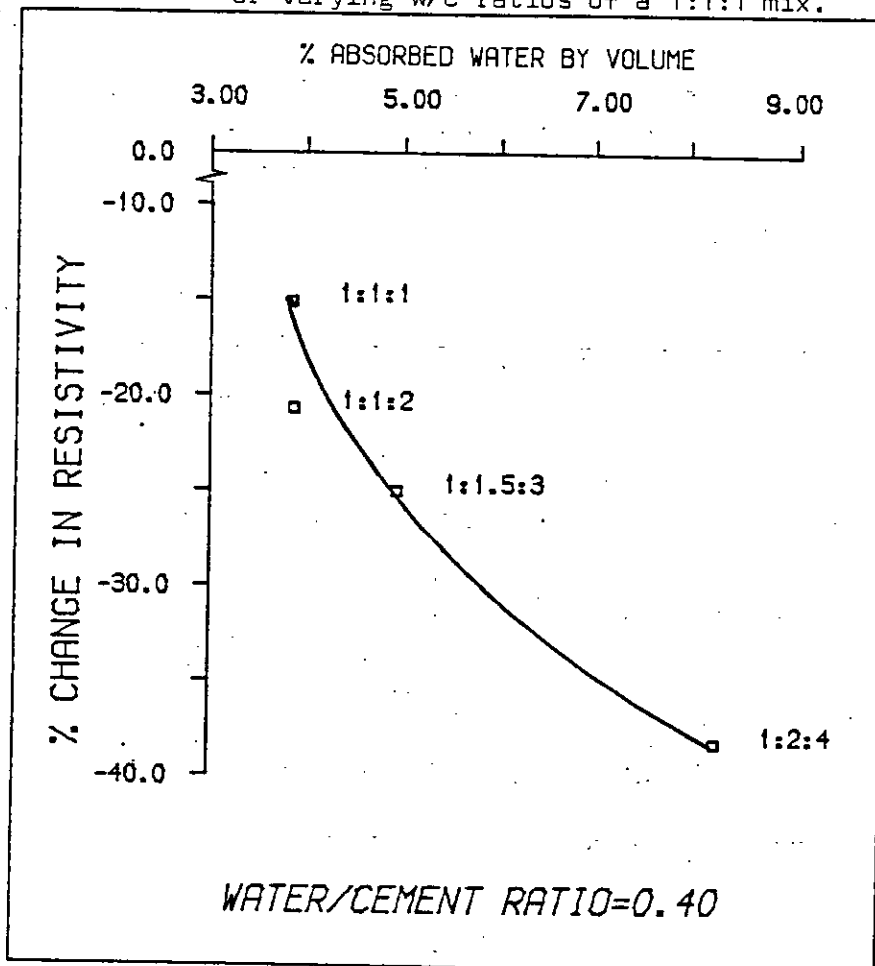


Figure 7.10 Effect of aggregate/cement ratio and water/cement ratio on the one-day discontinuity of concrete mixes



(a) For varying W/C ratios of a 1:1:1 mix.



(b) For different mixes with a 0.40 W/C ratio.

Figure 7.11 Resistivity changes between 1 and 1.5 days as a function of the percentage water absorbed by the concrete cubes during the 1 to 28 day curing period. -158-

voids. Special moulds were developed to allow for the inclusion of steel bars which could be removed once the concrete had set. The bars were individually insulated with rubber sheathing thereby allowing for resistivity measurements to be taken during the initial setting stages, see plate 7.1. The volume of steel and sheathing in each specimen accounted for a specific percentage, by volume, of air voids in the concrete.

Four specimens were made and tested:

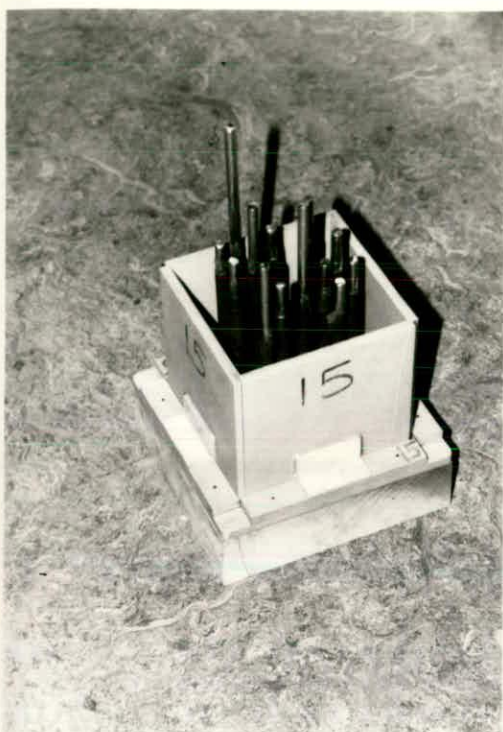
STANDARD MIX = 1:1.5:3 W/C = 0.50

	CUBE MASS
Specimen 1 STANDARD MIX	8.057 kgs
Specimen 2 STANDARD MIX + 5% aggregate	8.280 kgs
Specimen 3 STANDARD MIX + 5% air voids	7.702 kgs
Specimen 4 STANDARD MIX + 10% air voids	7.375 kgs

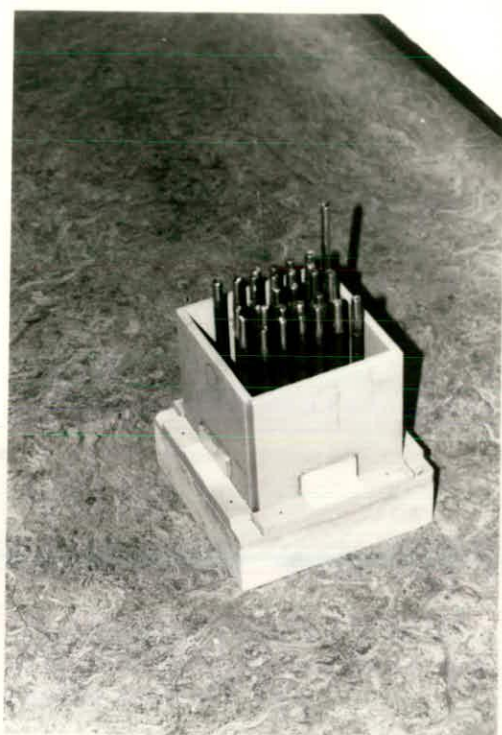
A direct comparison of specimens 2 and 3 shows a significant difference in mass between the specimen with 5% extra aggregate and that with 5% extra air voids. Their resistivity-time relationships, however, remain almost identical, see figure 7.12.

The resistivity of 10% extra voids specimen was not as high as expected. Due to the greater amount of rods, a higher degree of compaction was applied, resulting in a more conductive mix. Also, the narrower spacing between the bars prevented the larger particles from getting into the mould thereby varying the grading of the aggregate in the mix.

From a comparison of the resistivity measurements and masses, it was concluded that the existing apparatus was unable to distinguish between non-conductive solids and air voids in a concrete mix. Care has to be taken to vibrate all the specimens in order to achieve a good degree of compaction, errors would otherwise arise when relating resistivity to physical or mechanical characteristics.



a.



b.

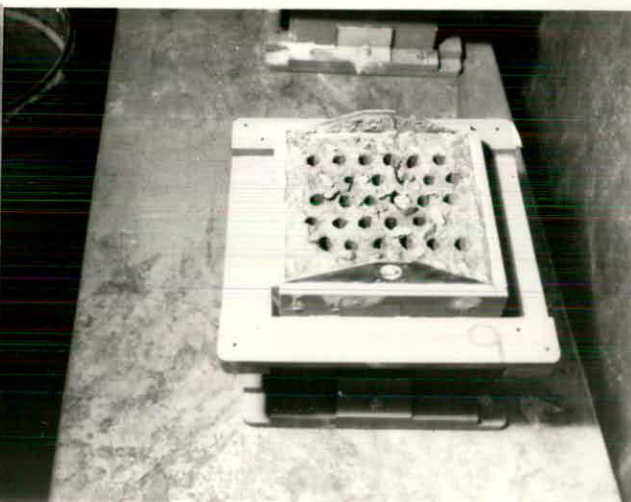
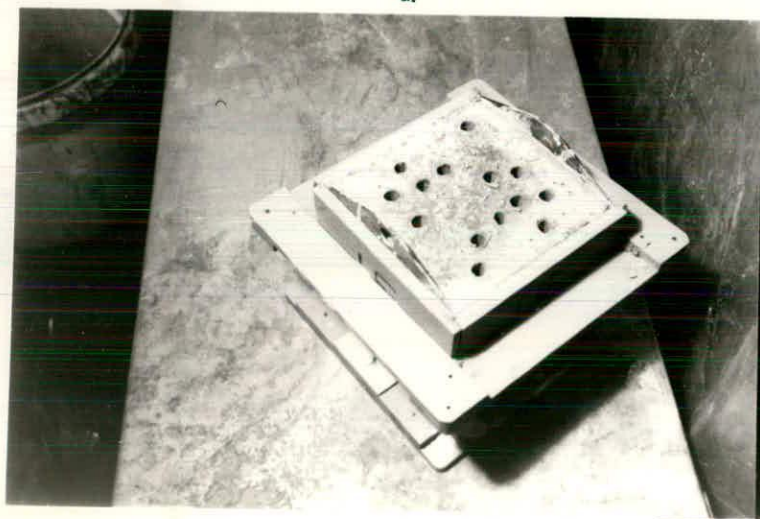
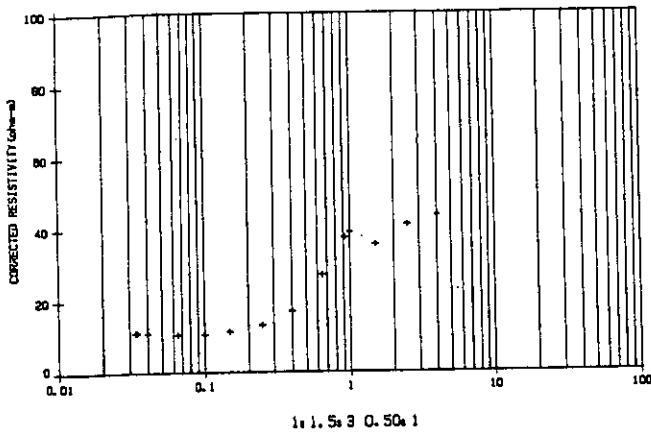


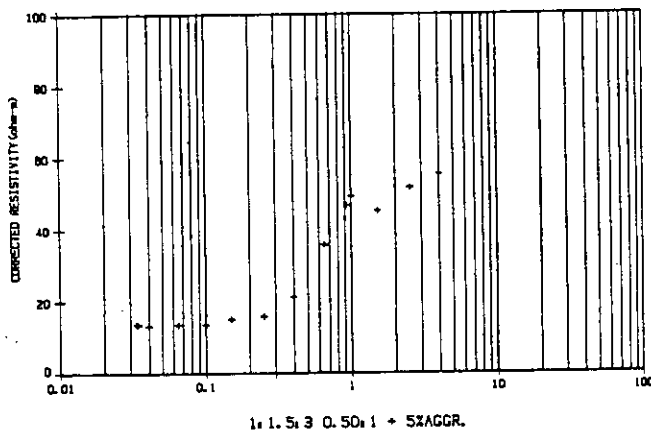
Plate 7-1 : Moulding arrangement for :

a. 5% air by volume

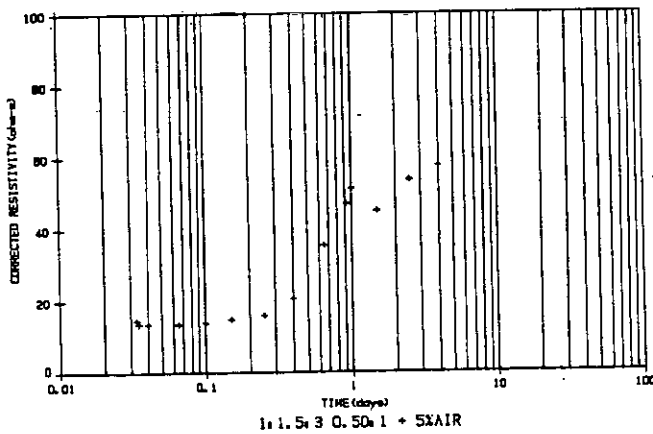
b. 10% air by volume



TIME	RES	TEMP	C	RES
0330	19.9	21.9		11.1
0340	19.9	20.4		11.0
0400	19.9	19.9		10.0
0650	19.9	19.8		10.0
1000	19.9	19.8		10.0
1500	11.2	30.4		11.3
2500	12.9	30.6		13.1
4000	16.5	21.0		16.9
6500	32.6	20.0		33.7
9230	36.6	21.4		38.8
10000	37.7	21.4		39.0
15000	34.6	21.1		40.4
50000	40.1	20.9		40.4
80000	43.0	20.4		43.4



TIME	RES	TEMP	C	RES
0330	14.0	18.3		13.0
0340	14.0	18.3		13.0
0400	13.6	18.2		13.1
0650	14.0	18.2		13.4
1000	14.0	18.2		13.4
1500	14.7	21.0		14.9
2500	16.1	19.2		15.6
4000	21.3	19.0		21.0
6500	35.3	20.0		35.5
9230	46.4	21.0		46.4
10000	46.0	21.4		49.0
15000	43.9	21.1		44.9
50000	50.5	20.9		51.4
80000	54.0	20.4		55.2



TIME	RES	TEMP	C	RES
0330	14.0	21.0		14.7
0340	14.0	18.5		13.7
0400	14.0	19.5		13.7
0650	13.6	19.9		13.6
1000	14.0	19.8		14.0
1500	14.7	21.0		14.9
2500	16.9	17.3		16.0
4000	21.7	18.1		20.7
6500	35.5	20.0		35.5
9230	49.3	21.0		47.0
10000	50.0	21.4		51.3
15000	44.9	21.1		44.9
50000	52.9	20.9		53.6
80000	57.1	20.4		57.5

FIGURE 7.12 : Effect of 5% extra air and 5% extra aggregate on the resistivity-time relationship of a concrete mix.



7.10 INVESTIGATION ON THE INFLUENCE OF AGGREGATE'S CONDUCTIVITY UPON MIX RESISTIVITY

7.10.1 Theoretical Approach

Patnode and Wyllie<sup>149</sup> have shown that Archie's equation for Formation Factor is valid only for non-conductive solids immersed in an electrolyte.

Archie's equation:-  $FF = \frac{\rho_c}{\rho_p}$  .....(7.3)

where

$\rho_c$  = Resistivity of concrete 100% saturated with paste,

$\rho_p$  = Resistivity of paste

If, however, the aggregate does contribute to electrical conduction<sup>149,156</sup>:-

$$\frac{1}{\rho_c} = \frac{1}{\delta_F} + \frac{1}{\delta_p} = \frac{1}{\delta_F} + \frac{1}{FF\rho_p}$$
 .....(7.4)

where

$\delta_F$  = Resistivity of conductive rocks as distributed

$\delta_p$  = Resistivity of the paste as distributed

$\delta_p = FF\rho_p$

where

$\rho_p$  = bulk paste resistivity

FF = Formation factor that would be found if none of the solids were conductive.

If we now consider a cement paste containing conductive solids:-

where

$\rho_s$  = Resistivity of conductive solids + electrolyte

$\rho_F$  = Bulk resistivity of conductive solids

$X_F$  = Volume fraction of conductive solids in the combination paste + solids

$\phi$  = Volume fraction of paste

$$\frac{1}{\rho_s} = \frac{X_F}{\rho_F} + \frac{\phi}{\rho_p} = \frac{1 - \phi}{\rho_F} + \frac{\phi}{\rho_p}$$

or,

$$\rho_s = \frac{\rho_p \rho_F}{\rho_p - \phi(\rho_p - \rho_F)} \quad \dots\dots(7.5)$$

Rearranging the expression:-

$$\frac{1}{\rho_s} = \frac{1 - \phi}{\rho_F} + \frac{\phi}{\rho_p} \quad \dots\dots(7.6)$$

Employing two values of  $\rho_s$  for two corresponding values of  $\rho_p$  we may solve the equation for the two unknown quantities<sup>156</sup>

$$\frac{1 - \phi}{\rho_F} \quad \text{and} \quad \phi$$

$$\text{Solving, } \phi = \frac{(\rho_p)_1 (\rho_p)_2 \{(\rho_s)_1 - (\rho_s)_2\}}{(\rho_s)_1 (\rho_s)_2 \{(\rho_p)_1 - (\rho_p)_2\}} \quad \dots\dots(7.7)$$

$$\text{and } \frac{1 - \phi}{\rho_F} = \frac{(\rho_p)_1 (\rho_s)_2 - (\rho_p)_2 (\rho_s)_1}{(\rho_s)_1 (\rho_s)_2 \{(\rho_p)_1 - (\rho_p)_2\}} \quad \dots\dots(7.8)$$

Where  $(\rho_s)_1$  and  $(\rho_s)_2$  are the resistivities of two concretes saturated with cement pastes of resistivity  $(\rho_p)_1$  and  $(\rho_p)_2$  respectively.

Substituting (7.7) and (7.8) back into equation (7.6) we obtain an expression for the resistivity of concrete in terms of the resistivity of the paste and that of the conductive aggregate fraction

$$\text{Also, if } \frac{1 - \phi}{\rho_F} < 0.02 \quad \frac{\phi}{\rho_p}$$

the error induced in the computation of  $\rho_s$  by neglecting the conductive solids would be less than 2% which is within permissible limits.

$$FF = \frac{\rho_c}{\rho_p} \quad (\text{For } \rho_c \text{ containing non conductive aggregate})$$

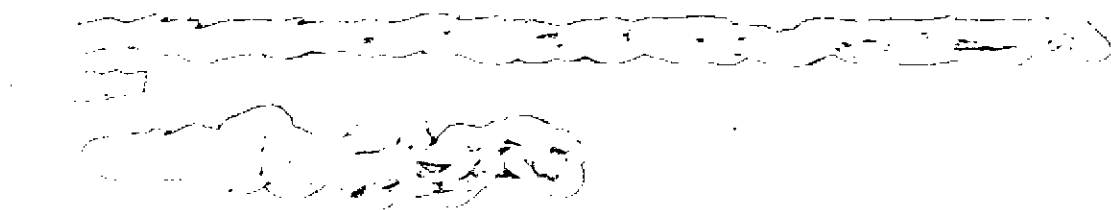
If, however,  $\rho_s$  does contain a fraction of conductive solids:-

$$\frac{1}{\rho_s} = \frac{1 - \phi}{\rho_F} + \frac{\phi}{\rho_p}$$

$$\text{Let } \frac{1 - \phi}{\rho_F} = k_1 \quad \text{and } \left(\frac{\phi}{\rho_p}\right) = k_2$$

$$\frac{1}{\rho_s} = k_1 + \frac{k_2}{\rho_p}$$

$$\frac{1}{\rho_s} = \frac{k_1 \rho_p + k_2}{\rho_p} \quad \rho_s = \frac{\rho_p}{k_1 \rho_p + k_2} \quad \dots\dots(7.9)$$



The above would therefore take into account the conductivity of the solid conductive fraction.

#### 7.10.2 Experimental Verification of Conductive Aggregate Criterion

Coarse and fine aggregates, as employed during the experimental programme, were mixed in a ratio of 2:1 in sufficient quantity to fill two 150mm moulds. To these were added two different salt water solutions, at a temperature of 20°C, whose resistivities had been previously measured. Salt water rather than cement water solutions were employed as the latter do not dissolve completely thereby making it impossible to maintain constant the proportion of solids in the mix. The following results were obtained:-

- $(\rho_p)_1 = 2.1 \text{ ohm.m}$       ) strong salt solution
- $(\rho_s)_1 = 3.8 \text{ ohm.m}$
- $(\rho_p)_2 = 150.7 \text{ ohm.m}$       ) weak salt solution
- $(\rho_s)_2 = 147.5 \text{ ohm.m}$

Substituting the above data into equations (7.7) and (7.8):-

$$\frac{1 - \phi}{\rho_F} = 0.00316 \quad \left(\frac{\phi}{\rho_p}\right) = 0.54601$$

$$\text{and } 0.00316 < 0.02 \frac{0.54601}{\rho_p}$$

As, in all the fresh cement pastes tested,  $\rho_p$  is approximately equal to 1.6 ohm.m:-

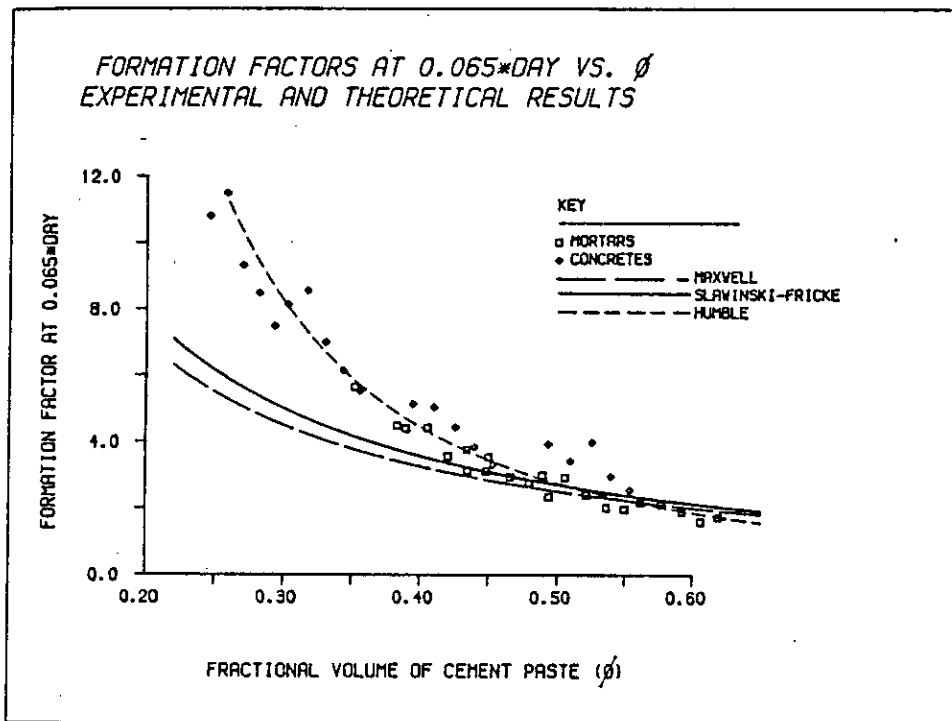
$$0.00316 < 0.02 \frac{0.54601}{1.6}$$

$$< 0.00682$$

The error due to conductive aggregates is less than 2%.

## 7.11 FORMATION FACTOR vs $\phi$ RELATIONSHIP

### 7.11.1 Comparing experimental results to theoretical curves



Maxwell - Formation Factor =  $\frac{3 - \phi}{2\phi}$

Fricke - Formation Factor =  $\frac{(2.4 - \phi)}{1.4\phi}$

Slawinski - Formation Factor =  $\frac{(1.3219 - 0.3219\phi)^2}{\phi}$

Humbles - Formation Factor =  $0.62\phi^{-2.15}$

Figure 7.13

From the concepts put forward in chapter five, the difficulties and dangers were appreciated which could be involved in treating concrete as a dispersion of electrically inert particles embedded in a conductive cement paste matrix. In the first instance, interparticle distance is too short for the material to be successfully treated as a dispersion also, aggregate particles are irregular in shape and size thereby making theoretical approaches very approximate.

The concept that hardening concrete could be best treated as a porous 'rock' rather than a dispersion was presented and a modified Archie's<sup>140</sup> equation, of the form  $FF = a\phi^{-m}$ , put forward as an initial model relating the formation factor FF to the fractional volume of cement paste. In order to test the goodness of fit of this relationship to experimentally obtained data the coefficients 'a' and 'm' were given values of 0.62 and 2.15 respectively as obtained in the course of an extensive study of rocks in the United States<sup>142</sup>.

The above four relationships were plotted against experimental results of fresh concretes and mortars and the quality of fit examined (Figure 7.13). The higher degree of fit of "Humble's relation"<sup>142</sup> is evident. Maxwell<sup>136</sup>, Fricke<sup>138</sup> and Slawinski's<sup>139</sup> curves proved to be reasonably accurate only for mixes with a high fractional volume of cement paste and therefore a higher degree of particle dispersion. The reason for their better fit to mortars, rather than concrete data, rests most probably in the fact that these equations were formulated for single sized, uniformly shaped particles. Also, for an equivalent fractional volume of aggregate, the grading of the aggregates in the concrete allows for a more effective packing arrangement than considered by the first three of the above formulae.

Having established Humble's generalized empirical approach to be the best fitting of all existing approximations, it was decided to accept its format but vary the coefficient ( $a=0.62$ ) and the exponent ( $m=2.15$ ) to best fit the results. Previous attempts to do this<sup>151</sup> for concrete, have over generalised the issue by ignoring (a) time dependency and (b) mix proportioning. It is by considering the effect of these factors on 'a' and 'm' that it is hoped new

light will be shed on the electrical resistivity characteristics of concrete and mortars.

### 7.11.2 Physical Explanation of Formation Factor Parameters 'a' and 'm'

Having established that the 'Formation Factor/Fractional volume of cement paste' is best expressed by a function of the form  $FF = a\phi^{-m}$ , the following is an explanation of the physical meaning of the 'a' and 'm' values. The significance of 'a' and 'm' as interpretation tools and the influence they have on the relationship are discussed.

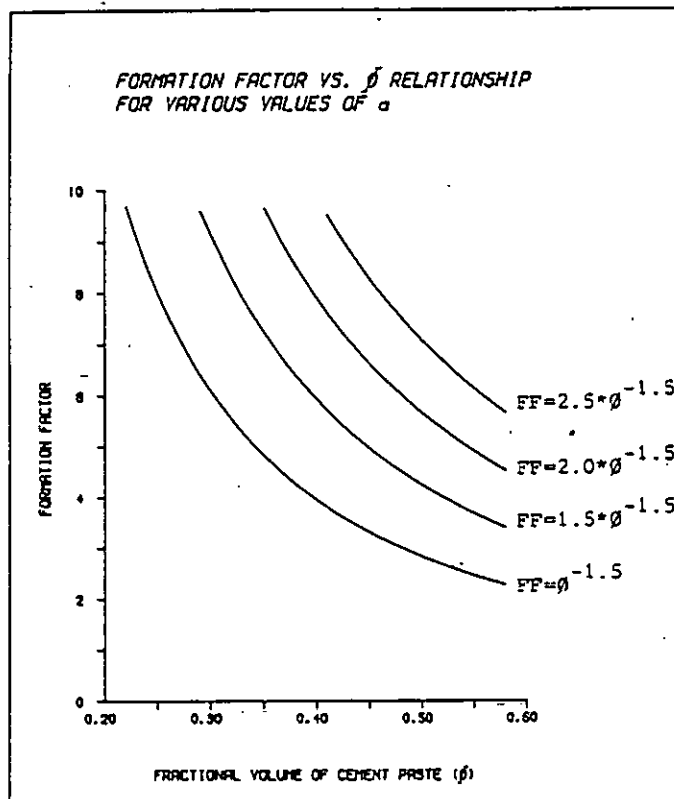


Figure 7.14

As shown in Figure 7.14, altering the value of 'a', whilst keeping 'm' constant, does not significantly alter the shape of the  $FF = a\phi^{-m}$  curve. For a constant fractional volume of cement paste, an increase in 'a' implies a higher FF value. An increase in

resistivity, whilst keeping the relative volumes of aggregate and paste the same, is caused by making it more difficult for the current to flow through the material. i.e. by employing a well graded aggregate fraction, conductive paths are more easily constricted.

Parameter 'a' is therefore a function of tortuosity and has been termed the Tortuosity Factor by Wyllie et al<sup>157</sup>. Like resistivity, permeability of a fluid through a porous formation is also dependent on pore geometry.

The Kozeny-Carman's equation, developed in 1950 to describe fluid flow through an unconsolidated porous media, is in agreement with the concept put forward above in that tortuosity of the pores affects the permeability of a fluid through a solid.

From Kozeny-Carman's equation,

$$\mu = \frac{\phi^3}{2.5(l_e/l)^2 s^2}$$

where

$\phi$  = Porosity,

$l_e$  = Effective distance electric current must travel

$l$  = External dimensions,

$s$  = Specific surface area,

$\mu$  = Permeability,

$(l_e/l)$  = Tortuosity

It can be seen that as Tortuosity, i.e. 'a', increases, permeability decreases even when there is no porosity variation or such variation is small. High values of 'a' are therefore the norm in geology when looking at low permeability rock formations.

Drawing a parallel between fluid permeability and electrical conductivity, it can therefore be postulated that an increase in tortuosity, for a fixed pore volume, has the effect of increasing the resistivity of a mix.

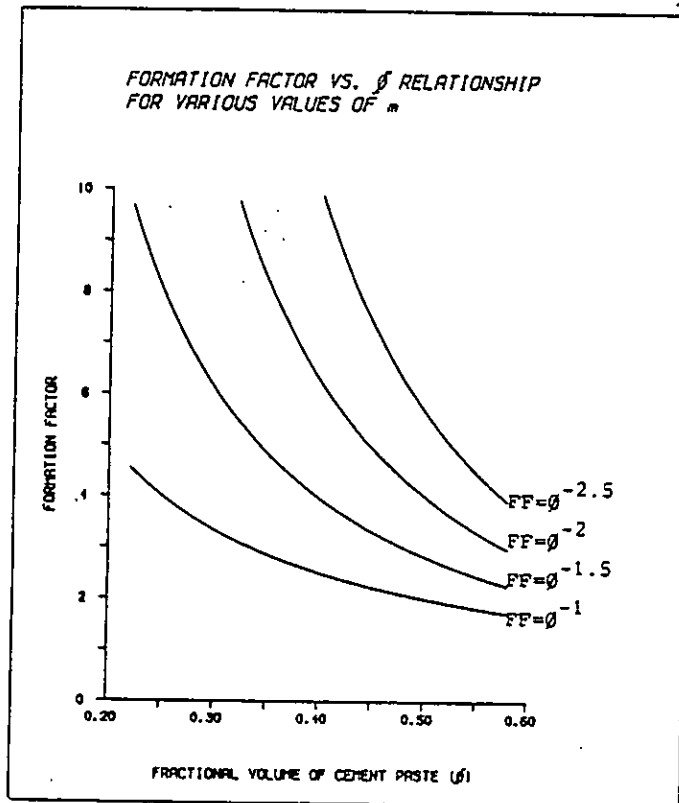


Figure 7.15

Figure 7.15 shows the marked influence that the 'm' exponent has in defining the shape of the FF versus  $\phi$  curve. At high values of 'm', the graph shows that, as the fractional volume of cement paste increases, the rate of decrease of Formation Factor diminishes; conversely, at low fractional volume of paste, slight increases in the paste content cause marked reductions in FF.

'm' has been termed the "Cementation Factor" by Sanyal et al 159, and geological data shows it decreasing with both permeability and porosity.

Parameters 'a' and 'm' should not be visualised as being unrelated; cementation, for example, whilst affecting the value of 'm', will also alter the Tortuosity ('a') of the conductive system.



As shown by Owen<sup>146</sup>, using synthetic pore models, an infinite number of combinations of Tortuosity and pore constriction are possible for any given porosity value.

### 7.11.3 Interpretation of Results

The following is a comparative assessment of the Formation Factor -  $\phi$  relationships obtained from experimental data at 0.065 and one-day.

Time = 0.065-day

MORTARS -

$$FF = 0.5455 \phi^{-2.27}$$

CONCRETES -

$$FF = 0.995 \phi^{-1.73}$$

TIME = 1.00-day

MORTARS:- W/C = 0.40 -

$$FF = 0.831 \phi^{-1.985}$$

W/C = 0.50 -

$$FF = 1.232 \phi^{-1.985}$$

W/C = 0.60 -

$$FF = 1.826 \phi^{-1.985}$$

CONCRETES:-

W/C = 0.40 -

$$FF = 0.295 \phi^{-2.447}$$

W/C = 0.50 -

$$FF = 0.447 \phi^{-2.447}$$

W/C = 0.60 -

$$FF = 0.676 \phi^{-2.447}$$

TIME = 0.065-day

MORTARS:- Ref. Figure 7.16

Linear regression of results yielded the following equation.

$$FF = 0.54455 \phi^{-2.27} \quad r^2 = 0.949$$

**NOTE:-** Mortars of low workability (S/C = 4.5  $\rightarrow$  6.0, W/C = 0.5) were not employed in the regression of the data as, in these cases, the fractional volume of the cement paste could not be determined accurately.

Figure 7.16 shows mortars, with equal sand/cement ratio but varying water/cement ratios, having a marked difference in both fractional volume of cement paste and the Formation Factor. This can be accounted for by the fact that, as the water/cement ratio

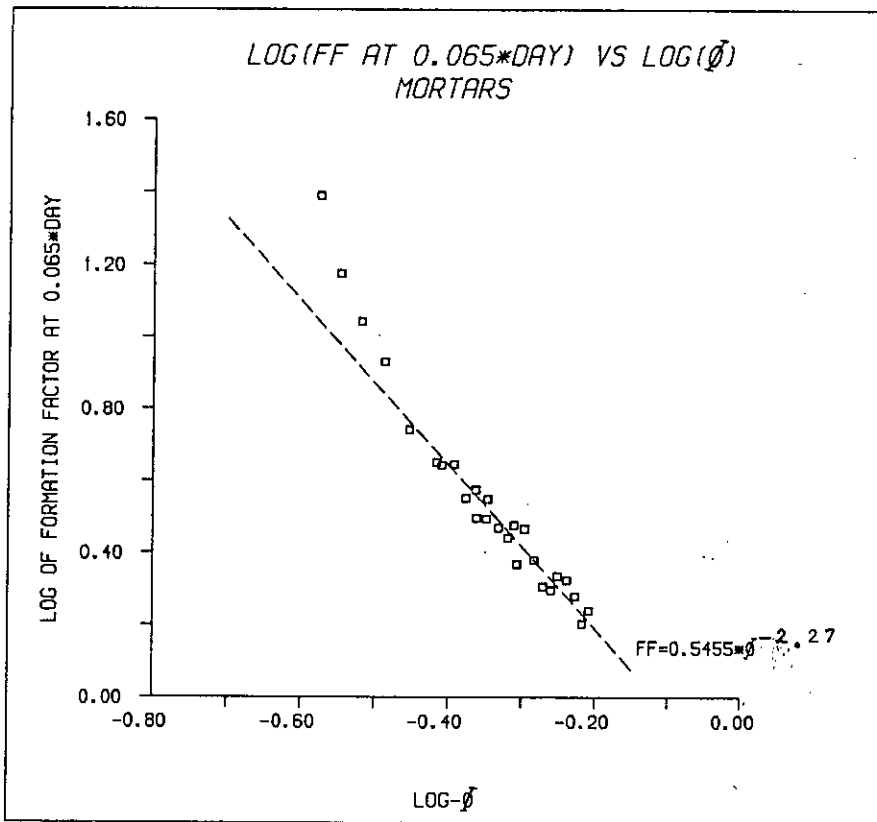


FIGURE 7.16

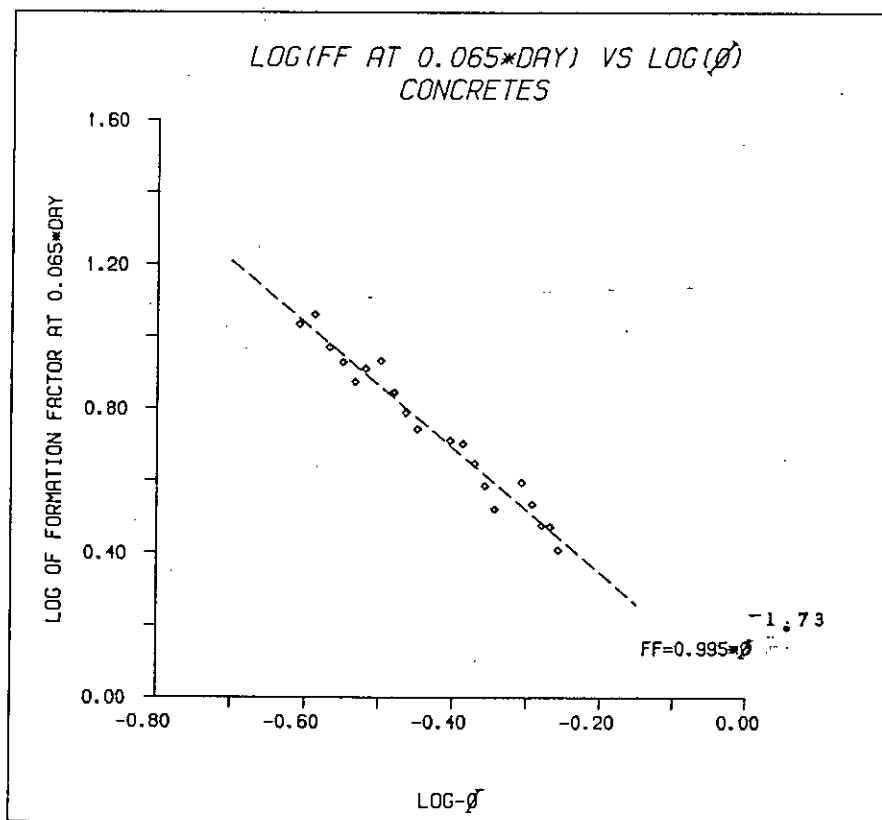


FIGURE 7.17

changes, so does, indirectly, the aggregate/cement ratio. i.e. for mix S/C = 1.5 and W/C = 0.4,  $V_p/V_m = 0.5608$  while, using the same mix but increasing W/C to 0.6,  $V_p/V_m = 0.6190$ .

$V_p$  = Volume of paste  
 $V_m$  = Volume of mortar

In this case, as in all the rest, the best fit line was observed not to pass through the origin (i.e. Formation Factor = 1 for a cement paste). This can be explained by the fact that the relationships, established for mixes with a uniformly distributed aggregate fraction, do not hold true when the aggregate is present in such low quantities to make tortuosity and constrictions effects negligible.

Concretes:-

Ref. Figure 7.17

$$FF = 0.995 \phi^{1.73} \quad r^2 = 0.967$$

In figure 7.17 it is evident that, for every mix, some points lie above and some lie below the best fit line. The points lying below the line are those with a high water/cement ratio whilst those above belong to mixes with a low water/cement ratio. This departure from linearity may be explained by the fact that the larger number and size of capillary pores present in pastes with a high water content, make them less susceptible to the 'constriction effect' of the aggregate.

The linear relationship at the setting stage is explained by the following:

$$FF = \frac{\rho_{mix}}{\rho_{paste}} \quad \rho_{mix} \propto \text{Amount of aggregate in the mix.}$$

$$\rho_{paste} \approx \text{Constant during setting.}$$

TIME = 1.00-day

Comparing Formation Factors obtained from fresh mixes to those from hardened ones, see figures 7.18 and 7.19, the following observations were made:-

- (a) Both the 0.065 and the 1-day graphs show a linear relationship within the workable range; however, as the material becomes

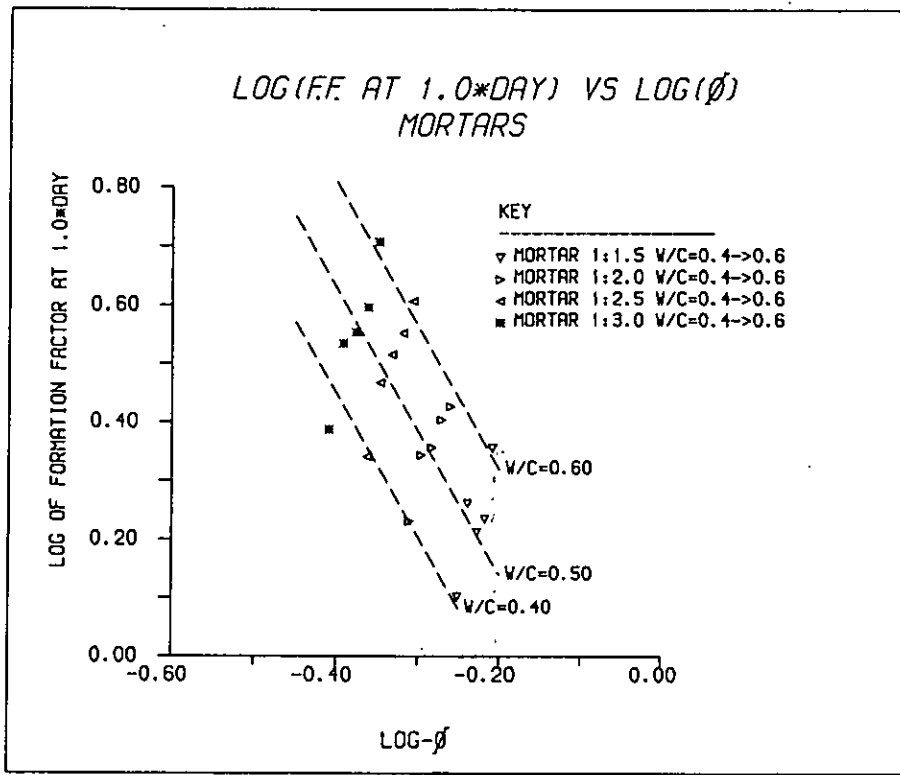


FIGURE 7.18

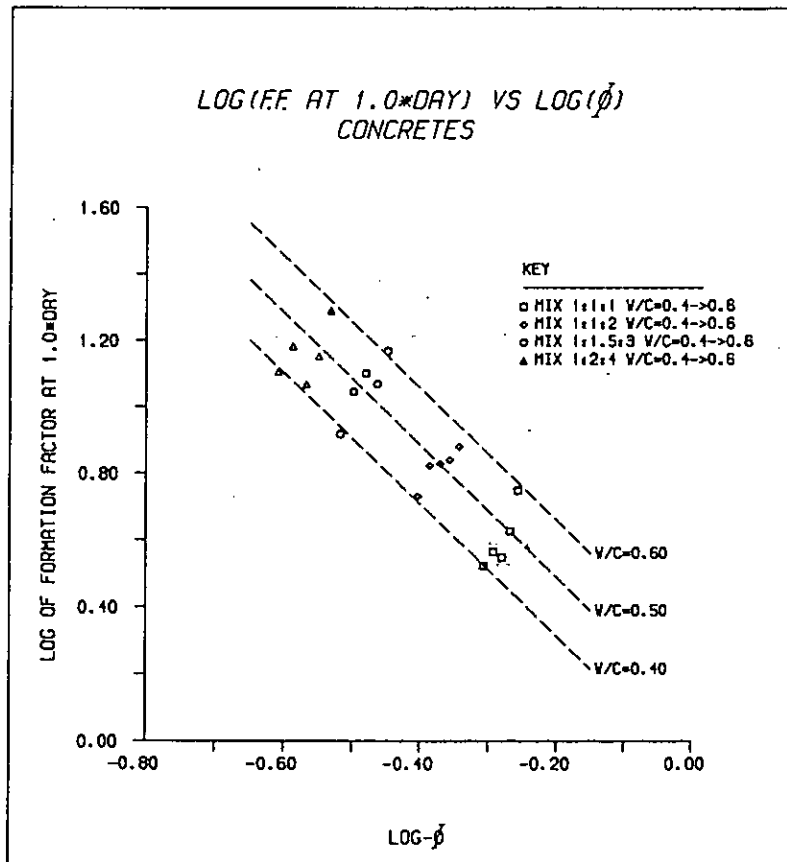


FIGURE 7.19

difficult to compact, and air is entrained, FF increases exponentially with a decrease in cement paste fraction, see figure 7.16.

- (b) Whilst in a fresh state, the Formation Factor parameter is related to the particle size and grading, and  $\phi$ , the fractional volume of cement paste.
- (c) There has been an increase in Formation Factor between the fresh and hardened states. This indicates that mixes have achieved higher resistivities than pastes of equal water/-cement ratio.
- (d) In the day-1 graphs, a family of lines developed, each corresponding to mixes with equal water/cement ratio. Mixes with higher water/cement ratios yielding higher Formation Factors than ones with low water/cement ratios.

It was therefore decided to investigate more closely the relative variations in resistivities of mixes and pastes during the fresh to hardened transitional stage.

#### 7.12 FORMATION FACTOR CHARACTERISTICS DURING INITIAL 24 HOURS

The main obstacle to the direct application of Archie's<sup>140</sup> equation to concrete lies in the time dependent characteristics of the material. McCarter<sup>133</sup> started from the presupposition that formation factor, by its own formulation, would be independent of the conductivity of the electrolyte, in the case of concrete, the cement paste.

In other words, that

$$\frac{d\rho_{\text{mix}}}{d_{\text{Time}}} = \frac{d\rho_{\text{paste}}}{d_{\text{Time}}} \quad \text{would hold true}$$

Theories put forward in chapter five do not agree with this concept. The rate of change of resistivity in a concrete mix, it was put forward, would be different from that of a cement paste of equivalent water/cement ratio and the extent to which the formation factor varies with time would be a function of both  $\phi$  and the water/cement ratio of the paste.

$$FF = \frac{\rho_{\text{mix}}}{\rho_{\text{paste}}} = f(\phi, w/c, \text{time})$$

The primary objective of this section is therefore to verify that resistivity changes in a mix during hydration are not solely dependent on the cement paste phase.

Figure 7.20 shows the variation in Formation Factor ( $\rho_{\text{mix}}/\rho_{\text{paste}}$ ) for a typical 1:1.5:3 concrete mix with a w/c ratio of 0.50. As can be seen, the Formation Factor does not remain constant over this period as has been assumed in the past<sup>133</sup> but, aside from being a function of  $\phi$  as in figures 7.16 to 7.19, it is also time dependent.

### 7.12.1 Effect of Mix Variations on the Time Dependent Characteristics of the Formation Factor

#### 7.12.1.1 Effect of varying the percentage aggregate by volume in the mix

See figure 7.21 for mortars and figure 7.22 for concretes. Note that all the mixes described in this section have a constant water/cement ratio of 0.50.

Both graphs show FF varying over the initial 24 hours for mixes of medium to high fractional volume of aggregate content. This reinforces the concept that resistivity changes occurring during the initial stages are not solely related to the cement paste.

A constant value of FF during the initial 24 hours implies that both the mix and the paste experience equal changes of resistivity during that period. i.e mix 1:1:1 in figure 7.22 and mixes 1:1.5, 1:2.0, and 1:2.5 in figure 7.21.

A noticeable increase in FF takes place during the setting stage and the early hardening period. This is subsequently followed by a more gentle and gradual decrease.

As the quantity of the aggregate present in the mix is increased, so is the proportion of constricted pores and the length of capillary paths. With reference to Figure 7.21 and Figure 7.22,

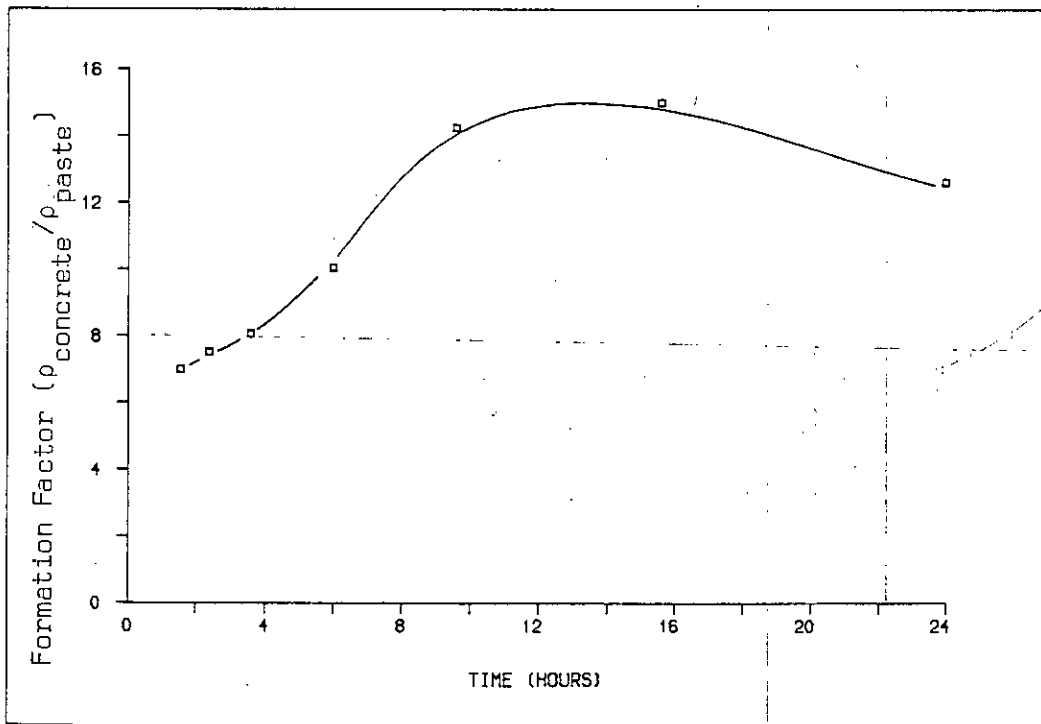
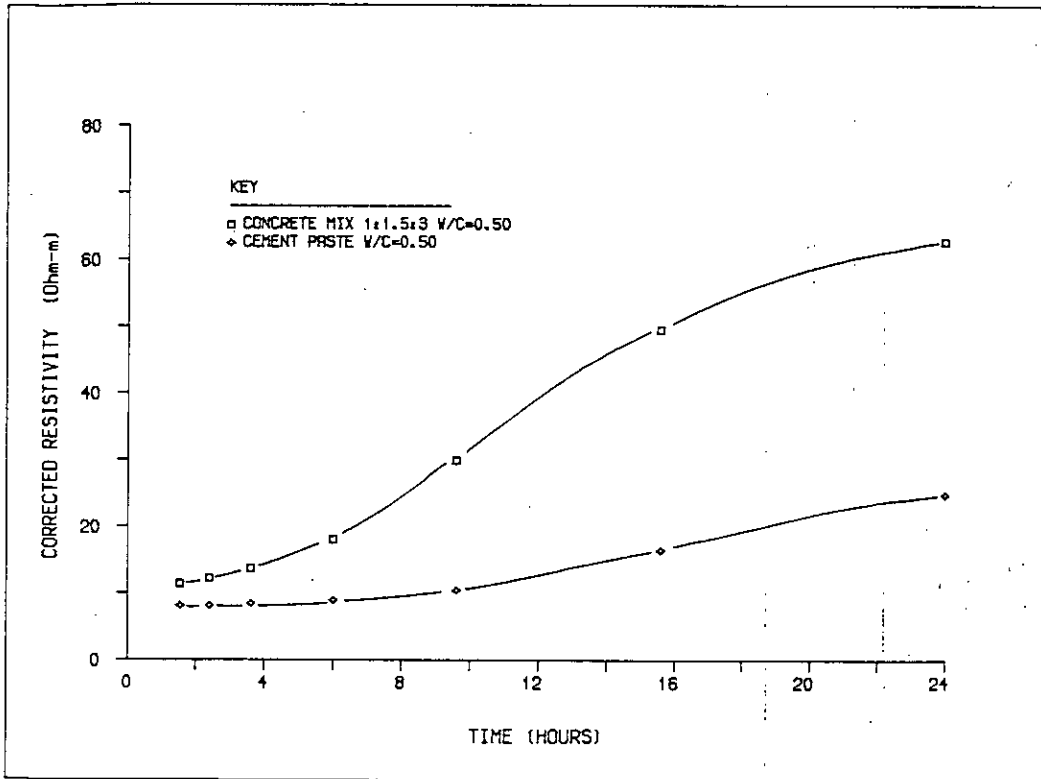


Figure 7.20 Resistivity and Formation Factor characteristics of a concrete mix during the initial 24 hour period. Note: The resistivity values of the paste (on upper diagram) have been multiplied by a factor of 5 prior to plotting.

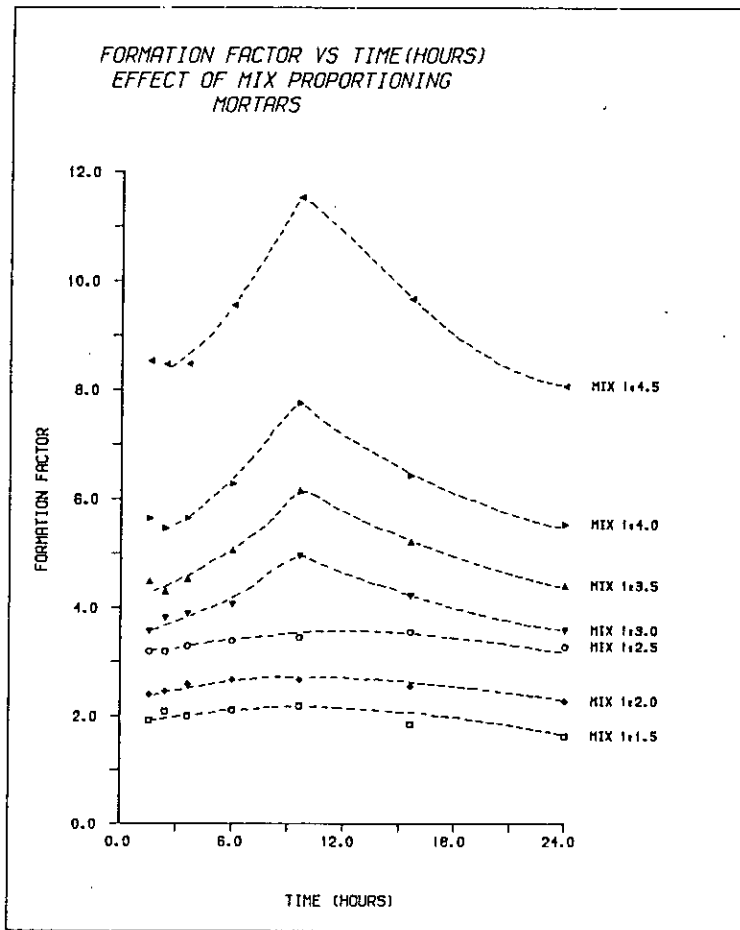


FIGURE 7.21

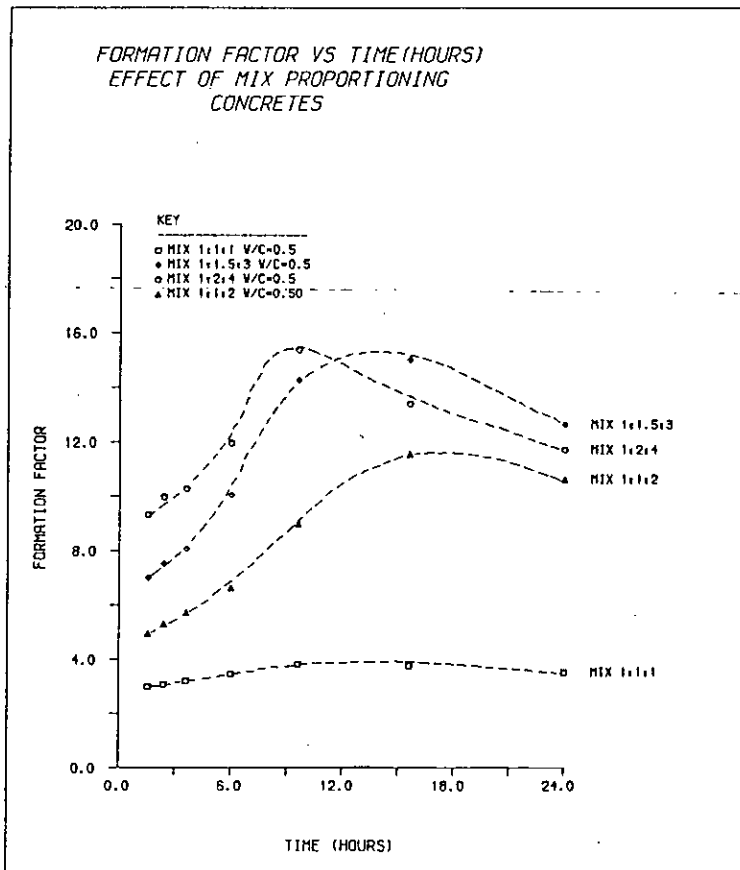


FIGURE 7.22



the more aggregate there is added to the system, the greater the shift from large to small pores, the bigger the increase in pore length and the more pronounced the difference in the  $dp/dt$  of paste and concrete.

#### 7.12.1.2 Effect of varying water/cement ratio

Ref. Figure 7.24 for concrete and Figure 7.23 for mortars.

Although the same trends are noted in concrete and mortars, they are much less accentuated in the latter.

In both Figures, there is a slight spread in the Formation Factor results obtained at the time of 1 hour and 30 minutes from the gauging of water. This is due to the fact that an increase in water/cement ratio causes:-

- (a) A decrease in the amount of aggregate present, and
- (b) A corresponding drop in the resistivity of the mix.

As hydration progresses, there is an initial increase in the Formation Factor, the rate of increase being higher for mixes with a low water/cement ratio.

Varying the water/cement ratio alters the pore size distribution of both paste and concrete mixes. A decrease in water/cement ratio results in a decrease in the number and size of large diameter capillary pores, whilst the opposite will happen for an increase in the water/cement ratio, (see figure 3.6).

#### 7.12.1.3 The effect of varying aggregate particle size on the Formation Factor-Time relationship

A series of tests was carried out to examine and assess the extent to which early age resistivity readings are influenced by the aggregates particle size.

As has been mentioned in section 7.7, the constricting of the capillary pores and the lengthening of their paths are physical effects of introducing aggregates to a water/cement paste. The first step in the experimental programme was to establish which fraction of the aggregates was the greatest contributing factor to this phenomenon. Test results showed that the size of the coarse

FORMATION FACTOR VS TIME (HOURS)  
EFFECT OF W/C RATIO  
MORTARS

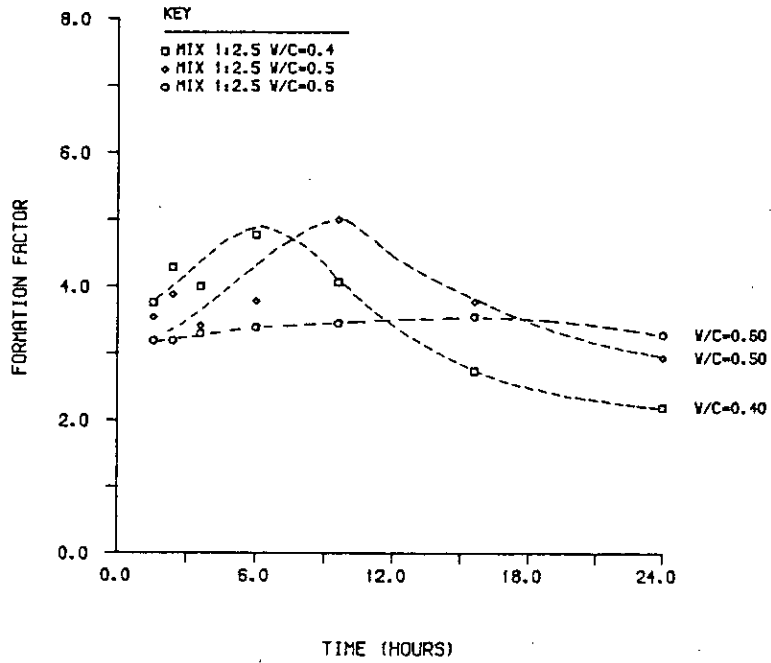


FIGURE 7.23

FORMATION FACTOR VS TIME (HOURS)  
EFFECT OF W/C RATIO

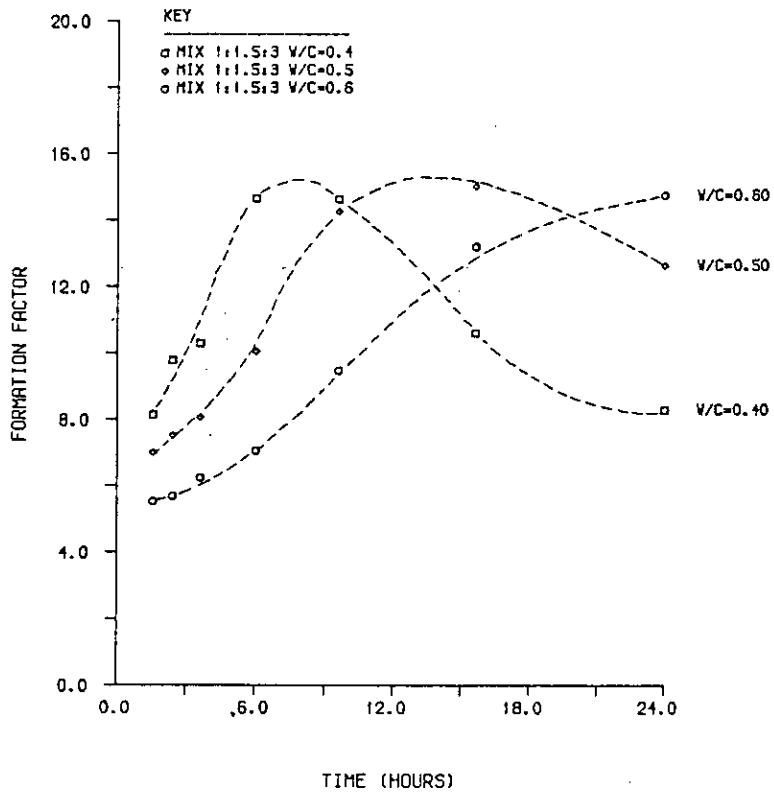


FIGURE 7.24

aggregates had no effect on the  $\rho$ - $t$  relationship but it was in fact the grading and the fractional volume of the finer graded sand which significantly altered it.

It was therefore decided to study the difference between concretes and mortars of equal total percentage aggregate by volume such as, for example, concrete mix 1:1.5:3 and mortar mix 1:4.5, (W/C = 0.50) See figures 7.21 and 7.22. Employing these two mixes to explain the general behaviour, the following observations were drawn.

- a) Mixes of equal volume of aggregate have an equal initial Formation Factor value.
- b) As hydration progresses the mortar showed an increase in Formation Factor from 8 to 12 whereas the concrete showed an increase of double this amount and raised to a Formation Factor of 16.
- c) After 24 hours the Formation Factor of the mortar dropped to 8 whereas that of concrete had a much slower decline and only dropped to 13.

The time dependence of the Formation Factor during the initial 24 hours cannot be attributed to absorption of water by the aggregates. If this was to be the case, mortars would have a higher increase in Formation Factor than the concretes given.

- a) The greater surface area offered by the smaller particles, and
- b) That the absorption coefficients of the two aggregate phases are not significantly different.

For mortars of low sand-cement ratios, i.e. mix 1:1.5, the percentage volume of sand is insufficient to induce constriction effects or increase  $l_e$  (the effective length of the conductive path) to any significant extent. This is reflected in the Formation Factor vs Time graph which shows the Formation Factor to be practically constant over the initial 24 hour period (see figure 7.21). In mortars of higher sand-cement ratios, however, the percentage volume of sand is sufficient to cause both a shift in the pore size distribution towards the smaller diameter range, and an increase in  $l_e$ .

Comparing a mortar and a concrete mix of equal fractional volume of cement paste, the latter will have bigger and less numerous aggregate particles and therefore a smaller variation in the pore sizes formed by the smaller grains. For a constant volume of particles, the smaller the particles the higher the variation in pore size distribution, towards the small diameter range, that they will cause. For an equivalent volume of aggregate, therefore, mortars attain the Formation Factor peak at an earlier age than concretes, see figures 7.21 and 7.22.

In concrete, the range of particle sizes contributes significantly to prevent segregation of the particles. The more uniform and effective packing which they provide induces bigger changes in the pore size distribution. The pore structure both in the early stages and later, during the hardening period, is therefore altered to a more significant extent.

### 7.13 DISCUSSION

The experiments carried out to investigate the conduction of electricity through cement pastes, mortars and concretes have been reported in this chapter.

Prior to the start of the main experimental work, investigations were undertaken into certain relevant properties of the constituent materials. Absorption tests (section 7.2) on the aggregates employed showed these to have a negligible effect on the water/cement ratio of the mixes employed. This would however, not necessarily be the case for all aggregates and a corrected water/cement ratio should be used where deemed necessary.

An investigation carried out on water/cement solutions of varying concentrations showed these to achieve electrolytic saturation of the aqueous phase at a percentage cement content corresponding to a water/cement ratio of 1.7. The implication being that, in the case of commonly employed concrete mixes ( $w_c < 1.00$ ), the conductivity of the fresh cement paste would be independent of the water/cement ratio. It is however possible

that the proportion of cement in solution required to achieve electrolytic saturation is dependent on the cement employed and a preliminary calibration check should be made when different cements are being used.

As described in chapter four, the rate at which the ions dissolved in the aqueous phase of the cement paste can transfer electrical charges, is related to the temperature at which the test is being carried out.

In this light, conductivity tests were carried out on an electrolytically saturated water/cement solution at different temperatures and, from the relationship  $\rho_T = \rho_0(1 + \alpha\Delta T)$ , a temperature coefficient of resistivity of 0.0216 was calculated. This value compares favourably with that of 0.022 obtained by McCarter et al<sup>131</sup> using pastes in a solid rather than a liquid state. This could be a pointer to the fact that the aqueous phase of the cement paste does not vary significantly in electrochemical properties of ionic composition during the hardening process.

The investigation of the conduction mechanism through cement pastes, mortars and concretes involved a study of the resistivity-time graphs for mixes of varying composition.

The conduction of cementitious mixes can, in the main, be attributed to the presence of an electrolyte filled porous structure in the cement paste. As described in chapter three (sections 3.3.3.1 and 3.3.3.2) the evolvment and nature of the porous structure is dependent on the hydration stage, the mineralogical composition of the cement employed, the water/cement ratio and the curing conditions.

During the course of the experimental programme both the composition of the cement and the curing conditions were, in as much as possible, maintained constant which meant that the only two variables affecting the pore structure in the cement paste were the hydrating stage and the water/cement ratio.

By measuring the resistivity during the course of hydration, it is therefore possible to indirectly monitor the rate at which the

capillary pores are gradually filled by hydration products.

Figure 7.4 shows the variation in resistivity of cement pastes as a function of time and water/cement ratio. The resistivity of the pastes during the setting stage coincided with the maximum conductivity of cement-water solutions obtained in the test for electrolytic saturation (= 1.6 ohm-m).

The end of the setting stage, when the pore volume is decreased by the development of hydrates, is marked on the resistivity-time graph by a rise in resistivity. It has been shown, experimentally that the electrical resistivity technique can be successfully employed to study the development of set in water-cement pastes and final setting times similar to those obtained using Vicat's apparatus were achieved.

Vicat's test is generally employed as a monitor for the setting characteristics of pastes of standard consistency. As a measure of the rigidity or stiffness of cement pastes with varying water/cement ratios it has, as yet, not been proven to be valid. Because of the sensitivity to changes in the effective cross-sectional area available for conduction, the electrical resistivity method might be better suited to establishing final setting times than mechanical techniques which have to rely on relatively large changes in the physical structure of the material.

Although previous research suggests that the test could be just as easily applied to concretes as pastes<sup>47,48</sup>, results obtained during the course of this experimental programme do not substantiate this. Aggregates, by increasing the tortuosity and the constriction of the pore network, make pores more liable to be segmented by hydration products. This causes an earlier increase in resistivity than would have been predicted from a cement paste of equivalent water/cement ratio.

An investigation was carried out to assess whether the presence of macro-voids could be discriminated from non-conductive solids by means of the resistivity technique. A test was devised whereby air-filled cylindrical cavities of known volumes were incorporated in the sample and a resistivity comparison made with a

similar mix where an equivalent volume of extra aggregate had been added. The resistivity-time graphs were found to be identical. Aside from confirming the fact that electrical conduction is through the cement paste matrix, the test showed the need for full compaction of the material if a meaningful relation between resistivity and physical characteristics were to be established.

It was noted, however, that an assessment could be made on the degree of compaction of the mix on the basis of a drop in resistivity which specimens underwent upon submergence at the water curing stage.

As concrete aggregates may be of low resistivity or may contain clay or silt fractions which would affect the conductivity of the system, a technique<sup>156</sup> to allow for this to be taken into account has been adapted for concrete. This method was also employed to check that the aggregate employed in the experimental programme made a minimal contribution to the conductivity of the mixes tested.

As put forward in chapter 5, the complexity and wide variation in the nature of pore systems in cement pastes precludes the mathematical development of a relationship describing the conductive mechanism through the material. The electrical resistivity of the various mixes has, therefore, been analysed in terms of the formation resistivity factors  $\rho_{\text{mix}} / \rho_{\text{paste}}$  which was found to best relate to  $\phi$ , the fractional volume of cement paste, by the empirical relation:-

$$FF = a \phi^{-m}$$

Though, by definition, the formation resistivity factor of an electrically inert porous medium (compacted aggregate) is independent of the electrical conductivity of the solution (cement paste), this was not found to be true in the case of hardening concrete mixes.

Results of the investigation were in agreement with the theories put forward in chapter five whereby aggregates, by affecting the tortuosity and by constricting the conductive paths, would affect the ease with which they would become blocked by

hydration products. This was demonstrated by analysing the variations in formation factor with time for mixes of varying proportions.

In the course of the investigation, emphasis has been placed on the effect of pore size distribution on the formation resistivity factor at varying water/cement ratios, fractional volumes of cement paste and ages. Although it is impossible at this point to determine quantitatively the relative role of each factor, a better qualitative understanding has been provided.

Because of the demonstrated dependence of resistivity on both mix proportions and the physical changes induced by the hydration process, it was decided to investigate the possibility of employing the technique to monitor these variations in concrete mixes.

#### 7.14 CONCLUSIONS

An extensive experimental programme was undertaken which confirmed the theories put forward in chapter 5, and showed the validity of the test methodology. The following conclusions were drawn from this study:-

1. For the aggregates employed absorption effects were not found to have an appreciable influence on the resistivity-time relationship.
2. Cement-water solutions, prepared using the same materials employed during the experimental programme, achieved electrolytic saturation at approximately 37% cement content.
3. The temperature coefficient of resistivity for an electrolytically saturated water/cement solution was established at 0.0216.
4. The end of setting and the beginning of the hardening stage is marked by an increase in resistivity.
5. As the cubes were submerged for curing, resistivity readings dropped as water permeated the air filled cavities. The size of the one-day discontinuity is related to the permeability and porosity of the specimens.
6. In the later stages of the submerged curing period the rate of increase of resistivity diminished and tended to a constant value.



7. A number of parameters were singled out from the resistivity-time graph to be employed in the analysis of the different mixes.
8. Whilst setting, all cement pastes maintained the same resistivity level of approximately 1.6 ohm.m.
9. An increase in the water/cement ratio results in larger and more numerous capillary pores, thus the amount of hydrates required to obstruct them increases accordingly. Mixes with high water content will, therefore, acquire a given resistivity value at a more advanced stage of hydration than those with less water.
10. Resistivity measurements allow for the determination of final setting time of cement pastes with an accuracy comparable to that of Vicat's test.
11. The presence of aggregate reduces the cross-sectional area available for electrical conduction thereby increasing resistivity.
12. Aggregates increase the tortuosity and the constriction of the network of conductive paths thereby making these more susceptible to being blocked off by hydration products. The net result is a shift in the position of the apparent setting time.
13. The magnitude of the  $\rho$ -t discontinuity caused by the ingress of water at the submerging stage is related to the degree of compaction of a concrete mix and the water/cement ratio of the cement paste phase.
14. The apparatus and method employed have proved unable to distinguish between air voids and the non conductive solids in the mix.
15. The apparatus is well suited to the study of commonly employed well-compacted concrete mixes. Further development is required to widen the scope of the test to include air entrained mixes.
16. The aggregates employed during the course of the experimental programme have been shown to contribute only a negligible amount to the overall conductivity of a concrete mix.

17. The Formation Factor expression  $FF = a\phi^{-m}$  has proved to be the closest fitting format to the experimental data. The physical significance of parameters 'a' and 'm' was investigated and their values adopted as an interpretation tool in the analysis of the data.
18. By monitoring the Formation Factor characteristics during the initial 24 hours, it was confirmed that the aggregates affect the apparent rate of hardening as monitored by resistivity measurements.
19. The extent to which the Formation Factor varies with time for a concrete mix is a function of both  $\phi$  and the water/cement ratio of the paste.
20. Resistivity measurements of mixes of equal water/cement ratio and equal percentage volume of aggregate, were found to be dependent on the aggregate particle size.
21. The electrical resistivity of concrete mixes was substantially affected by the fractional volume of cement paste, the aggregate size, water/cement ratio and degree of hydration indicating that those factors controlling physical characteristics, such as strength, also affect electrical resistivity.

**CHAPTER EIGHT**

**RELATIONSHIP BETWEEN RESISTIVITY AND PHYSICAL  
PROPERTIES OF CONCRETE MIXES**

## 8.1 INTRODUCTION

Following the fundamental investigation on electrical conduction through concrete, an experimental programme was undertaken to examine the possibility of employing the resistivity testing technique to monitor certain physical characteristics.

The chapter has been subdivided into two main sections:-

- (i) Relating Resistivity Measurements to Mix Proportions, and
- (ii) Relating Resistivity Measurements to Compressive Strength and Density.

The first stage of the investigation is concerned with the assessment of mix composition by means of the resistivity techniques and shows that by using predetermined relationships, both the percentage aggregate content by volume and the water/cement ratio can be found.

On the basis of the results obtained in this initial investigation, it was decided to assess the method's ability to monitor and predict the development of strength and the density of the material. This study makes up the latter section of the chapter.

Regression analyses were carried out using the statistical package Minitab<sup>157</sup> which, for a series of data, yielded the best fitting regression coefficients and the correlation coefficient  $r^2$ .

Although the regressed relationships obtained during the course of the study are unique to the particular materials employed in the experimental programme, the trends which have been established between electrical and physical parameters show great promise.

### (1) RELATING RESISTIVITY MEASUREMENTS TO MIX PROPORTIONS

The problem of characterising a concrete mix at a sufficiently early stage to allow for remedial measures to be taken is one which has received a considerable amount of attention (see section 2.4.2.2). The most important parameters in mix design, and also the ones which are most liable to error at the batching stage, are the aggregate/cement and the water/cement ratios. Given that these two variables are also those which have the greatest influence on the

conductivity of a concrete mix it was decided to concentrate efforts towards establishing the resistivity technique's ability to monitor aggregate content and water/cement ratio. A further objective was set - to attempt to characterise the mix at as early a stage as possible.

## 8.2 PERCENTAGE AGGREGATE BY VOLUME

It has been shown experimentally that fresh cement paste has a resistivity of approximately 1.6 ohm.m at a time of 0.065 of a day, a value which was found to be independent of water/cement ratio over the tested range (0.4 → 0.6), see figure 7.4.

It would be expected that, as aggregate is added to a cement paste, there would be a reduction in the cross-sectional area available for conduction and the resistivity of the mix would be correspondingly increased. One would also expect increasing 'tortuosity' and 'constriction' effects caused by the rise in aggregate content (see sections 5.4, 5.5 and 5.6) and therefore the relationship between the latter and resistivity measurements might not be linear in nature. Resistivity at 0.065 of a day (approximately one hour and 30 minutes) was chosen as the aggregate content monitoring parameter because:-

- (a) by then the hydrating process has advanced sufficiently for the cement to have achieved electrolytic stability,
- (b) the hardening proper, where differences in water/cement ratio result in different resistivities, has yet to commence.

### 8.2.1 Mortars

Figure 8.1 shows the relationship between the percentage sand by volume and the measured mortar resistivity at 0.065 of a day.

The sand/cement ratio of the mortars employed ranged from 1.5 to 4.5 and the water/cement ratio from 0.4 to 0.6.

As postulated in section 8.2, resistivity and rate of change of resistivity were found to increase with aggregate content and, by regressing the data, the following equation was obtained:-

$$\% \text{Agg} = 73.9 - 97.4 / \rho \quad \dots (8.1)$$

$$r^2 = 0.955$$

Table 8.1 shows, in the first three columns, the experimental data employed in the regression for the best fit line while the last two columns show the percentage of aggregate content (as obtained by substituting the measured resistivity values into the regression equation) and the percentage difference between the calculated and the actual aggregate contents.

-----				
		1		
ACTUAL		-----	%AGGREGATE	
%AGGREGATE	RESISTIVITY	RESISTIVITY	CALCULATED	%DIFFERENCE
-----				
38.1000	2.90000	0.344828	40.3129	5.80803
39.4000	2.60000	0.384615	36.4366	-7.52142
40.8000	3.10000	0.322581	42.4802	4.11827
42.3000	3.10000	0.322581	42.4802	0.42613
43.9100	3.40000	0.294118	45.2532	3.05909
45.0700	3.30000	0.303030	44.3849	-1.52002
46.4300	3.30000	0.303030	44.3849	-4.40464
47.8900	3.90000	0.256410	48.9268	2.16506
49.4300	4.30000	0.232558	51.2506	3.68322
50.6300	3.90000	0.256410	48.9268	-3.36390
51.0800	4.70000	0.212766	53.1788	4.10897
52.0100	4.50000	0.222222	52.2576	0.47604
53.4600	4.80000	0.208333	53.6107	0.28191
54.9900	5.20000	0.192308	55.1720	0.33095
55.1700	5.20000	0.192308	55.1720	0.00360
56.5300	5.10000	0.196078	54.8046	-3.05214
56.6200	5.90000	0.169492	57.3948	1.36849
57.9500	5.80000	0.172414	57.1101	-1.44928
59.4500	6.50000	0.153846	58.9191	-0.89309
61.0300	6.90000	0.144928	59.7879	-2.03515

Table 8.1 Comparison of actual % aggregate contents by volume to those extrapolated from equation 8.1

### 8.2.2 Concretes

Figure 8.2 shows the relationship between the percentage total aggregate by volume and the resistivity at 0.065 of a day.

The data, obtained from concrete mixes of varying proportions and water/cement ratios, was found to yield a best fitting regression relationship of the same form as that achieved in the regression of mortar results. The coefficients were, however, altered:-

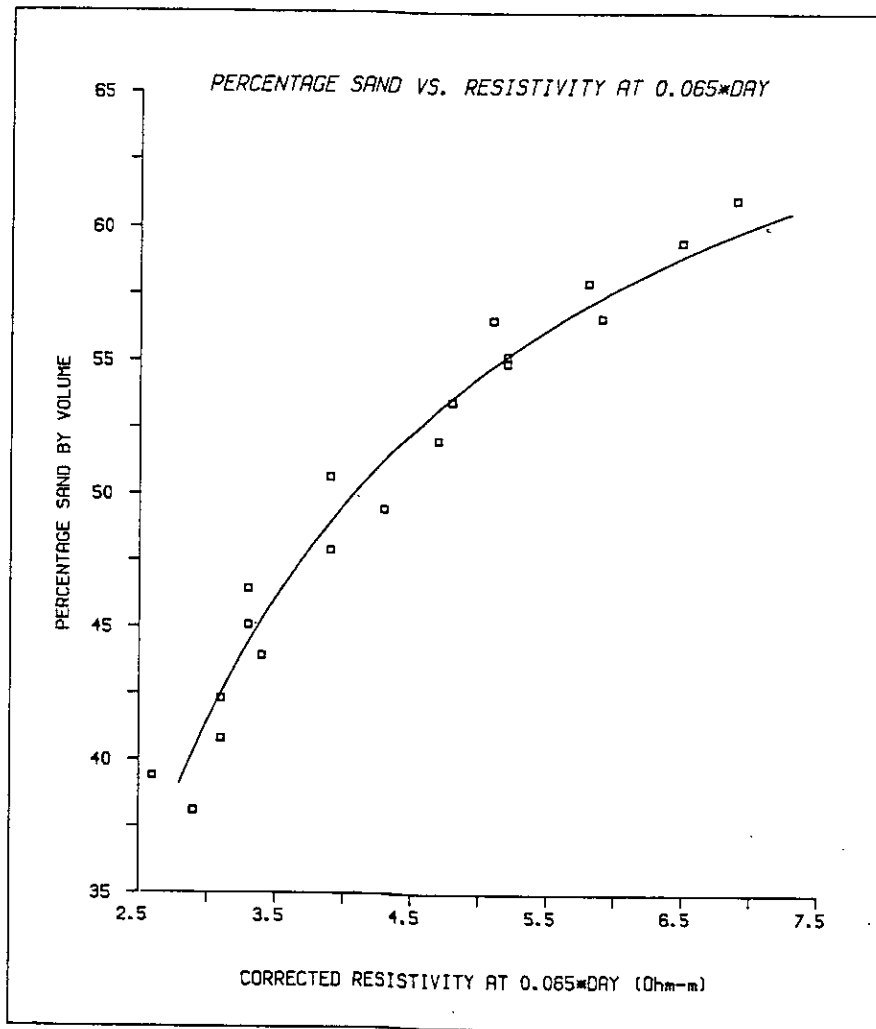


FIGURE 8.1

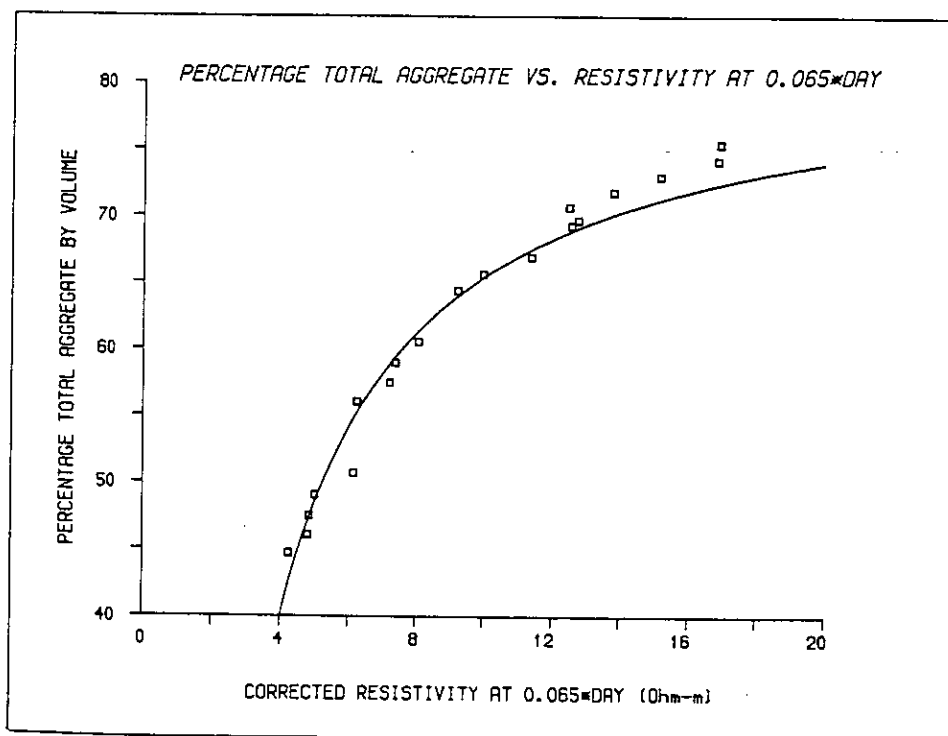


FIGURE 8.2

$$\text{Agg} = 82.5 - 171.1/\rho$$

.....(8.2)

$$r^2 = 0.93$$

As in the previous section, Table 8.2 shows the data employed for the regression and also the calculated values for aggregate content obtained by substituting actual resistivity values into equation 8.2.

-----				
1				
ACTUAL		-----	%AGGREGATE	
%AGGREGATE	RESISTIVITY	RESISTIVITY	CALCULATED	%DIFFERENCE
-----				
44.7000	4.2700	0.234192	42.5437	-4.8240
46.0700	4.8300	0.207039	47.1806	2.4108
47.5100	4.8700	0.205339	47.4711	-0.0820
49.0500	5.0300	0.198807	48.5865	-0.9450
50.7000	6.1700	0.162075	54.8595	8.2041
54.6700	5.5300	0.180832	51.6562	-5.5127
56.0300	6.2700	0.159490	55.3009	-1.3013
57.4600	7.2300	0.138313	58.9174	2.5364
58.9600	7.4000	0.135135	59.4600	0.8481
60.3000	12.5700	0.079554	68.9518	14.3479
60.5500	8.0700	0.123916	61.3760	1.3642
64.4000	9.2300	0.108342	64.0356	-0.5659
65.6500	10.0000	0.100000	65.4602	-0.2891
66.9500	11.4100	0.087642	67.5706	0.9269
69.7100	12.7700	0.078309	69.1645	-0.7825
70.6900	12.5000	0.080000	68.8757	-2.5666
71.8200	13.8200	0.072359	70.1806	-2.2827
72.9800	15.2000	0.065789	71.3025	-2.2986
74.1800	16.9000	0.059172	72.4326	-2.3556
75.4200	16.9600	0.058962	72.4684	-3.9136

Table 8.2 Percentage total aggregate by volume vs. resistivity at 0.065 of a day

The relationship between percentage aggregate by volume and resistivity has been shown to be represented by the equation  $\text{Agg} = \beta_0 + \beta_1/\rho$ ,  $\rho$  being the resistivity measured at 0.065 of a day. Although the form of the best fitting relationship was the same in the regression of both the mortars and the concretes, the obtained regression coefficients,  $\beta_0$  and  $\beta_1$ , differed. The reason for this lies in the amount of tortuosity and constriction in the pore structure caused by the difference in aggregate grading between the two materials. The effect of varying aggregate particle size on the



resistivity of a concrete mix has been discussed in detail in chapter five (section 5.5) and verified experimentally in chapter seven (section 7.12.1.3).

The high degree of fit of equations 8.1 and 8.2 to the mortar and concrete results is evident from the calculated correlation coefficients, 0.955 and 0.93 respectively, and also from the last column in Tables 8.1 and 8.2.

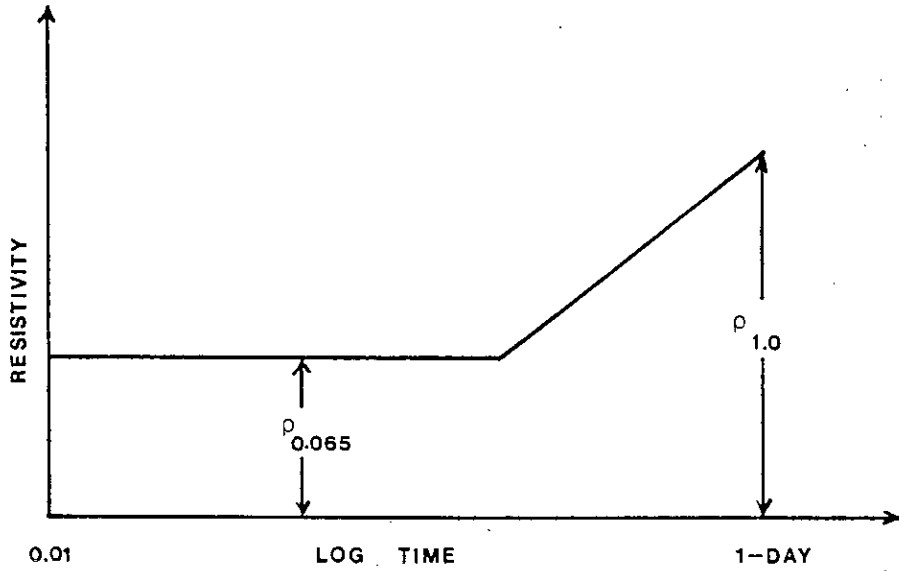
### 8.3 RELATING DAY-ONE RESISTIVITY PARAMETERS TO WATER/CEMENT RATIO

During the initial 24 hours of curing, the resistivities at two stages, 0.065 of a day and 1.0-day were chosen as w/c monitoring parameters for the following reasons:-

$\rho_{0.065}$ :- This parameter represents the resistivity at a very early age (approximately 1.50 hours). At this stage hydration has not yet advanced to the hardening stage and, for fully compacted mixes of equal cement type and equal curing conditions, the measurements will solely relate to the type and volume of aggregate present. As density measurements showed the mixes to be fully compacted, and curing conditions and aggregate type were maintained constant throughout,  $\rho_{0.065}$  only varied according to aggregate volume.

$\rho_{1.0}$ :- The value of this parameter represents the resistivity at that point in time at which hydration has proceeded furthest during the 24 hour air curing period.

$(\rho_{1.0} - \rho_{0.065})$ :- The rise in resistivity between the times of 0.065 of a day and 1.0 of a day is due to the formation of hydration products which effectively block the conductive paths through the cement paste fraction. The number and size of these conductive paths, capillary pores, is known to be dependent on the water/cement ratio, see figure 3.6. The higher its value, the larger and more numerous capillary pores are, and consequently, the lower the resistivity of the paste.



The water/cement ratio is therefore considered to be the greatest contributing factor affecting the rate of rise of resistivity with time during the latter part of the first day curing period.

$$\begin{aligned} W/C &= f\{(\rho_{1.0} - \rho_{0.065}) / (t_{1.0} - t_{0.065})\} \\ &= f(k(\rho_{1.0} - \rho_{0.065})) \end{aligned} \quad \dots(8.3)$$

Figure 8.3 below shows, however, how the value of  $(\rho_{1.0} - \rho_{0.065})$  is not a constant for equal water/cement in different mixes, but is also dependent on the percentage aggregate in the mix (monitored by  $\rho_{0.065}$ ), see also Table 8.3.

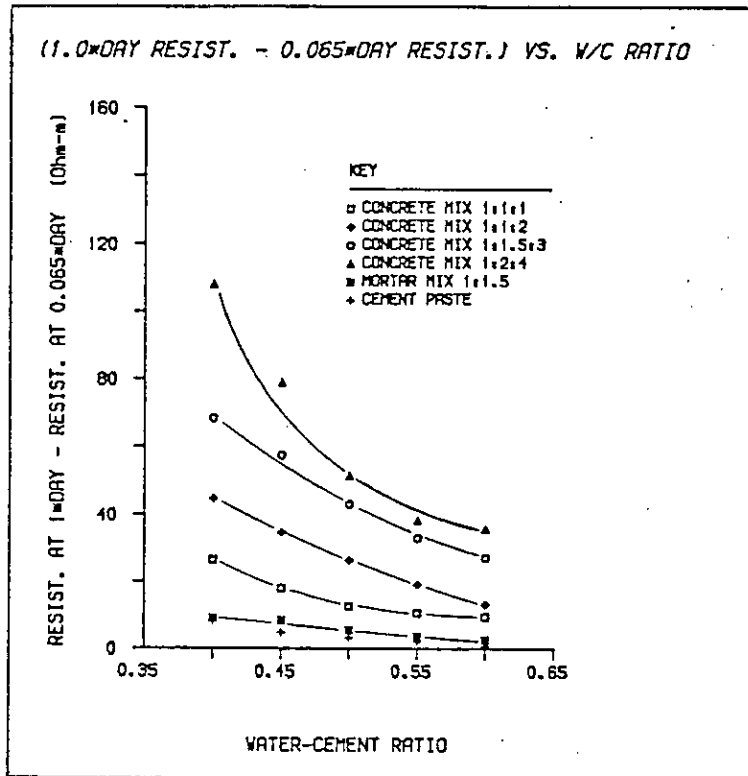


Figure 8.3 ( $\rho_{1.0} - \rho_{0.065}$ ) as a function of mix characteristics

CONCRETE MIX	W/C	( $\rho_{1.0} - \rho_{0.065}$ )	$\rho_{0.065}$
1:1:1	0.400000	26.4600	6.1700
	0.450000	18.0700	5.0300
	0.500000	12.6500	4.8700
	0.550000	10.6700	4.8300
	0.600000	9.5600	4.2700
1:1:2	0.400000	44.6300	8.0700
	0.450000	34.5700	7.4000
	0.500000	28.3000	7.2300
	0.550000	19.1300	6.2700
	0.600000	13.2000	5.5300
1:1.5:3	0.400000	68.3600	12.7700
	0.450000	57.4600	12.5700
	0.500000	51.3400	11.4100
	0.550000	33.0000	10.0000
	0.600000	27.1700	9.2300
1:2:4	0.500000	42.9700	15.2000
	0.550000	38.3000	13.8300
	0.600000	35.6700	12.5000

Table 8.3 ( $\rho_{1.0} - \rho_{0.065}$ ) as a function of both water/cement ratio and  $\rho_{0.065}$

It follows that, in order to predict successfully the water/cement ratios from resistivities obtained during the first day of curing, both  $(\rho_{1.0} - \rho_{0.065})$  and  $\rho_{0.065}$  must be employed as forecasting parameters.

Figure 8.4 shows the graph of  $(\rho_{1.0} - \rho_{0.065})$  as a function of  $\rho_{0.065}$  for the different concrete mixes tested. The lines, whose equations are given below, join up those points representing mixes of equal water/cement ratio.

W/C = 0.40	$Y = -100.91 + 70.78 \ln(x)$	$r^2 = 0.93$
W/C = 0.45	$Y = -62.41 + 48.84 \ln(x)$	$r^2 = 0.99$
W/C = 0.50	$Y = -41.65 + 34.40 \ln(x)$	$r^2 = 1.00$
W/C = 0.55	$Y = -30.43 + 26.72 \ln(x)$	$r^2 = 0.99$
W/C = 0.60	$Y = -24.03 + 22.60 \ln(x)$	$r^2 = 0.99$

where

$$x = \rho_{0.065}, \text{ and}$$

$$y = (\rho_{1.0} - \rho_{0.065})$$

See figure 8.4 for predicting curves.

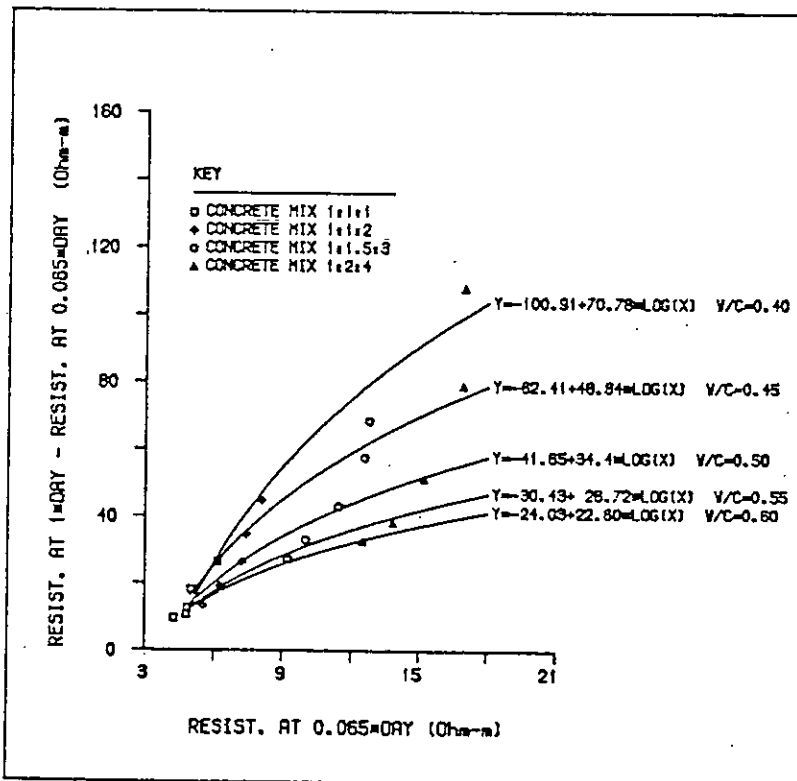


Figure 8.4 Predicting water/cement ratios from day-one resistivity parameters

As could be seen from figure 8.3, ( $\rho_{1.0} - \rho_{0.065}$ ) increases with aggregate content; in mixes which are rich in cement such as, for example, concrete 1:1:1 or mortar 1:1.5, this value is so low that it approaches that of a paste of equivalent w/c ratio. This is made more obvious in figure 8.4 where the water/cement ratio prediction curves converge to a common ( $\rho_{1.0} - \rho_{0.065}$ ) value at low aggregate/cement ratios. At such low aggregate contents, both 'tortuosity' and 'constriction' effects are of little consequence to the hardening rate as measured by resistivity readings, and the accuracy of prediction diminishes. The prediction curves are, however, more accurate within the range of commonly employed concrete mixes.

In the light of the conclusions drawn from chapter 7, i.e. that the electrical resistivity of a concrete mix is dependent on the chemical/geometrical characteristics of its constituents, it has to be noted that the relationships obtained above are unique to the materials employed during the course of the experimental programme.

Although meaningful and interesting trends have been established between resistivity parameters and mix proportions, regressed relationships might not be directly applicable to mixes of equal proportions but different constituent characteristics. Thus for the technique to be adopted as a site quality control technique, specific calibration would be required.

#### (ii) RELATING RESISTIVITY MEASUREMENTS TO COMPRESSIVE STRENGTH AND DENSITY

#### 8.4 RELATING COMPRESSIVE STRENGTH TO TIME AND RESISTIVITY MEASUREMENT

##### 8.4.1 Introduction

Early stripping and re-use of formwork or discontinuance of curing and protective measures are important from the standpoint of construction economics. The rate at which concrete develops strength is generally the limiting factor in such operations. Accordingly, the ability to monitor non-destructively, the strength development in a structure is often desirable.

There is a growing interest in the use of resistivity measurements as a way of investigating the characteristics of concrete. This is probably due to the fact that resistivity measurements taken at an early age may be used to predict the compressive strength of concretes at a later age. The early-age detection of faulty concrete, allowing prompt remedial actions to be taken, has long been a very desirable proposition. The major drawback in relating early age measurements to subsequent mechanical properties of the material is that the latter are very much dependent on curing conditions.

There are, however, situations where the concrete curing history is carefully controlled, such as pre-cast concrete works, thereby providing a suitable environment in which successful strength predictions could be made.

The data obtained from the laboratory tests on the different concrete mixes indicated that the resistivity technique for determining water/cement ratio and aggregate/cement ratio could be used directly to estimate the strength potential of a concrete mix.

To this end a series of experiments were undertaken to investigate the correlation between resistivity and compressive strengths of concrete mixes.

**Experimental Details:** Tests were carried out on concretes with different aggregate/cement ratios and different water/cement ratios.

(a) Variation in mix proportioning.

Mixes employed		
<u>1:1:1</u>	<u>1:1.5:3</u>	<u>1:2:4</u>
W/C = 0.5		

(b) Variation in water/cement ratio.

W/C employed		
<u>0.4</u>	<u>0.5</u>	<u>0.6</u>
Mix = 1:1.5:3		

Details of the mix proportions are shown in Table 7.1.

Aggregates employed for this series of tests consisted of 20 mm down Whinstone basalt and concrete sand, grading zone 2, see figure

6.5. The grading of the aggregate was the same for all the mixes. Dry aggregates and fresh Ordinary Portland Cement were employed throughout.

All materials were batched by weight.

Three 150mm concrete cubes were cast for each of the mixes and the cubes made and cured in accordance with the requirements of BS.1881 (see Appendix 1). Specimens were tested up to the age of 28 days.

#### 8.4.2 The effects of mix proportions and age upon changes in compressive strength ( $\Delta C.S.$ ) and resistivity ( $\Delta \rho$ )

During the course of the test programme a considerable amount of data was obtained relating resistivity and strength to age and mix proportions. Figures 8.5 and 8.6 show, respectively, the development of strength and resistivity with age, for the range of aggregate/cement and water/cement ratios tested.

It is the aim of this section to examine the effects of changes in mix proportions and age upon resistivity and compressive strength, and consequently upon the  $\Delta \rho / \Delta C.S.$  ratio.

##### 8.4.2.1 Effect of Mix Proportions

###### (a) Effects of variations in water/cement ratio

Comparing figures 8.5 and 8.6 (mix 1:1.5:3, w/c = 0.4, 0.5 and 0.6) it can be observed that, at a given age, the change in compressive strength and resistivity due to a change in water/cement ratio is greater at low than at high water/cement ratios. Change in strength due to a change in water/cement ratio is less at early than at late ages, this also appears to be true for resistivity. The reason for the similarity in response of both resistivity and strength to variations in water/cement ratio lies in their common dependence on the pore structure of the cement paste.

An increase in water/cement ratio causes a corresponding rise in the volume of capillary pores in the cement paste which both weaken the matrix and increase its ability to conduct electrical charges. Also, for any given mix, as hydration progresses the pores

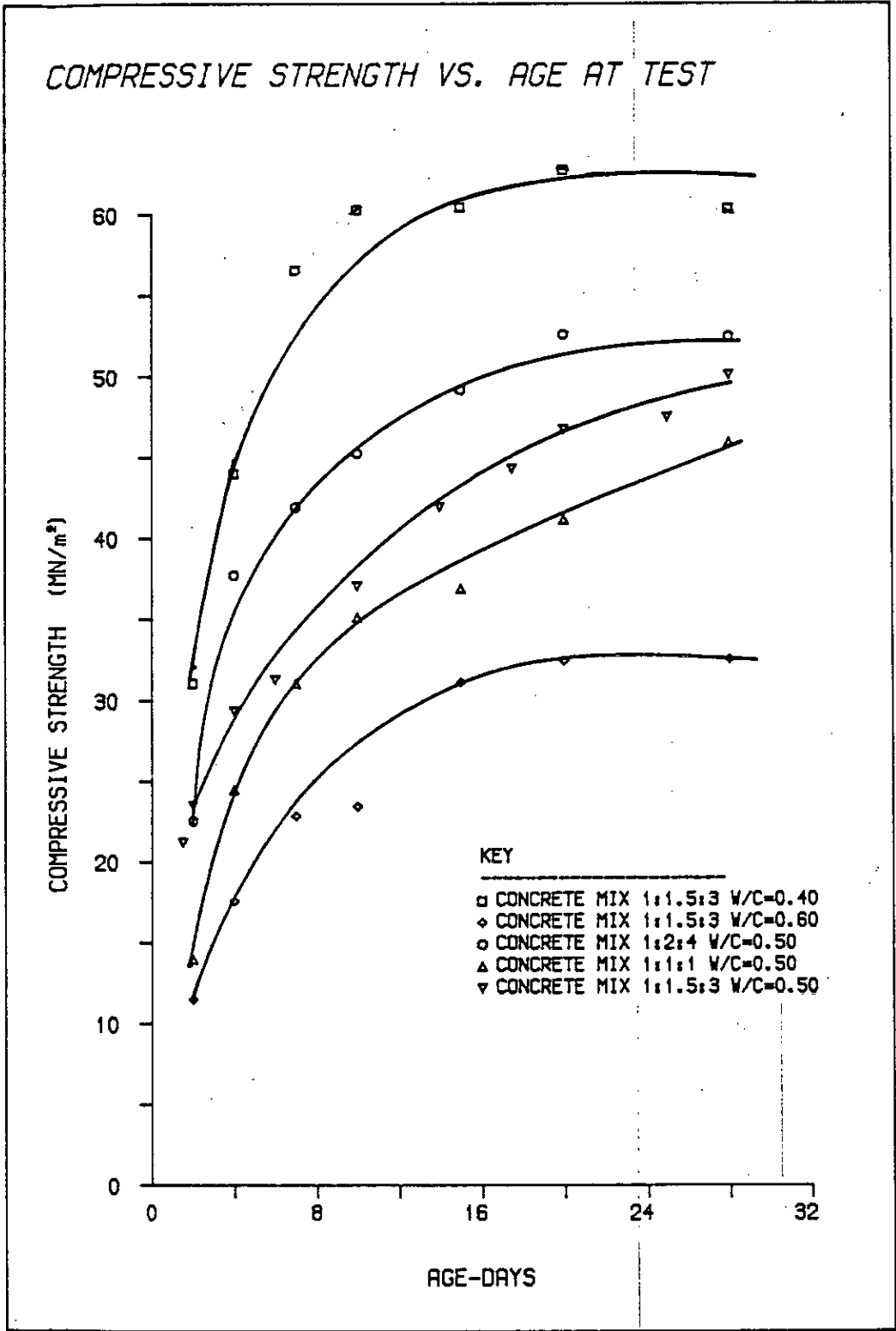


FIGURE 8.5



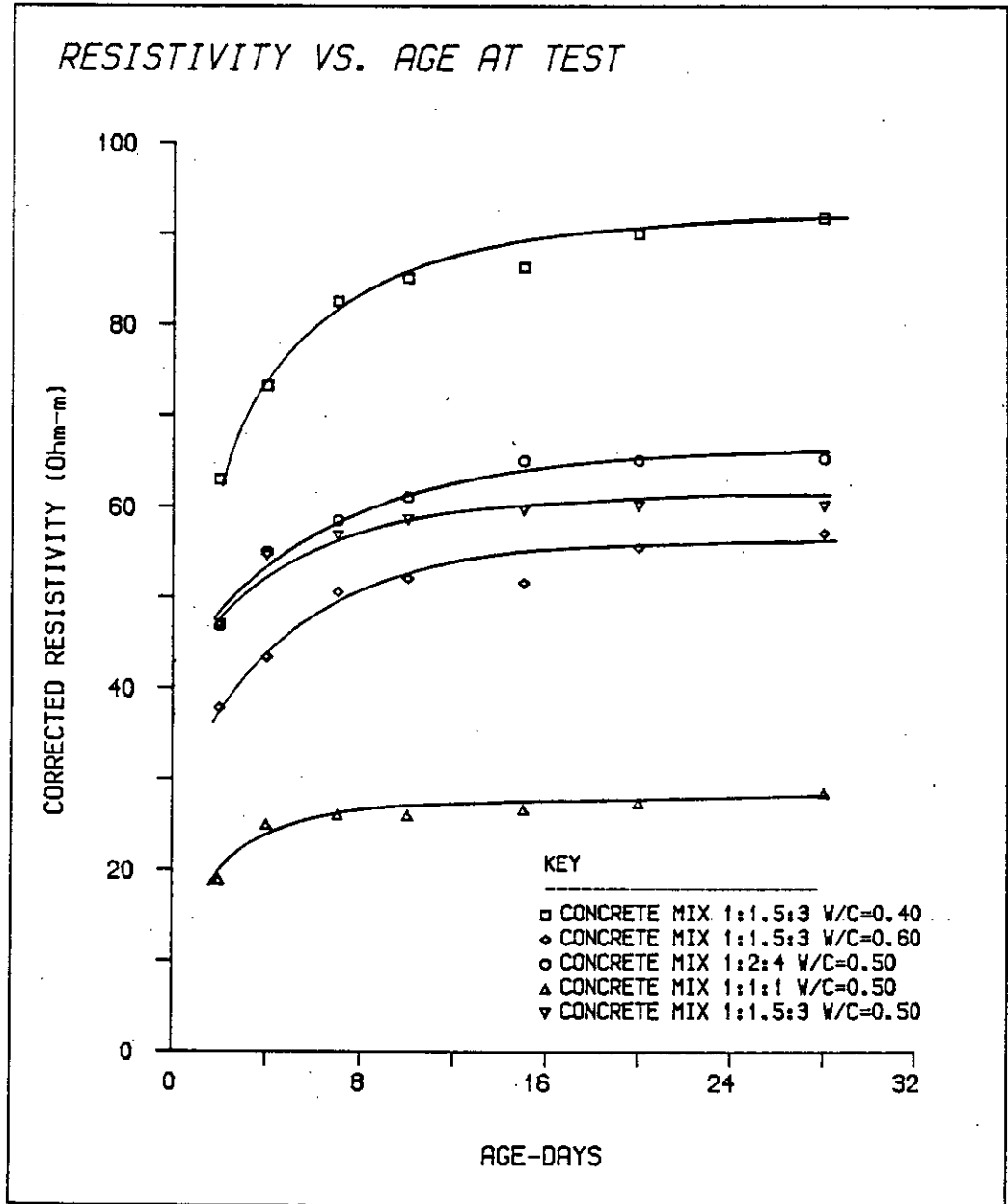


FIGURE 8.6

are gradually filled with hydration products thereby increasing both the strength and the resistivity of the material.

(b) Effects of variations in mix proportions for a constant water/cement ratio

An examination of the effects of varying the mix from 1:1:1 to 1:1.5:3 through to 1:2:4, for a constant water/cement ratio of 0.5, shows that an increase in aggregate/cement ratio has little effect on compressive strength when compared with a decrease in water/cement ratio. See mixes 1:1.5:3 and 1:1:1 (w/c = 0.50) in figures 8.5 and 8.6.

Although variations in aggregate content are secondary to water/cement ratio as strength affecting parameters, they are of equal, if not greater importance to the conductive characteristics of the material as the water/cement ratio.

The reasons for this, which lie in the effect of aggregates on the pore structure characteristics of the cement paste, have been described in detail in chapter five and verified experimentally in chapter seven (section 7.12.1.1).

(c) Effect of mix variations on the relationship between compressive strength and resistivity

Figures 8.5 and 8.6 show that there are strong similarities between the changes in resistivity and compressive strength due to a change in water/cement ratio. The difference in response of these two parameters to variations in aggregate content negates the application of a direct relationship between resistivity and strength when both variations in aggregate/cement and water/cement ratios are considered simultaneously.

#### 8.4.2.2 Effect of Age

It can be observed from figures 8.5 and 8.6 that the rate of change of both compressive strength and resistivity is greater in mixes of low water/cement ratio. It can also be seen that, after about twelve days, resistivity seems to reach a plateau whereas compressive strength continues to increase further, see figure 8.7.

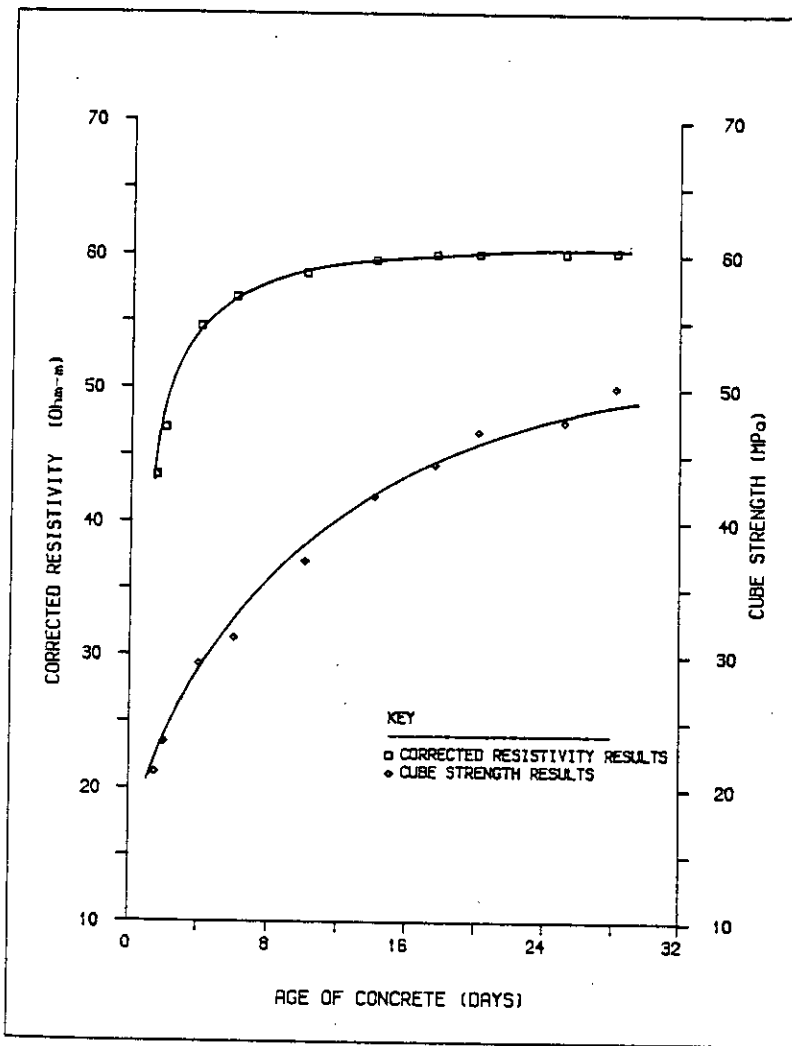


FIGURE 8.7 : Resistivity and cube strength as a function of the age of concrete.

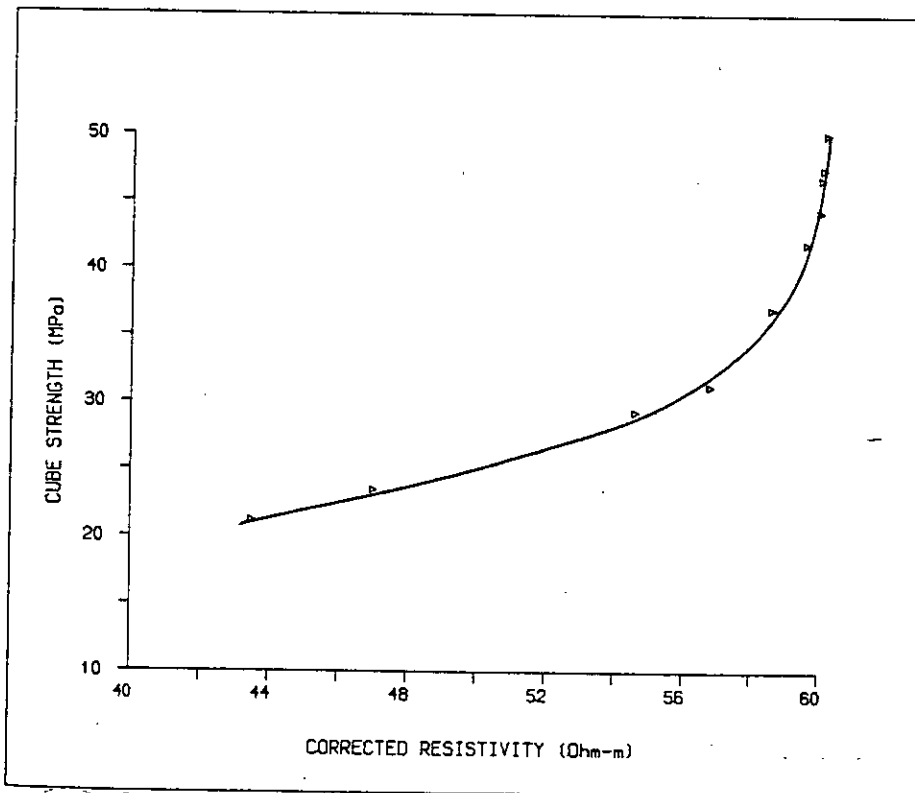


FIGURE 8.8 : Cube strength vs. resistivity.

This has been emphasised in Figure 8.8 where compressive strength has been plotted against resistivity. Here it can be seen that, at an early age, strength and resistivity are linearly related but, at later ages the rate of change in resistivity is much less than that of the compressive strength.

Bogue's graph, see figure 3.1, shows that, after the initial 7-day period, the rate of hardening of the Tricalcium Silicate phase starts to decrease while the Dicalcium Silicate begins to make a more significant contribution to the strength gaining process.

The levelling of the resistivity-time curve at the 28-day mark is to be attributed to the fact that, by this stage, all but the larger of the capillary pores have been obstructed by hydrates somewhere along their length. The presence of the large pores maintains resistivity levels constant while, the already segmented smaller pores, consolidate to form a denser monolithic structure thereby continuing to increase the compressive strength.

Predicting the compressive strength of an unidentified in situ concrete is therefore generally impossible, not only because of the relatively poor relationship between resistivity and strength in mature concrete but also because of the effect of the various mix constituents on this relationship.

#### 8.4.2.3 Relation between resistivity and compressive strength for a varying age

Figures 8.5 and 8.6 showed that strength and resistivity are each dependent on age, w/c ratio and a/c ratio and therefore, the relationship between compressive strength and resistivity must also depend on these three variables. In order to formulate a relationship between compressive strength and resistivity, which is based on age, the mix proportions must be constants.

Regression techniques were used to establish relationships between resistivity and compressive strength over the one to twenty-eight day period for various mixes.

C.S. =  $f(\rho, \text{age})$  was expressed by equations of the form:-

$$\text{C.S.} = \beta_0 + \beta_1 \rho + \beta_2 \text{ Days}$$

and the model tested with the experimental data shown in Table 8.4.

Prior to obtaining relationships for mixes of fixed proportions, it was decided to verify the postulated concepts that mix proportions have to be maintained constant for an accurate model to exist.

Test results of mixes with different aggregate/cement and water/cement ratios, Table 8.4 part (a) and (b), were regressed and a  $r^2$  value of 0.711 was achieved.

$$\text{C.S.} = 5.97 + 0.412\rho + 0.714 \text{ Days} \quad \dots\dots(8.4)$$

The accuracy of the prediction could be improved by only using the test results where the water/cement ratio was the same but the aggregate/cement ratio varied. See Table 8.4 part (b). This increased the  $r^2$  to 0.856.

$$\text{C.S.} = 13.1 + 0.273\rho + 0.877 \text{ Days} \quad \dots\dots(8.5)$$

Larger increases in  $r^2$  were achieved by employing only those test results where the aggregate/cement ratio was constant but the water/cement ratio varied. See Table 8.4 part (a). This yielded a correlation coefficient of 0.92.

$$\text{C.S.} = -18.2 + 0.829\rho + 0.325 \text{ Days} \quad \dots\dots(8.6)$$

Finally, test results were used where both the aggregate/cement ratio and the water/cement ratio were kept constant. As before compressive strength and resistivity results at varying ages were used and the  $r^2$  values, for the various aggregate/cement and water/cement ratios used, were found never to drop below 0.97. The following are examples of the regressed equations for two different mixes relating compressive strength to resistivity and time subsequent to batching.

Mix 1:1.5:3 W/C = 0.60

$$\text{C.S.} = -17.7 + 0.767\rho + 0.268 \text{ Days} \quad \dots\dots(8.7)$$

Mix 1:1.5:3 W/C = 0.40

$$\text{C.S.} = -64.2 + 1.52\rho - 0.514 \text{ Days} \quad \dots\dots(8.8)$$

Mix Proportions (By Volume) (1 part of cement employed throughout)			Time (Days)	Corrected Resistivity (Ohm-m)	Average Strength (MP <sub>a</sub> )
Coarse Aggregate	Sand	Water			
CONCRETES					
3	1.5	0.4	2	63	31.04
			4	73.27	44.00
			7	82.5	56.56
			10	85.1	60.30
			15	86.30	60.44
			20	90.00	62.74
			28	91.70	60.33
3	1.5	0.5	2	54	23.48
			4	58.5	29.33
			7	60	31.26
			10	61	37.04
			15	63	41.93
			20	63.5	46.74
			28	64	50.07
3	1.5	0.6	2	37.8	11.48
			4	43.43	17.55
			7	50.60	22.81
			10	52.10	23.41
			15	51.60	31.11
			20	55.5	32.44
			28	57.1	32.52

(a) water/cement ratio

Mix Proportions (By Volume) (1 part of cement employed throughout)			Time (Days)	Corrected Resistivity (Ohm-m)	Average Strength (MP <sub>a</sub> )
Coarse Aggregate	Sand	Water			
CONCRETES					
1	1	0.5	2	18.9	13.93
			4	24.98	24.45
			7	26.05	31.05
			10	26	35.11
			15	26.65	36.89
			20	27.4	41.15
			28	28.5	45.93
3	1.5	0.5	2	47	23.48
			4	54.6	29.33
			7	56.8	31.26
			10	58.6	37.04
			15	59.6	41.93
			20	60.06	46.74
			28	60.10	50.07
4	2	0.5	2	46.8	22.52
			4	55.0	37.70
			7	58.5	41.92
			10	61.03	45.26
			15	65.03	49.18
			20	65.10	52.59
			28	65.30	52.44

(b) mix proportions

TABLE 8.4 Increases in Resistivity and Compressive Strength with age as a function of (a) water/cement ratio and (b) mix proportions

The experimental results show that, for an accurate relationship to be established between C.S., resistivity and age, the mix proportions must be known.

It was also concluded from the investigation that in order to monitor strength from resistivity tests it would first be necessary to determine the resistivity-strength relationship for the particular mix under consideration. In view of this, the resistivity testing technique may have application to large concrete works as a quality control procedure where the effort involved in predetermining the resistivity-strength relationship would be considered viable.

The test would be of use as a non-destructive technique for monitoring the strength gained at various stages of the hardening process.

#### 8.4.2.4 Relation between resistivity at early ages and compressive strength at 28 days

In many circumstances it is very desirable to obtain early information enabling the potential strength of concrete in a structure to be predicted. This allows remedial action to be taken more easily than it could if defective concrete was detected only after a prolonged period of time.

If concrete is made with one type of cement and aggregate, it is normally found that concrete having the lowest strength at early ages will also have the lowest strength at late ages. For one type of cement and aggregate, it is found that fully compacted concrete having the lowest resistivity at early ages will have the lowest resistivity at late ages. The previous investigations, equations 84 to 88, whilst demonstrating reasonably good correlation between resistivity, strength and age at early ages, ie. less than 28 days, have also shown that resistivity is an insensitive index of strength at later ages. i.e. when the rate of change of resistivity is extremely low compared to that of compressive strength, see figure 8.7.

There has been a limited amount of research<sup>120,40</sup> on the applicability of early age resistivity tests for estimating the strength of concrete. It is therefore of interest to examine the relationship between resistivity at an early age and compressive strength at 28 days.

**Choice of Parameters:** Because of the effect that permeating water has on resistivity readings during the water curing stage, only measurements taken prior to this stage can be successfully employed as strength prediction parameters.

The significance of  $(\rho_{1.0} - \rho_{0.065})/\rho_{0.065}$  as a strength predicting parameter is:-

$(\rho_{1.0} - \rho_{0.065})$  is both an indicator of water/cement ratio and of aggregate content.

$\rho_{0.065}$  is an indicator of aggregate content.

Compressive strength =  $f(W/C, A/C)$

$(\rho_{1.0} - \rho_{0.065}) = f(\text{Water/Cement, Aggregate/Cement})$

$\rho_{0.065} = f(\text{Aggregate/Cement})$

Figure 8.9 shows compressive strength at 28 days plotted against  $(\rho_{1.0} - \rho_{0.065})/\rho_{0.065}$  for mixes with different aggregate/cement and water/cement ratios. Using regression techniques the linear relationship between resistivity measurements and compressive strengths was established. The relationship has a  $r^2$  value of 0.878.

$$\text{C.S.} = 8.97 + 9.72 (\rho_{1.0} - \rho_{0.065})/\rho_{0.065} \quad \dots\dots(8.9)$$

### 8.5 RELATIONSHIP BETWEEN RESISTIVITY MEASUREMENTS AND CONCRETE DENSITY

Since the electrical resistivity technique has been shown to be unable to distinguish between air voids and non-conductive solids (see section 7.9), mixes of low workability, and therefore containing entrapped air, have been discarded in the calculation of empirical relationships.



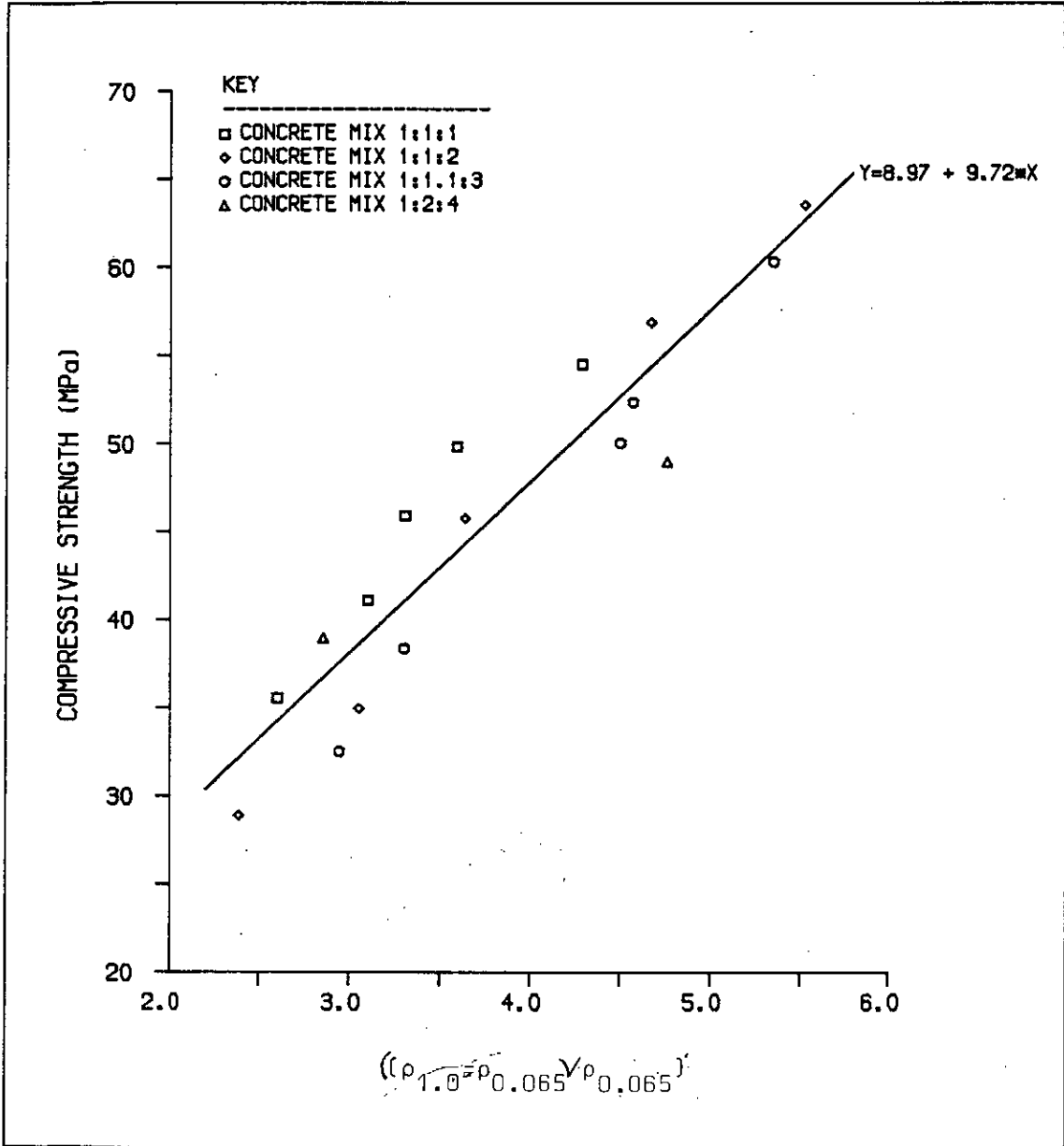


FIGURE 8.9 : 28-day cube strength vs. day-one resistivity parameters.

Workability can be defined as the amount of work required to compact fully a given concrete mix. As for practical purposes this is impossible to measure directly, the following approach to the problem was adopted:-

- (a) Given the mix proportions, and the specific densities of the individual constituents, a theoretical density was calculated.
  - (b) The actual density of the concrete at demoulding was measured.
- (Note: This does not take into account water loss during curing.)

The degree of compaction, hereinafter termed the compaction factor (C.F.), was then defined as the ratio of the actual density to that of the theoretical, fully compacted material.

The actual density of each mix was obtained from measured mass and dimensions of the cubes after the first day hardening and also after 27 further days of curing under water.

Given the mix proportions, and the specific densities of the individual constituents, the theoretical, fully compacted mix density was calculated by means of the following equations.

$$\frac{W}{1000} + \frac{C}{1000D_c} + \frac{A_1}{1000D_1} + \frac{A_2}{1000D_2} = V_{\text{mix}}$$

and  $D_{\text{mix}} = \frac{(W + C + A_1 + A_2)}{V_{\text{mix}}}$

where

W = mass of water (kgs)

C = mass of cement (kgs)

A<sub>1</sub> = mass of fine aggregate (kgs)

A<sub>2</sub> = mass of coarse aggregate (kgs)

V = volume of fully compacted mix (m<sup>3</sup>)

D<sub>c</sub> = Density of cement (Experimentally = 2990 kgs/m<sup>3</sup>)

D<sub>1</sub> = Density of fine aggregate (Experimentally = 2608 kgs/m<sup>3</sup>)

D<sub>2</sub> = Density of coarse aggregate (Experimentally = 2690 kgs/m<sup>3</sup>)

$$\text{Compacting Factor (C.F.)} = \frac{\text{Actual Density (After 1.0-day)}}{\text{Theoretical Density}}$$

**Best Fit Equations:** (The point defined by the mix 1:2:4 W/C = 0.4 was ignored as this mix could not be compacted fully and therefore yielded an excessively high resistivity value.)

Time = 0.065 day

$$\text{Density} = 2696 - 861 \phi \quad r^2 = 0.88 \quad \dots(8.10)$$

Time = 28.0 days

$$\text{Density} = 2724 - 836 \phi \quad r^2 = 0.86 \quad \dots(8.11)$$

With reference to figure 8.10:-

- (i) Apart from the mix 1:2:4, W/C = 0.4 all the mixes are fully compacted.
- (ii) A decrease in fractional volume of cement paste causes an increase in the density of concrete.
- (iii) For any given mix, a rise in water/cement ratio directly increases the fractional volume of cement paste.
- (iv) An increase in water/cement ratio results in a more porous cement paste and a correspondingly less dense concrete. This effect is more noticeable in concretes with a low aggregate/cement ratio.

**Density, Resistivity Relationship:** In a fully compacted mix, a loss in density can be attributed to either an increase in water/cement ratio or an increase in the proportion of aggregate in the mix (the density of the aggregate being normally lower than that of cement). Whilst an increase in aggregate content might only have a slight effect on the quality of the mix, an increase in water/cement ratio would cause a direct drop in the strength gaining characteristics and durability of the material.

Although not as sensitive to variations in water/cement ratio as strength testing, it is sometimes the case in Industry for density to be employed as a quality control parameter when the mix has not gained sufficient strength for it to be tested.

Electrical resistivity, like density, decreases with a drop in aggregate content or an increase in water/cement ratio, although, for a freshly batched concrete, the conductivity of the cement paste phase is constant for all commonly employed water/cement ratios (see figure 7.1).

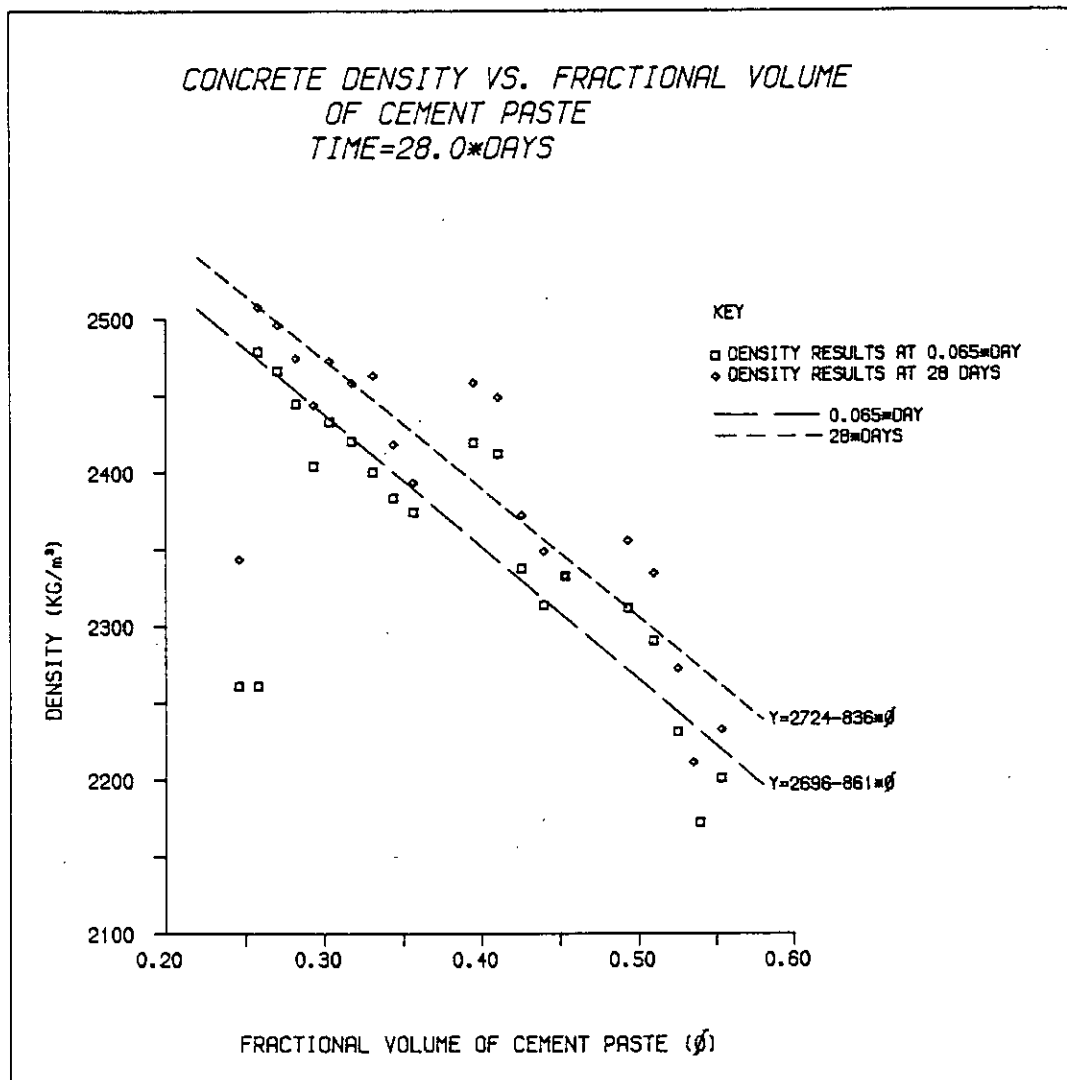


FIGURE 8.10

The technique is, however, very sensitive to the presence of macrovoids in the structure of the material (section 7.9) the most common cause of a drop in density.

Whether caused by a drop in compacting effort or a decrease in workability of the mix, macrovoids can have a serious deleterious effect on the quality of concrete. Strength and durability are both related to the density of the material.

A series of tests were therefore undertaken to assess the possibility of employing resistivity measurements to monitor density variations of fresh concrete. It was decided to measure the one-day rather than the twenty-eight day density because of the extra variation caused by water permeating the material during the water curing stage.

Figure 8.11 shows how resistivity of the concrete in a fresh state (0.065-day) relates to the one-day material density.

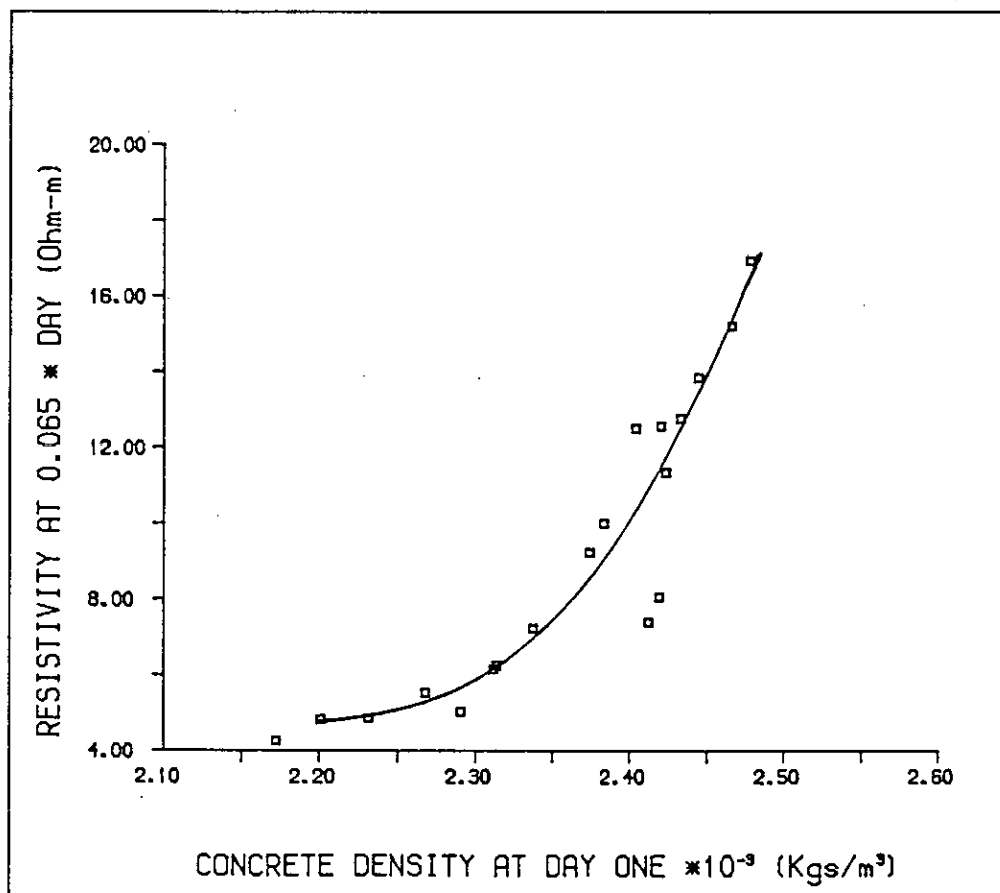


Figure 8.11 Resistivity of fresh concrete vs. its density at day-one

The quality of the relationship between density and resistivity was assessed using regression techniques.

Variables employed in the regression:

D = Density  $\times 10^3$  at one-day

$\rho$  = Resistivity at 0.065-day

A general form of the relationship describing the trend shown in figure 8.11 was found to be:

$$D = \beta_0 + \beta_1 \rho + \beta_2 \sqrt{\rho}$$

Regressing the data, the following equation representing the line shown in figure 8.11, was obtained:-

$$D = 1.33 - 0.0732\rho + 0.573\sqrt{\rho} \quad \dots(8.12)$$

$$r^2 = 0.898$$

The regular weighing of samples from a concrete mix is a tedious and often approximate manual task at many large concreting works. Electrical resistivity testing offers a secondary means of density control whilst its suitability to automation might be of use in increasing sample numbers and reducing the tedium of regular monitoring.

## 8.6 DISCUSSION

The experiments undertaken to investigate the relationship between the electrical resistivity and certain physical characteristics have been detailed in this chapter.

The bulk of the investigation was concerned with the study of resistivity-time graphs for concrete mixes of varying proportions. The experimental results confirm that variations in resistivity are related to the physical characteristics of the concrete through the changes in pores structure during the cement hydration process.

As was discussed in chapter 2, concrete can be more effectively and economically controlled by the regular testing of samples whilst still in a fresh state, than by cube testing. In the event of non-compliance with the specified mix, the concrete can be rejected

with minimum cost and delay. A series of tests were therefore carried out to investigate the possibility of employing electrical resistivity measurements to monitor the composition of fresh concrete mixes. The test results, in conjunction with mechanical properties such as strength and density, were analysed using regression techniques to determine the potential for resistivity measurements to be used as forecasting parameters for concrete properties.

In the investigation it is shown that, in order to establish the percentage aggregate content accurately, resistivity measurements obtained at an early age (less than an hour) would have to be employed. The reason for this being that the measurement of aggregate content is made on the basis of relative changes in effective cross-sectional area available for conduction. As hydration is allowed to progress, its by-products begin to interfere with the conduction process.

The opposite concept was however true in regressing water/cement ratio as a function of resistivity. Water/cement ratio was not found to affect the resistivity of cement pastes at an early age, its influence, however increasing with hydration time. The earliest accurate regression equations for water/cement ratios were obtained from day-one resistivity results.

The regression equation obtained for percentage aggregate content by volume and water/cement ratio are reported in section 8.2.

From the investigation it was concluded that resistivity offers a useful means of indirectly monitoring the aggregate content and the water/cement ratio, the two mix parameters which are generally regarded as most important in determining the mechanical characteristics of the hardened material. Given that relationships between resistivity and these mix parameters could be established, it was therefore thought possible to predict the strength of concrete using resistivity measurements.

From the experiments it was shown that both compressive strength and electrical resistivity of concrete depend upon the

three variables of age, water/cement ratio and aggregate/cement ratio. Regression analysis of the data showed the most accurate regression equations to be those relating to strength as a function of resistivity and age for a concrete of known mix proportions.

Strength and resistivity have a common dependence on the volume of hydrated gel in the cement paste, and thus they depend on the degree of hydration of the cement which is a time-dependent process.

The two resistivity parameters which had been employed to monitor aggregate content and water/cement ratio, were combined and related to 28-day cube strength as illustrated in figure 8.9. The relationship between early age resistivity measurements and 28-day compressive strength results showed an excellent correlation (see section 8.4.2.4) and it can be concluded that, in a general sense, a relationship between certain resistivity parameters and compressive strength exists and may be determined for any given set of conditions. However, any effort to apply such a relationship to concrete of unknown origin, for which the relationship has not been specifically determined, is likely to lead to large errors in strength estimation.

Another important physical characteristic of hardened concrete is its density. Whether caused by a drop in compacting effort or by a reduction in the workability of the mix, the presence of macrovoids can have a deleterious effect on the strength and durability of the mix.

A relationship regressed between density and resistivity of fresh concrete showed the test's ability to monitor changes in density as caused by variations in mix proportions. The sensitivity of the technique to increases in the volume of entrapped air voids (section 7.9) could be of practical use as a monitor for compaction and workability.

Figure 8.12 suggest a resistivity testing process which might be applied in practical situations. The flowchart details the information obtainable at various stages in curing using resistivity testing provided the initial effort has taken place to predetermine the various relationships.



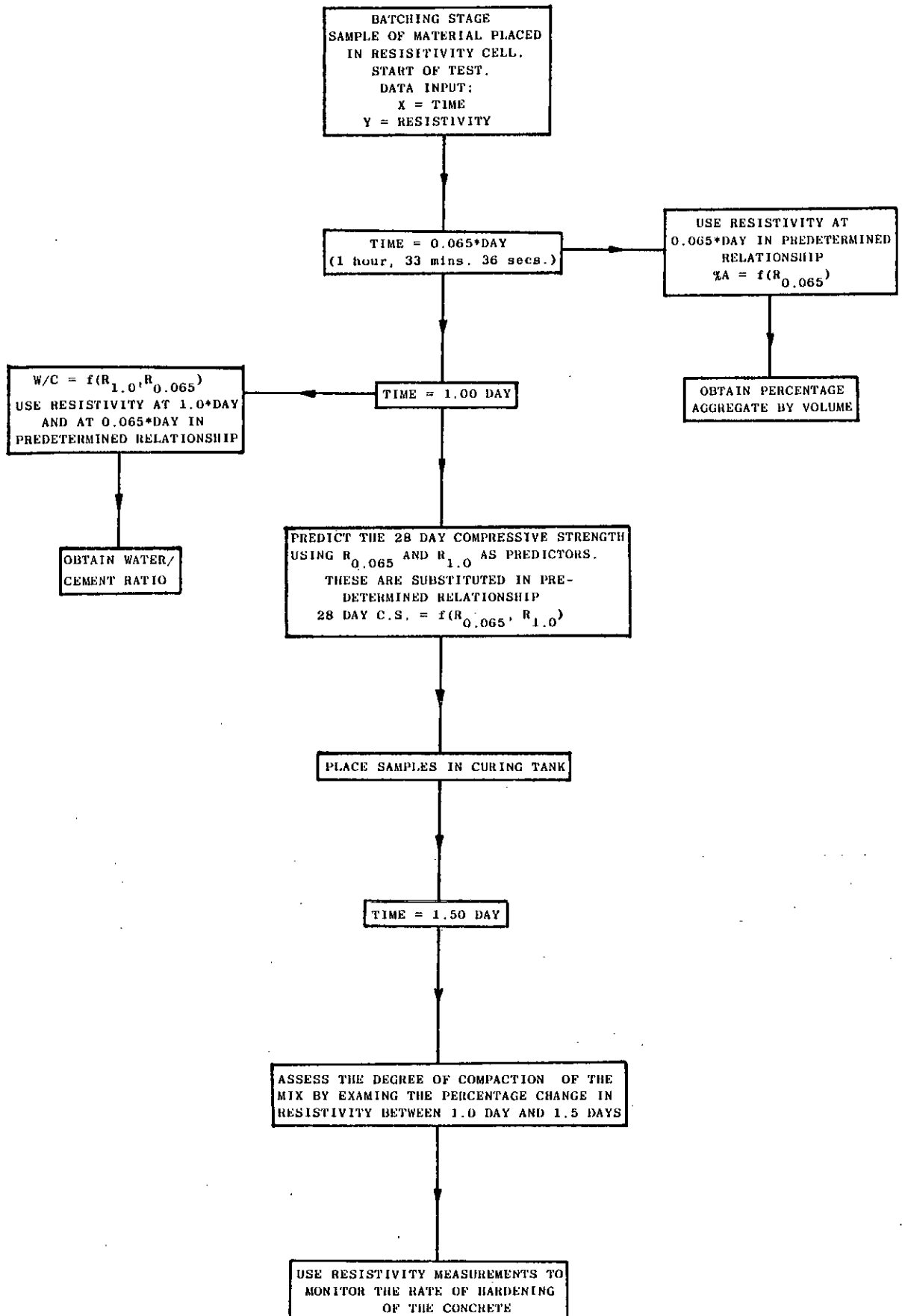


Figure 8.12 Operational flowchart of electrical resistivity testing process

## 8.7 CONCLUSIONS

Tests carried out to investigate the possibility of employing the electrical resistivity technique to monitor and predict mix proportions, compressive strength and density of concrete mixes have led to the following conclusions:-

1. Resistivity-time graphs were found to be unique to each particular concrete mix considered.
2. As a result of the dependence of resistivity on the effective cross-sectional area available for conduction and on the hydration process, the technique offers a useful means of indirectly monitoring the aggregate content and the water/cement ratio.
3. Resistivity readings obtained from fully compacted mixes during the initial setting stage were found to be independent of water/cement ratio and, for the cement and curing conditions employed, solely related to the aggregate content by volume.
4. The relationship between percentage aggregate content by volume and resistivity has been shown to be of the form

$$\text{Agg} = \beta_0 + \beta_1/\rho$$

where  $\rho$  = resistivity at 0.065 of a day.

5. Regression of the experimental data resulted in best fitting equations, of the form shown above, expressing aggregate content by volume as a function of resistivity. Correlation coefficients of 0.955 for sand/cement mixes and 0.93 for concretes were obtained.
6. As cement hydration progresses past the setting stage, resistivity measurements, although still related to the aggregate content, are increasingly affected by the initial water/cement ratio of the mix.
7. Employing  $\rho_{0.065}$  (monitor for aggregate content) and  $\rho_{1.0} - \rho_{0.065}$  (monitor for the rate of hardening) it has been possible to construct regression curves for water/cement ratio as a function of these two parameters.
8. On the basis of the technique's ability to monitor aggregate content and water/cement ratio which, for given materials and curing characteristics are the main strength affecting parameters, it has been possible to relate resistivity measurements to compressive strength.

9. Resistivity and compressive strength of a concrete mix of given material characteristics and curing history have been shown to be related to age, water/cement ratio and aggregate/cement ratio.
10. The changes in both resistivity and compressive strength are related to the nature and development of the capillary pore structure of the cement paste matrix.
11. A relationship can be established between the compressive strength of a concrete of known proportions, its resistivity and age. The relationship could be employed as an indirect, non-destructive means of monitoring the gain of strength of concrete during its early stages of hardening.
12. Resistivity measurements taken after 28 days of curing are a poor monitor of compressive strength because, while the latter continues to increase, electrical resistivity has, by that stage, levelled off to a constant value.
13. On the basis of a predetermined relationship, resistivity measurements obtained during the first day of curing can be employed to forecast the compressive strength of a mix at 28 days.
14. Resistivity may sometimes be used to estimate compressive strength provided considerable care has been exercised to establish a resistivity-strength relationship valid for the concrete to be tested.
15. The electrical resistivity techniques have been shown to be an accurate monitor of concrete density for mixes of varying proportions.
16. The sensitivity of the test to the presence of voids in a mix could allow for its use as a means of monitoring compaction and workability in a site environment.
17. The degree of accuracy attainable in the development of relationships between mix proportions and physical characteristics warrants further developments of the electrical resistivity technique as a general test method.

**CHAPTER NINE**

**THESIS OVERVIEW AND RECCOMENDATIONS**  
**FOR FURTHER RESEARCH**

## THESIS OVERVIEW AND RECOMMENDATIONS FOR FURTHER RESEARCH

This thesis is concerned with the possibility of using an electrical resistivity measurement technique in the testing of concrete mixes.

The study has been prompted by:

- (a) The need for a closer understanding of the conduction of electricity through a heterogeneous, time dependent material like concrete.
- (b) The continuous need for more accurate methods to assess the quality and forecast the behaviour of freshly mixed concrete.
- (c) The need to explore novel and alternative solutions to the problem, as developments in fields such as material science and instrumentation make techniques, such as electrical resistivity, more readily applicable to engineering research.

The variables which determine the electrical resistivity of a concrete mix have been investigated. Resulting from this investigation, certain precautions must be taken to ensure correct interpretation of results. Conclusions have been given at the end of each experimental section. With reference to the context of non-destructive testing the following main conclusions can be drawn from this study:-

- (a) An alternative method of testing concrete non-destructively has been investigated. The system variables have been analysed in detail and measurement procedures for concrete testing using this technique proposed. It was confirmed that the characteristic  $\rho$ - $t$  curve for a concrete mix gives qualitative indications as to the properties of the material.
- (b) An experimental investigation of the compositional variability of Ordinary Portland Cement has indicated substantial variations and, for research projects of this nature, a more uniform material would be desirable.
- (c) An investigation of the nature of the physical characteristics of concrete has confirmed these to be, in the main, dependent on the hydrated structure of the cement paste matrix. One of the most important properties of the cement paste phase, with regard to its mechanical characteristics, is its solid to pore volume ratio. As the water/cement ratio decreases, and as the hydration progresses, this ratio increases and so do the mechanical qualities of the material.

- (d) As a result of the poor conductive characteristics of the solids in concrete, it was confirmed that electrical conduction takes place, mainly, through the ion rich solution present in the pore structure of the cement paste matrix. As the solid to pore volume ratio varies, so does the effective cross-sectional area available for electrical conduction.
- (e) It was confirmed that the common dependence on the pore system provides the basis for the relationships between resistivity and concrete characteristics.
- (f) The electrochemical structure of the cement paste has been studied and the various factors affecting conductivity examined. A chemical analysis of a saturated solution has confirmed Calcium and Hydroxide to be the ions with, by far, the highest concentration levels.
- (g) A solution of Ordinary Portland Cement and water was found to achieve electrolytic saturation at a percentage cement content corresponding to a water/cement ratio of 1.7. Further addition of cement was found not to increase the conductivity of the solution. The resistivity of freshly mixed cement pastes can therefore be generally regarded as independent of water/cement ratio.
- (h) By testing an electrolytically saturated water-cement solution at different temperatures, the temperature coefficient of resistivity for cement paste employed,  $\alpha$ , was established at 0.0216.

$$\rho_0 = \rho_T(1 + \alpha\Delta T)$$

where

$\rho_T$  = measured resistivity,

$\rho_0$  = corrected resistivity, and

T = variation from temperature standard.

- (i) The need to test more than one sample of each mix for statistical significance, the number of mixes to be tested and the need for measurements to be taken at regular intervals during the hydration period, were all initial considerations which resulted in the development of an automated testing device. The apparatus, designed in conjunction with the Department of Electrical Engineering at the University of Edinburgh, is capable of monitoring up to 15 separate experiments and has allowed for the study of the resistivity-time relationship of over 200 specimens.

- (j) It has been *confirmed*, experimentally, that the electrical resistivity technique can be successfully employed to study the development of set in water-cement pastes and final setting times similar to those obtained using Vicat's apparatus were achieved.
- (k) The technique was not found to be directly applicable to the study of set in concrete mixes. The presence of aggregate in the paste causing a difference in the electrical response to the development of set.
- (l) A previously reported<sup>134</sup> anomaly in the  $\rho$ -t graph has been quantitatively analysed and shown to be caused by the ingress of water in the specimens at the water-curing stage. The magnitude of the discontinuity can be related to the compaction of the mix and the water/cement ratio.
- (m) The theoretical aspects of electrical conduction through concrete were analysed and verified by an extensive experimental programme.

Hydration time, water/cement ratio and aggregate/cement ratio were the three main variables whose effect on both electrical resistivity and physical characteristics of concrete mixes was investigated in the course of this study.

- (n) The electrical resistivity of concrete was *confirmed* to be best related to  $\phi$ , the fractional volume of cement paste, by the empirical relation

$$F = a \cdot \phi^{-m}$$

where  $FF = \text{Formation resistivity factor} = (\rho_{\text{mix}} / \rho_{\text{paste}})$

$a, m = \text{empirical parameters.}$

- (o) Although, by definition, the formation resistivity factor of an electrically inert porous medium (compacted aggregate) is independent of the electrical conductivity of the solution (cement paste), this was not found to be true in the case of hardening concrete mixes.

$(\rho_{\text{mix}} / \rho_{\text{paste}})$  was found to depend not only on  $\phi$  but also on the water/cement ratio and the stage of hydration.

- (p) An important conclusion drawn from the theory and experiments was that the presence of aggregate in the cement paste matrix, by increasing the tortuosity and constriction of the conductive paths, influences the ease with which they become obstructed during hydration. The net result being an increase in the rate of hydration, as monitored by resistivity measurements, with increased aggregate content.

- (q) Regression techniques were employed to study trends between resistivity parameters and physical variables such as aggregate content, water/cement ratio, strength and density. Results show that it is possible to detect compositional changes, and corresponding mechanical characteristics variations, provided the relationships employed have been specifically determined for the mixes and materials under consideration.
- (r) Although the actual relationships obtained are particular to the materials and procedures employed in the course of the experimental programme, the degree of correlation achieved in obtaining them is very encouraging.
- (s) The test shows promise as a means of studying the hydration process, as a check for mix proportions, as a monitor for the gain in strength and density, and as an early age predictor of compressive strength.
- (t) Because of its dependence on the porous structure of the cement paste, resistivity offers the advantage, over other non-destructive testing techniques, of providing a more direct means of monitoring the quality of concrete. Also, unlike other tests, it is not restricted to the monitoring of any one single characteristic. The fact that it is not based on a phenomenological approach but rather on a fundamental knowledge of the physical/mechanical properties of the material, make electrical resistivity a more flexible technique with a wider range of possible applications.
- (u) Although still at an early stage, research shows the potential for the use of the method in a diversity of situations, from the study of the basic properties of cements to the quality control of major concreting works.

#### RECOMMENDATIONS FOR FUTURE RESEARCH

The research detailed in this Thesis provides the background on which further work may be carried out on the effect of variables, other than mix proportions and age, upon the relationships between physical and electrical characteristics of concrete mixes. The effects of employing different materials, curing conditions and the use of admixtures must be examined to both achieve a better understanding of the conduction mechanism and to widen the scope of its applications.



Aside from strength, other important concrete properties which are affected by the volume and continuity of the pore network are permeability and durability. Further research is therefore required in the investigation of relationships between these two parameters and electrical resistivity.

At a more fundamental level, further analysis is needed on gel structure development and on the electrolytic properties of the aqueous phase in the hydrating cement paste.

The ultimate aim must be to relate the chemical composition of the unhydrated cement and the physical properties of the aggregates employed to the mechanical/electrical characteristics of the hydrating mix at any one time and for any given environmental set of conditions.

Another area which warrants further study is that of the continuous development of the apparatus. Although this project carried the automisation of the equipment a considerable way forward, further developments are envisaged. The equipment will be reduced in size and complexity of operation by building simpler, more compact units.

Although much of the preliminary research, e.g. laboratory investigation, system methodology has been extensively covered in this Thesis, the need to continue analytical and experimental investigations on the electrical resistivity characteristics of concrete cannot be overemphasized. Ultimately, the study should be carried out to the site environment.

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**APPENDIX ONE**

**RELEVANT BRITISH STANDARDS**

## RELEVANT BRITISH STANDARDS

### (A) CEMENT

- B.S. 12: 1978 Ordinary and rapid-hardening  
Portland cement.
- 4550: : Methods of testing cement.
- 4550: Part 0: 1978 General introduction.
- 4550: Part 1: 1978 Sampling.
- 4550: Part 2: 1970 Chemical tests.
- 4550: Part 3: 1978 Physical tests.

### (B) AGGREGATE

- B.S. 410: 1976 Test sieves.
- 812: Part 1: 1975 Method for sampling and testing  
of mineral aggregates, sands  
and fillers.
- 812: Part 1: 1975 Sampling, shape, size and  
classification.
- 812: Part 2: 1975 Physical properties.
- 812: Part 3: 1975 Mechanical properties.
- 812: Part 4: 1975 Chemical properties.
- 882,1201: Part 2: 1973 Aggregates from natural sources  
for concrete (including granolithic).

### (C) CONCRETE

- B.S. 1881: Methods of testing concrete.
- 1881: Part 1: 1970 Methods of sampling fresh  
concrete.
- 1881: Part 2: 1970 Methods of testing fresh  
concrete.
- 1881: Part 3: 1970 Methods of making and curing  
test specimens.



- 1881: Part 4: 1970 Methods of testing concrete for strength.
- 1881: Part 5: 1970 Methods of testing hardened concrete for other than strength.
- 1881: Part 6: 1971 Analysis of hardened concrete.
- 3148: 1980 Tests for water for making concrete (including notes on the suitability of the water).
- 4408: Recommendations for non-destructive methods of test for concrete.
- 4408: Part 1: 1969 Electromagnetic cover measuring devices.
- 4408: Part 2: 1969 Strain gauges for concrete investigations.
- 4408: Part 3: 1970 Gamma radiography of concrete.
- 4408: Part 4: 1971 Surface hardness methods.
- 4408: Part 5: 1971 Measurement of the velocity of ultrasonic pulses in concrete.
- 5328: 1976 Methods for specifying concrete.

**APPENDIX TWO**

**EXPERIMENTAL DESIGN**

**Figure A2.1: Diagram of Mould Elements**

Detailed drawing showing the four piece moulding assembly comprising the base plate, two wall pieces and the binding unit. The elements were fabricated entirely from P.V.C. (see section 6.4.2).

**Figure A2.2: Plan view of curing tank layout and heating system**

Plan view of the curing tank with two trays in position showing the thermostatically controlled water heating system. A detailed description of the system's operation is given in section 6.4.3.

**Figure A2.3: Schematic Diagram of Hoist and Tray Arrangement**

Side elevation of tray hoisting showing the computer controlled electric motor and the cams/microswitch assembly which governs the end positioning of the tray (see section 6.4.4).

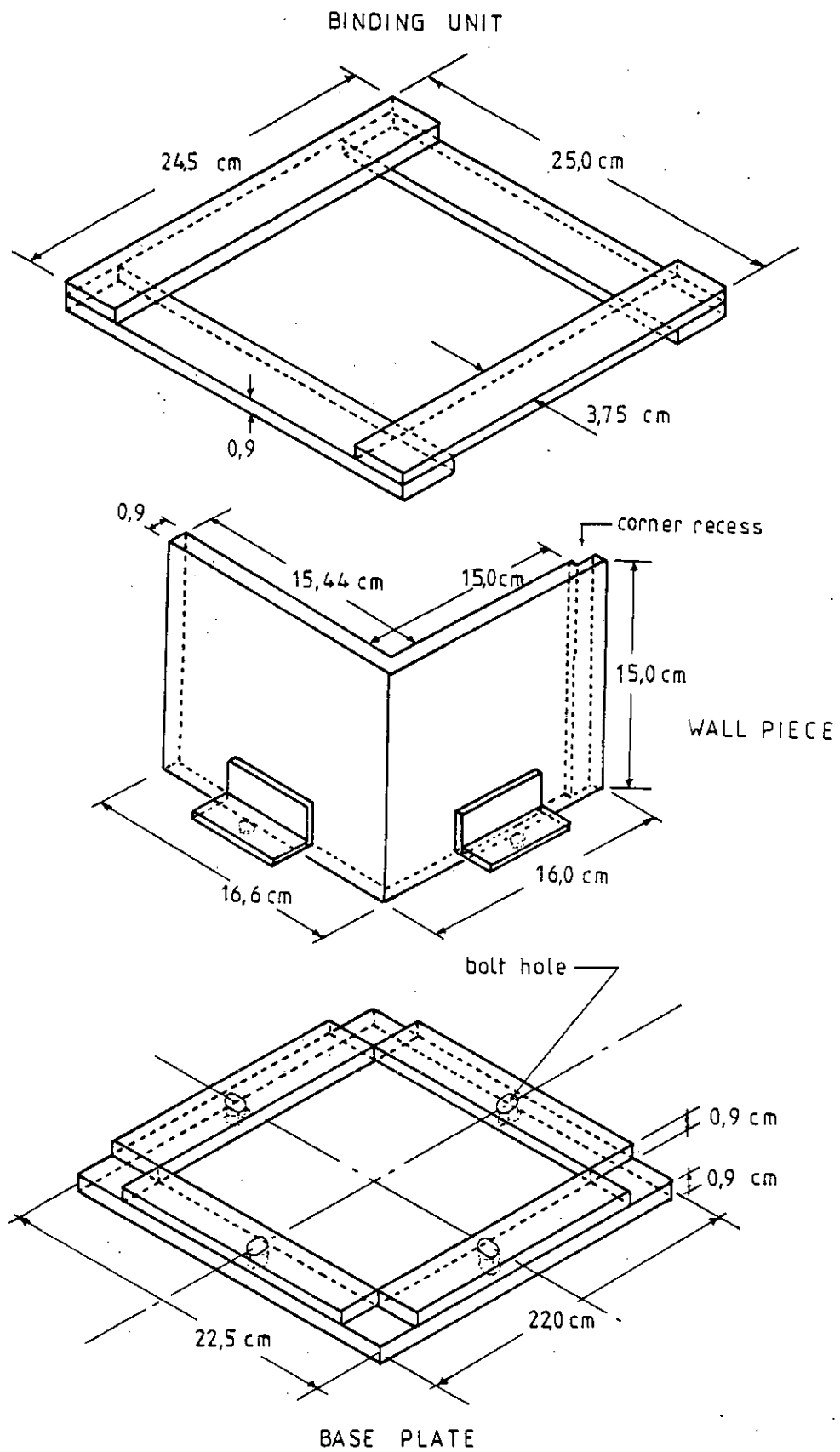
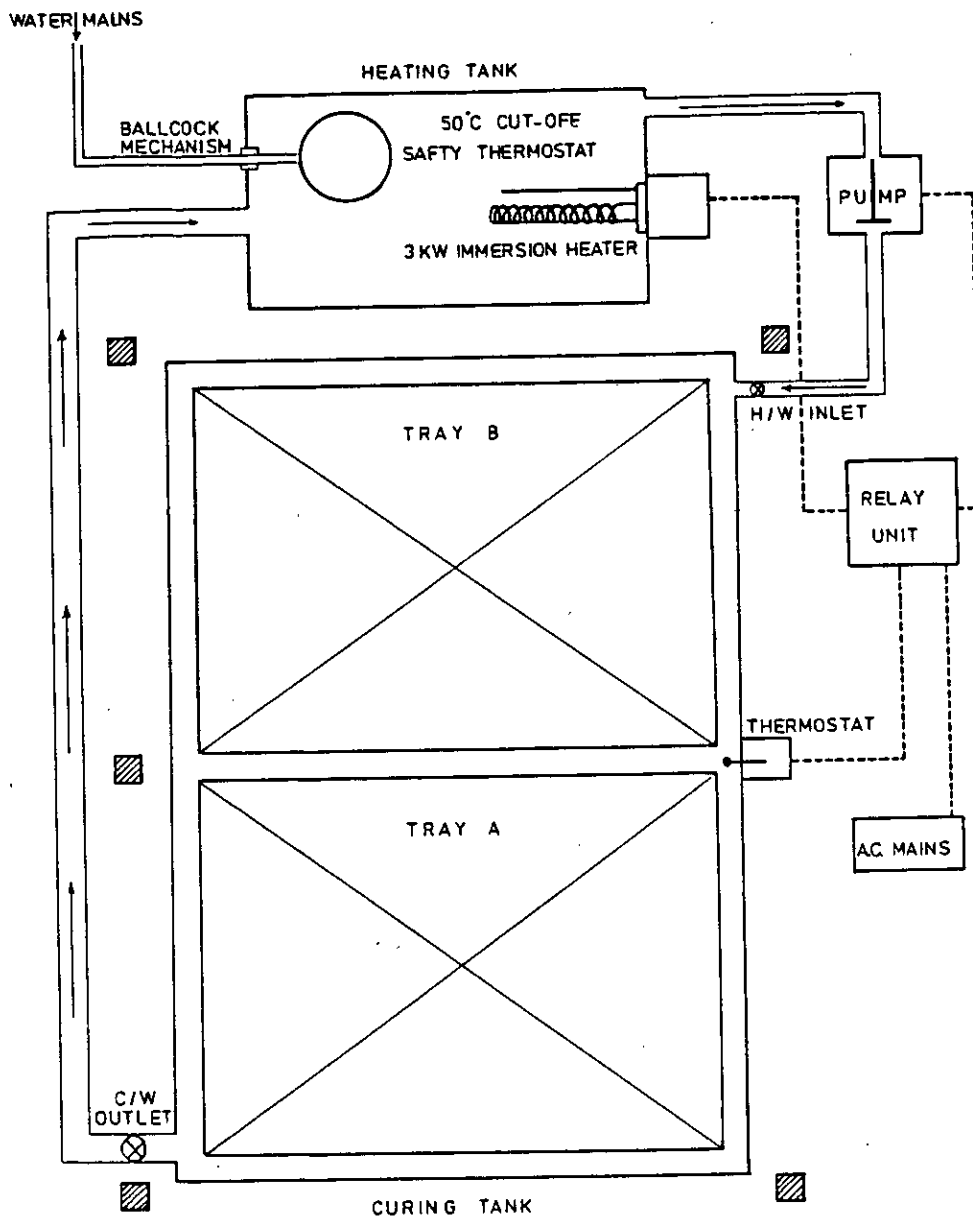


FIGURE A2.1 : Diagram of mould elements.



KEY

----- Mains Supply

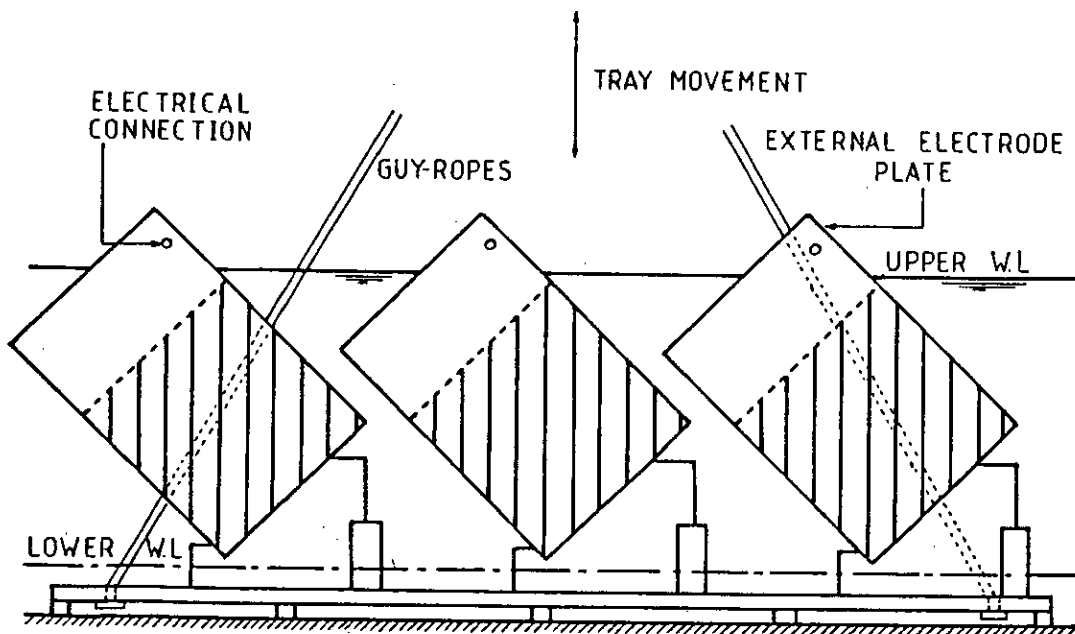
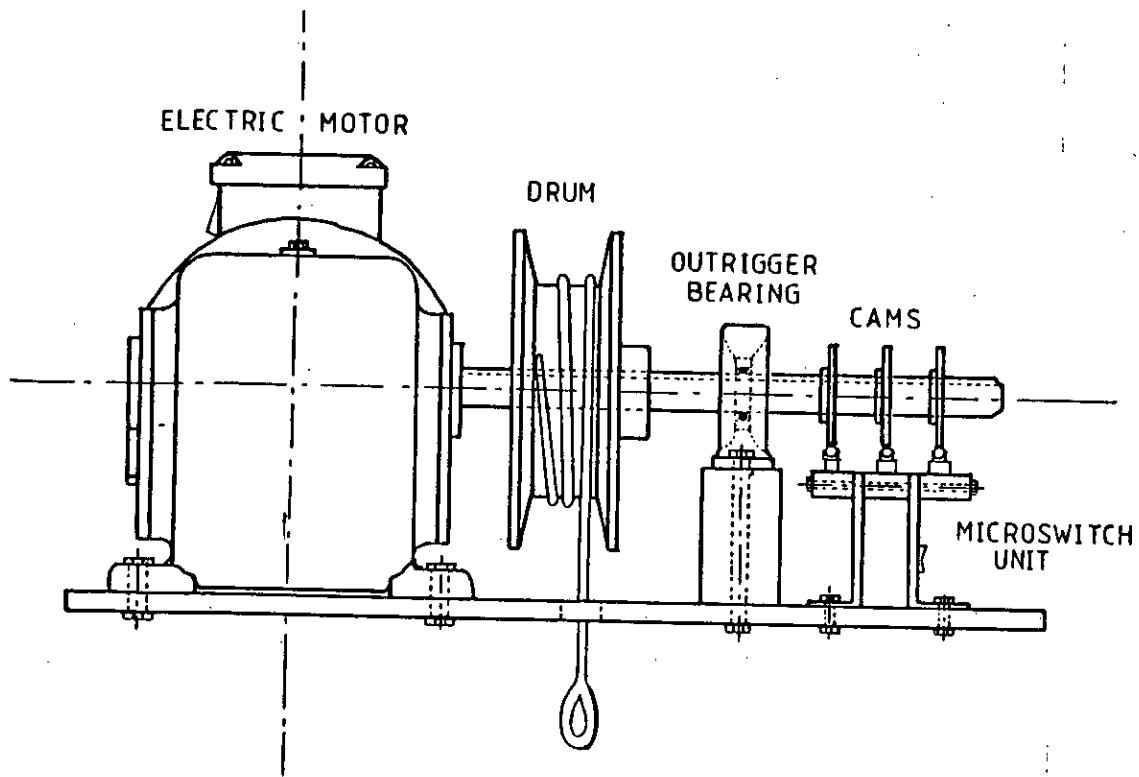
⊗ Hydraulic Valve

▨ Main frame supports

PUMP Stuart Turner 9.1 lts/min max. capacity

PLAN VIEW OF CURING TANK LAYOUT AND HEATING SYSTEM

FIGURE A2.2



SCHEMATIC DIAGRAM OF HOIST AND TRAY ARRANGEMENT

FIGURE A2.3

**APPENDIX THREE**

**TYPICAL EXPERIMENTAL OUTPUT**

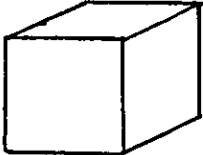
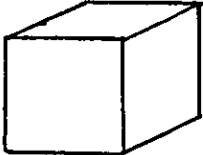
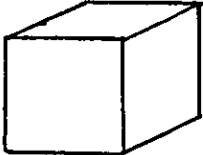
DATA TAPE NO. 1	DATE OF TESTS: - 28-6-83																				
LENGTH OF TEST: - 28 days	NO. OF CUBES= 3																				
START ADDS= 0/00	MIX= 1:1:2	W/C RATIO= 0.40:1																			
<p><b>MATERIAL CHARACTS.</b></p> <p>AGGREGATE: - 20mm down Whinstone</p> <p>SAND: - Concrete sand (Grading zone 2)</p> <p>CEMENT: - Ordinary Portland - sample 13</p> <p>WATER: - Tap</p>																					
<p><b>INFO. REGARD. DATA GROUPING</b></p> <p>NO. OF BATCHES: - 1</p> <p>BATCH CATALOGUE NO. 90</p>																					
<p><b>TEST INFO.</b></p> <table style="width:100%; border-collapse: collapse;"> <thead> <tr> <th style="width:15%;"></th> <th style="width:15%;">Sample</th> <th style="width:15%;">wt. at Day 1 (kgs)</th> <th style="width:15%;">wt. at Day 28 (kgs)</th> <th style="width:15%;">c.s. at day 28 (MPa)</th> </tr> </thead> <tbody> <tr> <td rowspan="3" style="text-align: center; vertical-align: middle;">  </td> <td>0/00</td> <td>8.147</td> <td>8.278</td> <td>63.78</td> </tr> <tr> <td>0/01</td> <td>8.093</td> <td>8.220</td> <td>61.33</td> </tr> <tr> <td>0/02</td> <td>8.257</td> <td>8.394</td> <td>65.55</td> </tr> </tbody> </table>					Sample	wt. at Day 1 (kgs)	wt. at Day 28 (kgs)	c.s. at day 28 (MPa)		0/00	8.147	8.278	63.78	0/01	8.093	8.220	61.33	0/02	8.257	8.394	65.55
	Sample	wt. at Day 1 (kgs)	wt. at Day 28 (kgs)	c.s. at day 28 (MPa)																	
	0/00	8.147	8.278	63.78																	
	0/01	8.093	8.220	61.33																	
	0/02	8.257	8.394	65.55																	

FIGURE A3.1 : Test details.



DATE 28-6-83  
 START 0/00 NUM 3  
 WATER:CEMENT 0.40:1  
 CEMENT:SAND:AGGREGATE 1:1:2  
 TEMP COEFFICIENT 0.022

SAMPLE NUMBER 1

TIME	RES	TEMP	C	RES
0.0135	6.3	20.6		6.4
0.0150	6.6	20.6		6.7
0.0250	7.2	21.4		7.5
0.0400	6.9	21.2		7.1
0.0650	7.6	21.6		7.9
0.1000	8.3	21.6		8.7
0.1500	9.0	22.0		9.4
0.2500	11.1	22.6		11.8
0.4000	16.4	22.8		17.4
0.6500	34.2	22.1		35.8
1.0000	48.8	21.3		50.2
1.5000	40.7	20.2		40.9
2.5000	46.4	20.4		46.9
4.0000	52.2	20.0		52.2
6.5000	58.3	20.4		58.9
10.0000	61.2	19.5		60.5
15.0000	63.2	20.4		63.8
25.0000	66.0	20.9		67.4

SAMPLE NUMBER 2

TIME	RES	TEMP	C	RES
0.0135	6.9	19.8		6.9
0.0150	6.9	20.0		6.9
0.0250	7.6	20.8		7.7
0.0400	7.2	20.9		7.4
0.0650	7.9	21.2		8.1
0.1000	8.3	21.6		8.6
0.1500	9.3	22.2		9.8
0.2500	11.1	22.9		11.8
0.4000	17.9	24.3		19.6
0.6500	37.0	23.5		39.8
1.0000	52.8	22.0		55.1
1.5000	42.9	19.5		42.4
2.5000	48.0	19.7		47.6
4.0000	54.4	19.1		53.3
6.5000	59.7	20.1		59.8
10.0000	63.0	19.5		62.3
15.0000	66.0	19.5		65.3
25.0000	67.5	19.3		66.4

SAMPLE NUMBER 3

TIME	RES	TEMP	C	RES
0.0135	6.6	19.7		6.6
0.0150	6.9	20.0		6.9
0.0250	7.2	21.0		7.4
0.0400	7.6	21.0		7.8
0.0650	7.9	21.4		8.2
0.1000	8.3	21.6		8.6
0.1500	9.4	22.4		9.9
0.2500	11.4	23.0		12.2
0.4000	17.9	24.1		19.6
0.6500	36.0	23.3		38.7
1.0000	50.9	21.7		52.8
1.5000	42.7	19.4		42.1
2.5000	48.0	19.7		47.6
4.0000	54.7	18.7		53.2
6.5000	59.5	19.5		58.8
10.0000	63.6	19.2		62.5
15.0000	66.6	19.3		65.6
25.0000	69.2	19.7		68.7

FIGURE A3.2 : Resistivity measurements as tabled by HP-85.

FIGURE A3.3 : Resistivity-time graph for initial 24-hour period.  
-245-

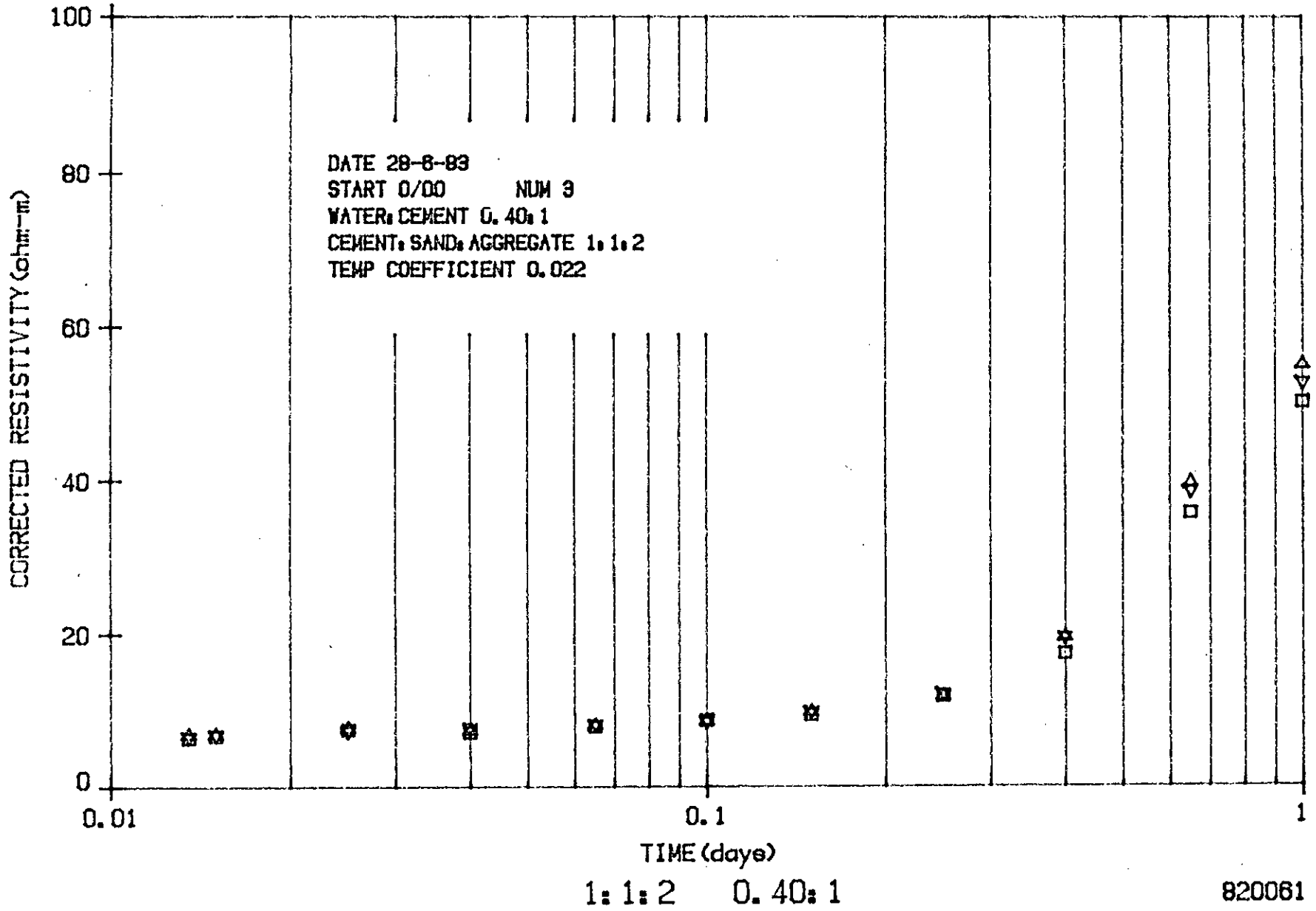
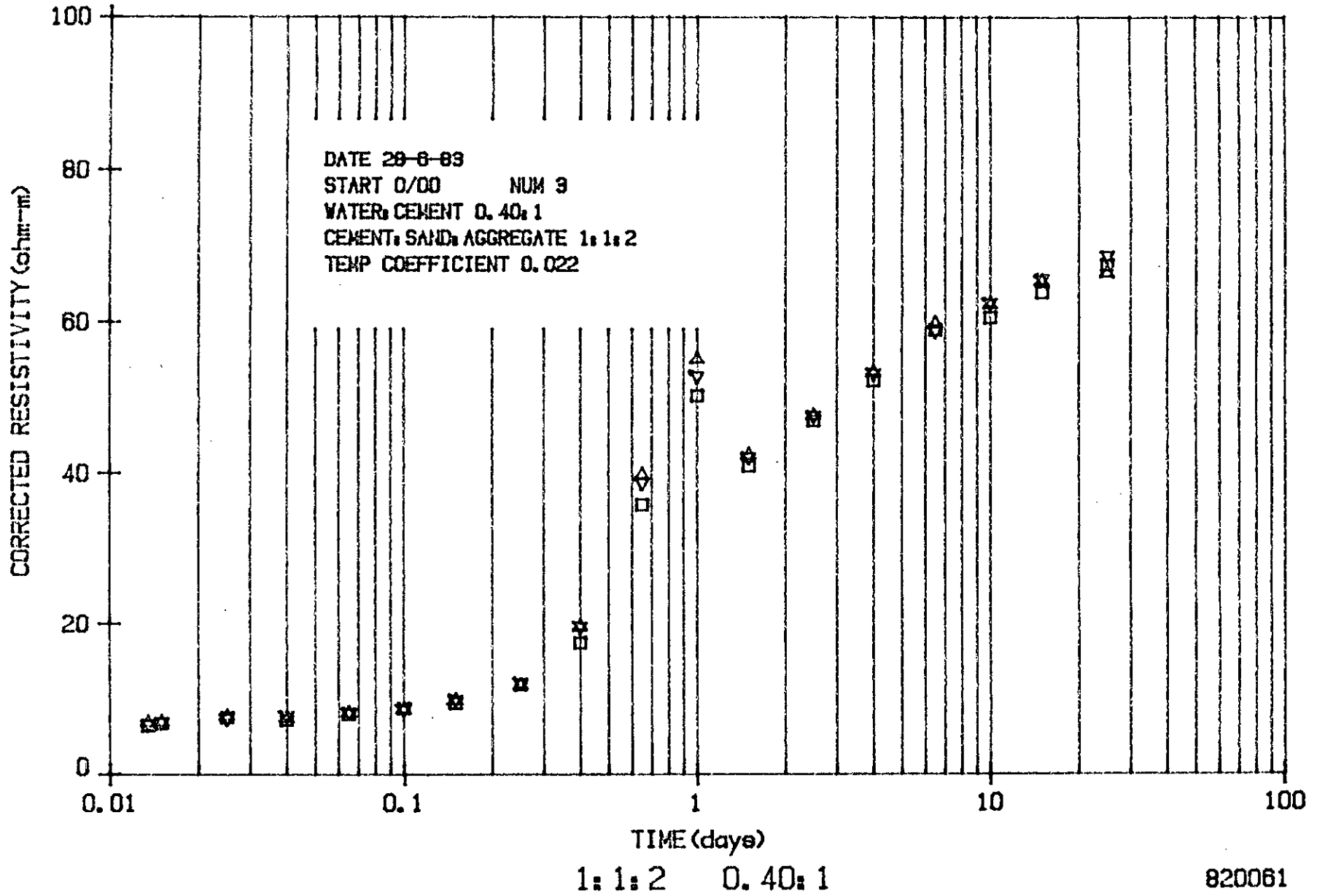


FIGURE A3.4 : Resistivity-time graph for 28-day test.  
-246-



**APPENDIX FOUR**

**DETERMINING THE SPECIFIC GRAVITY OF CEMENT**

DETERMINING THE SPECIFIC GRAVITY OF CEMENT  
(ORDINARY PORTLAND FROM THE DUNBAR WORKS)

The average density of the cement employed was determined by carrying out a series of liquid displacement tests in a density bottle using kerosene (paraffin oil).

Weight of bottle empty. =  $W_0$  gms

Weight of bottle filled with water. =  $W_1$  gms

Weight of bottle filled with kerosene. =  $W_2$  gms

Specific gravity of kerosene. =  $\frac{W_2 - W_0}{W_1 - W_0} = D$

Experimental s.g. for kerosene = 780 kgs/m<sup>3</sup>

(NOTE Specific gravity of kerosene quoted by "Science Data Book  
(Editor R.M. Tennent)" was 800 kgs/m<sup>3</sup>)

Weight of bottle & cement

=  $W_3$  gms

Weight of bottle & cement & kerosene.

=  $W_4$  gms

∴ Weight of cement.

=  $(W_3 - W_0)$  gms

∴ Weight of Kerosene equal in volume to cement.

=  $[(W_2 - W_0) - (W_4 - W_3)]$  gms

∴ Weight of water equal in volume to cement.

=  $\frac{(W_2 - W_0) - (W_4 - W_3)}{D}$

∴ Specific gravity of cement.

$$= \frac{(W_3 - W_0)D}{(W_2 - W_0) - (W_4 - W_3)}$$

Average specific gravity of 3 samples = 2.99

**APPENDIX FIVE**

**TABLES**

Mix Proportions (By Volume) (1 part of cement employed throughout)			Time (Days)	Corrected Resistivity (Ohm-m)	Average Strength (MP <sub>a</sub> )
Coarse Aggregate	Sand	Water			
CONCRETES					
3	1.5	0.4	2	63	31.04
			4	73.27	44.00
			7	82.5	56.56
			10	85.1	60.30
			15	86.30	60.44
			20	90.00	62.74
			28	91.70	60.33
3	1.5	0.5	2	54	23.48
			4	58.5	29.33
			7	60	31.26
			10	61	37.04
			15	63	41.93
			20	63.5	46.74
			28	64	50.07
3	1.5	0.6	2	37.8	11.48
			4	43.43	17.55
			7	50.60	22.81
			10	52.10	23.41
			15	51.60	31.11
			20	55.5	32.44
			28	57.1	32.52

(a) water/cement ratio

Mix Proportions (By Volume) (1 part of cement employed throughout)			Time (Days)	Corrected Resistivity (Ohm-m)	Average Strength (MP <sub>a</sub> )
Coarse Aggregate	Sand	Water			
CONCRETES					
1	1	0.5	2	18.9	13.93
			4	24.98	24.45
			7	26.05	31.05
			10	26	35.11
			15	26.65	36.89
			20	27.4	41.15
			28	28.5	45.93
3	1.5	0.5	2	47	23.48
			4	54.6	29.33
			7	56.8	31.26
			10	58.6	37.04
			15	59.6	41.93
			20	60.06	46.74
			28	60.10	50.07
4	2	0.5	2	46.8	22.52
			4	55.0	37.70
			7	58.5	41.92
			10	61.03	45.26
			15	65.03	49.18
			20	65.10	52.59
			28	65.30	52.44

(b) mix proportions

TABLE A5.1 Increases in Resistivity and Compressive Strength with age as a function of (a) water/cement ratio and (b) mix proportions



Mix Proportions (by Volume) (1 part of cement employed throughout)			Fractional volume of cement paste at demoulding	Formation factor at 0.065 * day	Formation Factor at 1.00 * day
Coarse Aggregate	Sand	Water			
PASTES					
0	0	0.25 0.30 0.35 0.40 0.45 0.50 0.55 0.60			
MORTARS					
0	1.5	0.40 0.45 0.50 0.55 0.60	0.56090 0.5700 0.59200 0.60600 0.61900	2.1656 2.1088 1.9018 1.5951 1.7365	1.2653 1.8254 1.6298 1.7166 2.2672
0	2.0	0.40 0.45 0.50 0.55 0.60	0.48920 0.50570 0.52110 0.53570 0.54930	2.9936 2.9252 2.3926 2.0245 1.9760	1.7041 2.2063 2.2736 2.5341 2.6721
0	2.5	0.40 0.45 0.50 0.55 0.60	0.43380 0.45010 0.46540 0.47990 0.49370	3.7580 3.5374 2.9448 2.7607 2.3353	2.1939 2.9365 3.2797 3.5695 4.0486
0	3.0	0.40 0.45 0.50 0.55 0.60	0.38970 0.40550 0.42050 0.43470 0.44830	4.3949 4.4218 3.5583 3.1288 3.1138	2.4388 3.4286 3.5815 3.9510 5.1012
0	3.5	0.50	0.38340	4.4885	4.4064
0	4.0	0.50	0.35240	5.6442	5.5332
0	4.5	0.50	0.32600	8.5276	8.0684
0	5.0	0.50	0.30330	11.0429	10.0604
0	5.5	0.50	0.28350	14.9693	14.3461
0	6.0	0.50	0.26620	24.4785	25.3923
CONCRETES					
1	1	0.40 0.45 0.50 0.55 0.60	0.49300 0.50950 0.52490 0.53930 0.53500	3.9299 3.4218 2.9877 2.9632 2.5569	3.3296 3.6667 3.5211 4.2234 5.5992
2	1	0.40 0.45 0.50 0.55 0.50	0.39450 0.41040 0.42540 0.43970 0.45330	5.1401 5.0340 4.4356 3.8466 3.3114	5.3776 6.6619 6.7465 6.9210 7.5830
3	1.5	0.40 0.45 0.50 0.55 0.60	0.30290 0.31700 0.33050 0.34350 0.36500	8.1338 8.5510 7.0000 6.1350 5.5269	8.2786 11.1159 12.6258 11.7166 14.7368
4	2	0.40 0.45 0.50 0.55 0.60	0.24520 0.25820 0.27020 0.28180 0.29310	10.8025 11.4966 9.3252 8.4847 7.4850	12.7480 15.2175 11.7042 14.2044 19.5020

TABLE A5.2 Formation Factor characteristics of mixes tested

Mix Proportions (by Weight) (1 part cement employed throughout)			Time (Hours)	Corrected Resistivity (Ohm-m)	Formation Factor
Coarse Aggregate	Sand	Water			
PASTES	0	0.40	1.56	1.57	1
			2.40	1.47	
			3.60	1.60	
			6.00	1.53	
			9.60	2.83	
			15.60	6.43	
			24.00	9.80	
0	0.45	0.45	1.56	1.47	1
			2.40	1.47	
			3.60	1.70	
			6.00	1.80	
			9.60	2.00	
			15.60	3.90	
			24.00	6.30	
0	0.50	0.50	1.56	1.63	1
			2.40	1.63	
			3.60	1.70	
			6.00	1.80	
			9.60	2.10	
			15.60	3.30	
			24.00	4.97	
0	0.55	0.55	1.56	1.63	1
			2.40	1.67	
			3.60	1.70	
			6.00	1.73	
			9.60	2.07	
			15.60	2.83	
			24.00	3.67	
0	0.60	0.60	1.56	1.67	1
			2.40	1.70	
			3.60	1.71	
			6.00	1.73	
			9.60	1.87	
			15.60	2.10	
			24.00	2.47	

FORMATION FACTOR CHARACTERISTICS DURING INITIAL 24 HOURS

TABLE A5.3

Mix Proportions (by Weight) (1 part cement employed throughout)			Time (Hours)	Corrected Resistivity (Ohm-m)	Formation Factor
Coarse Aggregate	Sand	Water			
MORTARS					
0	1.5	0.50	1.56	3.1	1.90
			2.40	3.4	2.09
			3.60	3.4	2.10
			6.00	3.8	2.11
			9.60	4.6	2.19
			15.60	6.1	1.84
			24.00	8.1	1.63
0	2.0	0.50	1.56	3.9	2.39
			2.40	4.0	2.45
			3.60	4.4	2.58
			6.00	4.8	2.67
			9.60	5.6	2.67
			15.60	8.4	2.54
			24.00	11.3	2.73
0	3.5	0.50	1.56	7.3	4.47
			2.40	7.0	4.29
			3.60	7.7	4.52
			6.00	9.1	5.05
			9.60	12.9	6.14
			15.60	17.2	5.22
			24.00	21.9	4.46
0	2.5	0.50	1.56	5.2	3.19
			2.40	5.2	3.19
			3.60	5.6	3.29
			6.00	6.1	3.38
			9.60	8.6	4.09
			15.60	12.5	3.78
			24.00	16.3	3.28
0	2.5	0.40	1.56	5.9	3.76
			2.40	6.3	4.29
			3.60	6.4	4.00
			6.00	7.3	4.77
			9.60	11.5	4.06
			15.60	17.6	2.73
			24.00	21.5	2.19
0	2.5	0.60	1.56	3.9	2.32
			2.40	3.9	2.29
			3.60	4.3	2.57
			6.00	4.8	2.77
			9.60	5.9	3.15
			15.60	7.8	3.71
			24.00	10.0	4.04

FORMATION FACTOR CHARACTERISTICS DURING INITIAL 24 HOURS

Mix Proportions (by weight) (1 part cement employed throughout)			Time (hours)	Corrected Resistivity (Ohm-m)	Formation Factor
Coarse Aggregate	Sand	Water			
CONCRETES					
1	1	0.50	1.56	4.87	2.98
			2.40	5.02	3.07
			3.60	5.43	3.19
			6.00	6.23	3.46
			9.60	8.05	3.83
			15.60	12.42	3.76
			24.00	17.50	3.52
2	1	0.50	1.56	8.07	4.94
			2.40	8.63	5.29
			3.60	9.70	5.71
			6.00	11.93	6.62
			9.60	18.68	8.92
			15.60	38.10	11.54
			24.00	32.70	10.61
4	2	0.50	1.56	15.2	9.32
			2.40	16.3	9.98
			3.60	17.5	10.27
			6.00	21.5	11.91
			9.60	32.2	15.36
			15.60	44.2	13.39
			24.00	58.2	11.70
3	1.5	0.50	1.56	11.4	7.00
			2.40	12.2	7.51
			3.60	13.7	8.00
			6.00	18.1	10.40
			9.60	29.9	14.42
			15.60	49.5	15.30
			24.00	62.7	12.68
3	1.5	0.40	1.56	12.77	8.13
			2.40	14.37	9.77
			3.60	16.47	10.29
			6.00	22.40	14.64
			9.60	41.37	14.61
			15.60	68.17	10.60
			24.00	81.13	8.28
3	1.5	0.60	1.56	9.23	5.53
			2.40	9.67	5.69
			3.60	10.43	6.24
			6.00	12.20	7.05
			9.60	17.70	9.46
			15.60	27.10	13.19
			24.00	36.40	14.74

TABLE A5.3 (cont.)

FORMATION FACTOR CHARACTERISTICS DURING INITIAL 24 HOURS