DETERIORATION OF CEMENT- BASED STRUCTURES: CHEMICAL INVESTIGATION OF CRACKS IN SANDCRETE BLOCKS

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BY

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DEGREE IN CHEMICAL ENGINEERING.

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DECLARATION

I, IBRAHIM MU'AZU (M. ENG/SEET/2001/2002/769), hereby declare that this research project Chemical Investigation of Cracks in Sandcrete Blocks, presented for the award of Master of Engineering in Chemical Engineering is purely and entirely my work and it has not been presented for any other degree elsewhere.

SIGNATURE

1/10/05

DATE

CERTIFICATION

This Thesis titled " Deterioration of Cement – Based Structures: Chemical Investigation of Cracks in Sandcrate Blocks "by IBRAHIM MUAZU meets the regulations governing the degree of M.ENG of The Federal University of Technology, Minna and is approved for its contribution to scientific knowledge and literary presentation.

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DEDICATION

This project is dedicated to my parent Mr. and Mrs. MUAZU SAIDU, my beloved wife SALAMATU IBRAHIM and my late son AL-MUKHTAR IBRAHIM.

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All praise is to Allah the almighty (S.W.T) who gave me the grace and wisdom to reach this of my academic career. May his peace and blessing be upon his prophet Muhammed (S.A.W)

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ABSTRACT

Cement–based structure stability could be characterized not only by their mechanical strength, soil geophysics and loading conditions, but also the resistance to various physical and chemical factors that could initiate its deterioration. The contribution of water and various water –based solutions in the environment, to cement based structure durability could be either physical as in seasonal flooding of the structure or chemical often initiated by the presence in the solution of organic, inorganic compounds and gases. Irrespective of the deterioration type, its destructive effect could be attributed to the build up of internal stress and weakening of the structure matrix. This work investigated the role of chemical changes in the sandcrete matrix in the formation and development of macrocracks in cement based sandcrete walls. Collected sandcrete samples were analyzed using titrimetric, gravimetric and instrumental methods. Results showed that the difference in percentage composition of SiO_2 at the top to bottom were high ranging from 76.72 to 80.30 % in all the sandcrete blocks samples analyzed. This indicates high permeability of ground level structure by the soil waters. The acidic nature of the soil in some areas with pH varying from 5.89-6.11 could seriously undermine the strength of the structures. The percentage compositions of CaO were generally very low ranging from 3.83 % at the ground to 4.44 % at the top level. Na_2O records high composition

at the top level declining steadily to the ground level (0.80 - 0.68 %). This may lead to Alkali Silica reaction. There are compositional differential horizontally and vertically across most points. This can be attributed to chemical reaction involved between the oxides of CaO, MgO, Na_2O, SO_3 with surrounding compounds in the presence of moisture, or poor cement quality, or probably cement with quality that does not comply to standard requirements.

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LIST OF ABBREVIATIONS

ASR	-	Alkali Silica Reaction
ILO	-	International Labour Organization
C-S-H	-	Calcium Silicate Hydrate
W/C	-	Water Cement Ratio
BS	-	British Standard
ASTM	-	American Standard for Testing Materials
LOI	-	Loss on Ignition
W	-	Weight
AAS	-	Atomic Absorption Spectrophotometer
NIS	-	Nigerian International Standard

CHAPTER ONE

1.0 INTRODUCTION

Concrete structures are hardly ever built under ideal conditions, so, for variety of reasons, defect may occur as the concrete is being cast or after some time (Allen et al. 1993). The factors that initiate corrosion of cement-based structure could be physical, chemical factors of the environment or chemical reaction occurring internally in cemented systems (Swamy, 1992). The compressive strength of cement-based structures is expected to increase steadily with age. However, this statement may not be true for many structures, especially those serving in aggressive environments. In some areas, most cement-based structures were found to develop micro-cracks within a few years of construction despite the fact that acceptable standards were observed (Odigure and Dickola. 2001). Various researchers have proved that long exposure of cement-based structures to aggressive medium containing acids, salt and alkalis immensely enhance the deterioration of their physicochemical and mechanical properties.

Reaction occurring internally, such as alkali aggregate reaction gives cause for concern, because this type of deterioration does occur even in non-aggressive environment. The potential for reaction is conditioned by chemical and mineralogical nature of the components, cement-aggregate-water composition as well as prevailing environmental conditions of temperature humidity e.t.c. The basic origins of the chemical potential involved in alkali-aggregate reaction is associated with the thermodynamically unstable nature of the reaction products , the siliceous aggregate material and alkaline mineral hydrates from the cement (ILO, 1987). These to a large extent define the cement base structure matrix rheology and morphology. For structures exposed to seasonal wetting or flooding by water, their chemical deterioration will depend on the extent of solubility of the mineral hydrates and possible migration through the various hardened matrix. Such migration of the salts or hydrates will be enhanced by contact of such structure to a wet earth/foundation.

1.1 Aims and Objectives of Study

The research work is aimed at investigating the contributions of chemical composition to cracks development in cement based structures .

The major objectives of study are:

1. To determine the percentage content of SiO_2,, MgO, Fe_2O_3, CaO, Loss on Ignition (LOI), Na_2O and Al_2O_3 present in sandcrete samples collected from four cracked walls.
2. To discuss the effect of these oxides as components of cements mineral hydrates on the sandcrete structures.

.2 Need for the Study

Building activities are on the increase in Nigeria. This is due to growth in population and consequently the need for an individual to own a house is on the increase (Sule, 2001). Because of this, houses are sited in industrial and non-industrial areas. Cement based structures constructed in industrial areas deteriorate with time as a result of various reasons such as improper mix, low quality of cement used and environmental effects. These factors have necessitated investigations to a certain the causes of such deterioration. This work is an attempt at finding the contribution of building material, chemical composition to the deterioration and possible crack formation in sandcrete structures.

1.3 Scope

This work is limited to four cases in Kaduna metropolis, capital of Kaduna State in Nigeria

1) Kurmin Mashi I and II along Nnamdi Azikiwe Road . The building has been in existence for the past six years , It was purposely constructed to be a market , but abandoned.
2) Ungwan Muazu I and II in Tudunwada. The building was also in existence for the past five years. It was built to be a market, but also abandoned .

Collections of samples were limited to both sides of the crack line.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 CEMENT

Cement can be defined as the hydraulic binding material used for construction purposes. Hydraulic means ability of substance to set or harden when exposed to moisture. The word "cement" originated from the Latin word "cementum" which was then Greek construction material. The history can be attributed to the work in 1824 of a British gentleman Joseph Aspidin (Kangiwa, 1995). Portland cement is the name given to cement obtained by intimately mixing together calcareous and argillaceous, or other silica, alumina and iron oxide bearing materials, burning them at clinkering temperature and grinding the resulting clinker(Neville and Brooks. 1994). Later in 1845 Isaac Johnson improved on the work of Aspidin by sintering certain proportions of calcareous and argillaceous materials at a higher temperature to obtain better qualitative products.

2.1.1 Hydration of Cement

This is the product of reaction of cement with water. In the presence of water, the silicates and aluminates of portland cement form products of hydration or hydrates, which in time produce a firm and hard mass. As stated earlier, the two calcium silicate (Ca_3S and Ca_2S) are the main cementitious compounds in cement, (Neville and Brooks. 1994). The former hydrates much more rapidly than the latter. The impure Ca_3S is known as ALITE and the impure Ca_2S as BELITE. The product of hydration of Ca_3S is the microcrystalline hydrate $C_3S_2H_3$ with same lime separating out as crystalline Ca $(OH)_2$, Ca_2S behaves similarly but clearly contains less lime. Nowadays, the calcium silicate hydrate are described as C-S-H. (previously reffered to as TOBERMORITE GEL). The approximate hydration reactions are stated below.

For Ca_3S

$$2Ca_3S+4.5H_2O \longrightarrow Ca_3S_2H_3+3Ca(OH)_2 \qquad 2.1$$

For Ca_2S

$$2Ca_2S+2.5H_2O \longrightarrow Ca_3S_2H_3+Ca(OH)_2. \qquad 2.2$$

The reaction of pure Ca_3A with water is very rapid and would lead to a flash set, which is prevented by addition of gypsum to the cement clinker.

The approximate reaction is stated below:

$$Ca_3A+6H_2O \longrightarrow Ca_3AH6. \qquad 2.3$$

2.1.2 Test on Cement

The quality of cement is vital for the production of good concrete, the manufacture of cement required stringent control. A number of tests are performed in the cement plant laboratory to ensure that the cement is of the desired quality and that it conforms to the requirements of the relevant national standards (Neville and Brooks. 1994). It is also desirable for the purchase or for an independent laboratory, to make periodic acceptance tests or to examine the properties of a cement to be used for some special purpose.

2.1.3 Characteristics of Cement

1) **Fineness:** This is one of the most important characteristics of cement as it influences the rate of cement reaction with water. It affects the reaction rate because of the fact that finely ground cement has more exposed surface area than a coarsely ground one.
2) **Soundness**: A sound cement is one whose paste (mixture of cement and water) sets and hardens without cracking or disintegration. Unsoundness is caused by hydration of free lime surrounded by cement particles. The surrounding cement particles prevent easy hydration of the lime during the normal setting period. Sudden hydration of the free lime particles causes expansion many times their sizes (Allen et al 1993), thus leading to cracking and disintegration of the concrete mortar. Too high magnesia content in cement could also cause unsoundness.

2.1.4 Storage of Cement

Cement can be kept in good condition for as long as required provided it is stored properly against moisture, which leads to lump formation (Allen, et al. 1993). To protect cement from lump formation, it must be stored in a cool and dry environment. One site, cement should be stored in a suitable building or covered with water proof material, the floor must also be provided with wooden platform, called, pallets that allows free movement of air.

Table 2.1 Typical Average Values of Compound Composition of Portland Cement of Different Types.

COMPOUND COMPOSITION (%)								
CEMENT	Ca_3S	Ca_2S	Ca_3A	Ca_4AF	$CaSO_4$	Free CaO	MgO	LOI
TYPE I	59	15	12	8	2.9	0.8	2.4	1.2
TYPE II	46	29	6 – 8	12	2.8	0.6	3.0	1.0
TYPE III	60	12	12–15	8	3.9	1.3	2.6	1.9
TYPE IV	30–35	40–46	5 – 7	13	2.9	0.3	2.7	1.0
TYPE V	45	36	4 – 5	12	2.7	0.4	1.6	1.0

2.1.5 Chemical and Mineralogical Nature of Cement

Portland cement which is commonly used in concrete is a manufactured product, made by calcination at high temperatures of naturally occurring raw materials. Limestone and clay are often used; they are intimately ground and fed into a rotary kiln. The kiln is frequently filled with a suspension, preheater to reduce the overall energy requirements of the process. A complex series of chemical reactions ensue as the feed materials are progressively heated; these reactions involve dehydroxylation, decarbonation and solid state reactions among the oxide components (Swamy, 1992). At the highest calcinations temperatures, typically 1450°C, smelting of some kiln charges and new mineral phases occur, not present in the original feed, develop in response to the high temperature regime. Reaction continues in the solid state but is generally facilitated by transport through a reactive high temperature melt.

The resulting product known as clinker is finally cooled to ambient temperature and ground to high specific surface area, typically 3000-5000 cm^2/g (Odigure, 2002). The micro structure and mineralogy of clinker are characteristic of a manufactured product, some show certain affinities to those observed four different kinds of crystals within reaction of cement clinkers and to those Tornebohm (1897) gave the names alite, belite, celite and felite. Although, the CaO content of Portland cement is very high, typically 63 – 69 % by weight, the fired product contains little free lime of about 1 – 3 % , and consists essentially of four phases. Two calcium silicate Ca_3S and Ca_2S, one aluminate, Ca_3A, and an aluminoferrite Ca_4AF. These formulae are idealized as other minor components also occur in the clinker feed such as MgO, Na_2O, K_2O, MnO e.t.c. The behavior of the alkalis Na_2O and K_2O is very important in this project work.

2.1.6 Alkali Nature of Cement

The alkali content of clinker may be differentiated broadly into two generic types: an alkali which is normally present and condensed on the surface of clinker grains, perhaps as sulphate salts; and an alkali which is locked into the crystal structures of the clinkers minerals. All their clinker minerals have some potential to retain alkalis in solid solution, but much of the sodium appears in Ca_3S (alite), while potassium tends to be more evenly distributed with Ca_2S (belite) and associated glass playing an important role as hosts (Swamy, 1992).

When alkali containing clinker is hydrated, surface and clinker alkalis behave differently with respect to their release rates. Alkali present as water soluble sulphates become available for solution almost immediately , whereas alkalis locked into clinker grains become available for solution rather more slowly in the course of hydration of their host minerals. The characteristic overall release rate therefore, may be variable from one cement to another depending on the distribution of alkalis between rapid release and slow release source as well as on total alkalis.

2.2 AGGREGATES

Crushed or uncrushed materials derived from natural sources such as rocks, gravel, boulders and sand for production of concrete are called aggregates.

There are two main groups in aggregate having particle size less than 5 mm and 90 to 100 % of which must be able to pass through 4.75 mm sieve. Natural sand , finely crushed stone and gravel are treated as fine aggregate (Allen et al. 1993).Coarse aggregate on the other hand, are the stone material most of which is retained on 4.75 mm sieve. Uncrushed gravel or stone are the usual coarse aggregate.

Aggregate, whether fine or coarse must be hard, durable, clean and free from coal and other mica, iron pyrites, shales, clay, alkalis and other organic material. There are other types too like broken brick-aggregate ,cinder and slag aggregate e.t.c which are used in concrete works, where- as only fine aggregate with either cement or lime are used in mortar for plastering and jointing.

Aggregate can be classified into 2 types.

1) Natural Aggregates: These consist of gravel obtained from natural sources like fits, river, ocean beds where they are deposited by alluvial and glacial action. Another source of natural aggregates is rock out crops, these used for concrete aggregate include granite, dolomite limestone e.t.c.

2) Artificial Aggregates: These are usually the inorganic by- product of some industrial processes. Example are expanded clay, foamed slag, iron ore shot,

3) barites, scrap iron e.t.c. many of these aggregate produce light weight concrete of adequate strength with good heat insulation properties.

2.2.1 Chemical and Mineralogical Features of Aggregates

Aggregate was originally viewed as an inert material dispersed throughout the cement paste largely for economic reasons. It is now know that aggregate is not truly inert and its physical, thermal and chemical properties influence the performance of concrete (Neville, 1988). SiO_2 comprises about 65 % by weight of the accessible portions of the earth crust. Therefore, SiO_2 occurs in the free state as a solid crystalline oxide and silicates, in which silicon and oxygen are combined with other element of which aluminum, magnesium, calcium, potassium, sodium, iron and hydrogen are the most important, it contains a basic building block which is having silicon at the centre of a tetrahedron of four oxygen; the four Si-O bonds are semi- covalent. The tetrahedrals are not isolated from each other, instead each oxygen is shared by linking equally to two other (Swamy 1992), geometrically very similar tetrahedral, so that, they would be fully cross linked into a three-dimensional frame work. It is a peculiarity of tetrahedrally linked frame works that a number of alternative frame work arrangements are possible, which differ from one another energetically.

Depending on temperature and pressure, several such frame works occur stably for SiO_2; Quartz is the most abundant and is a polymorph in which the cross link is strong, hence it is comparatively densed and relatively uncreative, being unaffected by the presence of most strong acids or alkalis. Their thermodynamic meta stability and comparatively open, disordered structures also give rise to an enhanced potential for reaction with cement alkalis. SiO_2 exhibit a unique structural relationship with that of ice water, the water molecules consist of a central oxygen around which are arrange in approximately tetrahedral array of two hydrogen and two electron pairs. The geometric similarity between SiO_4 and OH. e_2(where e_2 stands for two electron pairs) enable water to substitute to some extent in silica. In quartz the extent of such substitution is slight of the order of a few parts of H_2O per million (Swamy, 1992). However, in less crystalline silica the substitution may be much higher, ranging up to several percent or more. In effect, strong 'bonds; e.g. Si-O-Si, are broken hydroxylation replaced by more reactive Si-OH..... OH-Si bonds where represent a weak hydrogen bond. Such amorphous, hydrous silica are very reactive in the presence of alkali. Sediment from which a soil developed exerts a substantial influence upon soil chemical properties which have a variety of generic names: silica gel, Opal, Chert and Chalcedony.

Reactive silicious aggregates fall into two classes, the first class comprises mainly the crystalline, low-density polymorphs of SiO_2 as cristobalite and tridynite. The second comprises of the poorly ordered as well as the essentially non crystalline forms, such as opals, glasses, gels e.t.c.

Typical aggregate used for making concrete are graded according to sizes,concrete are made with aggregate particles sizes covering a range of sizes between 10 and 50 mm. The aggregate has important functions, for example, it helps control the shrinkage which would occur in cement-rich formulation. The setting of cement is strongly exothermic; by so doing, concrete made with amorphous materials, limestone aggregate etc tend to suffer a reduction in strength when subjected to high temperature (Pinfold, 1975).

2.3 CRACKS

These are openings which develop on the surface of walls, slabs, beams and columns as a symptom of failure which consequently may result to the collapse of the structure. There are cracks of various sizes and shapes which develop on any structure before collapse. The shape and size of each depends on the condition of exposure, and type of loading it is subjected to (Maren, 1995). Cracks in buildings occur due to one reason or the other. The rate of occurrence may differ from place to place depending on the degree of occurrence in construction, use of material and site investigation.

2.3.1 Types of Crack.

1. Plastic Shrinkage Cracks

If cracks appear in an exposed concrete surface very soon after it has been finished or even in some cases, before finishing is completed, they are termed as plastic shrinkage cracks (Allen et al. 1993). These are caused by rapid drying of the concrete surface while the body of the concrete is still plastic. They are usually discontinuous and they seldom extend to free edge. The most effective way of preventing their occurrence is by sheltering the surface from wind and sun during construction by covering it immediately after finishing. The remedial measures after the cracks have formed usually consist of sealing them against ingress of water by brushing in cement or low viscosity polymers.

2. Plastic Settlement Cracks

Concrete may continue to settle, especially in deep sections, after it has started to stiffen and anything that obstructs this movement, such as form work tie-bolts, may act as a wedge so a crack forms immediately over the obstruction. Cracks of this type are known as plastic settlement cracks.

They may be avoided by changing the concrete mix design and using air entrained concrete and, if they do start to form, they can be closed immediately the compacting the upper part of the concrete lifts. Remedial measures after the concrete has hardened consist of sealing the cracks with sealants in other to protect the reinforced bars.

3. Thermal Cracking.

Heat of hydration of cement raises the temperature of concrete so that it is usually slightly warmer than its surroundings when it hardens and in thick sections and with rich mixes the temperature rise may be quite considerable. As the concrete cools it would try to contract. If this contraction is restrained the concrete would be put into tension, and if the strain capacity of the concrete is exceeded as observed by Allen et al. (1993), crack would form. These thermal contraction cracks are often ascribed to as drying shrinkage. The risk of thermal cracking can be reduced by minimizing restraint to contraction and by delaying cooling until concrete is strong enough to resist the stresses induced.

2.3.2 Development of Cracks in Concrete

Latent defect may be caused by inadequacy of design material or construction which may not become evident until sometime after completion. The immediate mechanism of deterioration may be, for example chemical action or corrosion of re-enforcement but in a large proportion of cases, the fundamental cause can be traced back to something such as unrealistic detailing or poor workmanship. Deterioration of concrete may be due to chemical which reduces it resistance to sulphate, chloride, alkali silica reaction (ASR) e.t.c. attack (Allen et al, 1993 and Oyetola, 2000) or physical causes of corrosion of re-enforcement. Rust on steel occupies a volume several times greater than that of the metal from which it was formed, and this expansion can cause cracking and sapling of the surrounding concrete.

All concrete contains micro-cracks which do not affect the satisfactory performance of the concrete. However, macro-crack are due to shrinkage and excessive stress, and if they extend down to the steel can lead to loss of passivation leading to corrosion or the re-enforcement.

Shrinkage cracks can be due to high water-cement ratio (w/c) or the use of shrinkable aggregate and or inadequate curing of the concrete. There are difference of opinion regarding the maximum w/c ratio which should be specified (Allen et al. 1993). Believing that for any type of concrete containing steel reinforcement the w/c should not exceed 0.5 and for high quality concrete the w/c should be 0.4.

2.4 DETERIORATION OF CONCRETE

Deterioration of concrete mainly arises from 3 causes:

1) Physical damage caused by impact, abrasion, freeze-thaw (frost), thermal shock, high-velocity water.
2) Chemical attack, mainly on the cement paste, by external aggressive chemical compounds.
3) Reaction between the alkalis in hydrating cement and certain type of siliceous aggregates; this is known as alkali-silica reaction.

2.4.1 Physical Causes

When concrete is damaged by impact or abrasion, the cause is usually obvious and protection may be necessary as well as repair. Cracks that occur after construction, however are often more difficult to investigate. Over loading may produce cracks in tension zones of members, but other causes are probably more common. Drying shrinkage is a slow process in thick members, so it may lead to a gradual build-up of tensile stress if it is restrained(Perkins, 1997). The use of excessively wet, high shrinkage concrete mixes will aggravate matters.

Concrete may be damaged by environmental factors such as fire or frost. Concrete gradually loses strength with increase in temperature above 300^{O}C, the damage being greater with aggregate such as Quartzites with high coefficients of thermal expansion than these from limestone with lower thermal coefficients. Frost attack on concrete may take the form of spalling of the surfacing, or it may cause random cracking. Damage is made more likely by the use of de-icing salts. Physical processes of concrete deterioration are associated with chemical reaction (Goni and Sagrera, 1986), pocket that can fill water, or containing material that can fill water, or containing materials that can absorb water, are a source of trouble because the water in them will expand if it freezes, disrupting the surrounding concrete.

2.4.2 Chemical Attack on Concrete

Concrete are vulnerable to chemical attack by a wide range of chemical compounds in solution and this applies to both Portland cement concrete and high Alumina cement concrete. From a practical point .of view, the chemicals which are aggressive to concrete can be divided into 5 main categories:

1) Acid
2) Ammonium compounds
3) Magnesium
4) Sulphates
5) Alkali hydroxides.

The intensity of attack depends on a number of factors, the principal ones being the chemical composition of the aggressive agent, the concentration, the pH and the porosity & permeability of the concrete, the type of cement used, and the contact time.

There are vast number of chemical compounds used in industry and many of these are closely guarded trade secret. However (Perkins, 1997), some basic information is needed when considering the possible effect of a waste effluent on concrete sewer pipes to be specific. It is necessary for water authorities to obtain information on the constituents of a waste effluent which is discharged or intended to be discharged to public sewer. .

2.4.2.1 Attack by Acids

Chemical degradation is usually the result or attack on the cement matrix. Portland cement is alkaline, so it will react with acids in the presence of moisture and, in consequence, the matrix may become weakened and its constituents may leach out. Acidic ground water are potential cause of degradation of concrete foundations and these may be derived from decayed vegetation (humic acid) or from contamination of the ground from external sources. Dense concrete will be attacked only at the surface, so serious, damage may be slow to occur, but an acid resistant surface coating is the only complete protection (Allen et al. 1993).

2.4.2.2 Ammonium Compounds

Most ammonium compounds are aggressive to concrete, an exception is ammonium carbonate. The ammonium compounds used in the chemical fertilizer industry (sulphate, nitrate, super phosphate) cause serious deterioration of concrete in a relatively short time,. the actual time depends on the concentration and period of contact and the quality of the concrete in terms of porosity, permeability and cement content. The aggressive ammonium compounds react with the hydration products in the Portland cement. The damage to concrete caused by ammonium sulphate is mainly due to the expansion arising from the formation of calcium supho - aluminate, while ammonium nitrate and chloride solutions act rather like dilute acid by reaction with hydrated lime ($Ca\ (OH)_2$) in the cement paste.

The addition of condensed silica fines (dust) to Portland cement concrete would increase its resistance to attack by ammonium based fertilizer. High alumina cement is more resistant to ammonium compounds than Portland cement(Perkins 1997).

2.4.2.3 Magnesium Compounds

Magnesium chloride is aggressive to Portland cement but high alumina cement is reportedly immune to attack. magnesium sulphate is particularly aggressive to Portland cement but is less than ammonium sulphate. These attacks is due to the reaction with hydrated calcium silicate in addition to its reaction with calcium hydroxide and aluminates (Perkins, 1997). These reaction are known as double decomposition.

2.4.2.4 Magnesia (MgO)

The raw materials for cements usually contain certain amount of $MgCO_3$ which on burning dissociates to magnesium oxide (magnesia) and CO_2. The magnesia does not combine with the major oxides. Some of it is taken up in solid solution in the clinker minerals, and the remainder crystallizes as periclase MgO, the hydrate of periclase is similar to that of CaO (Soroka, 1979). Presence of too much magnesia may cause unsoundness. At the burning temperature of cement the magnesia is completely burnt and reacts with water very slowly over a period of years at ordinary temperature.

The magnesia content is limited to 4% by (BS 12). It should be pointed out that the magnesia content as such is not a good indication of potential unsoundness. As it has been pointed out some MgO is held in the solid solution by the clinker minerals and the remainder crystallizes as periclase only the presence of periclase causes unsoundness.

2.4.2.5 Sulphate (SO_3)

Most sulphates attack the hardened paste very severely. An exception is Barium Sulphate (barytes) which is almost insoluble in water and is not aggressive (Soroka, 1979). Other common sulphate likely to come into contact with concrete aside from the sulphates of magnesium and ammonium are calcium sulphate (gypsum) sodium and potassium sulphate on reaction with alumina produce sulpho- aluminate hydrate (Ettringite) in the form of needle-like crystals (Allen et al. 1993). These reaction increase the volume of the solid matrix which causes expansion. The expansion in turn causes cracking which under continuous and severe exposure may result in complete deterioration of the hardened paste. Cementitious repairs in this condition should use sulphate resisting cement, which has low Ca_3A content.

2.4.2.6 Sodium Hydroxide (caustic soda)

Solution of caustic soda up to about 10% will not adversely affect concrete but higher concentrations, particularly at elevated temperature, are likely to cause slow deterioration of concrete.

2.4.2.7 Distilled and Demineralized Water

It is surprising that very pure water should be aggressive to concrete, but this is due to the liquid's very high dissolving power. The main characteristics of this type of water are:

1) Very low calcium hardness

2) Very low dissolved solid

3) Low alkalinity

If the water contains dissolved carbon dioxide, thus bringing the pH down to below neutral point of 7, this will increase the potential for attack.

2.4.2.8 Moorland Water

Water from upland gathering grounds can have a long-term aggressive effect on concrete used to store and convey such water. The characteristics of such water are:

1) Low total dissolved solids

2) Low total hardness

3) Low alkalinity

4) Organic and other acid in dilute solution, some times sulphuric/sulphurous acids are present, derived from the breakdown of organic matter in peat and mashy ground area.

2.4.2.9 Role of Interfaces

The properties of concrete are influenced not only by properties of the constituent phases but also by the existence of their interfaces. To appreciate this we should note that the volume occupied by a properly compacted fresh concrete is slightly greater than would be the compacted volume of the aggregate which this concrete contains (Neville et al. 1994). This difference means that the aggregate particles are not in a point to point contact but are separated from one another by a thin layer of cement paste. The interface influences the modulus of elasticity of concrete. A very good interface prevent the development of MICRO-CRACKING. Under load, This micro-cracking develop progressively at interfaces, making varying angles with the applied stress.

Interfacial Effect

In the mixing and casting of the fresh concrete there are several changes, actual or potential introduced in the mix because of the characteristic of the interface. Thus, the shape of the aggregate affects the workability of the mix. There is less interference and friction between the particles of round aggregate than there is for irregular or angular aggregate which also have higher void ratio (Illston et al. 1979).

Another effect is water gain, in which sand and larger cement particles settle out from the larger stones to be replaced by finer particles and water moving up to form lenses under the stones. These become void after the concrete has hardened; i.e the proportion of entrapped air is increased with a consequent weakening of the concrete .

2.4.2.10 Sewage – Domestic and waste Effluents

Ordinary domestic sewage is not aggressive to Portland cement concrete. However, when sewage become septic due to relatively high temperatures and/or retention in sumps of pumping stations or long lengths of sewer with that gradients and low velocity, the hydrogen sulphide – sulphuric acid cycle can occur with disastrous results to any unprotected concrete above the top water line (Perkins, 1997).

The H_2S formed is converted to sulphuric acid, mainly by aerobic bacteria in the air space which has a high moisture content. The sulphuric acid thus formed attacks the cement paste in the concrete. The decomposition of the cement paste then results in the destruction of the concrete.

2.5 Alkali– Aggregate Reaction In Concrete

In general terms these reactions involve chemical interaction between alkali hydroxide which are usually derived from the cement used in the concrete and reactive components in the aggregate particles used. Research studies have shown that there are a number of different materials which produce chemical interactions in concrete which can be described as alkali-aggregate reactivity. One of these is alkali – carbonate reactivity, which is quite distinct from the alkali-silica reaction and was first described by Swenson in 1957 (Swamy, 1992). He described sections of concrete pavement in Kingstone, Ontario, as exhibiting excessive expansion which closed the joints and produced cracking of the slabs within 6 months of placing. The cracks defined roughly hexagonal areas 50-100 mm access and extended deeply into the concrete. Various types of alkali-carbonate reaction have also been reported; though not all of them appear to be expansive or deleterious. They may be classified into the following broad groups principally according to the type of reaction products they produce.

A. Carbonate reactions with calcitic limestone. Dark reaction rims develop within the margin of the limestone aggregate particles. These rims are more soluble in hydrochloric acid than the interior of the particle.

B. Reactions involving dolomitic limestone aggregate characterized by distinct reaction rims with aggregate. Etching with hydrochloric acid shows that both rim zones and the interior of the particles dissolve at the same rate.

C. Reactions involving fine-grained dolomitic limestone aggregate which contains interstitial calcite and clay. Reaction with alkalis produces a distinct dolomitised rim. Etching of reacted particles with dilute hydrochloric acid cleanly shows up a reaction rim, which in the majority of cases reported is found to be enriched in silica.

The third type of reaction appears to be the only type to produce significant expansion. The cause is not properly understood at present but Gillott, (1975) has suggested that the dedolomintisation of the crystals in the aggregate particles opens channels, allowing moisture to be absorbed on previously dry clay surfaces. The swelling caused by this absorption causes irreversible expansion of the rock and subsequent expansion and cracking of the concrete (Swamy, 1992). The reaction process is essentially one of dedolomintisation together with the production of brucite [$Mg(OH)_2$] and regeneration of alkali hydroxide and is believed to proceed in two stages, which may be expressed as follows:

$$CaMg(CO_3)_2 + 2ROH \longrightarrow Mg(OH)_2 + CaCO_3 + R_2CO_3 \qquad 2.4$$

And

$$R_2CO_3 + Ca(OH)_2 \longrightarrow 2ROH + CaCO_3 \qquad 2.5$$

Where R may represent sodium, potassium or lithium

disruption of the concrete. The expansion of individual rock particles suggests absorption of water on previously dry aluminosilicate surface in the microcrystalline portions of these rocks. This appears to be a direct relationship between the amount of micro-crystalline material, the porosity and the expansion of the concrete containing these aggregates. Complications in identifying alkali-silicate reactions of this type can arise in that alkali silicate reactions are sometimes also present in the concrete. The most common reaction between alkali hydroxides and siliceous material in the concrete aggregate is usually referred to as alkali-silica reactivity. It produces an expansive reaction product that can develop sufficient swelling pressure to crack and disrupt concrete. Typically the reaction progresses slowly so that it takes some years before expansion and damage to the structure become apparent.

2.5.1 The Alkali-Silica Reaction in Concrete

This reaction differs from the alkali-carbonate and alkali-silicate reactions in that, as a result of the reaction between the alkali pore fluids in the concrete and siliceous components of the aggregate particles, an alkali-silica gel is produced which is hydrophilic. As it absorbs moisture it increases in volume, thus generating pressures sufficient to disrupt the fabric of the concrete. The reaction may be considered to progress according to the following idealized equations.

$$4SiO_2 + 2NaOH \longrightarrow Na_2Si_4O_9 + H_2O \qquad 2.6$$

$$3SiO_2 + 2NaOH \longrightarrow Na_2Si_3O_7 + H_2O \qquad 2.7$$

However, the chemical composition of alkali-silica gel is variable and indefinite. Also, some research studies by Swamy, (1992) indicate that it is the OH^- concentration which is important to this reaction and that the alkali metal cation is only relevant in so far as it becomes incorporated into the gel.

A more satisfactory way of representing the chemical mechanism of the reaction is perhaps a two-stage process (Swamy, 1992).

(1). Acid-base reaction

$$H_{0.38}SiO_{2.19} + 0.38NaOH \longrightarrow Na_{0.38}SiO_{2.19} + H_2O \qquad 2.8$$

(2). Attack of the silicate bridges and disintegration of the silica

$$Na_{0.38}SiO_{2.19} + 1.62NaOH \longrightarrow 2Na^{2+} + H_2SiO_4^{2-} \qquad 2.9$$

In more general terms the reaction will proceed in stages, with the first stage being the hydrolysis of the reactive silica by OH^- to form an alkali-silica gel as indicated and later secondary overlapping in volume as a result (Swamy, 1992). The swelling pressure produced by the gel induces the formation of micro-cracks close to the reaction sites, and these propagate and coalesce to produce cracking within the fabric of the concrete and overall expansion of the structural element has developed these cracks provide access to the interior of the concrete and allow other deleterious mechanism to operate. Leaching by percolating water, often with the precipitation of calcium carbonate on surfaces, is common. The replacement of gel by ETTRINGITE [C_3A $3CaSO_4$ $3H_2O$] has also been observed in cracks, together with the development of secondary coarsely crystalline ettringite and secondary developments of portlandite [$Ca(OH)_2$] in the surrounding cement paste. In the majority of cases the sulphate levels in the concrete are normal, suggesting that an external source of sulphate attack has not caused the development of ettrinigite, but rather that the necessary sulphate is derived from within the concrete itself. The replacement of gel by ettrinigite also suggests that the alkali-silica reaction is well advanced before the formation of ettringite, which appears to develop prefentially in the gel (Marshall, 1995). A transport or sulphate ion along with water to the hydrating alkali-silica gel is the mechanism by which the ettringite crystals develop and grow. Their growth in fine micro-cracks and pores in the cement paste may exert sufficient presences within the concrete fabric to contribute to the observed expansion.

2.5.2 The Alkali Requirement in Alkali-Silica Reaction

The great majority of cases of concrete structures reported as showing deterioration due to alkali silica reaction were made using a high alkali cement content. Ordinary Portland cement will normally contain a small proportion of sodium and potassium present as sulphate and double sulphate (K, Na) SO_4, which tend to coat other clinker minerals, and also as minor constituents in the other cement minerals.

These alkalis are derived from raw feed materials used in the manufacture of the cement, usually the angillaceous fractions and the coal [if this is used for firing the kiln]. If the argillaceous material used as raw material contains mica or illicit clay, then clinker produced will be enriched in potassium, while if degraded feldspar is present the clinker may contain more sodium, potassium or both depending on the composition of the feldspar in the raw feed (Swamy, 1992). The final proportion of the alkalis present in a clinker will be dependent on the proportions of mica, illite or feldspar in the feed and upon the details of the cement manufacture in the particular plant concerned. The alkali phase tends to be a volatile fraction in the kiln environment, and some 50% will be volatilized during the burning process. Much of this alkali is redeposited in the chain section of the kiln and in the preheaters, dust precipitators and filters. In the interests of fuel economy and clean flue gases, recirculation's of the dusts is common practice in modern plants, but this procedure has an adverse effect on the alkali composition of the cement clinker.

In order to assess the total alkalis present in a cement or concrete in terms of sodium equivalent. This correlates the sodium and potassium oxides in terms of molecular proportions. The calculation of sodium equivalent is as follows:

$$\text{Sodium equivalent} = \text{wt\% } Na_2O + 0.658 \text{ wt \% } K_2O. \qquad 2.10$$

The chemical determination of these alkalis in samples of cement or concrete normally follows the nitric acid extraction procedure as outlined in BS 4550: Part 2 (1970), clause 16.2. In the analysis of concrete samples, it has been suggested that the acid extraction method is likely to give alkali concentrations higher than normally readily available for Alkali-Silica reaction in the concrete because additional alkalis may be extracted by the acid from aggregate minerals such as feldspars (Swamy, 1992). Although this may be correct it is generally considered that the acid extraction method is appropriate, and account is taken of the probability that it will tend to give a maximum figure for total alkalis in engineering judgments. The alkali-silica reaction cannot proceed in a concrete if the alkali concentration is below a certain threshold value.

Although the principal source of alkalis in concrete is the cement used, the addition of alkali to the concrete from other sources should not be overlooked. Normally, the mix water will contain significant alkali concentrations. However, where there is a possibility that sodium chloride will be incorporated into the mix, for example by the use of seawater, the alkali contributed by the sodium chloride should be incorporated into calculations for total alkali contents. There are recommendations that (Swamy, 1992), if alkalis from sources other than the cement exceed 0.2 kg/m^2 of the concrete, they should be taken into account when calculating the total reactive alkali available. Some mineral constituents of the aggregates, notably the micas, feldspar and illicit clays, contain alkali metal cations in their structure.

Opinions are divided as to whether alkali from these minerals become available for reaction within the concrete or are too firmly held within the crystal lattice (Swamy, 1992). It seems likely that where the aggregate have suffered a degree of geological weathering before being used as aggregate, the partial degradation of these minerals from this cause will facilitate the leaching of at least some of the alkali into the pore fluids of the concrete.

In addition to alkalis derived from the constituents of the concrete, consideration should be given to the absorption of alkalis by hardened concrete in contact with seawater, some ground water and other materials, such as de-icing salts. The adsorption of alkalis from such sources by a concrete will of course, depend primarily on the porosity and permeability of the concrete, and the length of time and the nature of the exposure to the alkali-containing fluids. Consideration must also be given to the mechanism during the adsorption process. An example of certain alkali-silica reactive concrete plinths for electrical transformers and other electrical power supply equipment in South-West England (Moore, 1978) is interesting in that detailed investigations of the alkali concentration of the affected concretes showed the top of the plinths to contain up to four times as much total-alkali as the lower parts which were in contact with moist ground. Two explanations for these observations have been purposed. The first is that moisture movement from the moist lower surfaces to the wind and sun dried top surfaces accounts for this alkali migration. An alternative or possibly complementary mechanism involves the electrical potential difference between the two faces of the slab induced by the electrical installations. The alkalis migrate through the pore fluid electrolyte to concentrate in the upper layers of the plinth.

2.5.3 Nature of Pore Fluid in Concrete

Since alkali aggregate reaction occurs in wet environments, it is not necessary to consider reactions in dry cement concretes, although the effects of periodic wetting and drying may be required to be assessed. When concretes are first mixed, their water-cement (w/c) ratio is an important design parameter. Typically, this may lie in the range 0.35 – 0.55. However, Swamy (1992) has suggested that the amount of water required for full hydration of the cement, calculated as w/c ratio, is substantially less about 0.24, therefore, water is normally present in well-cured concrete. A well-cured concrete thus consist of three phase: aggregate, together with unreacted blending agent (if present), cement hydration product and an excess of an aqueous phase.

During the initial stages of hydration the aqueous phase is most abundant, and it is more or less continuous in the sense that it wets the solid-phase grains. However, as hydration progresses the space occupied by the aqueous phase become increasingly filled with hydration products, such as C-S-H and Ca $(OH)_2$ and the remaining aqueous phase gradually becomes discontinuous. Cement paste itself is intrinsically porous, and considerable space remains to accommodate this fluid, hence termed PORE FLUID (Swamy, 1992). A paste made to very low w/c ratios may have little porosity in the range above 1 – 2 μm diameter (unless special steps are taken to entrain air), but much micro porosity normally still remains in the minor 1 – 2 μm diameter range, Ordinary concrete made to higher w/c ratios thus contain macro pores and, in the paste, micro-and meso pores which afford intimate contact between the pore fluid and hydration products as well as with aggregate particles. In mortars and concretes, pore fluid may also segregate at the aggregate cement hydration product interface, especially when mixed to w/c ratios above 0.35-0.4.

Pore fluid thus serves as a bridge between cement hydration products on one hand and aggregate on the other. Material transport or soluble species, such as alkali, SO_3, Cl ions e.t.c, occurs readily through the pore fluid. In principle, transport can also occur across solid-solid interfaces for example, between solid hydration products and aggregate but such reactions occur at a slower rate than through an aqueous phase, hence the importance of the latter in conditioning alkali aggregate reaction.

2.5.4 Chemical Mechanism of Alkali – Aggregate Reaction

Aggregate particles normally consisting of SiO_2, are thermodynamically unstable in the cement environment, as a result of reaction leading to a lowering of the free energy of the system. This reaction, or series of reactions, are accompanied by mass transport of OH^- and alkali ions. The pore fluid is in intimate contact with cement hydration products as well as with aggregate particles and it serves as the main agent of transport.

The details of reaction can be seen in more detail in Appendix B. The complex nature of aggregate surfaces and cement paste micro-structure shown in the figure (not drawn to an exact scale) is meant to convey the impression that many of the relevant events occur on an atomic scale (Swamy, 1992). The cement micro-structure is shown: it consist of solid hydration products, of which the drawing shows two features-platelets of $Ca(OH)_2$ and bundles of fibers or lath-like C-S-H structure. The hydration products are not normally space-filling, so considerable pore volume exist as meso and micro-pores. The microspores are not shown, but water-filled mesopores are suggested by areas of water molecules, depicted as H-O-H.

The surface of a silicious aggregate particle is also shown. Normally, its surface oxygen are hydroxylated, even in pore water, surface studies show that this disturbed region is normally several atoms or even tens of atoms deep. When aggregate are placed in a hydroxyl-rich medium, their potential to undergo further hydroxylation is enhanced. With well-crystallized quartz, this potential for further reaction exists, but the rate of hydroxylation is so slow as to be almost imperceptible on the normal engineering time scale. Temperature markedly accelerates the reaction: in hydrothermal conditions, finely ground but crystalline quartz is very reactive (Swamy 1992). However, susceptible aggregates typically react much more rapidly than crystalline quartz, even at normal ambient temperatures. A gel-like layer of indefinite constitution forms at the aggregate-cement paste interface. As this gel forms it imbibes water and soluble ionic species, principally Na^+, K^+ and Ca^{2+}. The gel is not very soluble, and generally concentrates around the margins of susceptible silicious aggregate particles, but softer gels may also be exuded by mechanical swelling pressures.

Thus three reactions occur. In the first stage the high pH fluid reacts with Si-O-Si bonds to form silanol bonds.

$$\mathrm{Si-O-Si + H_2O \longrightarrow Si{-}OH\text{---------}OH{-}Si} \qquad 2.11$$

Hydrous silica aggregates may already contain substantial silanol bonding. Those silacol groups are acidic in the Lewis definition, and participate readily in further reaction with basic cement pore fluids (Dunster et al, 1980). Thus, four-coordinate silicon with one coordination site already occupied by OH may be abbreviated as Si-OH, in the second stage it reacts further with hydroxyls:

$$Si-OH + OH^- \longrightarrow Si-O^- + H_2O. \qquad 2.12$$

Liberating more water in the process, the negatively charged $Si-O^-$ species attracts positive charges, mobile, readily abundant species such as sodium, potassium and calcium diffuse into the gel (Swamy, 1992) in sufficient numbers to balance the charge on the negatively charged grounds. Dent-Glasser, and Kataoka, (1981) represented the approximate stoichiometry as.

$$2H_{0.38}\,SiO_{2.19} + 0.38Na_2O \longrightarrow 2Na_{0.38}SiO_{2.19} + 0.38H_2O. \qquad 2.13$$

Where the charge composition is achieved by Na_2O although the implication is that other cations may also precipitate.

In the third stage more siloxane $Si-O-Si$ bridges are attacked.

$$Si-O-Si + 2OH^- \longrightarrow Si-O^- + O-Si + H_2O \qquad 2.14$$

At the limit of the process, some silica may pass into solution at high pHs the principal soluble silicate species is $H_2SiO^2{}_4$.

The gel itself has a significantly greater specific volume than that of the SiO_2 which it replaces and it is this which creates swelling pressures and expansion which are so characteristic of the alkali aggregate reaction. The extent of the swelling is difficult to predict, silica gels have water contents and densities which are variable over wide ranges, and the additional possibilities for imbibitions of water (Swamy, 1992), Na^+, R^+ e.t.c. cause its density and hence specific volume to vary. Silica gels may maintain a local steady state, quasi equilibrium with the local pore water. If the local pore water is diluted, as may occur by percolation of fresh water, the gel may spontaneously lower its ionic content during drying cycles. When local salt concentrations tend to increase in the pore fluid, the reverse reactions occur. The limits of wet-dry cycling are clearly important, because in structures cycling may induce high local concentrations of alkali and hence localized alkali aggregate attack, even through the mean alkali content of the bulk concrete may remain relatively low.

The absorbed properties associated with gels are sometimes confused with osmosis. It is true that the gel product of alkali aggregate attack is semi-permeable, and that it contains substantial alkali, calcium e.t.c., but it is not the alkali which initiates reaction. Rather it is the local concentration of OH^- ions and specific susceptibility of the aggregate particle that condition the potential for reaction. Osmosis as such, plays at most a minor part in the process. Imbibitions is the appropriate physical chemical process which governs the gel shrink swell properties (Swamy, 1992).

The indefinite constitution of gel-like reaction product has a number of consequences. Gels vary in physical properties, mainly as a function of their water content, from hard rigid materials to plastic, readily extendable substances. Examination of most real examples of alkali aggregate reaction shows that this is probably a feature of most real reactions, hard rigid gels are most likely to cause expansive force leading to cracking, while the more fluid, watery gels are readily extruded into cracks and may eventually emerge as exudates; soluble silicates species also migrate and subsequently reprecipitate, mainly in zones where local pH is lowered as occurs during carbonations (Ballum and Alexander, 1980).

CHAPTER THREE

3.0 METHODOLOGY

Samples were collected from non industrial areas of Kaduna, the capital of Kaduna State in Nigeria , specifically from the Kurmi Mashi I ,& II and Ungwan Mua'zu I &II areas. The structures are located in abandoned market site constructed five and six years ago respectively .Samples were collected from three levels on both sides across and along the cracked line. The control samples were also collected from structure located in the same areas . Additional experiments were conducted on local indigenous cement , ground soil and sand commonly used in the production of sandcrete blocks in the area. The experimental methods used were the titrimetric , gravimetric and instrumental .All the experiment were conducted as specified in the BS 4550 part 11 (1970), BS 1377 part 3 (1990) and ASTM 114-67 (1970) .The analysis were carried out to determine the percentage content of SiO_2 , CaO, MgO, Al_2O_3, Fe_2O_3, Na_2O and Loss on Ignition (LOI) in the samples.

3.1 CHEMICAL ANALYSIS OF SAMPLE

The sample was crushed and ground; it was then sieved on a #120 British mesh sieve. 1.00g of the powdered sample was thoroughly mixed with anhydrous sodium carbonate in a platinum crucible and was fused in the furnace at 1000°C for 90mins. It was then allowed to cool. Dilute HCl was used to loosen the cake formed from the crucible. The resulting solution was filtered. The residue was dried in the oven and treated as silica. The filtrate was treated with hydrogen peroxide and precipitated with dilute ammonia. The precipitate was filtered and collected as ammonia group of oxides [R_2O_3]. The filtrate was further used for the determination of calcium oxide and magnesium oxide (Besset et al. 1978).

3.2 Determination of Silica

The residue mentioned in section 3.1 was dried in the oven for silica determination. The sample placed in a platinum crucible weighed W_1. When burnt in the furnace at 1000°C for 1hr and was cooled in a dessicator it weighed W_2. The SiO_2 obtained contain small impurities which was treated in the crucible with 1 or 2 ml of water, 2 drops of dil. H_2SO_4 and 20 ml of HF and continuously evaporated to dryness. The Residue was heated at 1000°C for 30mins, cooled and weighed W_3. The difference between this weight and weight previously obtained represents the weight of SiO_2.

Calculations:

$$\text{Silica (\%)} = \frac{(W_1 - W_2) - W_3}{W} \times 100 \qquad 3.1$$

Where W_1 = weight of silica and insoluble impurities (grams)

W_2 = weight of impurities (grams)

W_3 = weight of silica recovered from Iron and Aluminum Oxide (grams)

W = weight of sample taken (grams)

3.3 Determination of Combined Ferric Oxide and Alumina

5 ml of HCl was added to the filtrate reserved in section 3.1 after separating silica. Few drops of methyl red indicator was added and treated with NH_4OH drop wise until the colour of the solution becomes distinctly yellow, a drop is added in excess for confirmation.

The solution containing the precipitate was heated to boiling for 5 minutes. This was filtered using medium textured filtered paper and hot solution of ammonium nitrate (20 g/l) was used in washing the precipitate for at least 2 – 3 times.

The filtrate was set aside and the precipitate was transferred with the same beaker where the first precipitation was effected. The precipitate was dissolved by using hot HCl and stirred by thoroughly macerating the paper. The solution was diluted to about 100 ml and re-precipitation was carried out as done previously. The solution was filtered and the precipitate was washed with about four 10 ml portion of hot NH_4NO_3 (20 g/l) solution.

The precipitate was placed in a weighed platinum crucible and the paper was slowly heated until it charred. This was finely ignited to constant weight at 1050 to 1100 ^{O}C. The ignited residue was weighed as the combined oxide and Alumina known as R_2O_3.

Calculation

$$R_2O_3\ (\%) = \frac{S_i \times 100}{W} \qquad 3.2$$

Where S$_i$ = weight of crucible (grams)

W weight of sample taken (grams)

3.4 Determination of CaO

10 ml of the aliquot was taken from the R_2O_3 filtrate in 250 ml volumetric flask, 40 ml of distilled was added followed by the addition of 10 % KOH, 3 ml of hydroxyl ammonia chloride, few drops of mixture of KCN and calcium as indicator was then titrated with 0.02M of EDTA.

The CaO was determined from:

$$\%CaO = \frac{100 \times 0.02\text{MEDTA} \times \text{Titre Value} \times \text{MM}_{\text{CaO}} \times \text{Vol. Of Stock Soln.}}{\text{Wt. of sample} \times \text{vol. Of aliquot}} \qquad 3.3$$

3.5 Determination of MgO

10 ml of the aliquot was taken from the R_2O_3 filtrate in the 250 ml volumetric flask, 40 ml of distilled water was added, followed by the addition of 30 ml of magnesium buffer, 2-3 drops of mixture of KCN and magnesium as indicator was then titrated with 0.02M of EDTA.

The MgO was determined from:

$$\%MgO = \frac{100 \times 0.02\text{MEDTA} \times \text{Titre Value} \times \text{MM}_{\text{MgO}} \times \text{Vol. Of Stock}}{\text{Wt. of sample} \times \text{vol. Of aliquot}} \qquad 3.4$$

3.6 Determination of Fe_2O_3

The solution (100 ml) from silica filtrate (made up to 250 ml) was cooled to $10^{O}C$ and a freshly prepared and filtered 5ml aqueous cupferron solution added. These were rigorously stirred until no further formation of brown precipitate. After stirring, this was filtered through Whatmann No. 41 filter paper and the precipitate was washed several times with 10 % by volume of HCl containing 1.5 g of cupferron per litre and the precipitate was washed twice with 5 M ammonia to remove excess cupferron and finally washed once with water.

The precipitate and filter paper was transferred to a weighed crucible and ignited at $1000^{O}C$ to a constant weight. The ignited residue was weighed as Fe_2O_3.

The Fe_2O_3 was determined from:

$$Fe_2O_3\ (\%) = \frac{\text{Weight of precipitate}}{\text{Weight of sample}} \times 100 \qquad 3.5$$

3.7 Determination of Alumina (Al_2O_3)

The percentage Al_2O_3 was calculated by deducting the percentage of Fe_2O_3 from the percentage of combined ferric oxide and Alumina i.e.

$$Al_2O_3 = R_2O_3\ (\%) - Fe_2O_3\ (\%). \qquad 3.6$$

3.8 Determination of Na_2O and K_2O

0.1 g of the powdered sample was taken into a teflon beaker, then 10 ml of HF and 4 ml of perchloric acid was added and heated on a sand bath. Warm distilled water was added and filtered into 100 ml plastic absorption spectrometer for the determination of sodium oxide. The same digested sample was used for the determination of K_2O using A.A.S. Sodium hollow cathode lamp and potassium hollow cathode lamp were put in the A.A.S. at the time of the determination respectively. Working standard solution of sodium in the range 0.4 – 1.4 ppm and potassium in the same range were prepared for use in the A.A.S.

$$\text{ppm} = \frac{\text{conc. x Dilution factor x Volume of stock soln}}{\text{Wt of sample}} \qquad 3.7$$

3.9 Loss on Ignition

1.00 g of sandcrete samples was weighed in a platinum crucible at a temperature of 25 °C. This was heated in a muffle furnace at temperature between 900 – 1000 °C, cooled and weighed (W_1). The loss in weight was checked by a second heating at same temperature for 5 minutes and the content reweighed. This was repeated until a constant weight was attained (W_2). The loss on weight was recorded as the loss on Ignition. The percentage loss on ignition was calculated thus:

$$\Delta W = W_1 - W_2 \qquad 3.8$$

$$\%\ \text{loss of ignition} = \frac{\Delta W}{W_1} \times 100 \qquad 3.9$$

Where, W = weight of sample taken (gram)

3.10 Determination of SO_3

1.00 g of sandcrete sample was taken, 25 ml of cold water and 5 ml of HCl was added while the mixture was stirred vigorously. The solution was heated for complete decomposition of sample and diluted to 50 ml. The solution was digested for 15 minutes at a temperature just below boiling point. This was filtered through a medium-texture paper and washed thoroughly with hot water. The filtrate was diluted to 250 ml and heated to boiling, 10 ml of hot $BaCl_2$ (100 g/l) was added and boiling continued until the precipitate was well formed. The solution was digested for 8 hours at temperature just below boil and the volume of solution was maintained between 225 – 260 ml by adding water when necessary.

The solution was filtered through a retentive paper and washed thoroughly with hot water. The filter paper containing the precipitate was weighed in a platinum crucible and charred without inflaming and finally ignited at a temperature of 800-900 ^{0}C until constant weight was attained, it was cooled in a desiccators and weighed as SO_3

The SO_3 was determine from:

$$SO_3(\%) = \frac{\text{Weight of Ignited Residue} \times 34.3}{W} \qquad 3.10$$

Where, W = Weight of the sample (gram)

Conversion factor for $BaSO_4$ to Sulphate (SO_3) = 0.343

3.11 Determination of the pH value

Initial sample was obtained and air dried by spreading out on a tray exposed to air at room temperature. The sample was sieved through 2 mm test sieve. The material passing through the 2 mm test sieve was divided by successive riffling through 15 mm divider which produce a representative test of 30 g to 35 g.

30 g of soil sample was placed in a 100 ml beaker, where 75 ml of distilled water was added and stirred for few minutes which then covered allowed to stand for 8 hours. The suspension was stirred before testing, the pH meter was calibrated by using standard buffer solutions, following the procedure recommended by the manufacturer. The electrode was washed with distilled water and was immersed in the soil suspension. Three readings of the pH of the suspension were taken with brief stirring between each reading. The electrode was removed from the suspension and washed with distilled water. The pH meter calibrations were re-checked against one of the standard buffer solutions (BS 1377 – Part 3 1990).

CHAPTER FOUR

4.0 EXPERIMENTAL RESULTS

The results obtained from the experimental work are as follows:

Table 4.1: Chemical Composition of Sandcrete Samples from Cracked Zone I

	Percentage Composition (%)						
		Ground level		Middle level		Top level	
Compound	Ground soil	Left side	Right side	Left side	Right side	Left side	Right side
SiO_2	51.76	78.49	78.48	77.00	77.10	76.72	76.80
CaO	1.50	4.04	4.05	5.56	5.55	5.98	5.96
MgO	0.19	0.18	0.18	0.16	0.162	N.D	N.D
Al_2O_3	21.43	3.55	3.53	3.02	3.00	2.98	2.98
Fe_2O_3	8.98	3.09	3.06	2.99	2.97	2.95	2.95
Na_2O	0.75	0.68	0.67	0.77	0.77	0.81	0.80
K_2O	1.82	0.435	0.435	0.423	0.422	0.391	0.393
SO_3	0.01	0.145	0.149	0.151	0.153	0.161	0.161
LOl	13.56	9.44	9.44	9.89	9.83	10.01	10.00

ND = Not detected

Ground soil pH = 7.11

Table 4.2: Chemical Composition of Sandcrete Samples from Cracked Zone II

		Percentage Composition (%)					
		Ground level		Middle level		Top level	
Compound	Ground soil	Left side	Right side	Left side	Right side	Left side	Right side
SiO_2	49.41	78.5	78.63	78.63	78.70	77.52	77.52
CaO	1.42	4.10	4.09	4.00	4.01	5.49	5.49
MgO	0.20	0.16	0.16	0.15	0.15	0.001	N.D
Al_2O_3	22.91	3.36	3.31	3.08	2.99	2.86	2.85
Fe_2O_3	9.57	2.98	2.98	2.87	2.88	2.73	2.74
Na_2O	0.71	0.68	0.69	0.77	0.77	0.81	0.81
K_2O	1.71	0.423	0.424	0.412	0.411	0.393	0.393
SO_3	0.05	0.151	0.15	0.156	0.155	0.160	0.161
LOI	13.76	9.57	9.56	9.93	9.93	10.04	10.03

ND = Not detected

Ground soil pH = 7.13

Table 4.3: Chemical Composition of Sandcrete Samples from Cracked Zone III

		Percentage Composition (%)					
		Ground level		Middle level		Top level	
Compound	Ground soil	Left side	Right side	Left side	Right side	Left side	Right side
SiO_2	73.42	80.30	80.30	79.40	79.40	79.30	79.30
CaO	3.08	3.83	3.82	4.25	4.26	4.44	4.44
MgO	0.60	0.17	0.17	0.15	0.14	N.D	0.002
Al_2O_3	6.15	3.02	3.00	3.00	3.01	2.98	2.97
Fe_2O_3	3.99	2.98	2.98	2.94	2.95	2.89	2.88
Na_2O	0.85	0.64	0.65	0.68	068	0.70	0.71
K_2O	1.98	0.425	0.426	0.414	0.414	0.400	0.401
SO_3	1.13	0.153	0.154	0.16	0.16	0.165	0.167
LOI	9.31	8.57	8.44	8.98	8.98	9.13	9.12

ND = Not detected

Ground soil pH = 5.89

Table 4.4: Chemical Composition of Sandcrete Samples from Cracked Zone IV

		Percentage Composition (%)					
		Ground level		Middle level		Top level	
Compound	Ground soil	Left side	Right side	Left side	Right side	Left side	Right side
SiO_2	72.89	79.67	79.65	79.67	79.64	79.29	79.30
CaO	3,.18	3.92	3.92	4.10	4.11	4.35	4.36
MgO	0.62	0.18	0.18	0.16	0.16	0.002	0.001
Al_2O_3	6.24	3.32	3.33	3.23	3.23	3.33	3.31
Fe_2O_3	3.97	2.79	2.79	2.69	2.70	2.61	2.61
Na_2O	0.82	0.71	0.72	0.75	0.75	0.801	0.805
K_2O	1.82	0.35	0.349	0.341	0.342	0.34	0.34
SO_3	0.92	0.154	0.155	0.16	0,1.6	0.175	0.i73
LOI	9.58	8.89	8.90	8.90	8.91	9.10	9.10

Ground soil pH = 6.11

Table 4.5: Chemical Composition of Sandcrete from Uncracked Zone

	Percentage Composition (%)		
Compound	Ground level (%)	Middle level (%)	Top level (%)
SiO_2	80.80	80.10	80.00
CaO	4.49	4.87	5.02
MgO	0.542	0.501	0.492
Al_2O_3	4.15	4.02	3.98
Fe_2O_3	3.19	3.14	3.00
Na_2O	0.74	0.78	0.80
K_2O	0.54	0.53	0.49
SO_3	0.170	0.172	0.169
LOI	5.38	5.85	6.01

Table 4.6: Chemical Composition of Ashaka Cement (Typical Indigenous Cement Brand)

	Percentage Composition
Compound	Ashaka (%)
SiO_2	26.03
CaO	62.00
MgO	0.82
AL_2O_3	3.80
Fe_2O_3	2.4
Alkalis	1.05
SO_3	1.65
LOI	2.25

Table 4.7: Chemical Composition of Sharp Sand from Kaduna Used Predominantly for Sandcrete Block Production and Mortar.

	Percentage Composition
Compound	Sand (%)
SiO_2	81.63
CaO	1.68
MgO	0.52
AL_2O_3	4.89
Fe_2O_3	3.20
Na_2O	0.37
K_2O	0.45
SO_3	0.03
LOI	7.23

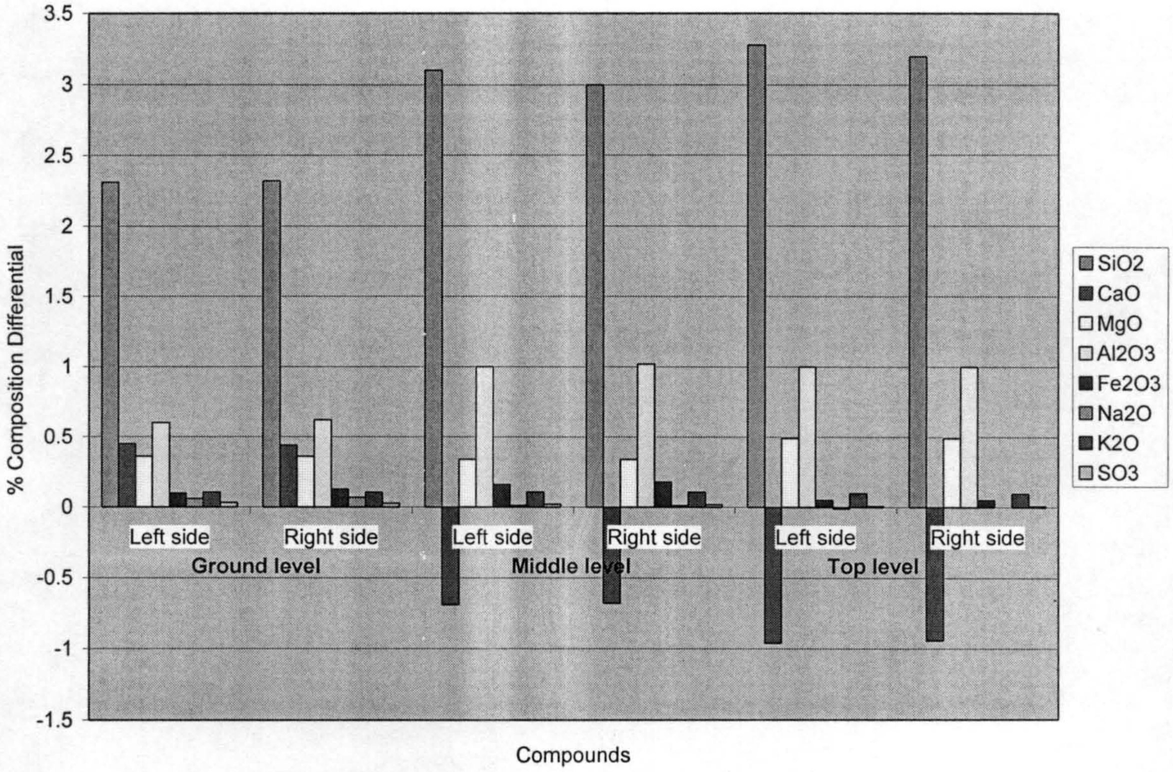

Figure 4.1: The Chemical Composition % difference between Table 4.1 and 4.5

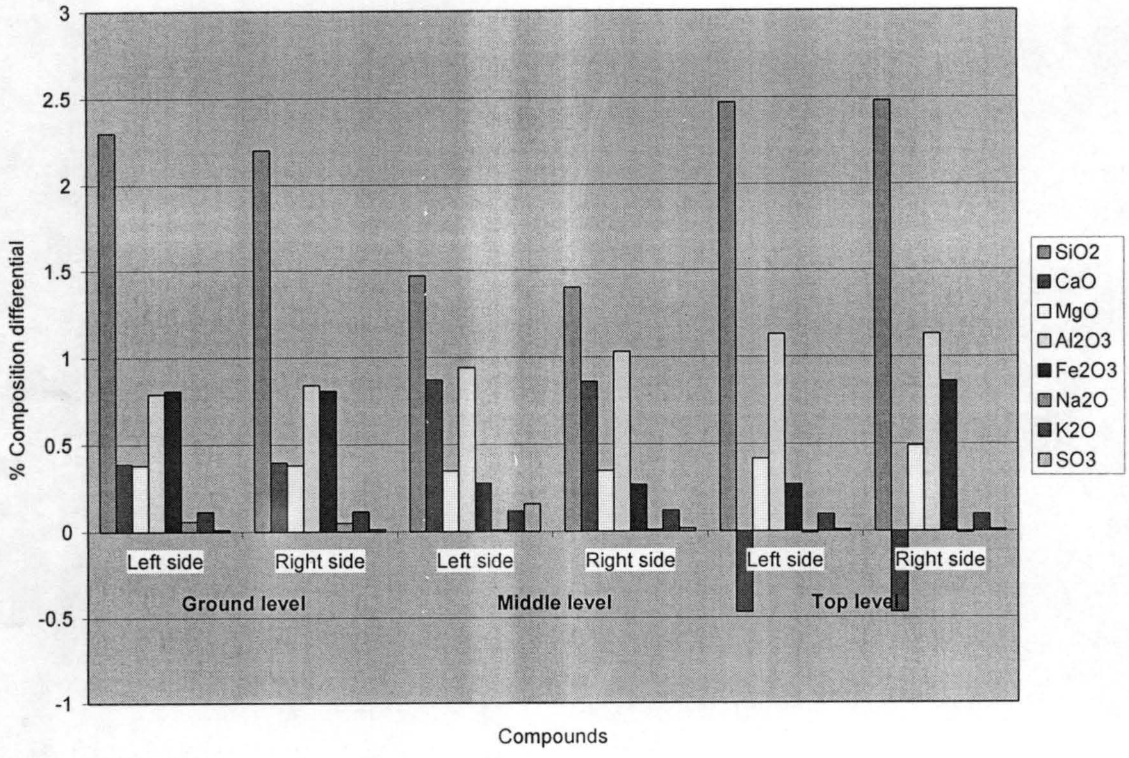

Figure 4.2: The Chemical Composition % difference between Table 4.2 and 4.5

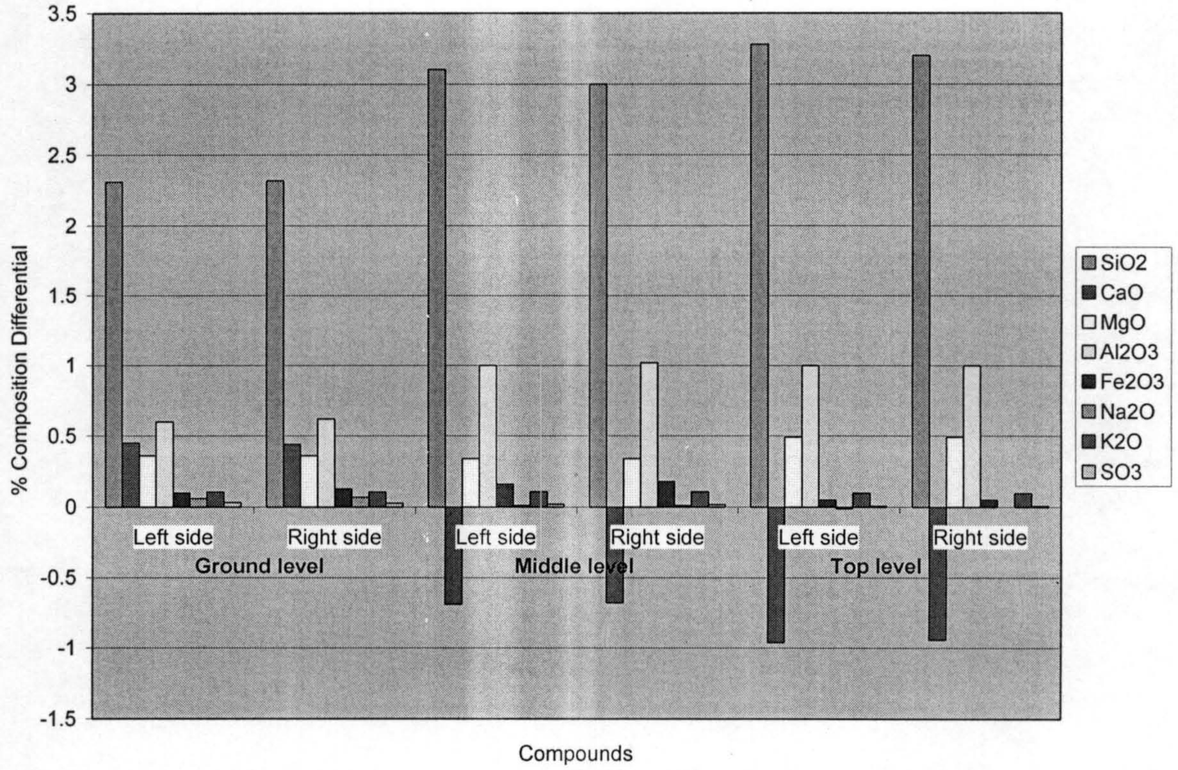

Figure 4.1: The Chemical Composition % difference between Table 4.1 and 4.5

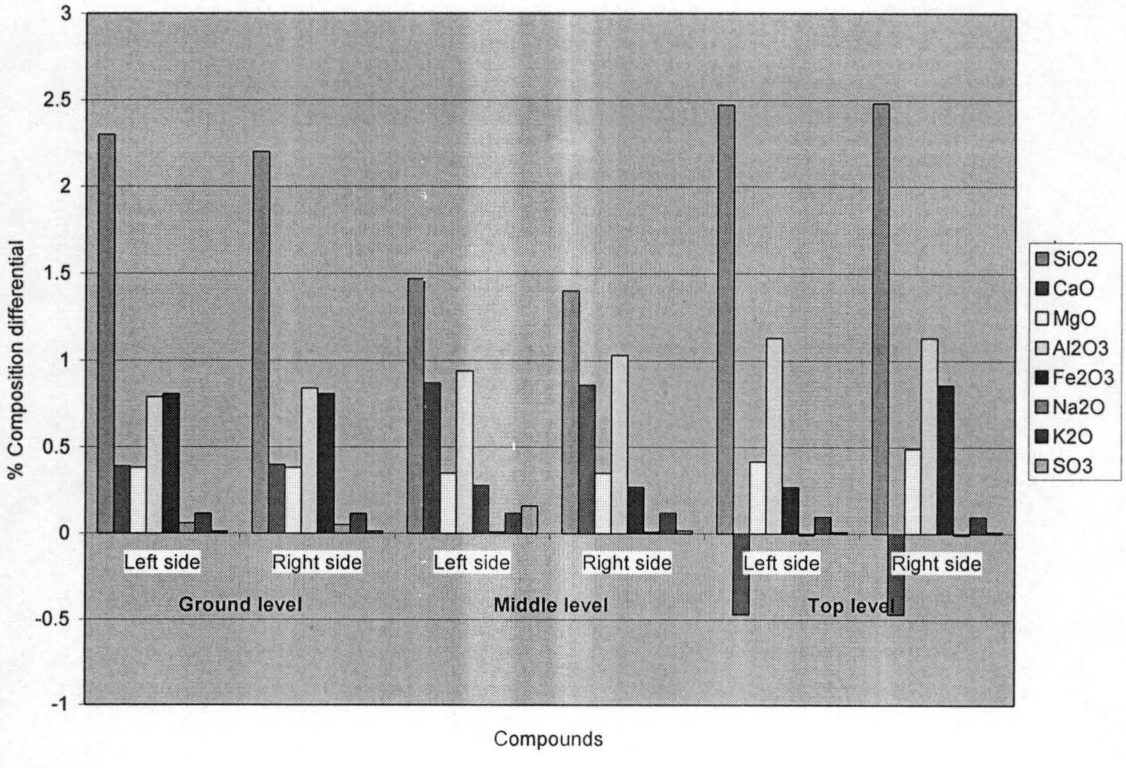

Figure 4.2: The Chemical Composition % difference between Table 4.2 and 4.5

4.1 DISCUSSION

Experimental results presented in Table 4.1-4.7 show the percentage compositions of the various based structures. The two ground soil sample gotten from the area in Ungwan Mu'azu Kaduna representing Zone III and IV of Table 4.3 and 4.4 respectively were found to be humus in nature. The data presented in Table 4.1 shows that the percentage compositions of SiO_2 is relatively high in all the samples varying from 78.49% in ground level to 76.72% of top level of the left hand side of the crack line, Table 4.2-4.5 follow the same patterns.

This pattern of high SiO_2 concentration in all levels is a prerequisite to high permeability as such moisture within the matrix of the sandcrete carries some of the load when under stress. Gradual dissipation of the stress in water would lead to time dependent strain which would be observed as creep and strain for wet sandcrete as opposed to dry sandcrete The loss of this water by drying causes reduction in the Elasticity of Young Modulus (Illston et al, 1979). As a result cracks are liable to form under pressure.

pH tests carried out on ground soil of Table 4.3 and 4.4 shows that the dark and sandy soil is humus in nature and slightly acidic with pH 5.89 and 6.11 respectively. In the presence of moisture and long exposure of the structure to soil water, the acids could react with cement mineral hydrate especially

$Ca(OH)_2$ to produce soluble calcium salt and water. The leaching of the resultant products from the hardened matrix exposes it to further attack, leaving behind a weak structure. It is also well documented that such pH considerably enhances metal reinforcement corrosion.

The data in Table 4.1 to 4.5 shows that the concentration of sodium oxide (Na_2O) in the samples was highest at the top level compared to the middle and ground level. This could be attributed to high moisture diffusivity due to wind and sun dried top levels which accounts for this alkali migration as was observed by Swamy (1992); leaving the lower level with low alkali concentration. The presence of high alkali at the top level of a building would pave way for alkali silica reaction; this is when a reactive siliceous component of the aggregate reacts with alkali to produce a gel which is hydrophilic. On absorption of moisture, the gel increases in volume, thus generating pressure sufficient to disrupt the fabric of the sandcrete structure.

From Tables 4.1 to 4.4, the percentage content of calcium oxide (CaO) at the ground level was always the lowest while the top level was the highest. It varied from 3.83 % at ground level to 4.44 % at the top level. This difference could be attributed to gradual leaching of the mineral hydrates from the structure Matrix, or reaction of CaO with ground H_2O to produce additional $Ca(OH)_2$, which can react with acidic ground water. The leaching process leaves behind a spongy matrix and enhances ground water diffusion into the structure. Figures 4.1 to 4.4 show that when compared to the uncracked wall shows that the CaO content was relatively low. The cracking process might have also be enhanced by the poor aggregate cement ratio used for the production. With this, the cement content could not ensure proper binding of the fine aggregates. Consequently the physico-mechanical properties of the sandcretes produced would be very poor.

The percentage composition of Al_2O_3 and Fe_2O_3 generally followed a decreasing order from ground to top level, specifically, in Table 4.1; Alumina at ground level is 3.55% of the left side declining to 2.98% at top level left side. In the same Table, Fe_2O_3 reads 3.09% on ground level of left side declining also to 2.95% at the top level left side. In Table 4.2 to 4.4, alumina and ferrous concentration follows the same pattern. All this conformed to the standard presented by Neville et al (1994). But the difference noticed in percentage concentration of ground to top level might be attributed to improper mixing of the aggregate/water/cement ratio, which can cause sliding of individual molecules or group of molecules past one another resulting into slippage, and then cracks are formed. Figure 4.1 to 4.4 shows that alumina and ferrous contents differ from the standard sample value for the ground. Middle and to top level.

The ground soil of Table 4.1 present percentage composition of alumina, and ferric oxide to be 21.43 % and 8.98 % respectively, this is because the soil is clayish in nature. Table 4.2 follows the same pattern. The ground soil of Table 4.3 also present alumina and ferrous to be 6.15% and 3.99% respectively, in this case the soil is humus.

From all the Tables 4.1 to 4.4, magnesia was found to be minima with majority of it coming with the sand. Specifically Table 4.1 shows that MgO at ground level is 0.18% and not detected at the top level. Table 4.6 shows that the percentage concentration of the cement merges well with NIS 367-2 1997.

The data presented in Table 4.7 shows that the sand percentage compositions [SiO_2 is 81.63:% calcium oxide is 1.68 % followed by Al_2O_3 and Fe_2O_3 which are 4.89 and 3.20 % respectively] were within limit (ILO, 1987).

In Table 4.1 and 4.2, the result for loss on ignition (LOI) at top levels left side vary from 10.01% and 10.04% respectively, this loss of impurities could be sufficient enough to cause plastic deformation in amorphous materials, because impurity atoms occupies a lattice site normally occupied by an atom of the host lattice (Srivastava, 1991), which result to crack formation.

As temperature is raised material under load cause no permanent deformation at room temperature starts to creep. Its shows that deformation continues with time. Here up North, where temperature is generally high, thermal cracking may arise from great temperature differential through the hardened sandcrete and the ambient air temperature as observed by Yvonne (1996). It also affects the Elastic of Modulus of hardened cement paste as higher temperature gives lower Elastic Modulus. The temperature dependence may well be related to the increased mobility of the moisture at higher temperature, giving some loss of stiffness in the solid structure.

In conclusion, to avoid weakness of sandcrete structures ,usage of high quality cement and also adhering to standard cement/sand ratio. Secondly, choice of location for construction should be considered to avoid building on acidic ground soil. Lastly, choice of aggregates can greatly minimize cracks at the interface when angular aggregates are used, rather than smooth and rounded aggregates.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

Based on the chemical analysis conducted on various samples from different locations, the following points were drawn.

1. Percentage composition of SiO_2 is high in all the sandcrete samples varying from 80.3 % at the ground level declining steadily upward to 79.3 % at the top level. This indicates high permeability of ground level structure a prerequisite for crack development.
2. The slightly acidic nature of the soil at Ungwa Muazu I and II (pH 5.89 and 6.11 respectively) could seriously affect the structure strength as a result of acidic-alkaline reactivities. These would enhance the development of cracks.
3. The concentration of Na_2O was lowest at the ground level and rises steadily to the top level. This high concentration of Na_2O might initiate alkali silica reaction and consequently leads to the formation of cracks.
4. The percentage concentration of calcium oxide is very low ranging from 3.83 % at the ground level to 4.44 % of the top level. This might be as a result of reaction between CaO and ground H_2O to produce additional $Ca(OH)_2$ or poor quality cement used and probably non-compliance to sand/cement standard ratio.

5. Difference in composition of some oxides such as Fe_2O_3 taken from the same strata might be due to improper mixing. This enhances material slippage and consequent formation of cracks.

RECOMMENDATION

The following views are recommended for further research on this subject;

1. Analysis based on X-ray diffraction (XRD), X-ray flouresence (XRF) and Atomic Absorption Spectrophotometer (AAS) should be used as a comparative tool to titrimetric and gravimetric method, this would give and accurate judgment of result.
2. Electro microscopic investigation of thin layers should be looked into.
3. The progress of cracking can also be detected by ultrasonic and acoustic emission tests.

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APPENDIX A

CALCULATION OF CHEMICAL COMPOSITION OF OXIDES

1. % silica determination

$$= \frac{(W_1 - W_2) - W_3}{W} \times 100$$

$$(0.8005 - 0.523) + 0.5074 \times 100$$

$$78.49\%$$

2. % CaO determination

%CaO

$$= \frac{100 \times 0.02\text{M EDTA} \times \text{Titre Value} \times \text{MM}_{(\text{CaO})} \times \text{Vol. of Stock Solution}}{\text{Wt of Sample}_{(\text{Mg})} \times \text{Vol. of aliquot}}$$

$$= \frac{100 \times 0.02 \times 1.442 \times 56.08 \times 250}{100 \times 10}$$

$$= 4.04\%$$

3. % MgO determination

$$\%\text{CaO} = \frac{100 \times 0.02\text{M EDTA} \times \text{Titre Value} \times \text{MM}_{(\text{MgO})} \times \text{Vol. of Stock Solution}}{\text{Wt of Sample}_{(\text{Mg})} \times \text{Vol. of aliquot}}$$

$$= \frac{100 \times 0.02 \times 0.089 \times 40.3 \times 250}{100 \times 10}$$

$$= 0.18\%$$

4. % Fe_2O_3 determination

$$Fe_2O_3\ \% = \frac{\text{wgt of ppt}}{\text{wgt of sample}} \times 100$$

$$= \frac{0.0309 \times 100}{1}$$

$$= 3.09\%$$

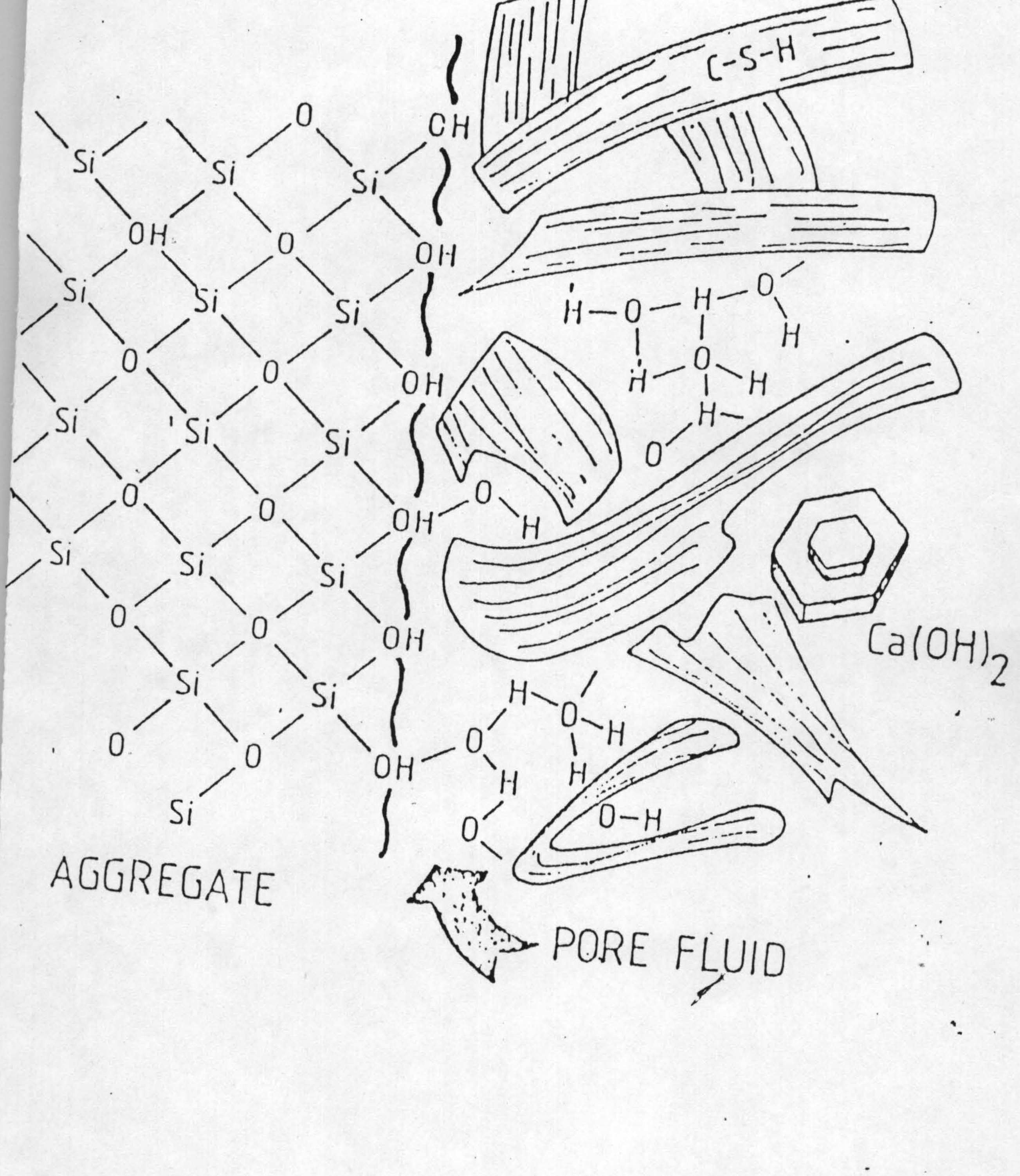
5. % Al_2O_3 determination

$$Al_2O_3\ \% = R_2O_3\ \% - Fe_2O_3\ \%$$

$$= 6.64 - 3.09 = 3.55$$

APPENDIX B

THE ALKALI-SILICA REACTION IN CONCRETE



APPENDIX C

The Difference between Data in Table 4.1 and 4.5 (%)

	Ground level		Middle level		Top level	
Compound	Left side	Right side	Left side	Right side	Left side	Right side
SiO_2	2.31	2.32	3.1	3.0	3.28	3.2
CaO	0.45	0.44	-0.69	-0.68	-0.96	-0.94
MgO	0.362	0.362	0.341	0.339	0.492	0.493
Al_2O_3	0.6	0.62	1.00	1.02	1.00	1.0
Fe_2O_3	0.1	0.13	0.16	0.18	0.05	0.05
Na_2O	0.06	0.07	0.01	0.01	-0.01	0
K_2O	0.105	0.105	0.107	0.108	0.099	0.097
SO_3	0.031	0.027	0.021	0.019	0.008	0.008

The Difference between Data in Table 4.2 and 4.5 (%)

	Ground level		Middle level		Top level	
Compound	Left side	Right side	Left side	Right side	Left side	Right side
SiO_2	2.3	2.2	1.47	1.4	2.47	2.48
CaO	0.39	0.4	0.87	0.86	-0.47	-0.47
MgO	0.382	0.382	0.351	0.351	0.419	0.492
Al_2O_3	0.79	0.84	0.94	1.03	1.13	1.13
Fe_2O_3	0.81	0.81	0.28	0.27	0.27	0.86
Na_2O	0.06	0.05	0.01	0.01	-0.01	-0.01
K_2O	0.117	0.116	0.118	0.119	0.097	0.097
SO_3	0.011	0.012	0.16	0.017	0.009	0.008

The Difference Between Data in Table 4.3 and 4.5 (%)

	Ground level		Middle level		Top level	
Compound	Left side	Right side	Left side	Right side	Left side	Right side
SiO_2	0.5	0.5	0.7	0.7	0.7	0.7
CaO	0.66	0.67	0.62	0.61	0.58	0.58
MgO	0.372	0.372	0.351	0.352	0.492	0.492
Al_2O_3	1.13	1.15	1.02	1.01	1.00	1.01
Fe_2O_3	0.21	0.21	0.21	0.20	0.11	0.12
Na_2O	0.1	0.09	0.1	0.1	0.1	0.09
K_2O	0.115	0.114	0.116	0.116	0.09	0.089
SO_3	0.017	0.016	0.012	0.012	0.004	0.002

The Difference Between Data in Table 4.4 and 4.5 (%)

	Ground level		Middle level		Top level	
Compound	Left side	Right side	Left side	Right side	Left side	Right side
SiO_2	1.13	1.15	0.43	0.46	0.71	0.70
CaO	0.57	0.57	0.77	0.76	0.67	0.66
MgO	0.362	0.362	0.341	0.341	0.49	0.491
Al_2O_3	0.83	0.83	0.79	0.79	0.65	0.67
Fe_2O_3	0.4	0.4	0.46	0.45	0.39	0.39
Na_2O	0.03	0.02	0.03	0.03	-0.001	-0.005
K_2O	0.19	0.191	0.189	0.188	0.15	0.15
SO_3	0.016	0.015	0.012	0.012	-0.006	-0.004