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# Preliminary Studies of Ca-Al-Layered Double Hydroxide (Ldh) and Its Effect on the Compressive Strength in Concrete

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## Authors' contributions

This work was carried out in collaboration among all authors. Author UDA designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors KO and EIO performed the calculation and statistics involved in the work then authors IEC and CUM managed the managed the literature searches. All authors read and approved the final manuscript.

### Article Information

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# ABSTRACT

This research work involved the preliminary studies of CaAI-LDHs using co precipitation method and applying it in the preparation of concrete to evaluate the compressive strength in grade 15 concrete. Slum and compressive strength test were carried out on the concrete control and concrete mixed with CaAI-LDH, Scanning electron microscope (SEM) and X-Ray Diffractogram (XRD) was carried out on the synthesize LDH, concrete control, and concrete mixed with LDH.

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From the results obtained in the work failure load test, it was observed that there was an increase by 62.60% in the compressive strength of concrete mixed with 30kg of CaAl-LDHs, the SEM micrograph also shows the increase in the density and the less porosity of the concrete mixed with CaAl-LDH which explained the increase in the compressive strength of the concrete. CaAl-LDH has therefore shows a promising effect on the increase on the compressive strength of concrete.

Keywords: Double layered hydroxide; scanning electron microscope; X-ray diffractogram; concrete.

## 1. INTRODUCTION

Concrete is the second most widely used resources worldwide after water, it is used in the construction of bridges, roads, buildings, tunnels, Dams etc. The use of poor quality concrete could lead to loss of life, property, resources and the waste of time and energy. In any form of with constructional work, concrete high compressive strength with better workability property is very important, this has given rise to researchers finding ways to improving the quality of concrete for better workability, such as the modification of the starting materials in concrete formulation or addition of additives to the concrete. One class of concrete additives is hardening accelerators, which speed up concrete hardening process and improve the compressive and flexural strength of concrete. In modern concrete construction, hardening accelerators are one of the most widely used concrete additives. Traditional inorganic potassium or sodium based hardening accelerators have many disadvantages, for instance, their durability is not high and they suffer from leaching of the salt. Chloride salts cause rusting of the steel bars used in reinforced concrete and sulfate salts may promote craze cracking of the concrete. Organic additives such as triethanolamine and triisopropanolamine, readily suffer from escape from concrete environment involved. The development of new types of CaAl-LDHs as a hardening accelerator for concrete has therefore attracted considerable attention in recent years.

Layered Double Hydroxide (LDHs) is one of such nano-ordered layered compounds. Layered double hydroxides, also known as hydrotalcite or anionic clay are mineral and synthetic materials, which deserve much attention in recent years. The general formular for layered double hydroxides is given as;

 ${M^{2^{+}}_{1-x}M^{3^{+}}_{x}(OH)_{2}}^{x^{+}}(A^{n^{-}})_{x/n}YH_{2}O$ 

Where,

 $M^{2^{+}}$  is a divalent met al such as Ca  $^{2^{+}}$ , Mg  $^{2^{+}}$ , Zn  $^{2^{+}}$  etc

 $M^{3^{+}}$  is a trivalent met al such as  $Al^{3^{+}},\ Co^{3^{+}},\ Cr^{3^{+}}$  etc

 $A^{n^{-}}$  is an anion such as Cl<sup>-</sup>, NO<sub>3</sub><sup>-</sup>, CO<sub>3</sub><sup>2-</sup> etc x is given by  $M^{3^{+}}/M^{2^{+}} + M^{3^{+}}$  with values that varies from 0.25 to 0.33 [15]

The structure of layered double hydroxides can be best explained analogous to mineral brucite  $\{Mg(OH)_2\}$ . Brucite consist of hexagonal close packing sites occupied by divalent ion. The figure below shows a schematic diagram of the brucite-like structure of layered double hydroxide.

In layered double hydroxides, some of the divalent cations of these brucite like sheets are isomorphously substituted by trivalent cations, making it very similar to the AFm phase occurring in hydrated cements being composed of positively charged layer  $\{Ca_2AI(OH)_6\}^+$  [18]. occupancy of the trivalent The ions generates a net positive charge on the mixed metal hydroxide layers and anions are present in the inter layers' space to compensate the positive charge. In natural layered double hydroxides, these inter lavers' anions are usually carbonate, chloride etc. On the other hand, the electric charge of the layers and inter layers' ions is just the opposite of that found in silicate clay (cationic clays), for all this reason, the materials are usually known as Layered Double Hydroxide, anionic clay or hydrotalcite [15]. As an anionic clay, the inter layer anions are easily exchange by various organic and inorganic ions. Replacing capability of the inter layer ions makes it to have a broad field of applications such as high performance catalytic materials or catalyst support, adsorbent, separation materials, nano-composite material engineering, optics, additives in plastics as flame retardants, biological and pharmaceutical materials [18]. Another useful application of layered double hydroxide which has received considerable attention is their ability to be used as hardening accelerator in concrete. Layered double hydroxides are relatively simple to produce.

### 1.1 Properties and Applications of Layered Double Hydroxides

Layered double hydroxides present a great number of properties due to its varied compositions and methods of synthesis. Below are few explanation of the properties of layered double hydroxides.

#### 1.1.1 Anion exchange capacity

The anion exchange capacity (AEC) depends on the metallic cation ratio, the ability of the involved anion in stabilizing lamellar structure and molecular mass of the cations and anions involved. As reported by Leroux and Besse [10], anion exchange capacity values might change between 200 and 450cmol<sub>c</sub>kg<sup>-1</sup>. Values below 200cmol<sub>c</sub>kg<sup>-1</sup> are not possible, once the M<sup>2+</sup> and M<sup>3+</sup> ratio is very low to support layered double hydroxide structure.

#### 1.1.2 Colloidal properties

The small particle size and low charge density of some layered double hydroxide are important for systems with colloidal characteristics and/or delamination (stacking structure loss). The large size of the host anion often causes interlayer diffusion problems. Thus, in order to overcome these problems, colloidal and/or delaminated systems, which are formed from layered double hydroxides, allow a better arrangement of the lamellae between host and anion. Several studies have reported the formation systems with colloidal features by combining layered double hydroxides and organic molecules [10,19]. From colloidal suspensions of layered double hydroxides, Gardner et al. [6] have produced continuous and transparent films. Layered double hydroxides of hydrophobic nature and colloidal characteristics were prepared from the combination of hydrotalcite and anionic surfactants, such as heptanes, benzene, toluene and propanol. Leroux et al. [9] performed the delamination of Zn<sub>2</sub>Al-LDH intercalated with dodecyl suphonate. Organically modified layered double hydroxide suspended in butanol resulted in the formation of highly stable translucent colloidal solutions. Hibino and Jones [8] performed lavered double hydroxide delamination intercalated with amino acids and observed excellent results when layered double hydroxides were intercalated with glycine in formamide solutions. As stated by these authors, the process of delamination does not result from the driving force generated by solvent inclusion. However, it is considerably influenced by solvent boiling point. Solvent with high dielectric constant are known to facilitate this process. The enlargement of alcohol alkyl chain provides an increase in the boiling point with consequent decrease of the dielectric constant.



Fig. 1. A schematic diagram of brucite-liked structure of layered double hydroxide (LDH)

### 1.1.3 Memory effect

An unusual property of layered double hydroxide is that when they release their interlayer anions and are expose to water and other source of anions the LDH structure can be reformed through the sorption of anion and water. This process is known as the reformation or memory effect.

## 1.1.4 Thermal stability

Thermal stability is another important property of lavered double hydroxides. Thermal characterization of these materials was performed by Thermogravimetric Analysis (TGA) and Differentia Scanning Calirometry (DSC) techniques. Thermal decomposition temperatures are dependent on several factors such as, LDH crystalinity, nature, cation molar ratio and the interlayer anion type (organic or When organic anions are within inorganic). LDHs layers, resulting in intercalated nanomaterials, it usually shows higher thermal stability relative to free organic anion. Another hypothesis is that layered double may act as a protective layer, delaying the byproduct crystallization and inducing unusual solid formation after heat treatment under inert atmosphere.

#### 1.1.5 Applications of layered double hydroxides

Layered double hydroxide are promising layered materials due to some of their interesting properties, such as thermal stability, ability to intercalate different types of anion, biocompatibility, flexible tenability etc. below is some of the applications of Layered Double Hydroxide.

#### 1.1.6 Water treatment

Waste water often contains oxyanions such as F<sup>-</sup>, Cl<sup>-</sup>, Br<sup>-</sup>, which are harmful to both human and wildlife. Enhanced ability to remove oxyanions is of importance in environmental protection. Structured layered double hydroxides are one new type of promising material due to their high ability to capture organic and inorganic anions which can be use in water treatment. Calcined LDH powder is also an important type of material used in water treatment, since LDH can afford mixed metal oxides and as a special property called memory effect. The main advantages of LDHs over the conventional anionic exchange

resins include their higher anion exchange capacity for certain oxyanions and their good thermal stability.

Furthermore, LDHs can be fully regenerated in a short time for reuse. Lv et al. [16], used MgAl-NO<sub>3</sub>-LDH powder and calcined powder to remove F-, Cl-, Br- and I- and concluded that the rate constant for exchange of nitrate anions by halide decreases in the order F-> Cl-> Br->l<sup>-</sup>, following pseudo-second order kinetic models.

## 1.1.7 LDHs in biology and medicine

LDH materials, being unstable in acidic conditions, do not survive for long in stomach. However, given a suitable enteric coating, slowrelease of drugs into small intestine could be realized leading to effective delivery of fragile genetic materials into cells. Antisense DNA, a potential gene specific therapeutic agent, can be intercalated in LDH to form Bio -LDH nanohybrid which protects intercalated antisense molecule from degradation and also improves cell penetration. Bio-LDH nanohybrid also avoids specific aptameric effects (leading to nonspecific binding of antisense oligonucleotides). Once LDH antisense hybrids entered cell, hydroxide layers are removed by dissolution in lysosomes, where pH is slightly acidic and encapsulated biomolecules are released into cell. Powdered LDHs have demonstrated to be one type of important and green carrier or host for genes and drugs due to excellent biocompatibility and nontoxicity. MgAI-LDH is used as an important component of drugs, or as nanocarriers for delivery of drugs and genes into cells. Wei et al. [17] demonstrated that LDH was able to be used as an effective nanocarrier by greatly enhancing the thermal and photo-stabilities for L-Dopa and L-Tyrosine, which are both unstable agents in transport. Comprehensive storage or investigations have been performed in Choy's group on the toxicity of LDH nanoparticles in vitro and in vivo in practical biology application. Interestingly Lv's group recently reported an efficient LDH-based delivery for siRNA to mammalian cells in vitro. A pronounced downregulation of protein expression upon LDH mediated siRNA transfection of HEK293T cells was observed.

# 1.1.8 Catalyst

LDH powder, LDH film, and activated rehydrated LDH (RLDH) structures are well known as effective solid catalysts. Zhang et al.[13] used the

Cu<sup>2+</sup> base LDH powder as a catalyst for wet oxidation, and reported that the array of active Cu<sup>2+</sup> centers on the surface of CuZnAI-CLDH is ordered net shape, which is influenced by the effect of ordered cross trap. The advantages of novel LDH films are able to overcome the problems of use of powdery catalyst on an industrial scale, such as high pressure drop and difficult catalyst separation. Lv et al. [16] reported that the activated MgAI-LDH film was promising to be used as precursor to synthesize monolithic catalyst for the aldol condensation of acetone and other base catalyst reaction. For the interesting 3DOM LDH structure, Geraud et al. [7] demonstrated that the activated MgAI-LDH, with decatunstate anion intercalated exhibited the enhanced photocatalytic activaty for the photodegredation of 2, 6-dimethylphenol with control of the corresponding coprecipitated LDH material, due to the high specific surface area of the 3DOM structure.

# 1.1.9 Additive in flame retardant and concretes

Powdered LDH are one type of additives in concrete. Basically, CaAl-LDH has a general formula very similar to the AFm phase occurring in hydrated cement. being composed of positively charged layers [Ca<sub>2</sub>Al(OH)<sub>6</sub>]<sup>+</sup> and negatively charged interlayers [X<sup>-</sup>nH<sub>2</sub>O]<sup>-</sup>. AFm phase is generally believed to act as a crystal seed accelerating cement hydration, CaAl-LDHs have been therefore, proposed as a potential concrete hardening accelerators.

Powdery LDH is also commercially promising as an additive in flame retardants. Many flame retardants are harmful, having being liked to liver, thyroid, reproductive/developmental and neurological effects. Currently, halogen free alternatives are one active research area. LDHs and cationic clay (e.g montmorillonite) have been widely investigated as additives in this context compared to other flame retardants, LDH is a new type of material due to high smoke suppression, non-toxicity or low toxicity. LDH is currently explored as a second generation flame retardants with enhanced properties by either modification of the lavers or intercalation of different anions. Ling et al. [11] found that a borate-pillared MgAI-LDH with ethylene vinyl acetate (EVA) as the polymer component was a promising flame retardant. By tuning the

composition of the layers, the flame retardancy of LDHs was steered.

### 1.2 Concrete

The importance of concrete in modern society cannot be overestimated. Concrete structure is found everywhere such as in buildings, roads, bridges, dams etc. there is no escaping the impact concrete on man's everyday life. The use of poor quality concrete for structural and conventional works may cause loss of lives and properties, so the use of quality assurance of concrete has become an important and critical factor.

Concrete is a composite material which is made up of a binder and fillers. The binder (cement and water) glues the fillers (gravels and coarse aggregate) to form a synthetic conglomerate in fluid form which can easily be moulded. The composition of concrete is discussed below.

## **1.2.1 Composition of concrete**

#### (i) Cement

Cement is a binder, a substance used for construction that sets, hardens and adheres to other materials to bind them together. Cement is seldom used on its own, but rather to bind sand and gravel together. Cement mixed with fine aggregate produces mortar for masonry, or with sand and gravel to produce concrete. Cement is the most widely used material in existence and is only behind water as the planet's most consumed resources. Cement used in construction are usually inorganic, often lime or calcium silicate based, and can be characterized as either hydraulic or non-hydraulic, depending on the ability of the cement to set in the presence of water.

Non-hydraulic cement, such as slaked lime does not set in wet conditions or under water. Rather, it sets as it dries and reacts with carbon dioxide in the air. It is resistant to attack by chemicals after setting. Hydraulic cement (e.g Portland cement) set and become adhesive due to a chemical reaction between the dry ingredient and water. The chemical reaction results in mineral hydrates that are not very water soluble and are so guite durable in water and safe from chemical attack, which allows setting in wet conditions or under water and further protect the hardened material from chemical attack.

The raw materials used in the manufacture of cement consist mainly of Lime, Silica, Alumina, Iron oxide, Magnesia, Gypsum etc. these compounds interact with one another in a kiln at temperature of around 1400-1600 °C to form a series of more complex products like tricalcium aluminate (Ca<sub>3</sub>Al<sub>2</sub>O<sub>6</sub> or C<sub>3</sub>A), belite or dicalcium silicate (Ca<sub>2</sub>SiO<sub>4</sub> or C<sub>2</sub>S), Alite or tricalcium silicate (Ca<sub>3</sub>SiO<sub>4</sub> or C<sub>3</sub>S), tetra calcium aluminoferrite (Ca<sub>4</sub>Al<sub>2</sub>Fe<sub>2</sub>O<sub>10</sub> or C<sub>4</sub>AF) and gypsum (CaSO<sub>4</sub>.2H<sub>2</sub>O or CSH<sub>2</sub>). The percentage composition of cement ingredients is given as;

SN	Ingredient	Percentage in Cement	
1	Lime	60-65	
2	Silica	17-25	
3	Alumina	3-8	
4	Magnesia	1-3	
5	Iron Oxide	0.5-6	
7	Calcium Sulphate	0.1-0.5	
8	Sulphur Trioxide	1-3	
9	Alkaline	0-1	

#### **Functions of Cement Components**

The main features of cement ingredients along with their functions and usefulness or harmfulness are given below;

- a) Lime: lime is calcium oxide CaO, which is gotten from calcium carbonate, chalk, shells, shale or calcareous rock. The presence of lime in a sufficient quantity is required to form silicates and aluminates of calcium. Deficiency in lime reduces the strength of the cement while excess of lime makes cement unsound and causes cement to expand and disintegrate.
- b) Silica: Silicon dioxide SiO<sub>2</sub> is usually from sand, old bottles, clay or argillaceous rock. Silica impart strength to cement, silica is usually present in cement as dicalcium and tricalcium silicate.
- c) Alumina: Also known as Aluminium oxide Al<sub>2</sub>O<sub>3</sub>, is gotten from bauxite, recycled aluminium or clay. Alumina imparts quick setting on cement, clinkering temperature is lowered by the presence of requisite quantity of alumina. Excess alumina weakens the cement
- d) Iron Oxide: Iron oxide Fe<sub>2</sub>O<sub>3</sub> gotten from iron ore, scrap iron, fly ash or clay, imparts colour to cement it also act as flux, at a very high temperature, it imparts into the chemical reaction with calcium and aluminium to form tricalcium aluminoferrite.
- e) **Calcium sulphate:** this is present in the form of gypsum (CaSO<sub>4</sub>.2H<sub>2</sub>O), it slows down or retard the setting action of cement.
- f) Magnesia: Magnesium Oxide (MgO) should not be present in more than 2% in

cement. Excess magnesia will reduce the strength of cement.

g) Magnesia: This is also known as magnesium oxide MgO, it is usually present in small quantity, and it imparts hardness and colour to cement. Excess of magnesia reduces the strength of cement.

#### (ii) Water

Water is another key ingredient in the making of concrete, which when mixed with cement forms a paste that binds the aggregate together. The water causes the hardening of concrete through a process called hydration, which is a chemical reaction in which the major compounds in cement form chemical bonds with water molecules and become hydration product.

There are two ways in which water react with the complex compounds formed in cement during hydration. In the first, a direct addition of some molecules of water takes place, this is being considered as a true reaction of hydration. The second type of reaction with water is known as hydrolysis, in which its nature can be illustrated using the  $C_3S$  hydration equation:

The reaction of  $C_3S$  with water continue even when the solution is saturated with lime and the resulted amounts of lime precipitate in crystal form  $Ca(OH)_2$ . Calcium silicate hydrate remains stable when it is in contact with the solution saturated with the lime. Calcium silicate hydrate hydrolyzed when being in water-some of lime form, and the process continues until the water saturate with lime. If the calcium silicate hydrate remains in contact with water it will leave the hardened compound only as hydrated silica due to the hydrolysis of all the lime.

The rates of chemical reactions of the main compounds are different:

- Aluminates react with the water in the beginning
- Affect the route of the chemical reactions at early periods of hydration.
- Silicates affect the later stage reactions.

The main hydrates of the hydration process are:

 Calcium silicate hydrate, including hydrated products of C<sub>3</sub>S and C<sub>2</sub>S.

Hydration of calcium silicate  $C_3S$  takes about one year or more. This initial gel forms an external layer over  $C_3S$  causing the delay of the reaction. After few hours, this initial C-S-H undergoes hydrolysis to form the second product of the gel CSH. The full hydration of  $C_3S$  can be expressed approximately following the equation;

 $2C_3S + 6 \longrightarrow C_3S_2H_3 + 3Ca(OH)_2$ 

 $C_3S$  contribute most to the strength development during the first four weeks.

The equation of reaction of C<sub>2</sub>S is given as

2C<sub>2</sub>S + 4H----- C<sub>2</sub>S<sub>2</sub>H<sub>3</sub> + Ca(OH)<sub>2</sub>

The reaction of  $C_2S$  is slower than that of  $C_3S$  and the amount of  $Ca(OH)_2$  produce from its hydration is less.  $C_2S$  influences the gain in strength from 4 weeks onward. At the age of about one year, the two compounds contribute approximately equally to ultimate strength.

(2) Tricalcium aluminate hydrate.

 $C_3A$  contribute to the strength of the cement paste at one to three days and possibly longer, but causes retrogression at an advanced age, particularly in cement with a high  $C_3A$  content.

(a) 
$$C_3A + 6H_{----}C_3AH_6$$

(3) The role of  $C_4AF$  in the development of strength of cement is not clear till now.

Since calcium silicate ( $C_3S$  and  $C_2S$ ) are the main cement compounds (occupies about 75% of cement weight), they are responsible for the final strength of the hardened cement paste. The

rate of hydration decreases continuously with time and the size of unhydrated cement particles decreases. For instance, after 28 days in contact with water, grains of cement have been found to have hydrated to a depth of only 4 micro meters, and 8 micro meters after a year. This is due to:

- Accumulation of hydration products around the unhydrated cement grains which lead to prevent water from channeling to them
- Reduction of the amount of water either due to chemical reaction or evaporation.
- Reduction of the amount of cement due to reaction.

The progress of hydration of cement can be determined by different means, which are;

- 1. The measurement of the amount of  $Ca(OH)_2$  in the paste resulted from the hydration of the silicates.
- 2. The heat evolved by hydration.
- 3. The specific gravity of the paste.
- 4. The amount of chemically combined water.
- 5. The amount of unhydrated cement present (using x-ray quantitative analysis)
- 6. Indirectly from the strength of the hydrated paste [14].

The amount of tricalcium aluminate ( $C_3A$ ) present in most cement is comparatively small but its behavior and structural relationship with the other phases in cement makes it of interest. The tricalcium aluminate hydrate forms of prismatic dark interstitial material in the form of flat plates individually surrounded by the calcium silicate hydrate. The reaction of pure C<sub>3</sub>A with water is very violent with evolution of large amount of heat, forming calcium aluminates hydrate in the form of leaf hexagonal crystals. In Ordinary Portland Cemenet (OPC), this reaction leads to immediate stiffening known as flash setting. Gypsum, added to the clinker through grinding process cause delaying the reaction of with water  $C_3A$ by its reaction with C<sub>3</sub>A to form insoluble calcium sulfo aluminate  $(3CaO.Al_2O_3.3CaSO_4.30-32H_2O)$ ettringnite-around C<sub>3</sub>A particles, which permits enough time for the hydration of C<sub>3</sub>A that its reaction is slower than  $C_3A$  and permits the occurring of natural setting. But eventually tricalcium aluminate hydrate is formed, although this was preceded by a metastable 3CaO.Al<sub>2</sub>O<sub>3</sub>CaSO<sub>4</sub>.12H<sub>2</sub>Oproduce at the expense of original high-sulphate calcium sulphoaluminate.

The reaction of gypsum with  $C_3A$  continues until one of them is exhausted, while  $C_3S$  continue in hydration. If  $C_3A$  is exhausted before gypsum, the surplus gypsum expands and become an agent that assist the disruption and deterioration of cement paste. If gypsum is exhausted before  $C_3A$ , the remaining  $C_3A$  begins in hydration, which is represented as;

 $C_3A + 6H_{-----}C_3AH_6$ 

Calcium aluminium hydrate  $C_3AH_6$  is stablecubical crystal with high sulphate resistance, although ocuurs in many forms before transforming into the stable state. It is probably forming hexagonal crystals ( $C_4AH_8$ ,  $C_4H_{10}$ ,  $C_4H_{12}$ ) before the cubical crystals. When the hexagonal crystals expose to sulphate (inside concrete from sand or external from soil or ground water) react, it forms calcium sulphoaluminate with increase in volume, depending on the amount of remaining aluminates and the concentration of the sulphates crack and deteriorate the hardened concrete.

The transformation of the calcium aluminates hydrate from the metastable hexagonal form to the stable cubical form is accompanied with change in density and the size of the crystal, leading to decrease in the late ages strength of the cement paste due to:

- Lose of adhesion and cohesion in the microstructure.
- Increase in porosity of the hardened cement paste.

The present of  $C_3A$  in cement is undesirable, it contributes little to the strength of cement except at early ages (1-3 days) and when hardened cement paste is attacked by sulphates, expansion due to the formation of calcium sulfoaluminate from  $C_3A$  may result in disruption of the hardened cement paste [14].

Hydration of cement can be presented by the following schematic diagram



Basic elements of cement

## (iii) Aggregates

Aggregates are chemically inert solid bodies held together by cement. Aggregates come in various shapes, sizes and materials ranging from fine particles of sand to large coarse rock and gravel.

# 1.3 Calcium Silicate Hydrate (C-S-H) Structure

Calcium silicate hydrate (C-S-H) which is formed as a result of the reaction of Calcium Oxide (CaO), Silicate (SiO<sub>2</sub>), and water comprises 60% of volume of cement paste. C-S-H is the most important composition of cement structure. Understanding and predicting the behavior of cement paste is best explained at the nano up to the micro structure of C-H-S. C-H-S is the main factor associated with the binding phase of cement paste and also responsible for the important properties such as strength and shrinkage.

The molar ration of CaO to SiO<sub>2</sub> (C/S ratio) in C-S-H is one of the major criteria in ascertaining and controlling the properties of a calcium silicate hydrate system. This value varies from 1.2 to 2.1 in hydrated silicate phases and has an average of about 1.75. The C-S-H system may be divided into low and high lime content categories partitioned by the C/S ratio of about 1.1 where chemical and physical properties change noticeable. The state of water in a C-H-S system is also vaguely defined. Water can be present within the interlayer structure of C-S-H (either in the form of H<sub>2</sub>O or OH<sup>-</sup>). Water molecules can also be physically absorbed on the surface of solid phases.

There are several ordered calcium silicate hydrates that are structurally related to the C-H-S. Tobermorite and Jennite (with approximate stiochiometry of  $C_5S_6H_5$ and  $C_9S_6H_{11}$ respectively), for example, have a defined crystal structure and have been studied for many years as possibly analogues to C-S-H. The reaction between lime and silica in excess water result in the formation of tobermorite-like and jennite-like systems most commonly known as C-H-S(I) and C-H-S(II). These hydrates can also be prepared through mixing sodium silicate and calcium salt in aqueous solution, although they are less crystalline. These phases pure materials are relatively easy to produce and are convenient for systematic research work on C-S-H.

### 1.3.1 C-S-H nanostructural models

The nanostructure of C-H-S has been the subject of much research, yet is still not clearly understood with suggested models, ranging from colloidal to layer-like. One of the first physical models was proposed by Powers and Brownyard [3]. They describe C-S-H as a colloidal material. In this model the gel particles are held together mainly by van der Waals forces and the space between them is called "gel porosity" which is accessible only by water molecules. A more comprehensive model was developed later by Feldman and Sereda [5] base on extensive experimental studies of hydrated cement system. The role of water in this model is explained in more detail and the changes in the mechanical properties of C-H-S related to water content can be easily described. The main feature of their model is concerned with the layered nature of the C-H-S. Structural roles that are assigned to the interlayer water of the C-S-H. exhibit irreversible behavior during the adsorption and desorption process.

Advancement in experimental techniques, has led to the development of new models. Jennings' colloidal model feature globules of about 5nm in diameter for C-H-S and proposes the existence of intraglobular pores (IGP) and small gel pores. The viability of using a layered model for the C-S-H in cement paste seems, however, also plausible according to recent work, which used helium inflow technique as a nanostructure probe along with XRD to follow the changes at the nano-level in the properties of C-H-S, a layered semi-crystalline material. The helium inflow results are analogues to those for C-H-S in hydrated Portland cement. They can be best explained by a layered model for C-H-S in cement paste. The Jennings colloidal model is essentially a hybrid where the globules are comprised of C-H-S layers. The layered model is incompatible with the colloidal model in its explanation of physio-chemical and engineering behavior. The colloidal model neglects the structural role of inter layer water in cement paste as evidenced by the corresponding behavior of synthetic C-S-H. A primary difference is rooted in the inability of the colloidal model to separate the reversible and irreversible thermodynamic aspect of sorption phenomena. It is unable, for example, to rigorously explain the elastic and viscoelastic behavior and their dependence on relative humidity.

#### 1.3.2 Mechanical properties of C-S-H

The mechanical properties of phase pure C-S-H systems have rarely been studied. The intrinsic modulus of elasticity of C-S-H appears to be independent of its C/S ratio and degree of polymerization. Nanoidentation methods have been employed in order to study the elastic character of C-H-S nanoparticles. Two types of C-S-H. Two types of C-S-H are suggested, they are low and high density C-S-H phase with volume fraction of 30 and 70% in hydrated ordinary Portland cement. They have a mean stiffness of about 21.7 and 29.4GPa respectively.

Dynamic molecular modeling and free energy minimization techniques have also been used by Manzan et al. [12] to estimate the elastic properties of C-S-H. It has been reported that the average Young's modulus (E) increases with the increase in the C/S ratio of the C-S-H. A recent study by Beaudoin et al. [2], demonstrated that the modulus value of the C-S-H increases with the increase in the mean silicate chain length.

# 1.3.3 Intercalation of the interlayer spaces between the C-S-H sheets

It is also possible for organic molecules such as layered double hydroxides to intercalate the interlayer spaces of C-S-H. Evidence for the intercalation process is primarily based on the expansion of the basal-spacing of synthetic C-S-H as indicated in X-ray diffraction spectrum. The degree of expansion varied depending on the C/S ratio, synthesis method (in-situ or exchange), type of molecules (non-ionic, anionic or cationic), concentration and PH. Small shift in the basal spacing of C=S-H could be interpreted as evidence of partial intercalation at layer ends.

# 2. MATERIALS AND METHODS

### 2.1 Materials

The following Material were used for Synthesis of CaAl-LDH.

- 1. Calcium Chloride dihydrate (CaCl<sub>2</sub>.2H<sub>2</sub>O), 98%
- 2. Aluminium Chloride hexahydrate (AICl<sub>3</sub>.6H<sub>2</sub>O) 97%
- 3. Ethanol (C<sub>2</sub>H<sub>5</sub>OH), 98%
- 4. Sodium Chloride (NaCl), 99.5%
- 5. Nitrogen gas (N<sub>2</sub>)
- 6. Tetraoxosulphate (iv) acide (H<sub>2</sub>SO<sub>4</sub>), 99%
- 7. Deionized Water

- 8. Sodium Hydroxide (NaOH)
- 9. Sand
- 10. Cement
- 11. Engine oil

All the reagents were purchased from Loba Chem sale representatives.

## 2.2 Co-precipitation Method

The co-precipitation method is relatively the simplest and most commonly used methods for the preparation of layered double hydroxide, and is the only method that will be describe in detail here. Co-precipitation method is done in three different forms: Co-precipitation at (i) increasing PH (ii) decreasing PH and (iii) at constant PH [1,4]. In the variable PH method, a solution containing salts of the divalent and trivalent cations is added to an alkaline solution containing the anion that will be interleaved. In the co-precipitation at constant PH approach, the dissolved divalent and trivalent cations, the interlayer anion, and alkaline solution are all combined at the start time. The co-precipitation at constant PH requires the use of a more sophisticated experimental apparatus, but it results in particles with greater uniformity. As PH is increased, the cations react with OH and interlayer anion generating layered double hydroxides, which has very low solubility and therefore precipitates. These reactions are performed under strong stirring and require a further purification step to remove any remaining counter ions. Unfortunately, layered double hydroxide prepared by co-precipitation method often suffers from poor crystalinity and the presence of impurities. In contrast a method like the urea method (which is in many ways similar to co-precipitation method) allows better control of particle size and higher crystalinity than other methods. However, the urea method is only suitable for preparation of layered double hydroxide with high charge density, meaning it cannot be use to prepare layered double hydroxide containing Cu<sup>2+</sup> or Cr<sup>3+</sup>.

All materials with analytical purity were purchased from Pyrex and used without further purification. A batch of calcium aluminium layered double hydroxide was prepared by the Co-Precipitation method as describe below.

A mixed metal solution was prepared by dissolving calcium chloride dihydrate (7.8g) and aluminium chloride hexahydrate (6.44g) in a 2:1 ratio respectively, with  $Ca^{2+}$  concentration of

0.66M in a mixture of ethanol and deionised water (100mL) in 1:4 ratio respectively producing an acidic PH of 2.1. A caustic solution was prepared by slowly dissolving sodium hydroxide (8.0g) and sodium chloride (8g) in ethanol/deionised water (100ml) in 1:4 ratios respectively producing an alkaline solution of PH 12.2. Both the caustic solution (100ml) and the mixed metal solution (100ml) was added drop wise at the rate of a drop per four seconds with continuous stirring. After which, a layered double hydroxide was formed as white precipitate with a PH of 11.9. The mixture was covered and left in an ovum maintained at 110°C for 18 hours. The lavered double hydroxide precipitate was collected by vacuum filtration and washed with deionised water (50ml). The wet layered double hydroxide was dried in an ovum for 16 hours maintained at a temperature of 60°C, the dried CaAl-layered double hydroxide had reduced in size considerably and was weigh to give 6.8g and had an appearance of a flaky white solid which was further grinded. The procedures were repeated for about 14<sup>th</sup> times.

# 2.3 Concrete

In its simplest form, concrete is a mixture of cement, water, sand and coarse aggregate like gravels. The purpose of which a concrete is used is determined by the nature of the proportion mixture of the concrete ingredient.

There are basically six major types of concretes which are produced depending on the structural usage, they are as follows;

- 1. Grade 10 Concrete. The grade 10 concrete is in the ratio of 1: 3 and 6 of cement, sand and gravel respectively, it is usually used for the construction of patio slabs pathways and non-structural work.
- Grade 15 Concrete. The grade 15 concrete is in the ratio of 1: 2 and 4 of cement, sand and gravel respectively, it is used for floor binding and pavement kerbs.
- Grade 20 Concrete. The grade 20 concrete is in the ratio of 1: 1.5 and 3 of cement, sand and gravel respectively, i.e 10Kg of cement, 15Kg of sand and 30Kg of concrete are mixed together with water using a mixer. This type of concrete is used for domestic floors, foundations where the weight of structure will be lighter, workshop bases, garages, driveways, internal floor slabs etc.
- 4. Grade 25 Concrete. The grade 25 concrete is in the ratio of 1: 1 and 2 of cement, sand

and gravel respectively, i.e 10Kg of cement, 10Kg of sand and 20Kg of gravel are mixed together with water using a mixer. This type of concrete mixture is used for the construction high story buildings.

- 5. Grade 30 Concrete. The grade 30 concrete is used in the construction of pathways and roadways (this is the lowest grade concrete mix that can be used for this purpose), it is more durable than the other grades, it is weather-resistant and can take heavy road traffic.
- Grade 35 Concrete. This is used for commercial structures, creating external walls, slabs and structural pilling.
- Grade 40 Concrete. This is used for commercial construction sites, creating foundations and beams for structural support and roads. Grade 40 concrete can withstand chemical corrosion also.

Note: 0.5-0.6% volume of cement gives the volume of water to be used for all types of concrete mixture.

## 2.4 Preliminary Synthesis of Grade 15 Concrete for Control

The cement, sand and gravel were purchased in a construction merchant at Igue – Iheya guarters, Ovia North East Local Government Area of Edo state. The sand and gravel were spread and allow to dry under room temperature for a day without any modification of the cement. The grade 15 concrete used for control was prepared by a mixture of 4kg of cement, 8Kg of sand, 16Kg of gravel and 2.4 litres of water using the mixing machine. A compact test was done on the concrete, nine oily moulds were then filled with the concrete and were placed on a vibrator table for evenly distribution. The concrete inside the mould were de-moulded after a day and were submerged in a water bath for curing to occur. A compressive test results were carried out on three concrete blocks for 7, 14 and 28 days respectively.

# 2.5 Preliminary Synthesis of Grade 15 Concrete Mixed with Different Proportions of Layered Double Hydroxides

The same cement, sand and gravel were used for the control were also used for the making of the concretes although mixed with different weights of layered double hydroxides. Three different grade 15 concretes mixed containing 10g, 20g and 30g of layered double hydroxides respectively were prepared using the same process as that of the control.

The first concrete mixture contains of 4 kg of cement, 8 Kg of sand and 16 Kg of gravel, 2.4 litres of water and 10g of layered double hydroxides. A slum/compact test was carried out and recorded for the first mixture, which was used to mould nine concrete blocks. The second concrete mixture contain 4kg of cement, 8kg of sand, 16kg of gravel, 2.4 litres of water and 20g of double layered hydroxides. A slum/compact test was done on the mixture which was also used to mould another nine concrete blocks. The third concrete mixture contain 4kg of cement, 8kg of sand, 16kg of gravel, 2.4 litre of water and 30g of layered double hydroxides. A slum/compact test was also carried out on the mixture and was used to mould another 9 concrete blocks.

The twenty-seven concrete blocks were demoulded after a day and were all submerged in a water bath for curing to occur. Compressive tests were carried out on the 7<sup>th</sup>, 14<sup>th</sup> and 28<sup>th</sup> day after submersion on nine concrete blocks for the stated days. The nine concrete blocks comprise of three concrete blocks from the different concrete blocks mixtures.

# 3. RESULTS AND DISCUSSION

#### 3.1 Slump Test

The table shows the result for the slump test of the concrete control and concrete mixture with the different proportions of layered double hydroxides (LDH).

The concrete slump test is also known as the compact test or slump cone test is used to determine the workability or consistency of concrete mix. The slump test is the simplest workability test for concretes, it is usually done using simple equipment that are relatively cheap, easy to use and provides immediate results. Due to this fact, it has been widely used for workability test since 1922.

Generally concrete slump value is used to find the workability, which indicates water cement ratio, but there are various factors including properties of materials, mixing methods, dosage, admixtures etc. which also affect the concrete slump value. Factors which influence the Concrete Slump test

- (a) Material properties like chemistry. fineness. particles size distribution. moisture content and temperature of cementitious materials, size texture combined grading, cleanliness and moisture content of the aggregates.
- (b) Chemical admixtures dosage, type, combination, interaction, sequence of addition and its effectiveness.
- (c) Air content of concrete.
- (d) Concrete batching, mixing and transporting methods and equipment.
- (e) Time since mixing of concrete at the time of testing.
- (f) The amount of free water in concrete.
- (g) Temperature of the concrete.
- (h) Sample of concrete, slump-testing technique and the condition of test equipment. Procedure for Concrete Slump Cone Test.
- (a) The internal surface of the mould was clean and oil applied.
- (b) The mould was placed on a smooth horizontal non-porous base plate.
- (c) The mould was filled with the prepared concrete mix in 4 approximately equal layers.
- (d) Each layer was tamping with 25 strokes of the rounded end of the tamping rod in a uniform manner over the cross section of the mould, for the subsequent layers, the tamping was penetrated into the underlying layer.
- (e) Excess concrete was removed and level the surface with a trowel.
- (f) The mortar or water leaked was cleaned out between the mould and the base plate.
- (g) The mould was raised from the concrete immediately and slowly in vertical direction.
- (h) Measured slump was the difference between the height of the mould and that of height point of the specimen being tested.

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 The above operation was carried out at a place free from vibrations or shock and within a period of 2 minutes after sampling.

# 3.2 Types of Concrete Slump Test Results

- (a) True Slump. True slump is the only slump that can be measured in the test. The measurement is taken between the top of the cone and the top of the concrete after the cone has been removed. The true slump test has a value that ranges from 20 to 60 mm. If a slump value is lower than 20mm. it therefore means that the water added is too small compare to the quantity of cement, so also for a slump value above 60mm indicates much water compare to the quantity of cement.
- (b) Zero Slump. Zero slump is an indication of very low water-cement ratio, which result in dry mixes. This type of concrete is generally used for road construction.
- (c) Collapse Slump. This is an indication that the water-cement ratio is too high, i.e. concrete mix is too wet or it is a high workability mix, for which a slump text is not appropriate.
- (d) Shear Slump. The shear slump indicated that the result is incomplete, and concrete be retested.

# 3.3 Weights, Failure Load and Compressive Test of the Control and Concrete Mix

The tables below show the weight, failure load and the compressive strength of the concrete control for day 7, 14 and 28 respectively.

# Table 1. The results for the slump test of the concrete control and concrete mixture with the proportions of layered double hydroxide (LDH)

Control (mm)	Concrete mixed with10g of LDHs (mm)	Concrete mixed with 20g of LDHs (mm)	Concrete mixed with 30g of LDHs (mm)
25.00	50.00	40.00	38.00

# Table 1a. The weight, failure control and the comprehensive strength of the concrete controlfor 7 days

Concrete Control	Weight (Kg)	Failure Load (K/N)	Compressive Strength(N/MM <sup>2</sup> )
Sample 1	2.601	228.9	10.17
Sample 2	2.562	227.1	10.09
Sample 3	2.573	228.4	10.15
Average	2.579	228.1	10.14

# Table 1b. The weight, failure control and the comprehensive strength of the concrete controlfor 14 days

Concrete Control	Weight (Kg)	Failure Load (K/N)	Compressive Strength(N/MM <sup>2</sup> )
Sample 1	2.611	251.9	11.20
Sample 2	2.620	254.0	11.29
Sample 3	2.401	244.2	10.85
Average	2.616	243.0	11.24

# Table 1c. The weight, failure control and the comprehensive strength of the concrete controlfor 28 days

Concrete Control	Weight (Kg)	Failure Load (K/N)	Compressive Strength(N/MM <sup>2</sup> )
Sample 1	2.599	330.8	14.70
Sample 2	2.641	331.0	14.71
Sample 3	2.651	334.0	14.84
Average	2.630	331.9	14.75

The tables below show the weight, load failure and the compressive strength of concrete mixed with 10g, 20g and 30g of layered double hydroxides.

Concrete mixed with 10g LDH	Weight (Kg)	Failure Load (K/N)	Compressive Strength(N/MM <sup>2</sup> )
Sample 1	2.592	229.7	10.21
Sample 2	2.611	227.9	10.13
Sample 3	2.618	229.4	10.19
Average	2.607	229.0	10.18

Table 2a. The weight, failure control and the comprehensive strength of the concrete controlmixed with 10g of layered double hydroxide for 7 days

 Table 2b. The weight, failure control and the comprehensive strength of the concrete control mixed with 10g of layered double hydroxide for 14 days

Concrete mixed with 10g of LDH	Weight (Kg)	Failure Load (K/N)	Compressive Strength(N/MM <sup>2</sup> )
Sample 1	2.619	255.9	11.37
Sample 2	2.604	254.7	11.32
Sample 3	2.640	256.2	11.39
Average	2.621	255.6	11.36

Table 2c. The weight, failure control and the comprehensive strength of the concrete control mixed with 10g of layered double hydroxide for 28 days

Concrete mixed with 10g LDH	Weight (Kg)	Failure Load (K/N)	Compressive Strength(N/MM <sup>2</sup> )
Sample 1	2.562	334.8	14.88
Sample 2	2.611	337.1	14.98
Sample 3	2.613	331.9	14.75
Average	2.595	334.6	14.87

Table 3a. The weight, failure control and the comprehensive strength of the concrete control mixed with 20g of layered double hydroxide for 7 days

Concrete mixed with 20g LDH	Weight (Kg)	Failure Load (K/N)	Compressive Strength(N/MM <sup>2</sup> )
Sample 1	2.602	231.1	10.27
Sample 2	2.617	229.9	10.21
Sample 3	2.599	232.4	10.33
Average	2.606	231.1	10.27

 Table 3b. The weight, failure control and the comprehensive strength of the concrete control mixed with 20g of layered double hydroxide for 14 days

Concrete mixed with 20g of LDH	Weight (Kg)	Failure Load (K/N)	Compressive Strength(N/MM <sup>2</sup> )
Sample 1	2.599	255.9	11.37
Sample 2	2.762	254.7	11.32
Sample 3	2.791	256.2	11.39
Average	2.777	255.6	11.36

Table 3c. The weight, failure control and the comprehensive strength of the concrete controlmixed with 30g of layered double hydroxide for 28days

Concrete mixed with 20g LDG	Weight (Kg)	Failure Load (K/N)	Compressive Strength(N/MM <sup>2</sup> )
Sample 1	2.622	335.7	14.92
Sample 2	2.638	361.1	16.05
Sample 3	2.601	359.0	15.96
Average	2.630	360.1	16.01

Concrete Mixed with 30g	Weight (Kg)	Failure Load (K/N)	Compressive Strength(N/MM <sup>2</sup> )
Sample 1	2.700	408.1	18.14
Sample 2	2.688	410.1	18.23
Sample 3	2.679	408.9	18.17
Average	2.689	409.0	18.18

# Table 4a. The weight, failure control and the comprehensive strength of the concrete control mixed with 30g of layered double hydroxide for 7 days

 Table 4b. The weight, failure control and the comprehensive strength of the concrete control mixed with 30g of layered double hydroxide for 14 days

Concrete mixed with 30g LDH	Weight (Kg)	Failure Load (K/N)	Compressive Strength(N/MM <sup>2</sup> )
Sample 1	2.789	413.3	18.37
Sample 2	2.799	415.9	18.48
Sample 3	2.801	414.6	18.43
Average	2.796	414.6	18.43

# Table 4c. The weight, failure control and the comprehensive strength of the concrete controlmixed with 30g of layered double hydroxide for 28 days

Concrete Control	Weight (Kg)	Failure Load (K/N)	Compressive Strength(N/MM <sup>2</sup> )
Sample 1	2.723	480.0	21.33
Sample 2	2.769	479.2	21.30
Sample 3	2.699	415.0	18.44
Average	2.730	331.9	21.32

The table below compares the average compressive strength of the control concrete with concrete mixed with different proportion of Layered Double Hydroxide.

### Table 5. Comparasion of the average compressive strength of the control concrete with concrete mixed with different proportion of layered double hydroxide

Days	C/S of control (N/MM <sup>2</sup> )	C/S of concrete with 10g of LDH (N/MM <sup>2</sup> )	C/S of concrete with 20g of LDH (N/MM <sup>2</sup> )	C/S of concrete with 30g of LDH (N/MM <sup>2</sup> )
7	10.14	10.18	10.27	18.18
14	11.24	11.36	11.36	18.43
28	14.75	14.87	16.01	21.32

From the Table 5 the results show that there is no significant difference between the compressive strength of concrete mixed with 10g and 20g of layered double hydroxide and that of the compressive strength of the concrete control in the 7, 14 and 28 days of curing, while there is a significant difference in the compressive strength of concrete mixed with 30g layered double hydroxide compared to that of the compressive strength of the concrete control.

It was observed that the compressive strength of the concrete mixed with 30g of layered double hydroxide was increased by 62.62%. The Scanning Electron Microscope (SEM) micrographs in the appendix in Figs. 1 and 2 show the effects of the CaAI-LDHs additives on the morphology of the cement concrete. It can be seen that the cement concrete containing the LDHs is denser and less porous than that without the LDHs. The porosity in cement paste can be classified into two main groups: capillary pores and gel pores. The capillary pores, which are the remnants of water-filled spaces between hydrated cement grains are relatively large and can be observed by SEM. The effects of capillary pores on the compressive strength and permeability of concrete are more significant than those of gel pores. The micrographs in Fig. 3 suggest that there are fewer capillary pores in the cement concrete containing the LDH additives, which is responsible for its increased compressive strengths compared to the cement concrete without LDHs.

The compressive strength of any material is defined as the resistance to failure under the action of compressive forces. Especially for concrete, compressive strength is an important parameter to determine the performance of the service material during conditions. The compressive strength of concrete cube test about most provides an idea of the characteristics of concrete. Concrete compressive strength for general construction varies from 15MPa (2200 psi) to 30 MPa (4400 psi) and higher in commercial and industrial structures.

Compressive strength of concrete depends on many factors such as water-cement ratio, cement strength, quality of concrete material, and quality control during production of concrete etc.

# 3.4 Procedures for Failure Load and Compressive Strength Test of Concretes Cubes

The concrete was poured in a mould of size 15cm x 15cm x 15cm and tampered properly so as not to have any void using a vibrating tables. After 24 hours the moulds were removed and the test specimens were put in water for curing. The top surface of the specimens was made even and smooth, this was done by putting cement paste and spreading smoothly on the whole area of specimen.

These specimens were tested by compression test machine for failure load after 7 days, 14 days and 28 days of curing. Load was gradually applied at the rate of 140 kg/cm<sup>2</sup> per minute till the specimen fails. Load at the failure divided by the area of specimen gives the compressive strength of concrete. The sequential steps that were carried out during the concrete cube testing are as follows;

- 1. Removal of the specimen from water after a specific curing time was done.
- 2. Wiping out of excess water from the surface of the specimen
- 3. The dimension of the specimen was taken to the nearest 0.2m.

- The specimen was placed on the compression machine in such a manner that the load was applied to the opposite side of the cube.
- 5. The specimen was aligned centrally on the base plate of the machine.
- 6. The movable portion of the machine was put into rotation by hand so as to touch the top surface of the specimen.
- The load was applied gradually without shock and continuously at the rate of 140 kg/cm<sup>2</sup>/minute till the specimen fails.
- 8. The maximum load was recorded.
- 9. Minimum of three specimens were tested at each selected age.

If strength of any varies by more than 15 percent of average strength, result of such specimen was always rejected. Average of three specimens gives the crushing strength of the concrete.

# 3.5 Factors Affecting Compressive Strength Test Results

Concrete compressive strength is affected by many factors, such as quality of raw materials, water/cement ratio. coarse/ aggregate ratio, age of concrete. fine compaction of the concrete, temperature, relative humidity, curing of concrete, machine and man.

# 3.5.1 Quality of the raw materials

**Cement:** Provided the cement conforms to the appropriate standard and it has been stored correctly (i.e. in dry conditions), which makes it suitable for concrete to use.

**Aggregate:** Quality of aggregates, its sizes, shape, texture, strength etc determines the strength of concrete. The presence of salts (chloride and sulphate), silt and clay also reduces the strength of concrete.

**Water:** Frequently the quality of the water is covered by a clause stating "the water should be fit for drinking". This criterion though is not absolute and reference should be made to respective codes for testing of water construction purpose.

Water / Cement Ratio: The relation between water cement ratio and strength of concrete is shown in the plot as shown below.



Free water/cement ratio

The higher the water/cement ratio, the greater the initial spacing between the cement grains and the greater the volume of residual voids not filled by hydration products.

#### 3.5.2 Coarse/fine aggregate ratio

The following points should be noted for coarse/ fine aggregate ratio:

- If the surface area of the aggregate has increased, the water demand will also increase.
- If the proportion of fines is increased in relation to the coarse aggregate, the overall aggregate surface area will increase.
- Assuming the water demand has increased, the water cement ratio will increase.
- Since the water cement ratio has increased, the compressive strength will decrease.

#### 3.5.3 Aggregate/cement ratio

The following points must be noted for aggregate cement ratio:

- If the volume remains the same and the proportion of cement in relation to that of sand is increased the surface area of the solid will increase.
- If the surface area of the solids has increased, the water demand will stay the same for the constant workability.
- Assuming an increase in cement content for no increase in water demand, the water cement ratio will decrease.
- If the water cement ratio reduces, the strength of the concrete will increase.

#### 3.5.4 Age of concrete

the degree of hydration is synonymous with the age of concrete provided the concrete has not been allowed to dry out or the temperature is too low. In theory, provided the concrete is not allowed to dry out, and then it will always be increasing albeit at an ever reducing rate. For convenience and for most practical applications, it is generally accepted that the majority of the strength has been achieved by 28 days.

#### 3.5.5 Compaction of the concrete

Any entrapped air resulting from inadequate compaction of the plastic concrete will lead to a reduction in strength. If there was 10% trapped air in the concrete, the strength will fall down in the range of 30 to 40%.

#### 3.5.5.1 Temperature

The rate of hydration reaction is temperature dependent. If the temperature increases the reaction also increases. This means that the concrete kept at higher temperature will gain strength more quickly than a similar concrete kept at a lower temperature. However, the final strength of the concrete kept at the higher temperature will be lower. This is because the physical form of the hardened cement paste is less well structure and more porous when hydration proceeds at faster rate.

#### 3.5.5.2 Relative humidity

If the concrete is allowed to dry out, the hydration reaction will stop. The hydration reaction cannot proceed without moisture.

#### 3.5.5.3 Uring

It should be clear from what has been said above that the detrimental effects of storage of concrete in a dry environment can be reduced if the concrete is adequately cured to prevent excessive moisture loss.

3.5.5.4 Men

Mould preparation, sampling, compaction, demoulding, curing, handling, identification and testing.

3.5.5.5 Machine

Capacity, stability, accuracy, hardness of machine, platens and calibration.

# 4. CONCLUSION

The use of co precipitation method in synthesis of CaAl-Layered Double hydroxide in ethanol and water mixture which was used as an additive in concrete shows that the higher the weight of layered double hydroxide mixed with concrete reduces the curing time of concrete and also increases the compressive strength of concrete. Further studies should be done on the long term effect of the concrete mixed with layered double hydroxide.

# **COMPETING INTERESTS**

Authors have declared that no competing interests exist.

# REFERENCES

- 1. A De Roy, Claude F, Khalid EM, JP Besse. Anionic clay: treads in pillaring chemistry. Expanded clays and other microporous solids. 1992;108–169.
- 2. Beaudoin JJ, Raki L, Mitchell LD. Layered double hydroxide like materials nanocomposites for use in concrete. Cem. Concr. Res. 2004;34:1717-1724.
- Brownyard TL, Powers TC. Studies of the physical properties of hardened Portland cement paste. ACI J. Prod. 1946;43:101,249,469,549,669, 845,933.
- Cavani F, Trifiro F, Vaccari A. hydrotalcitetype anionic clays, preparation, properties and application. Catal Today. 1991;11(2):173.
- 5. Feldman RF, Sereda PJ. A model for hydrated Portland cement paste and its practical implications. Eng. J. Can. 1970;53:53-59.
- 6. Gardner E, Huntoon KM, Pinnavaia TJ. Direct synthesis of alkoxide-intercalated

derivatives of hydrotalcite-like layered double hydroxides: Precursors for the formation of colloidal layered double hydroxide suspensions and transparent thin films. Adv Mater. 2001;13:1263-1266.

- 7. Geraud E, Prevot V, Ghanbaja J, Leroux F. Macrospically ordered hydrotalcite-type materials using self-assembled colloidal crystal template. Chem Mater. 2006;18:238-240.
- Hibino T, Jones W. New approach to the delamination of layered double hydroxides J. Mater Chem. 2001;11:1321-1323.
- J Leroux, JP Besse. Polymer interleaved layered double hydroxide: A new emerging class of nanocomposites. Chemistry of Materials. 2001;13(10):3507–3515.
- 10. J Leroux, JP Besse. Biopolymer immobilization during the crystalline growth of layered double hydroxide. Journal of Solid State Chemistry. 2004;117(1):245– 250.
- Ling S, Dianqing L, Jianrong W, Sufeng L, David GE, Xue D. Synthesis, flameretardant and smoke-suppressant properties of a borate-intercalated layered double hydroxide. Clays and Clay Materials. 2005;53(3):294–300.
- Manzano H, Dolado JS, Guerrero A, Ayuela A. Mechanical properties of crystalline calcium silicate hydrates, comparison with cementitious C-S-H gels. Phys. Status Solidi. 2007;204: 1775-1780.
- M Zhang, Bin G, Ying Y, Mandu I. Phosphate removal ability of biochar / MgAI – LDH ultrafine composite prepared by liquid phase deposition. Chemosphere. 2013;92(8):1042–1047.
- 14. Odawood AO. Concrete technology lecture note, department of civil engineering, Misan University; 2016.
- Sandip K, Asish M, Chapal KD. Layered double hydroxide modified into linseed compounds for polyurethane elastomer nanocomposites. Polimery. 2012;57(1):18– 24.
- S Lv, Wei Z, Hui M, Wenfang S. Preparation and properties of polymer / LDH nanocomposite used for UV curing coatings. Progress in Organic Coatings. 2009;65(4):450–456.
- Wei T, Bi Yan C, Jian-Zhong L. Genome engineering Escherichia coli for L-DOPA over production from glucose. Scientific Reports. 2016;6(1):1–9.
- 18. Y Kuang, Lina Z, Shuai Z, Fezhi Z, Mingdong D, Sailong X. Morphologies,

preparations and applications of layered double hydroxide micro -/ nano structures. Materials. 2010;3(12):5220 – 5235.

 Y Zhao, Feng Li, Rui Zhang, David GE, Xue D. Preparation of layered double – hydroxide nanomaterials with a uniform crystallite size using a new method involving separate nucleation and aging steps. Chemistry of Material. 2002;14(10):4286–4291.



# APPENDIX

FOV: 537 µm, Mode: 15kV - Map, Detector: BSD Full, Time: MAY 2 2019 15:11



Fig. 1. SEM Result for layered double hydroxide Disabled elements: O

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and the way of the	Element	Element	Element	Atomic	Weight
	Number	Symbol	Name	Conc.	Conc.
and the Barris and the	20	Ca	Calcium	27.33	48.16
	6	С	Carbon	54.09	28.57
	14	Si	Silicon	12.03	14.86
	13	Al	Aluminium	3.19	3.79
North and the second second	12	Mg	Magnesium	1.10	1.17
	11	Na	Sodium	0.96	0.97
	26	Fe	Iron	0.34	0.84
	15	Р	Phosphorus	0.47	0.64
	25	Mn	Manganese	0.21	0.51
	19	K	Potassium	0.28	0.48

FOV: 537 µm, Mode: 15kV - Map, Detector: BSD Full, Time: MAY 2 2019 15:03



Fig. 2. SEM Result of concrete control after 28 days of curing Disabled elements: O

in the second	Element	Element	Element	Atomic	Weight
i and a state in the second	Number	Symbol	Name	Conc.	Conc.
1 A State of the s	20	Са	Calcium	27.62	51.18
	6	С	Carbon	63.44	35.22
	14	Si	Silicon	3.59	4.66
Par a la la	30	Zn	Zinc	0.78	2.37
	13	Al	Aluminium	1.50	1.87
	11	Na	Sodium	1.04	1.10
and the second se	19	K	Potassium	0.56	1.00
	26	Fe	Iron	0.37	0.94
	12	Mg	Magnesium	0.77	0.87
the second s	23	V	Vanadium	0.33	0.77
15kV - Map MAY 2 2019 14:47					

FOV: 537 µm, Mode: 15kV - Map, Detector: BSD Full, Time: MAY 2 2019 14:47



## Fig. 3. SEM Result for concrete mixed with 30g of LDH after 28days of curing

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