RESEARCH THESIS

INVESTIGATE THE USE OF SINTERED WATER TREATMENT SLUDGE AS AN INTERNAL CURING FINE AGGREGATE FOR INTERNAL CURING CONCRETE

Pradeep K. I.

198058F

Supervised by: Prof. W.K. Mampearachchi

Degree of Master of Science (Major component of Research)

Department of Civil Engineering

Faculty of Engineering

UNIVERSITY OF MORATUWA-SRI LANKA

June 2021

RESEARCH THESIS

INVESTIGATE THE USE OF SINTERED WATER TREATMENT SLUDGE AS AN INTERNAL CURING FINE AGGREGATE FOR INTERNAL CURING CONCRETE

Pradeep K. I.

198058F

Supervised by: Prof. W.K. Mampearachchi

This Research Thesis submitted in partial fulfilment of the requirements for the degree of Master of Science (Major component of Research)

Department of Civil Engineering

Faculty of Engineering

UNIVERSITY OF MORATUWA-SRI LANKA

June 2021

DECLARATION

I, Pradeep K. I. declare that the research report entitled “Investigate the use of water treatment sludge as an internal curing fine aggregate for internal curing concrete” submitted by me to Department of Civil Engineering, University of Moratuwa in partial fulfilment of the requirement for the award of the degree of Master of Science (Major component of Research) is carried out by me under the supervision of Prof. W. K. Mampearachchi. This is my own work and is submitted in belief that it does not include any material previously published except for those to which acknowledgement is made in the text.

Also, I hereby grant to University of Moratuwa the non-exclusive right to reproduce and distribute the thesis, in whole or in part in print, electronic or other medium. I retain the right to use this content in whole or part in future works.

……..
Pradeep K. I.
198058F

Date; 02/04/2023

The undersigned hereby certified that I have read and recommended the thesis for the acceptance in partial fulfilment of the requirements for the Degree of Master of Science (Major component of Research)

……....
Prof. W.K. Mampearachchi
Senior Lecturer,
Transportation Engineering Division,
Department of Civil Engineering,
University of Moratuwa.

Date; 02/04/2023

ACKNOWLEDGEMENTS

I would like to express my sincere gratitude to my supervisor Prof. W. K. Mampearachchi for providing his invaluable guidance, comments and suggestions throughout the duration of this research study. And also, my special thanks to sir for constantly motivating me to work harder to complete the research project successfully.

Next, I principally am thankful for Accelerating Higher Education Expansion and Development (AHEAD) Operation of the Ministry of Higher Education funded by the World Bank who supported me financially to complete this research study.

Further, my special thanks is extended to Dr. S. U. Adikari who facilitated the use of the Ceramic and Heat treatment laboratory in the Department of Material Science and Engineering. And also, the non-academic staff Mr. Mihranga, Mr. Karunarathne and Mr. Rathnayake of the Department of Material Science and Engineering who supported to conduct experiments during the practical sessions. Non-academic staff (Mr. Uditha, Mr. Pathum, Mr. Roshan, Mr. Lanka, Mr. Leenus, Mr. Piyal) of Department of Civil Engineering whose valuable efforts helped me succeed in my experiments. Another special thanks should go to Head of the Department of Earth Resources Engineering- Dr. D.G. I. Samaradivakara who permitted the use of the rock mechanics laboratory. And also, the non-academic staff Mr. Perera of the Department of Earth Resources Engineering who supported me for experiments during the practical sessions.

Moreover, my special thanks to Plant Managers of Kadana, Biyagama, Kaltuwawa and Labugama water treatment plants who supported me to obtain raw materials for the research. And also, the Plant manager of Dankotuwa Porcelain-Mr. Vinoba and Mr. Demal supported me in the manufacturing of aggregates in a large scale. The plant manager of Sumagi tile factory-Mr Chanaka, Director and deputy director of Lanka Refractories Ltd-Mr Piyasiri and Mr. Kelum, General Manager of Midaya Ceramics (Pvt) Ltd-Mr Anura who gave invaluable support towards me in manufacturing a large quantity of aggregate should be highly admired.

Finally, I would also like to acknowledge Dr. Hasitha Bandara who supported me by sharing knowledge, theory on the research, and my friend Mr, T. Tharshigan who supported me all the time in any practical for the success of this research, and all the Civil Engineering staff and students for their cooperation in numerous ways.

CONTENT

FIGURES

TABLES

KEYWORDS

ICFA ; Internal Curing Fine Aggregates

ICC ; Internal Curing Concrete

ECC : External Curing Concrete

NCC ; Not Curing Concrete

WTP ; Water Treatment Plant

WTS ; Water Treatment Sludge

ABSTRACT

The thesis reports the investigation carried out in developing an internal curing fine aggregate using water treatment sludge, and studying the changes of concrete properties in using developed fine aggregates in internal curing concrete. The water treatment sludge is converted to burnt clay chips through the process of clay mixing, sintering and crushing. Initially, Thermogravimetric analysis was carried out to identify the thermal behavior of sludge. Also, an X-ray fluorescence test was conducted to identify the chemical composition of the sludge. The firing temperature was made to range from 800^0C to 1300^0C at 100^0C intervals. Water absorption, Water desorption, and Relative density tests were conducted on the developed fine aggregates which were heated to different temperatures to observe the physical properties and requirements in order to satisfy the internal curing property. Furthermore, Scanning Electron Micrographs (SEM) analysis was followed to observe the microstructure of the fine aggregates. To carry out the above experiments, the temperature range which had to be maintained in the sintering process was between 975^0C and 1200^0C. The water absorption of the developed ICFA ranged from 15%-25% for the optimum temperature range, while water desorption rate was recorded to be 90%-95% under the 94% relative humidity at 25^0C room temperature. The relative density of the selected ICFA was recorded to range between 1.7 and 2.0, while the bloating coefficient ranged between 0.8-0.9 for the optimum heating temperature. The latter part of the research was focused on studying the improved properties of ICC using developed ICFA. The mix design was done for grade 30 concrete by referring to BS8110-1983 and ASTM-C1761M codes. Workability, compressive strength and drying shrinkage was compared with normal concrete. Workability and the compressive strength of ICC displayed higher values than that of ECC. Moreover, a significant value deviation was observed in the drying shrinkage of ICC: a 50% reduction in 28 days. Initial studies on water treatment sludge and improved properties of developed ICC confirmed that the sludge can be used to develop an ICFA and that it can be utilized in the construction industry.

1 INTRODUCTION

1.1 GENERAL

In the recent past, the demand for construction materials has highly increased. In order to balance the demand, a significant amount of attention needs to be given towards developing a sustainable material using industrial waste. Due to the scarcity of natural resources and the adverse environmental impacts associated with the use of natural resources, most countries have been investigating on the development of an alternative material that can be employed in the construction industry. Researches have attempted to use industrial sludge as a construction material by making it undergo different processes.

Worldwide, a substantial amount of industrial sludge, such as sludge from the brewing industries, paper sludge and co-generation ashes of paper mills, sludge generated from copper slag recycling processes, sewage from waste water treatment plants, textile laundry sludge, and water treatment sludge from drinking water treatment plants only to list a few, are being dumped in disposal yards daily. This has led to the relevant authorities having to face disposal issues due to the immense quantity generated.

Most researchers have found several ways to either reuse or develop them to use as construction materials. Certain types of sludge can be utilized for partial replacement of clay from bricks and ceramics, for the development of light weight aggregates and as an internal curing agent for concrete. Also, to further the purpose of converting waste into a value-added product, various methodologies have been adopted.

The development of fine aggregates using the above-mentioned sludge types are also significantly prominent among researchers, owing to the scarcity of fine aggregates and the ability to minimize the adverse impact on the environment caused by sand mining from river beds. Among these sludge types, water treatment sludge was selected in this study to develop fine aggregates that show internal curing characteristics.

Internal curing of concrete gives solutions to some prevailing problems in the construction industry. Simply put, internal curing means the curing of concrete; water is supplied internally by water reservoirs in the freshly casted concrete. Water reservoirs are the aggregates which can store water and release water when needed for cement hydration. An effective cement hydration process occurs through internal curing as the water reservoirs can be distributed throughout the concrete uniformly.

Water treatment sludge collected from Kadana and Biyagama water treatment plants were taken for the study. The authorities have stated that 6 - 10m^3 of sludge is generated daily. However, the amount of sludge generated during rainy days is twice as much of that generated in a normal day. Furthermore, the only way of disposing the sludge is open dumping in landfills. Biyagama water treatment plant disposes those sludge daily as pressed blocks, while the Kadana plant stores the sludge in sludge lagoons and disposes them once a month. Thus, a huge quantity of sludge is misspent without reaping any benefit.

To further the research, physical, mechanical and chemical properties of water treatment sludge were studied. The initial feasibility study of water treatment sludge conducted to develop it as a fine aggregate was successful, and the study revealed that the developed fine aggregates have internal curing properties. In the latter part of the study, internal curing concrete was produced to investigate and identify the structural properties, and it can be noted that satisfactory results were obtained.

The importance of this research can be brought down to the very fact that the huge quantity of sludge which is not so far in use can be turned into a value-added product, which in turn solves the prevailing problems germane to external curing as well as those that may come up in the future and offers a solution for the disposing of waste issue faced by the relevant authorities.

1.2 PROBLEM STATEMENT

This research study aims to solve several problems in the construction industry. Some of the core problems are the scarcity of fine aggregates and the how the extracting of fine aggregates affects the environment. The current demand for sand in the construction industry is over 7.5 million cubic meters per year in Sri Lanka [1]. River sand mining adversely affects the natural equilibrium and is associated with even more environmental problems. Therefore, the need for an alternative for sand has become a principal topic in the construction industry.

The other issue in focus is that concerning waste materials from factories. The disposal of sludge waste from water purification plants is becoming a huge issue for related authorities. In 2015, National Water Supply Drainage Board-Sri Lanka produced 590 million m^3 of drinking water per year. Normally 2% of water production is produced as sludge and it is a bulk amount that cannot be borne by their land area [1]. These authorities also make monetary payments to several parties to dispose the water treatment sludge, and disposes the sludge to landfills without making any use out of it.

Yet, there is no developed fine aggregate in Sri Lanka that meets the internal curing property requirements needed for cement hydration. Most countries use natural aggregates that have porosity and have developed light weight aggregates that have both absorption and desorption properties.

Though this may be the case, there is no existing substance that can fulfill all the requirements of internal curing as one aggregate which can be utilized to produce effective internal curing concrete.

Upon considering the 3 issues afore mentioned, the following objectives were established in order to initiate the research.

1.3 OBJECTIVES

Following points are the main objectives of the research,

- Developing a fine aggregate from water treatment sludge to enhance the internal curing characteristics.
- Checking the possibility of using the developed internal curing fine aggregate in the production of concrete.

1.4 SIGNIFICANCE OF THE RESEARCH

An internal curing fine aggregate using water treatment sludge will replace the portion of fine aggregates in concrete production. Part of the fine aggregates replaced by ICFA in concrete will help mitigate the environmental problems. Another main advantage of ICFA is that it will be a solution for the disposal of water treatment sludge generated in vast quantities by plants.

Considering the internal curing property of the aggregate, it is noted that it directly affects the concrete strength and shrinkage. Concrete gains its strength while reducing shrinkage due to the effective cement hydration process.

1.5 SCOPE OF THE STUDY

This study has been limited to water treatment sludge. Even though there are several properties in the construction materials, this study focuses on improving internal curing properties. In addition, the possibility of using the developed internal curing fine aggregate in the construction industry has been tested only for concrete.

2 LITERATURE REVIEW

2.1 CURING OF CONCRETE

American Concrete institute describes curing as “Action is taken to maintain moisture and temperature conditions in a freshly placed cementitious mixture to allow hydraulic cement hydration and pozzolanic reactions to occur so that the potential properties of the mixture may develop.” Curing starts immediately after the placing and finishing of concrete.

Concrete curing enhances the cement hydration process to achieve the desired concrete strength while improving the microstructure by developing better hydrate gels and solid masses. Curing, controls the temperature and moisture loss which occur due to solar radiation and wind, while improving the durability and reducing cracks in concrete. So, properly cured concrete shows its effective performance by maintaining an adequate amount of moisture along with continued cement hydration and strength development.

Concrete mixtures having a low water -cement ratio may require special curing methods. Due to the cement hydration process, decreasing of internal relative humidity causes self-desiccation (internal drying) when water supply fails. This may lead to the achieving of desired concrete properties. Additionally, water loss may be a cause linked to the shrinking of concrete while generating concrete tensile stresses. Consequently, all exposed surfaces have to be protected against moisture evaporation. Therefore, curing of concrete is an essential requirement in the construction industry.

2.1.1 METHODS FOR CURING

Concrete curing can be divided into two major parts and curing methods available under each category is shown in Figure 2.1.

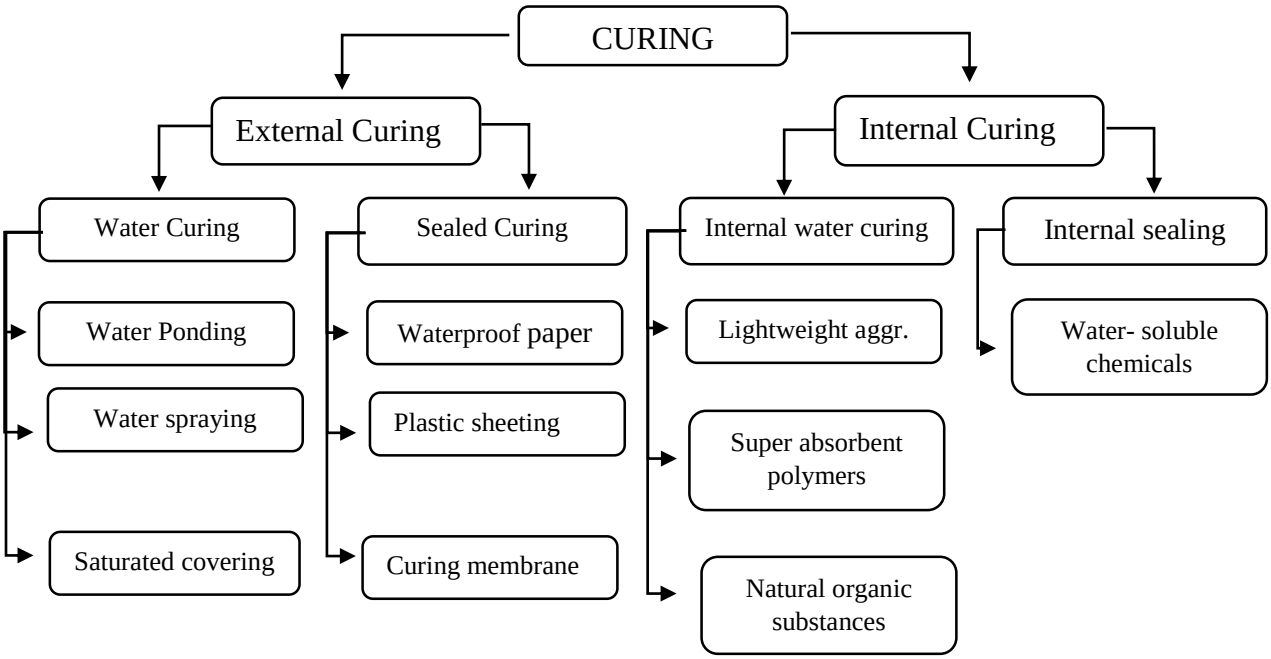

Figure 2.1; Concrete curing methods

2.2 EXTERNAL CURING

Generally, external curing is the conventional curing type in the construction industry. The most suitable curing method depends on few factors: the kind of materials used, construction methods, and what the predicted use of hardening the concrete is. In most cases, application of curing compounds is done by covering the newly casted concrete by impermeable sheets [2].

Ponding is used for flat surfaces by making dikes using soil or sand around the casted concrete surface. It needs a considerable amount of labour and supervision. So, it is used for only minor jobs. Water fogging and sprinkling are much better, if the humidity level is low with ambient temperature well above the freezing point. It raises the relative humidity in the air while slowing down the evaporation of surface water. One of the main disadvantages of sprinkling is the cost for the method and requirement of good supervision.

Fabric coverings such as rugs, cotton mats and fabrics which can retain moisture are also used for curing. It should be applied soon after the hardening of concrete. Wet coverings like earth, sawdust, and sand are more effective for small jobs. However, the use of these materials will discolour the concrete surface.

Another important method of curing is covering the fresh concrete with waterproof paper. It will enhance the cement hydration by avoiding the moisture loss. This method is important as it does not require the addition of water periodically.

Application of plastic sheets is also yet another concrete curing method. It is used to avoid moisture loss and can be applied to any shape of concrete easily. The procedure is same as the procedure followed in the laying of water proof paper.

Membrane curing compounds can be used to minimise the water evaporation by enhancing the cement hydration. Those compounds can retain 80% of relative humidity on the concrete surface for seven days. Normally, 250ml of compounds per square meter is used for curing [2].

Even though there are a lot of ways for concrete curing, any method entails one or two disadvantages. Insufficient moisture inside the concrete will affect the cement hydration. Plastic shrinkages, temperature imbalances cause thermal cracks to occur, if water is not supplied periodically. Cost for curing compounds, impervious papers, and other materials related to curing and labour, supervision costs are also another major disadvantage in external curing. Further, external curing is effective only for several millimetres from the top of the concrete surface. Therefore, any external curing method is not an air tight solution for the internal drying of concrete.

2.3 INTERNAL CURING

Internal curing is an alternative method for conventional curing methods, as well as that it is recognized as a new construction technology. American Concrete Institute states that internal curing is "Supplying water throughout a freshly placed cementitious mixture using reservoirs, via pre - wetted lightweight aggregates, that readily release water as needed for hydration or to replace moisture lost through evaporation or self-desiccation." [3]

The first publisher, Paul Klieger had published a paper on internal curing: "Lightweight aggregates absorb considerable water during mixing which apparently can transfer to the paste during hydration." In 1991, Robert Philleo who is a concrete technologist wrote, "Either the basic nature of Portland cement must be changed so that self-desiccation is reduced, or a way must be found to get curing water into the interior of high strength structural members." [3] Considering both concepts, many researchers have started to investigate internal curing using pre-wetted light weight aggregates. Later, researchers had started to find materials that can perform as internal water reservoirs. They had found out that super absorbent polymers, and water absorbed wood fibres can also be used as internal curing agents.

2.3.1 Theory behind Internal Curing

The requirement of internal curing is directly due to the basic nature of cement hydration reactions. Due to the reactions between cement and water, gel and crystalline hydration products are formed. Water which is required into those products needs less space than which water volume in its real

form. Thus, chemical shrinkage will start with in the mixture as the products which are generated need less volume than reactants [3].

Before setting the concrete, an equivalent physical shrinkage will take place due to the above-mentioned chemical shrinkage inside the fresh concrete microstructure. When the cement paste gets hardened and develops a finite resistance to deformation, with the lack of an extra source of water self-desiccation occurs and results in partly filled pores.

The Young's equation by Alberty and Daniels describes the relationship between partially filled pores and surface tension, size of the largest partially filled pore.

$$\sigma = (-2\gamma cos\theta)/r \qquad (2.1)$$

Where,

σ ; Capillary pressure
γ ; surface tension of the pore solution
θ ; contact angle
r ; pore radius

As per *Equation 2.1*, it is fair that 4 variables should be changed to reduce the capillary stresses inside the cement mixture. Past research papers indicated that the capillary stresses can be minimized by reducing the surface tension using a shrinkage-reducing admixture and increasing the volume of the pores being emptied, by supplying water ponds in larger sized pores to the mixture, which will finally improve the internal curing.

The reduction of internal relative humidity will be subject to self-desiccation and capillary stresses. The Kelvin equation describes the relationship between the internal relative humidity of concrete mixture and capillary pressure.

$$\sigma = [RTln(RH)]/V_m \qquad (2.2)$$

Where,

R ; Universal gas constant
T ; Absolute temperature
RH ; Relative humidity
V_m ; molar volume of pore solution

Internal relative humidity of the concrete can be measured using *Equation 2.2*. Relative humidity of the cement-based system directly affects the strength, thermal characteristics and rate of cement hydration. Bents and Weiss have combined both *Equation 2.1 and 2.2*, which gives a relationship

between relative humidity versus radius of pores which are being emptied [3]. So, a proper curing method is required to maintain internal relative humidity, to reduce early age cracking.

2.3.2 Importance of Internal Curing

Generally, the strength of concrete is inversely proportional to the water-cement ratio. As the amount of water is low for high strength concrete, un-hydrated cement particles may return in the cement-based system. Those concrete mixtures having a low w/c ratio lower than 0.42, may not effectively complete a hydration process due to the lack of moisture [4]. To achieve such a high strength, all the cement particles have to be fully hydrated. Therefore, proper curing techniques should be adopted to complete the hydrating process [5]. According to the concept of internal curing, there is no other way for hydration of cement molecules inside the concrete microstructure.

Mitigating early age crack formation and reducing the long-term drying shrinkage are also other benefits present in internal curing [6]. Delatte clearly derived from their study that internal curing minimizes the permeability while increasing the concrete durability [7]. Weiss also found through his experiments, that the service life of the concrete is increased by internal curing with an improved hydration process. According to field and laboratory experiments by Friggle and Reeves, there is a small amount of reduction in unit weight and elastic modulus when there is a significant reduction in shrinkage of the internal curing concrete [8]. Schlitter also discovered that internally cured concrete reduces the elastic modulus while increasing the strength [9]. In 2011, Ozyildirim had found that internal curing reduces the thermal expansion of the concrete system [10].

Many researchers have conducted plenty of field and laboratory experiments to identify the benefits of internal curing, and it is clear that, all the above variations in concrete will increase the performance of concrete.

2.3.3 Internal Curing Aggregates

Different types of internally cured aggregates are used in the construction industry. The fundamental requisite that internal curing aggregates fulfil is the ability to store water in its pore structure. Those aggregates act as a water reservoir to supply water for cement hydration process. Water gets stored in the aggregate due to the availability of cells in the structure. Those voids can retain water, because of the capillary interaction with the cell membrane and the surface tension of water.

The principle behind water absorbent polymers is that there are a number of cross-linked polymer chains which have negatively charged molecules. As the water particles are polar and have minuscule charges, water particles get attracted to the polymer chains, and also since they are negatively charged, water is held in the polymer network by hydrogen bonding [11].

There are materials with chemically bound water, physically absorbed water and physically held water which can be used as an internal curing aggregate.

Superabsorbent polymers can store a large amount of water without dissolving in water. According to the Jensen study, theoretically the maximum water capacity of superabsorbent polymers is 5000 times its own weight [12]. A study of mixture proportions for superabsorbent polymers has also concluded that compressive strength of concrete is highly dependent on the amount of polymer content.

According to the "RILEM State of the art report" study, recycled aggregates have internal curing properties [3]. The aggregates have a high capacity of water absorption than normal aggregates because recycled aggregates have cement mortar in their outer shells. Furthermore, Kim and Bentz found that there is a considerable amount of reduction in autogenous shrinkage. However, recycled aggregates cannot be used as internal curing aggregates because of them having a poor water desorption property. Since the outer shell of recycled aggregates have the particles in original concrete, it consists of tiny pores. Technically, the water travels from large cells to small cells due to the capillary forces. The presence of micro sized pores in the recycled aggregates as in original concrete, causes a reduction of its functionality as an internal curing aggregate.

Light weight aggregates (LWA) are more popular in the construction industry due to their low bulk density. Different research groups have discovered that light weight aggregates show internal curing aggregate properties due to their pore structure. During the past few decades, Weber S. and Reinhardt have experimented about LWA as internal water reservoirs and have manufactured internal curing concrete successfully [13]. LWA are available in four types which can be categorized as natural, by-products, manufactured structural and insulating aggregates. All the types can be used as internal curing aggregates if they can store and release water during the hydration process. Natural LWAs like pumice, perlite and volcanic cinders have been tested by researchers to discern whether they can be used as ICA, and the results have been positive.

Some clay types expand when heated up to a specific temperature. Due to the burning of organic matters and gases, a pore structure is made in the clay particles. After the sintering process, ceramic pellets will be encompassed with high porosity, high water absorption and low compressive strength. Thus, it can be stated that researches have successfully produced these artificial light weight aggregates as internal curing aggregates.

2.3.4 Requirements of ICA

The two basic requirements needed to act as an ICA are water absorption and water desorption properties. According to ASTM C1761M "Standard specification for lightweight aggregate for internal curing of concrete", the ICA shall have a 72-h absorption not less than 5%. If water absorption is higher in ICA, then it reduces the amount of internal curing aggregate volume for concrete. According to a study about the absorption property of internal curing LWA, researchers identified that the 24hr absorption of different types of fine LWA vary between 6% - 31%. The expanded slates have a 6% to 12% water absorption, the expanded shale have a 24hr absorption between 10% - 20% while expanded clays have a 24hr absorption rate between 15% - 31% [14].

Even if the aggregate can store water meeting the required limits, but cannot desorb water during the cement hydration process, the aggregate cannot be used as an ICA. Complying with the ASTM C1761M, the ICA shall release at least 85% of absorbed moisture at 94% relative humidity.

Past research papers have mentioned that water is not only the vital requirement for internal curing, but also the capability of partial distribution of such water inside the concrete mixture. For this matter, it is better to use fine aggregates instead of coarse aggregates as an internal curing aggregate [14]. Owing to the fact that fine aggregates will distribute internally stored water throughout the concrete microstructure uniformly.

According to the absorption tests by Holm et al, water absorption rate is specific to each type of aggregate based on the pore size, pore continuity and pore distribution. Moreover, in compliance with the Bentz and Weiss review on internal curing, the graph in which internal relative humidity versus kelvin radius is plotted reveals a significant fact: the minimum pore size of ICA to maintain internal relative humidity around 94% should be around 100nm [3]. Simply put, an internal curing aggregate should have minimum 100nm diameter pores to release water at 94% internal relative humidity.

2.3.5 Internal Curing Concrete

The pre-eminent factor of concrete is compressive strength. ICC may increase the compressive strength because of the effective cement hydration process while reducing early age shrinkage cracks as the required amount of water needed for hydration have been supplied by internal water reservoirs.

Even though the most prominent internal curing aggregate is Light weight aggregates, recent study on physical properties of internal curing concrete reveals that reduction of 7day tensile strength can be observed in samples that have 25% of LWA replacement as an internal curing agent [15]. And also, further increment of LWA has decreased the concrete strength. It is due to the presence of mechanical properties in LWA than in conventional aggregates. Likewise, several researchers have

experimented and mentioned that reduction in the compressive strength of concrete because the internal curing agents are strength wise weaker than conventional aggregates. However, Rao has found in his research on ICA that, compressive strength increases up to 5% to 10% while flexural and splitting tensile strength of the concrete increases up to 5% [16].

According to Golias's study, mortar mixtures were prepared with internal curing aggregates to observe the effect of curing conditions on compressive strength of concrete [14]. He found that internal curing water will perform at the latest age by showing that the samples can be stored in a dry environment such as one with 50% relative humidity in the area. He also discovered that there is strength gain in internally cured mixtures than in mixtures which utilized conventional curing methods.

Permeability is a major factor affecting the durability of concrete. Generally, major durability problems occur as normal concrete shows higher permeability due to the shrinkage cracks. According to the recent study by Rao, 60% reduction in shrinkage cracks indirectly reduces the permeability of concrete due to internal curing [16]. Thus, internal curing governs the increase in the durability of concrete.

Henkensiefken has studied about the plastic shrinkage of ICC in comparison to normal concrete. In consequence with the results, if the sufficient amount of LWA (18%) can be used as an ICA for the concrete, it can eliminate plastic shrinkage cracking completely under normal environmental conditions [17]. Thus, more and more researchers have started to find the property changes in internal curing concrete.

For the above factors, it is essential to use the correct proportions of internal curing agents with the concrete, for otherwise it will reduce the performance of concrete. ASTM C1761M, indicates the proportion of internal curing aggregates that needs to be replaced with normal fine aggregates.

2.4 Identification of locally available waste materials

Several types of waste materials are being generated during different industrial processes in Sri Lanka. The quantity of those waste materials and removal of them are huge problems to the related authorities. As a consequence, numerous researches are being carried out to develop a value-added product using the waste.

It is observed that water treatment sludge, fly ash, bottom ash, sewage sludge, textile effluent sludge, recycled mortar, waste ceramics and waste clay tiles are some of the main industrial wastages generated with in Sri Lanka.

Also, Lakvijaya coal power plant at Norochchalai is the largest coal power plant in Sri Lanka while being the pioneer in the supplying of 50% of electricity used in the country. Fly Ash and Bottom Ash are two main by-products of coal combustion. Fly Ash and Bottom Ash are being directly disposed to a dumping yard. Annual production of fly ash is about 150,000 tons and a small amount of the ash is used by INSEE group for their cement manufacturing process. Except for that, all the excess amount of fly ash and bottom ash are moved to a dump yard. There is only a limited space in the dump yard, and the dust emitted to nearby villages cause serious health issues. In addition, the toxicity of certain fly ash can be a problem since it can mix with soil in the dumping yard. Even though a number of researches have been carried out to discover how to use waste to develop a value-added product they still have not succeeded.

The textile industry is a main pioneer of the Sri Lankan economy. Large quantities of water and chemicals are consumed during the dyeing and washing of clothes in the textile industry. The chemicals and dyes which get mixed with water pose a significant pollution hazard and it needs to be treated and disposed to the water reservoirs. So, any textile factory which processes dyeing or washing have a waste water treatment plant. The sludge which is an inevitable solid waste from these waste water treatment plants is categorized as toxic substances by authorities. Currently, a huge amount of sludge generated from the countrywide plants are all being dumped in protected areas at the plant premises. Since the amount of sludge generated is quite high, authorities try to find ways to reuse or give a value to the waste. Recently, many researchers have studied about finding a way to give value to sludge by improving the sludge as fertilizers to cultivate lands, as palletized aggregates to concrete, for ceramics items and clay bricks [18].

Large quantities of construction industry debris have been disposed to landfills at the end of lifetime period of buildings. As a consequence, scholars have researched on the reusing of recycled aggregates and recycled mortar. According to some research findings, those aggregates have a relatively high-water absorption capacity than normal aggregates because their outer shells consist of stone particles and cement mortar. However, those recycled aggregates and mortar cannot be used alone since they display poor desorption capabilities. Since the outer shell of recycled aggregates have the particles in original concrete, it consists of tiny pores. Technically, water travels from large cells to small cells due to the capillary forces. Presence of micro pores in the recycled aggregates similar to those in original concrete, makes them less useful as internal curing aggregates.

Bringing attention towards the Ceramic industry in Sri Lanka, it can be observed that it is a huge exporting industry in which a number of new ceramic items and ornaments are finished day to day. Around 5% of the daily production is removed as ceramic waste according to the data of Dankotuwa

porcelain. Some companies reuse this waste by mixing them again to the raw material. However, some are disposed since the clay types have changed their phases. Those ceramic waste can be used in construction industry as a fine aggregate since it has a high strength as conventional aggregates. Waste clay tiles are also another locally available material which can be developed to be used in the construction industry. Several researchers have conducted studies on the properties of concrete by adding those tile waste as a coarse aggregate. High water absorption in the waste can be noted as one good property aiding the decision as to whether it can be used as an internal curing agent.

2.5 Study on Water Treatment Sludge

Raw water extracted from surface water contain a wide variety of organic and inorganic contaminants and suspended solid particles. Most of the surface WTP is based on the steps such as Coagulation, Flocculation, Sedimentation and Filtration and is typically followed by aeration, and proceeded by a disinfection producing a huge amount of sludge to obtain the quality of drinking water. *Figure 2.2* shows the dried water treatment sludge generated in Kadana water treatment plant.

Figure 2.2; Dried sludge in sludge lagoons of Kadana Water Treatment Plant

According to the 2015 data analysis, National Water Supply Drainage Board-Sri Lanka produces 590 million m^3 of drinking water per year. Normally 2% of the water production is produced as sludge [1]. The removal of the sludge is becoming a huge problem to the local authority as well as for other countries due to the large quantity. Several countries discharge that sludge to the water reservoir again without any treatment. However, it leads to an increase in the concentration of aluminium in water, aquatic organisms and even human bodies. Some medical researchers have found that the aluminium's contributory influence to children's mental retardation. Certain countries have even opted to removing the sludge to landfills. *Figure 2.3 and 2.4* shows the removal of sludge to landfills. Yet, the severity of heavy metals and aluminium concentration is similar as the above since it can be

mixed with the soil. Therefore, a proper method of discharging and managing should be arranged to dispose the sludge without causing any environmental hazards.

Figure 2.3; Land fill of Kadana Water Treatment Sludge

Figure 2.4; Removal of Water Treatment Sludge to Dumping Yards

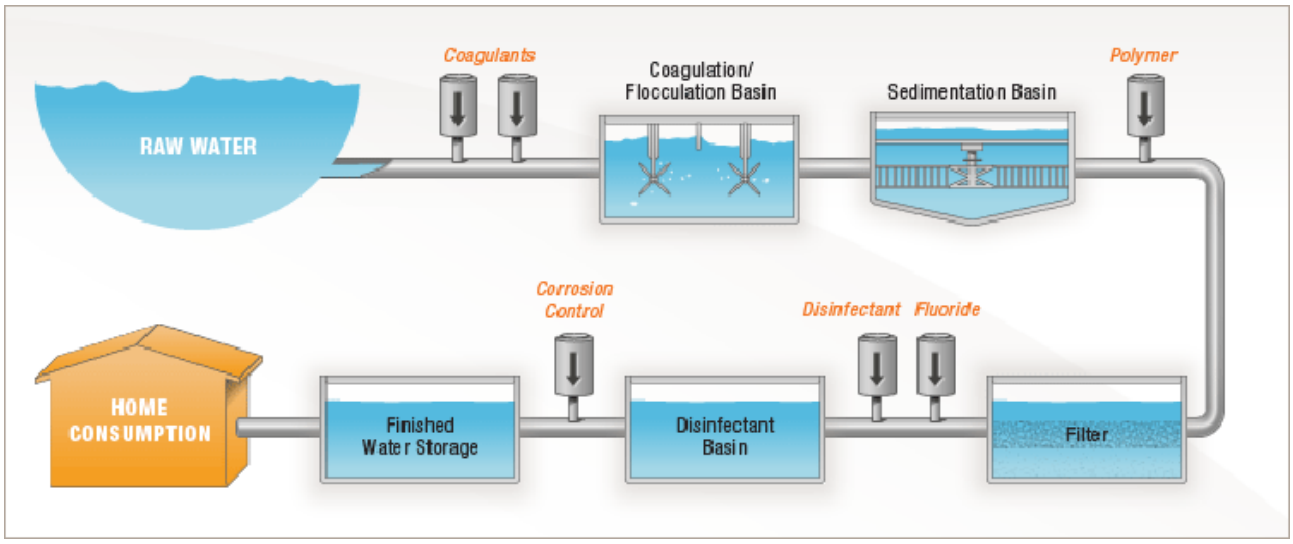

Figure 2.5; Flow chart of a conventional water treatment plant

Figure 2.5 shows the steps of a conventional treating process. Firstly, raw water which is extracted from the reservoir is pumped to the first mixing chamber through screening to remove large debris such as rags, plastics, paper and metals to avoid damage to pumps. The second step of the WTP is pre chlorination and the mixing of coagulant for coagulation. Lime (Ca(OH)2) is added to the raw water to adjust the pH value to 6.2-6.5 in pre-chlorination. Afterwards, Alum (AL_2SO_4) is added to the pH corrected water for coagulation in the mixing chamber.

The next steps of the process are flocculation and clarification. Variable speed motors are set up for flocculates. Several sedimentation methods are used in the flocculation tanks and those are collected by the sludge hoppers and pumped to a sludge thickener. Then the processed water is moved to the sand filters for filtration. The final stage of the process is disinfection or post chlorination. Lime is added to the final treated water to avert the contamination of pathogens and indirectly prevents waterborne diseases while maintaining the pH value between 6.5 to 6.7. Then the treated clean water is stored in large tanks and distributed to the population.

Sludge which is produced from the clarifier and filtration is collected in the sludge balancing tank. A polymer (polyacrylamide-powder form) is added to the liquid to further the formation of flocs, removal of excess moisture and reduction of the volume of sludge. That thickened sludge is then moved to a sludge decanter or sludge lagoon to remove excess moisture.

In Sri Lanka there is no other way of removing sludge than landfills. Yet worldwide, most countries have started to research on sludge to add a value and they have succeeded in several instances. Brazil has developed a composite based on water treatment sludge with sawdust and has used it as coarse lightweight aggregate for concrete. In Taiwan, WTS is used as a raw material for brick production.

Researchers in Egypt have found that it can be used to cast bricks by WTS and burning of ashes. Physical, chemical and mechanical properties which are similar to clay open a path for that sludge to be developed in to a value-added product.

2.6 Internal Curing Aggregate production

Several countries have used natural aggregates which show internal curing properties for ICC, while some countries have researched on developing a structure to retain water to act as an internal curing agent using naturally available raw material which cannot be directly used as an internal curing aggregate. A limited number of methodologies concerning the development of an ICA have been published by researchers and this chapter determines what methodology should be adopted in converting water treatment sludge in to an internal curing aggregate.

Most countries have researched on shale, clay types and slags to develop ICA by following a sintering process, as the heating can change its phase and structure due to chemical reactions, burning of inorganic matters and emission of various type of gases. The materials which can develop a porosity inside the structure will fulfil one characteristic for internal curing. During the heating process, some raw materials release gases and expand while some accumulate space inside the structure by burning organic matter. At a specific temperature, raw material gets melted and change its phase to a plastic form not showing further clay properties and that will be beneficial to retain strength properties.

2.6.1 ICC Production Process

Normally, in the production stage, following steps have been followed by most researchers.

I. Raw material extraction (Mining)
II. Seasoning (Drying under sun light or mechanization)
III. Grinding or Pulverizing
IV. Blending or crushing
V. Sieving (to remove debris and to retain required particle size)
VI. Palletisation or casting plates
VII. Heating
VIII. Cooling
IX. Crushing
X. Screening

Selected raw materials were extracted from clay mines and the seasoning was done to remove excess moisture for the purpose of the next step of crushing it into clay powder, or to retain a certain amount of moisture for the step of palletizing. As well as that, due to the absence of clay properties of some

raw materials, usually they are blended with other clay materials to achieve a homogenous clay mass. Subsequently, different sizes of clay pallets were palletized mechanically and were moved for the sintering process. The firing temperature and the firing environment, and rates were decided according to the thermos- gravimetric analysis of the material.

2.6.2 Firing Process

A major part of the production line of ICA is the firing or heating step. Some countries have used a rotary kiln for this step while some are using large scale gas or electric tunnel furnaces. The heating rate, and heating environment depends on the material and manufacturer's needs.

During the firing period, clay undergoes different chemical reactions at different stages. Some researchers have decided firing curves during the process. One research paper describes its firing stages as "The pallets were pre-dried overnight in an oven for 105^0C before sending to the furnace. Later the firing temperature was held to 550^0C for 1hour to ensure the removal organic matters and another one hour at 900^0C to avoid in black coring effect and complete oxidation. Finally, pellets were heated to 1135^0C for 4hours and cooling rate was delayed to support for recrystallization" [18]. Hung and Hwang described in their paper that firing pattern as "the temperature was increased at a fixed rate of 5^0C/min and kept for 10-12min at 1200^0C and set for cooling in the furnace" [19].

2.6.3 Effects of Firing

As per past research revelations, clay undergoes several milestones during the period of the sintering process. Releasing of physically bound water before 150^0C, vaporization of sulphur materials at 400^0C, releasing of chemically bound water at 600^0C, formation of CO_2 at 700^0C and gas formation due to Fe_2O_3 at 1100^0C can be identified in general.

Number of chemical reactions occurs at several heating stages according to its chemical composition. Generally, those stages can be found by thermo-gravimetric analysis and differential thermal analysis. The exothermic peaks, endothermic peaks, weight loss and heat flow variations with time, gives a clear clarification on how raw material changes with temperature. Chemical compositions of the raw material can be found by the X-ray fluorescence test. A rough estimation for heating temperature, environment and rate can be found through those analysis.

3 EXPERIMENTAL DESIGN OF THE STUDY

3.1 Introduction

This research is an attempt to develop an economical and effective internal curing aggregate using water treatment sludge and to use it with concrete, and to study the properties. All the test procedures and methodology regarding how the aggregate was developed to fulfil the requisites of internal curing properties and areas such as strength gain, shrinkage variation and durability of concrete is described in this chapter.

3.2 Selection of Materials

The water treatment sludge is generated in Western province of Sri Lanka (Water treatment plants : Kadana, Biyagama, Labugama and Kaltuwawa) were selected for the study. Initially, the sludge was tested to identify the properties,whether they can be developed as an ICA and to decide which methodolgy should be adopted to manufacture it.

Figure 3.1A; Biyagama WTS

Figure 3.1B; Kadana WTS

Figure 3.1C; Kaltuwawa WTS

Figure 3.1D; Labugama WTS

Figures 3.1 A-D shows the several types of water treatment sludge which were collected from water purification plants in the Western province of Sri Lanka. The colour, moisture content, chemical properties and mechanical properties are different from each other.

3.2.1 Tests on Atterberg Limits

The atterberg limits of WTS were determined BS 1377-2-1990 to identify the liquid limit and plastic limit [20]. It is because a mixture with a moisture content above the liquid limit cannot retain its shape due to the paste being in a liquid state. On the other hand a mixture with a moisture content below the limit of plasticity will easily crumble [18]. Therefore, the moisture content of the sludge paste is highly consequential during the manufacturing process.

3.2.1.1 Determination of the liquid limit

The Casagrande type of test is followed in this study for two main types of tests. As per the standard, the clay particles should be less than 0.425mm. The sludge was dried and crushed to powder form using a laboratory ball mill. Afterwards, the powder was seived by a 0.425mm seive, obtaining the sludge powder that gets sieved for the experiment. Next, the sludge paste was prepared by adding water to 300g of sludge powder. It is esssential to consider whether to add more water at the beginning or not.

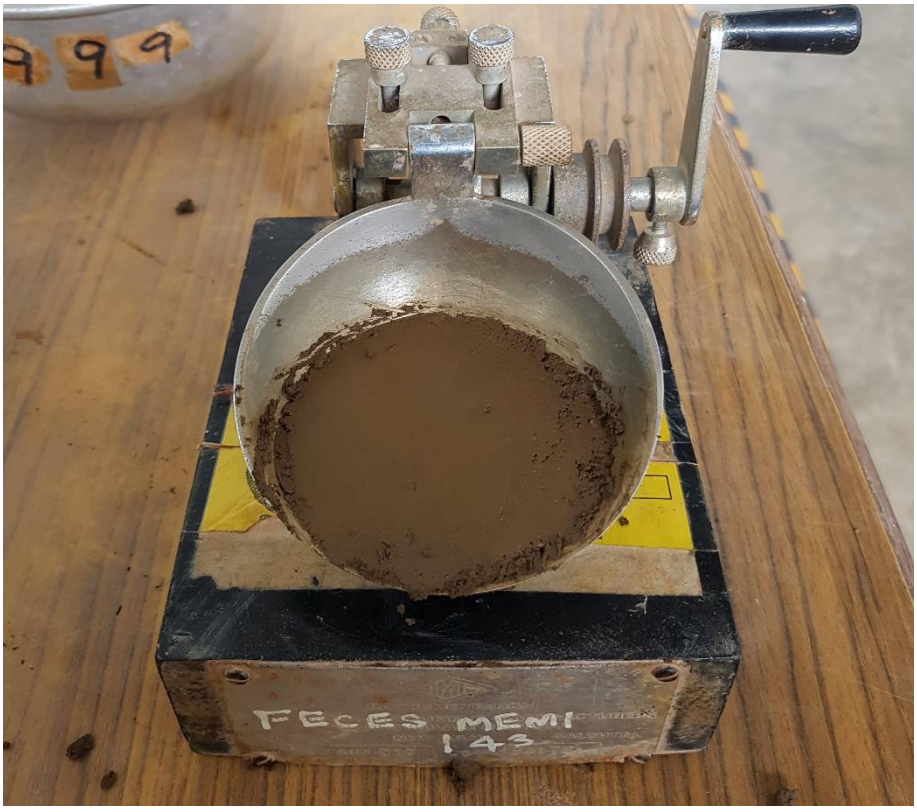

Figure 3.2; Casagrandes Apparatus for Liquid Limit

The sludge paste has to be placed on the cup of the apparatus and the clay paste has to be levelled as shown in *Figure 3.2.* Then the soil paste in the cup was divided into two parts using the groovong tool from the top part of the cup to the down at once. Afterwards, the cup was lifted and dropped by turning the crank handle at a rate of 2 rotations per second. Then the rotations were counted until the cut equal parts of the clay touched each at the bottom of the cut groove, afterwards the bumps count were recorded. Two more samples were tested according to the above procedure for the same water content. Moisture content was measured in each test by collecting 10g of sample from each.

The procedure was followed for 3 moisture contents and the number of bumps for each moisture content were obtained. The british code has mentioned that " The amount of water added shall be such that when four or more moisture contents are plotted they are evenly distributed over the range of 50 bumps to 10 bumps." Therefore, it is a fact of great significance to be considered when adding water to the mixture.

Finally, all the moisture contents were calcualated for each paste and a graph was plotted by drawing the best straight line to the moisture content versus bumps count. Liqiud limit was obtained by the graph which corrosponded to the 25 blows standard.

3.2.1.2 Determination of the plastic limit

In accordance with the standard, the clay particles should be less than 0.425mm. The sludge was dried and crushed to powder form using a laboratory ball mill. Then raw material was seived by a 0.425mm seive and the passing sludge powder was taken for the experiment. Afterwards sludge paste was prepared by mixing powder with water.

Figure 3.3; 3mm sludge thread for plastic limit test

A sample of 20g from the sludge paste was prepared and rolled between the palms until the heat of the hands reached the clay thread which led to the identification of cracks on its surface. Subsequently, this sludge thread was subdivided into two samples which had a mass of 10g each. Again these subsamples were divided into four or more less equal parts. Afterwards, these parts were treated by rolling them to threads about 6mm in diameter. Finally, uniform pressure was applied, rolling them to reduce the thread diameter to about 3mm as shown in *Figure 3.3*.

The British standard states that the first crumbling point is the plastic limit. The moisture content of the crumbled thread is the plastic limit.

3.2.2 Thermal Analysis

Thermal behaviour of the water treatment sludge was examined by Thermal Gravimetric Analysis (TGA), Differential Thermal Analysis (DTA) and Differential Scanning Calorimeter (DSC) analysis. The TGA – DTA instrument consisted of sample/reference caps, a balance, thermocouple berms, a furnace and horizontal purge gas flow. The instrument was set up by balancing the sample and reference caps by placing a mass of sludge around 20mg and another platinum cap, them being the reference material to observe the thermal behaviour.

Figure 3.4; TGA-DTA Apparatus

Figure 3.5; 20mg sludge Holder for TGA testing

The instrument was set to the following parameters according to findings of previous research [21][22].

Heating Environment; Normal atmosphere

Heating Rate; 10ºC/min

Operating heat range; 0 - 1300 ºC

3.2.3 X-ray fluorescence Test

The chemical composition of the water treatment sludge was observed using an x-ray fluorescence spectrometer which built in accordance with the principle of individual atoms, can emit x-ray photons of a characteristic energy or wave length. The primary x-radiation which is generated by the spectrometer will hit the atom of the clay particles, generating fluorescent radiation. Those fluorescent radiations are read by the detector while the radiation data are converted to compositions of the clay particles by an electronic device.

For the XRF test, a sample has to be homogenized, pulverized and pressed in to a pallet with or without a binder. A soil sample of 1kg mass was dried and crushed to powder form using the laboratory ball mill. Afterwards, the powder was sieved by a 0.06mm sieve and the retaining particles were removed. A 150-200mg sample was taken and pressed to prepare a 25mm diameter pellet for the test. Then, the experiment was continued by inserting the prepared pallet to a spectrometer, procuring the results.

3.3 Production of internal curing aggregates in laboratorial scale

According to the two methods of collecting sludge in water treatment plants which are mentioned under Chapter 01, two procedures were followed for each type of sludge to cast samples for the heating process.

The dried sludge collected from the Kadana water treatment plant (sludge lagoon method) was crushed to powder form using the laboratory ball mill (*Figure 3.6*).

Figure 3.6; Laboratory Ball mill

Later, the crushed sludge particles were sieved by a 0.6mm sieve and the clay particles that passed through the sieve were collected (*Figure 3.7*) for further testing. Sludge paste (*Figure 3.8*) was prepared by adding water to the water soil ratio of 0:4. The water soil ratio was selected by the results of Atterberg limits that is a moisture content value between liquid limit and plastic limit. Cylindrical

samples 17mm in diameter and 80mm in length were prepared using a laboratory piston press (*Figure 3.9*). The specimens (*Figure 3.10*) were kept to dry for 72 hours in a normal atmosphere and then kept in a furnace for 24hours at a temperature of 105^0C to remove the moisture content and to prevent shrinkage due to the moisture content.

Figure 3.7; Sludge Powder (Particle size < 0.6mm)

Figure 3.8; Sludge Paste

Figure 3.9; Laboratory made Piston

Figure 3.10; 17mm diameter cylindrical sludge samples

Sludge (Biyagama) which were extracted using the sludge decanter method, showed about 85% of moisture content. That sludge was laid on the floor and cut in to 75mm x 75mm sizes as shown in *Figure 3.11* and *Figure 3.12* and kept for 30 days to dry. Afterwards, the dried sludge plates were used for the next step.

Figure 3.11; Wet sludge laid on floor and cut to 75 x 75mm

Figure 3.12; Dried sludge plates

Subsequently, both types of specimens were kept in a laboratory furnace (*Figure 3.13*) for sintering. The specimens which are shown in *Figure 3.10* were heated at temperatures ranging from 800^0C to 1300^0C at 100^0C intervals, utilizing the slow heating method at an increment of 5^0C/min with a soaking time of 30 minutes and was left in the furnace to cool.

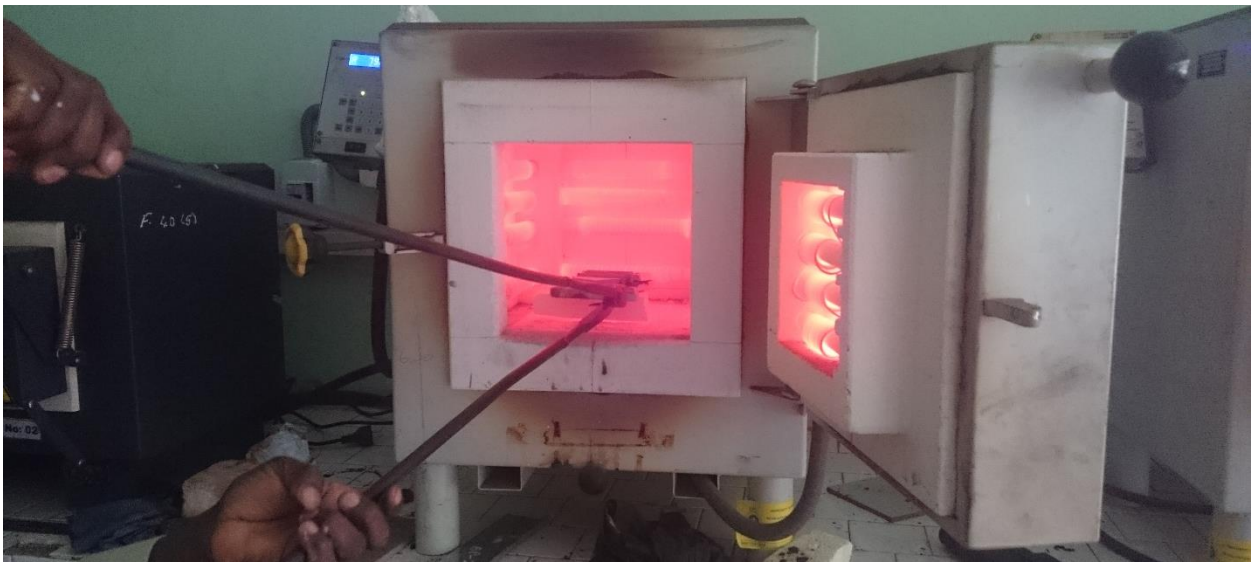

Figure 3.13; Laboratory Furnace

Figure 3.14; Sintered sludge cylinders for 800^0C to 1300^0C temperatures

Figure 3.14 shows the sludge cylinders which were heated at various temperatures. Afterwards, the sintered clay samples were crushed into fine sizes and sieved using 4.75mm and 0.6mm sieves to obtain the aggregate size fraction ranging between 4.75-0.6mm, to avoid loss of aggregates in wiping. *Figure 3.17* shows the prepared fine aggregates for different temperature levels.

Figure 3.15; Laboratory Jaw Crusher

Figure 3.16; Crushed fine aggregates

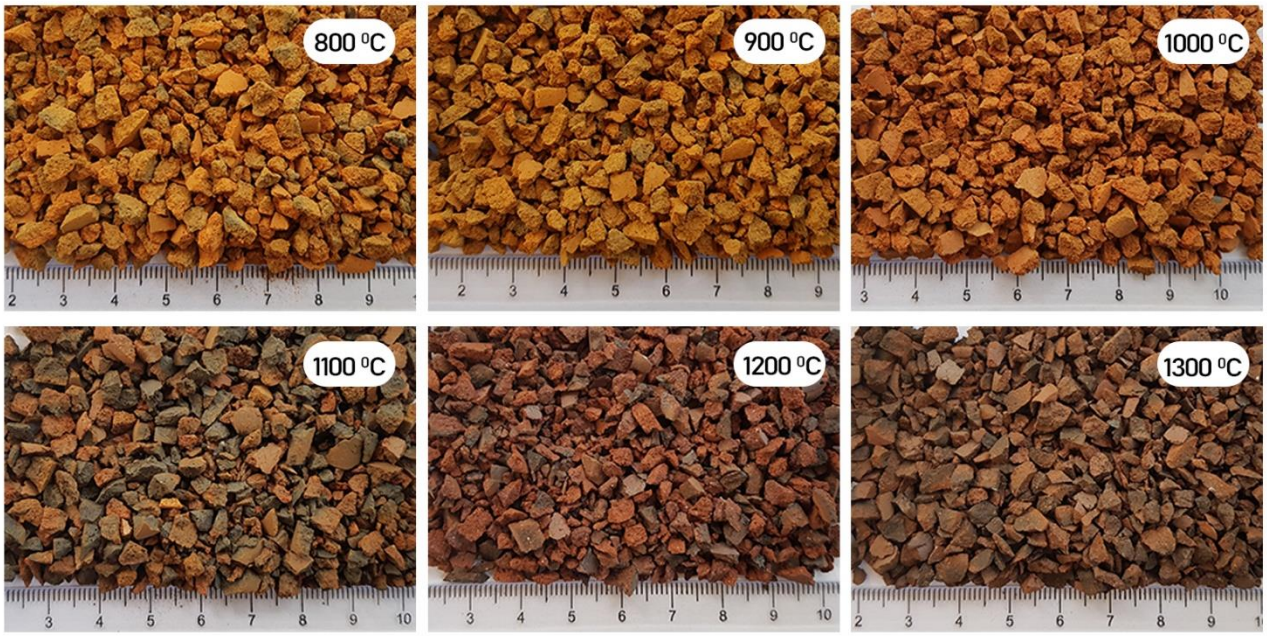

Figure 3.17; Crushed fine aggregates

3.4 Bloating coefficient

Sludge paste was prepared using Kadana water treatment sludge which was dried and sieved by a 0.6mm sieve to remove larger clay particles. The sludge paste was kept for 1.0-1.5hr to distribute the internal relative humidity evenly in a desiccator.

The cylindrical specimens were prepared with the dimensions of 17mm and 80mm which represent the diameter and height respectively, using the laboratory piston press. Then, the specimens were kept for 48 hours in a normal atmosphere to remove excess moisture inside the sludge. Then, sludge cylinders were kept under the temperature of 105^0C in a laboratory oven for the further removal of moisture inside it. Afterwards, the dimensions of the specimens were measured using a Vernier calliper to calculate the volume prior to heating. Subsequently, dry specimens were moved to the laboratory kiln and fired to heating temperatures ranging from 800^0C to 1300^0C at steps of 100^0C. Factors such as slow firing method at a heating rate of 5^0C/min, soaking time of 30minutes and furnace cooling was considered during the sintering process. After the specimens cooled down, again the dimensions were taken using the Vernier calliper to calculate the volume proceeding the heating process or at the end of the bloating effect.

The bloating coefficient of the sintered sludge cylinders (*Figure 3.18*) were calculated using *Equation 4.1* and the sludge was categorized as mentioned in Chapter 4.

Figure 3.18; Sintered sludge cylinders for different temperatures

3.5 Water Absorption

The sintered clay specimens which were heated to 800^0C, 900^0C, 1000^0C, 1100^0C, 1200^0C and 1300^0C were crushed to a fine aggregate size and were sieved, employing a 4.75mm sieve. The aggregate size between 4.75mm to 0.6mm were used for the test to minimize the loss of fines which occur when wiped by paper to achieve a surface saturated dry condition.

The paper towel method was used in determining the surface dry condition. Around 500g of the crushed aggregates for each temperature were soaked in water for 24hours, 48 hours and 72hours. Afterwards, wet aggregates were taken out from the water and spread on a paper to enable it to reach to surface dry condition as shown in *Figure 3.19*. Once it was discernible that no more water can be absorbed by the paper, it was decided that the aggregates had reached the surface saturated dry condition. (It can be determined by the visual observation of the paper: whether its colour changes or not).

Figure 3.19; Water saturated fine aggregates for water absorption test

Thereafter, the mass of the surface saturated aggregates was recorded. Also, the aggregates already measured for their weights were kept in a laboratory oven for 24hours at a temperature of 105^0C to

measure the dry mass of the fine aggregates. Water absorption capacity of the aggregate specimen were calculated as per the ASTM-C1761/C1761M "Standard specification for Lightweight Aggregate for Internal Curing of Concrete".

3.6 Water Desorption

The sintered clay specimens which were heated up to 800^0C, 900^0C, 1000^0C, 1100^0C, 1200^0C and 1300^0C were crushed to the fine aggregate size and were sieved through a 4.75mm sieve. The aggregate size between 4.75mm and 0.6mm were used for the test to minimize the loss of fines resulting from being wiped on paper to achieve surface dry condition.

The crushed aggregates were soaked in water for 72hours until it reached such a point that water absorption rate became zero or till no water was being absorbed furthermore. Subsequently, the wet aggregates were taken out from the water and laid on paper to obtain the Surface Saturated Dry (SSD) condition for the water desorption test. Those SSD aggregates were stored in a laboratory humidity chamber (*Figure 3.21*), having a relative humidity of 94% under the temperature of 24^0C. A potassium nitrate solution was used to balance the relative humidity at 94% as specified in ASTM standards. A Humidity meter which shown in *Figure 3.20* was used to measure the relative humidity in the humidity chamber.

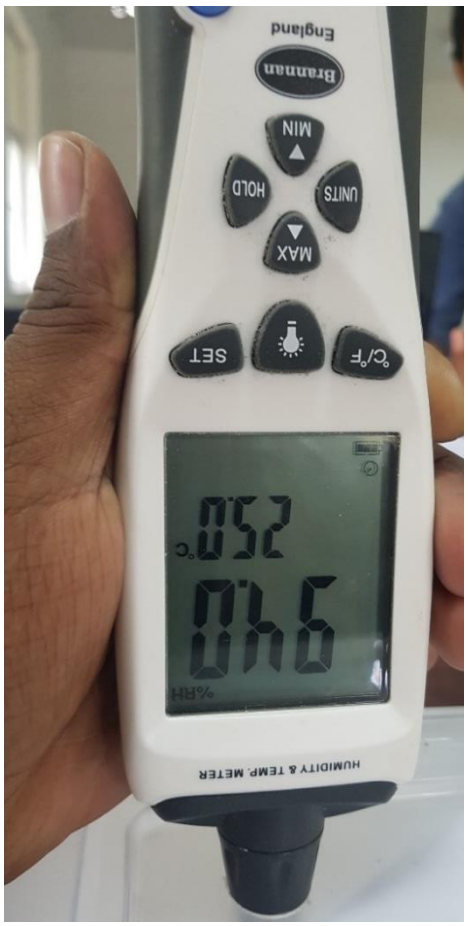

Figure 3.20; Humidity meter

Figure 3.21; Humidity chamber-Laboratory

Mass of the aggregates were measured day by day up to 28 days from the start date of the test. After the 28th day, aggregates were kept under a temperature of 105^{0}C for 24hours to measure the dry weight. Water desorption capacity of aggregate specimens were calculated as per the ASTM-C1761/C1761M "Standard specification for Lightweight Aggregate for Internal Curing of Concrete" [23].

3.7 Relative Density

The crushed aggregates were soaked in water for 72hours so that water absorption rate reached zero or till no furthermore absorption of water took place. Afterwards, the wet aggregates were taken out from water and laid on a paper to obtain the surface saturated dry condition and the weights of SSD aggregates were measured and were recorded.

Figure 3.22; Pycnometer

Pycnometer (*Figure 3.22*) method was followed to measure the relative density of aggregate specimens. As per the standard, A 1L jar was used for the test which is recommended for fine aggregates. Initially, the jar which is shown in *figure 3.22* was filled with room temperature water. It

is essential to confirm that the wall of the jar is free from water bubbles and that the water level reaches the top of the pycnometer. Afterwards, the surface of the jar and pycnometer was wiped out to eliminate surface moisture and the mass of the filled jar was measured and recorded.

Next, pycnometer was filled partially with water at a temperature of 23 +- 2^0C. The quantity of surface saturated aggregates which was measured earlier for SSD weights were introduced to the jar and the loss of any particle was avoided. Then, the jar was filled with water up to about 90% of the capacity, shaking the pycnometer to remove air bubbles. Thereupon, water was filled to the highest point of its capacity. Subsequently, surface of the jar and pycnometer was wiped out to remove any surface moisture and the mass of the filled jar was measured and recorded.

Finally, the aggregates were taken out from the pycnometer and were kept under temperature of 105^0C for 24hours in the laboratory oven to measure the dry weight of the specimen. Relative density of aggregate specimens was calculated as per the ASTM-C1761/C1761M "Standard specification for Lightweight Aggregate for Internal Curing of Concrete" using the above recorded data.

3.8 Microstructure of developed aggregates

Scanning electron microscope (SEM) analysis is a method to observe the surface of a sample up to a resolution better than 1nm. SEM is a type of electron microscope that can obtain images of a microstructure by scanning the surface with focused electron beams.

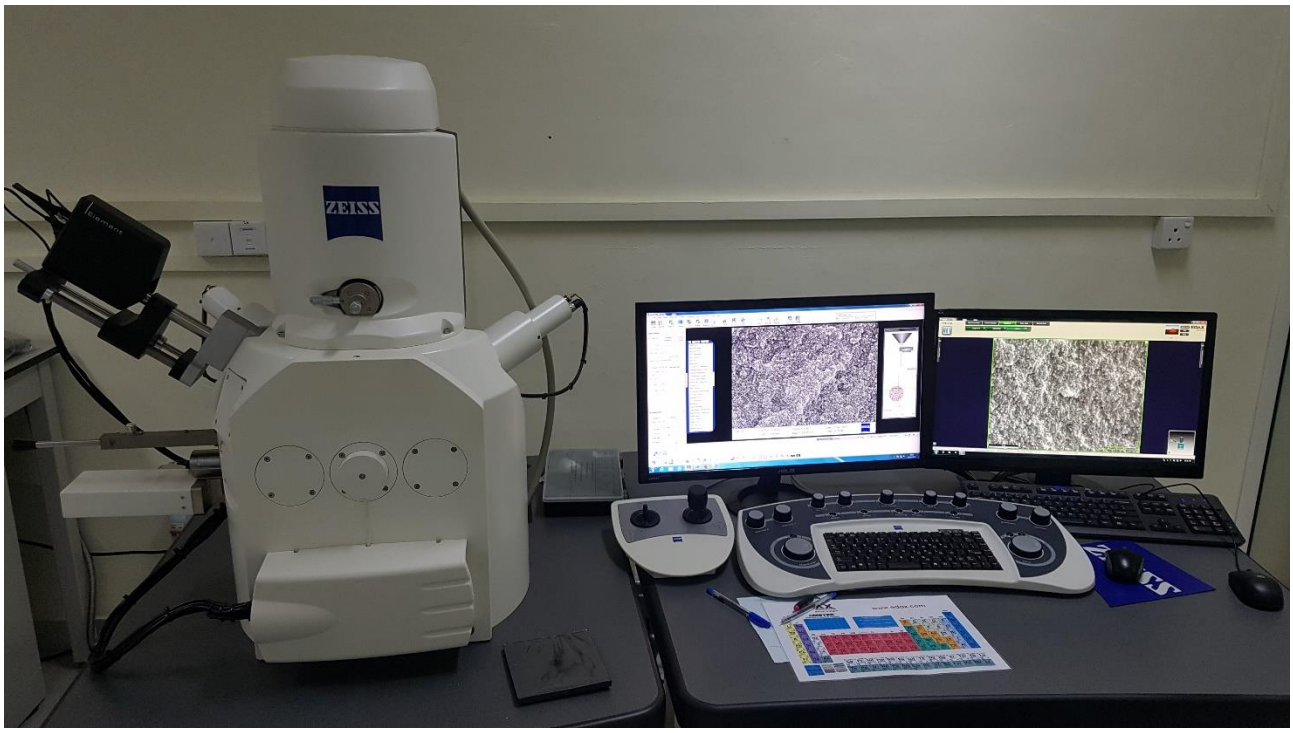

Figure 3.23; Scanning Electron Microscope Setup I

Preparation of the sample to take images in the analysis is more important than other instances. The specimen should be prepared to a size which can be placed on the stage. Those specimens should be prepared to increase its electrical conductivity. A slice of the specimen was prepared to about 5mm size in length and width. Since sintered sludge particles are non-conductive material, the surface of the specimen was painted using material capable of conducting electricity, employing the setup as shown in *Figure 3.24*. The selected gold was coated on to the surface by a low vacuum sputter coating.

Figure 3.24; Scanning Electron Microscope Setup II

Afterwards, the coated specimen was mounted rigidly on a specimen holder and was inserted to the instrument (*Figure 3.23*). When the primary electron beam which is emitted by an electron gun in the instrument touches the sample, the electrons lose energy through absorption and random scattering. The reflection of high-energy electrons due to the energy exchange between the electron beam and the specimen were detected by the instrument's detectors and transmitted to the computer through electronic amplifiers to create images.

Black and white images of the surface of the aggregates under a magnification of 10,000 were taken for each temperature to compare and analyse the sizes of the pores and to determine at which temperatures the specimen changes its microstructure.

3.9 Production of Internal Curing Aggregates in industrial Scale

After all the initial testing and laboratory experiments, manufacturing of a large amount of internal curing aggregates using sludge extracted from the Kadana water treatment plant was decided on. At an early stage, the equipment and methodologies adopted in ceramic industry and clay industry were studied. Visits were paid to Dankotuwa ceramic factory, Sumagi tile factory and Lanka Refractories (Pvt) Ltd to discover how raw materials convert to ceramic items, raw clay materials to roof tiles, clay bricks, and the process was concluded in converting that sludge into internal curing aggregates.

Firstly, the dried sludge was extracted from the sludge lagoon and was sun dried to achieve a moisture content of about 10%. Secondly, the sludge was introduced to an industrial crusher (*Figure 3.25*) to crush the sludge to a finer size. Thirdly, that sludge powder was sieved using a no.40 mesh (0.6mm sieve) to remove larger particles as shown in *Figure 3.26*. Next, the sieved sludge powder was mixed with water (water soil ratio; 0.05). Water was added to the sludge powder through water sprinklers and it was sieved again through the mesh and kept for 1.0-1.5hours to obtain a homogeneous wet sludge powder.

Figure 3.25; Sludge milling machine

Figure 3.26; Crushed sludge sieving

Figure 3.27; Sludge Plate molding machine

Figure 3.28; Fresh Sludge Plates

Later, the homogenous sludge mixture was moved to an industrial hydraulic press as shown in *Figure 3.27* (Maximum power of 5000KN) to cast plates with the dimensions of 300mm x 300mm x 20mm (width x length x thickness). Then, the casted plates were kept for 120 hours or 5 days under a normal atmosphere to remove the additional moisture in the plates. Subsequently, those sludge plates were stacked to an industrial furnace to heat up to a temperature of 1000^0C. The heating curve of the furnace is illustrated in the *Figure 3.29*.

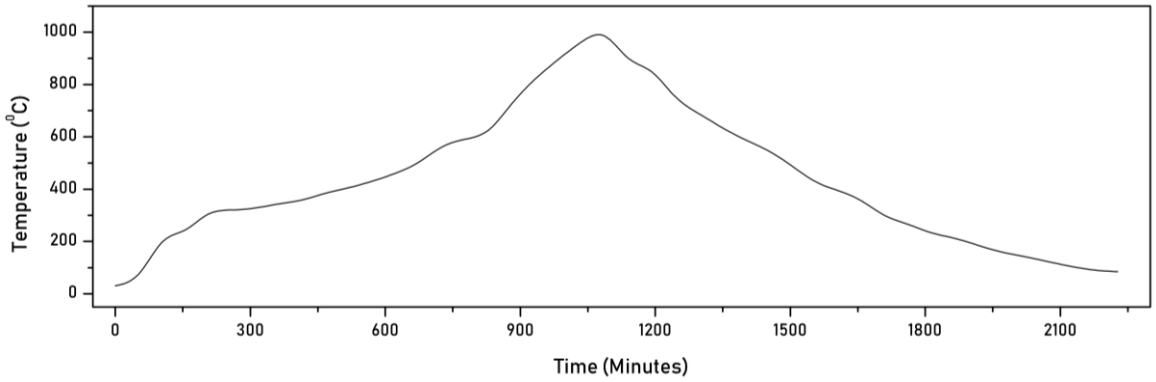

Figure 3.29; Heating Curve

Figure 3.30; Sintered sludge plates

As the last step of the process, the sintered sludge plates were moved to an industrial jaw crusher (*Figure 3.31*) to crush the plates to a fine aggregate size. The crushed particles were sieved through a 4.75mm mesh and the larger particles were moved to the crusher. The particles less than 4.75mm were sieved through a 0.6mm sieve to remove the dust particles from the developed fine aggregates.

Figure 3.31; Industrial Jaw Crusher

3.10 Internal Curing Concrete

After all the initial lab experiments on the developed aggregates and the large quantity production of internal curing aggregates, the final part of the study deals with the comparison and observation of the characteristics of ICC which were casted by newly developed aggregates with conventional concrete. The strength class of G30 was selected to cast both conventional and internal curing concrete.

Figure 3.32; Internal Curing Concrete Cube

3.10.1 Mix proportioning for Concrete

Mix proportioning to enumerate the required amount of ICFA were calculated as per the ASTM-C1761/C1761M "Standard specification for Lightweight Aggregate for Internal Curing of Concrete" [23].

At the outset, the mix design for G30 normal concrete according to the BS1411 were calculated and the data were illustrated in *Table 4.1*. As per the standard, *Equation 3.1* was used to calculate the amount of developed aggregate needed for internal curing.

$$M_{LWA} = C_f \times CS \times \alpha_{max} / S \times W_{LWA} \quad (3.1)$$

Where,

M_{LWA} = Mass of oven dry ICFA required, kg/m^3

C_f = Cement content, kg/m3

CS = Chemical shrinkage of cementitious at 100% hydration, kg of water/ kg of cement

α_{max} = Maximum degree of hydration of cement (0 to 1.0)

S = Degree of saturation of developed aggregate (0 to 1.0)

W_{LWA} = Mass of water released by ICFA

The numerator of the equation 3.1 represents the amount of water required to fill the empty capillary pores in cement paste which resulted due to chemical shrinkage. Chemical shrinkage in concrete defines that reduction in volume of cementitious paste that occurs during hydration because the hydration products occupy less space than the volume occupied by the reactants. Chemical shrinkage of Portland cement can be estimated from the phase composition. α_{max} stands for the maximum potential degree of hydration if all the water supply by the internal curing aggregate were available for cement hydration and no lost through evaporation.

The denominator is the amount of water released from the aggregate per unit mass of internal curing aggregate in the oven dry condition.

Complying with the standards, data from the mix design and previous studies, the values for chemical shrinkage of cement, maximum degree of cement hydration were considered as 0.07 and 1.0 respectively. The degree of saturation of pre wetted ICA was taken as 1.0 while the amount of water desorbed (W_{LWA}) was calculated by multiplying water desorption percentage value of 0.92 with water absorption capacity value of 0.24. Cement content for the G30 concrete mix design was calculated as 410 Kg/m^3.

The amount of fine aggregates needed to be replaced is equal to the calculated amount of internal curing aggregate.

3.10.2 Casting Concrete

Initially, quantities of raw materials were measured to cast normal concrete as shown in *Figure 3.33*. 20mm size coarse aggregates were used while river sand was used as the fine aggregate. Ordinary Portland cement belonging to the strength class 42.5KN was used as the cementitious material. Portable concrete mixer was used to mix the ingredients. As the first step, coarse aggregates and fine aggregates were inserted to the hopper and was mixed for 2-3 minutes. Subsequently, cement was added and the mixture was blended for about 2-3minutes to mix all ingredients homogeneously. Finally, a quantity of water was instilled into the mixture, and it was blended for 3-5minutes. The fresh concrete mixture prepared so was then moved to check fresh concrete properties, hardened properties and durability properties.

Figure 3.33; Raw materials to cast concrete

The calculated mass of oven dried internal curing aggregates was soaked in water for 48hours before mixing it with the internal curing concrete. Then, soaked wet aggregates were moved from the water container to remove the extra amount of water from the surfaces of the aggregates and to achieve surface saturated dry condition. *Figure 3.34* shows how wet aggregates were laid to achieve such condition.

Figure 3.34; Surface Saturated ICFA

Similarly, as for normal concrete preparation, coarse aggregates and fine aggregates were blended in the first instance using a concrete mixer for 2-3minutes. Next, surface saturated dry aggregates were inserted to the hopper and blended for 2-3 minutes, and whether all the internal curing aggregates have mixed uniformly in the mixture was checked as illustrated in *Figure 3.35*. Later the measured quantity of cement was mixed with the paste for 2-3 minutes. Following that, the water was instilled to the mixture and blended for 3-5 minutes. Afterwards, the prepared fresh internal curing concrete was moved to check for certain properties: fresh properties, hardened properties and durability properties.

Figure 3.35; Homogeneous mixture with ICFA

3.10.3 Workability

Slump test was conducted for fresh concrete to check the workability properties as per BS1881-102 code [24]. Following equipment was used for the test were according to the British standards: a mould having a top diameter of 100mm, base diameter of 200mm and 300mm in height, a tray with the dimensions of 900mm x 900mm x 50mm representing height width and depth respectively, and a tamping rod 16mm in diameter and 600mm in height.

First and foremost, internal surface of the mould was cleaned and oil was applied. Next, the tray was placed on a horizontal, undisturbed surface and the mould was kept in the middle of the tray. Secondly, fresh concrete was added to the mould in three layers, in such a way that each layer could be tamped to one-third of the mould's height. Each layer was tamped with 25 strokes, employing a tamping rod while maintaining the uniform distribution of strokes throughout the layer. It is to be ensured that the tamping rod does not reach the layer underneath when tamping the foremost layer. After the 3rd layer, the top of the concrete was levelled by rolling the tamping rod. Then, all the extra concrete mixture which fell around the mould and tray was removed and cleaned. Afterwards, the mould was raised vertically in 5-10s in such a way to minimize lateral and torsional movement to fresh concrete as per the code. The cycle time of the procedure has to be around 150s. Instantly, the slump was measured by calculating the difference between the mould's height and the highest position of the flowed concrete specimen as shown in *Figure 3.36.*

Figure 3.36; Measuring the slump

The test was performed for both the conventional concrete mixture and internal curing concrete.

3.10.4 Compressive strength

Hardened properties of concrete were measured by following the compressive strength. 150mm cubes were casted and tested for normal concrete and internal curing concrete as per the BS1881-108 and BS1881-116 codes [25] [26].

Initially, moulds (150x150x150mm) as shown in *Figure 3.37* were cleaned and oil was applied. Then those moulds were kept on a vibrating table (surface should be horizontal), and concrete was poured to the moulds layer by layer. Approximately a 50mm deep concrete layer was laid 3 times. Each layer was compacted for 25-30seconds by using the vibrating mode of the table. The vibration time was selected so that the concrete surface acquired a smooth finish in accordance with that mentioned in the code. Likewise, after finishing all 3 layers, the top of the concrete surface was smoothened using the trowel and all the unwanted concrete particles around the mould were cleaned out.

Figure 3.37; Cube Casting moulds (150x150x150mm)

Figure 3.38; Concrete Cubes

Figure 3.39; Compressive strength test machine

Subsequent to the passing of 24 hours after the cube casting, moulds were removed carefully and a part of the cubes (*Figure 3.38*) was put into a curing tank while the other part was kept outside for no curing which was to be casted using normal concrete. The internally cured concrete cubes were also kept outside to check the strength after 7 days and 28 days.

Reaching the final stage of the experiment, cubes were placed and cantered to the loading pads in the testing machine (machine was set to comply with BS 1881-115) as shown in *Figure 3.39*. Load was applied to cubes at a rate of 0.2-0.4 N/mm^{2}s as per the code parameters. Lastly, the maximum load machine which gives the that is applied to the concrete specimen, was recorded.

3.10.5 Concrete Drying Shrinkage

The drying shrinkage of internal curing concrete was obtained according to the ASTM C157 code [27]. Accordingly, the test specimen size was selected as a 75mm square cross-section with a 285mm length because all of the aggregates in the mixture passes through a 25mm sieve.

Three prismatic cubes were casted by placing the concrete to mould in 2 equal layers. Each layer was compacted using the vibrating table for 25-30 seconds. Gauge studs were fixed to the two ends of the mould before the starting of the pouring. After 24 hours from the concrete pouring, test specimens

were demoulded, and conventional concrete specimens were submerged in a curing tank while ICC test cubes were kept under a normal atmosphere.

In the end, test specimens were tested on the 3rd, 7th, 14th, 21st and 28th days to obtain the following parameters to calculate the drying shrinkage using *Equation 3.2* as per the standards.

$$Lx = (CRD - initial\ CRD) \times 100 / G \quad (3.2)$$

Where,

Lx = length change of the concrete bar, %

CRD = difference between the comparator reading of the concrete bar and the reference bar

G = the gage length (250mm)

Figure 3.40; Concrete bars for drying shrinkage test

4 ANALYSIS AND DISCUSSION OF RESULTS

4.1 Physical, Chemical & Thermal properties of water treatment sludge

Prior to starting the manufacturing of fine aggregates using water treatment sludge, the physical properties, chemical properties and the thermal behaviour were observed to decide whether it can be developed as an ICA, and to further work out a path for the methodology to be adopted regarding how it can be developed.

4.1.1 Atterberg Limits

The Atterberg limits of the WTS were figured out to estimate a moisture content for the casting of laboratory sludge specimens from Kadana water treatment sludge. The water content was measured according to Atterberg limits because the sludge paste having a moisture content below the limit of plasticity can crumble easily while a moisture content above the liquid limit interferes with the paste's capability to retain its shape [18]. If the amount of water content is low, the material gets stiff and hardens during compaction to cast sludge specimens. Also, if the amount of moisture is raised in the sludge paste, it is workable and eases the process of compaction to cast sludge specimens, thus resulting in the increase of the dry densities of those specimens.

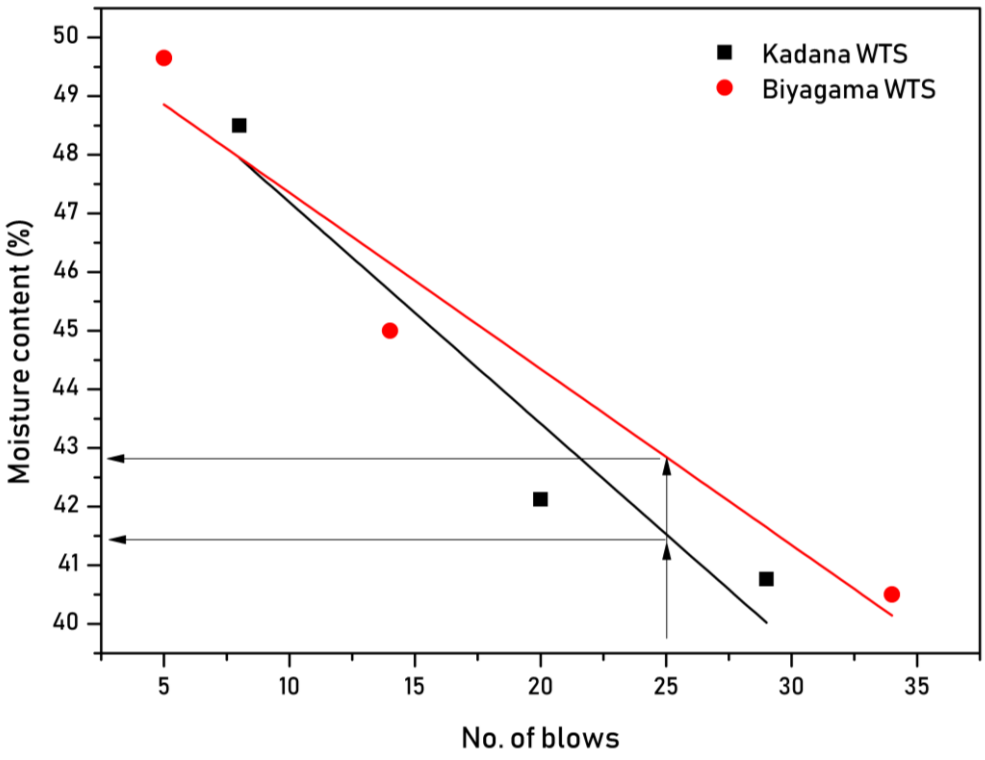

Figure 4.1; Graph-Moisture content versus number of blows

Table 4.1; Atterberg Limits

Type	Liquid Limit	Plastic Limit	Plasticity Index
Kadana	41	32	9
Biyagama	43	34	9

Figure 4.1 shows the 'moisture content versus number of blows' graph which is plotted to estimate the liquid limit. Estimated Atterberg limits (LL, PL & PI) are illustrated in the *Table 4.1*. Considering estimated Atterberg limits, Biyagama sludge has slightly higher values of LL and PL than the values of Kadana sludge.

Following the discovery of the optimum moisture content to cast laboratory sludge specimens, a water/sludge ratio of 40% by mass, (which is a value between liquid limit and plastic limit) was used to prepare the sludge paste for facilitating the compaction during sludge specimen casting to avoid above circumstances.

4.1.2 Thermo gravimetric Analysis of Kadana and Biyagama Sludge

Thermal behaviour of the two sludge types analysed by TGA and DTA graphs employing Kadana and Biyagama sludge are shown in *Figure 4.2 and 4.3*. The weight loss due to free water evaporation, release of chemically bound water, decomposition of organic matters and fragmentation of carbonates was identified by exothermic peaks and endothermic peaks.

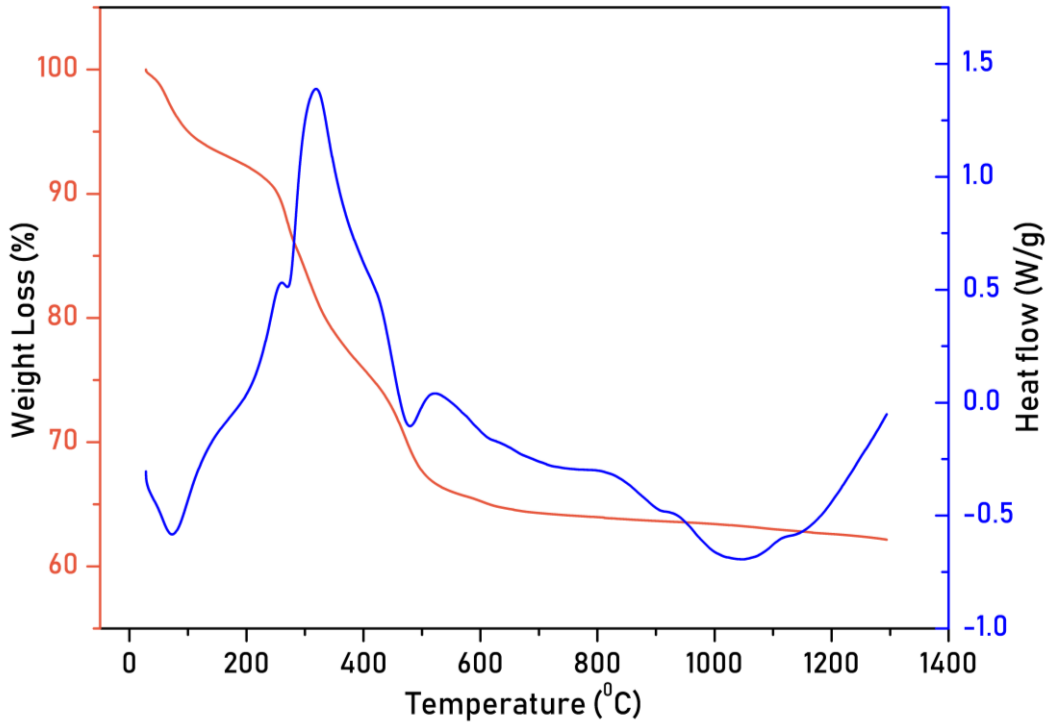

Figure 4.2; Graph-TGA & DTA curves of Kadana WTS

According to the graph, the first endothermic peak is at 73.19^{0}C and it is related to the release of physically absorbed water. 8% of weight loss is due to this dehydration process. Furthermore, endothermic process is followed by exothermic process which reaches a peak at 319.08^{0}C. It is assumed to be the exothermic effect, owing to the decomposition of organic matter. Mass loss due to the burning materials from water treatment sludge is about 15%. Furthermore, the endothermic process occurring at a maximal value of 479.85^{0}C may be due to the release of chemically bonded water and is associated with the kaolinite de-hydroxylation. Mass loss during the process is 4.5%. Afterwards, an exothermic process can be found with a maximum temperature of 523.87^{0}C which is due to the decomposition of the other small fragments of materials which have less biodegradability. The mass loss of 1.9% can be observed in the process. WTS melts at the 1047.24^{0}C showing the peak of the endothermic process [28].

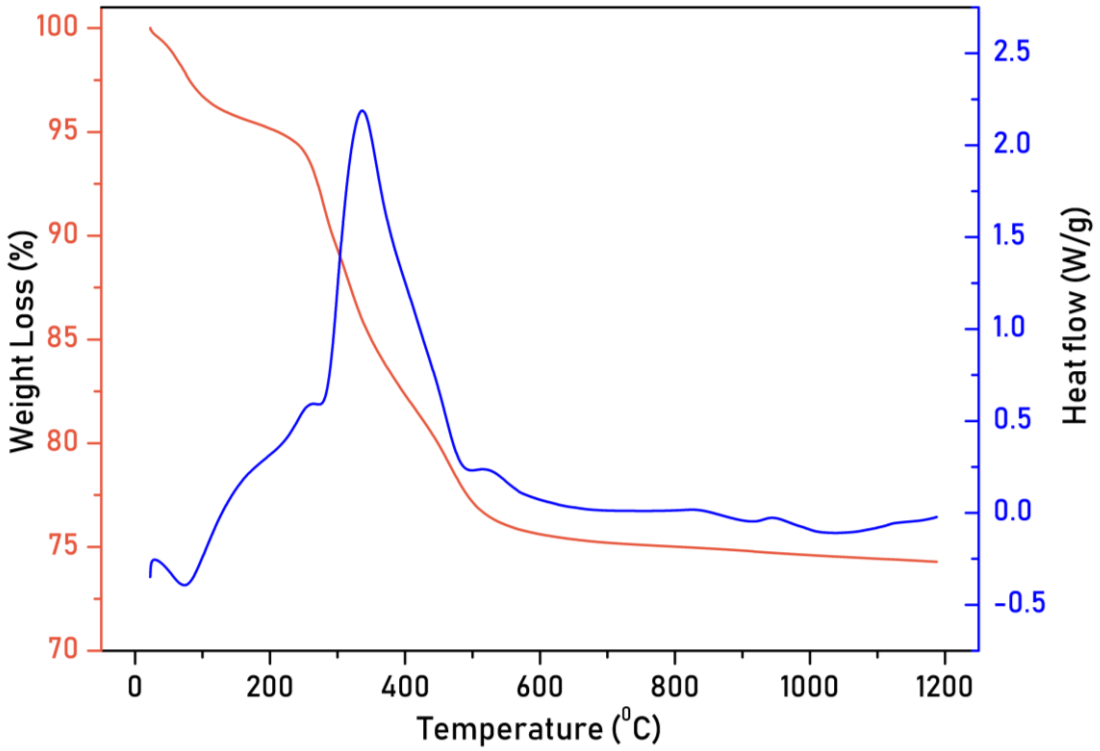

Figure 4.3; Graph-TGA & DTA curves of Biyagama WTS

In accordance with the *Figure 4.3*, first endothermic peak can be observed at 72.81^{0}C and it is related to the release of physically absorbed water. 3.5% of weight loss is due to this dehydration process. Moreover, the endothermic process is followed by the exothermic process which appears to peak at 336.7^{0}C. It is assumed to be an exothermic effect due to the decomposition of organic matter. Mass loss due to the burning of materials from water treatment sludge is about 16%. An endothermic process follows this, occurring at a maximal value of 498.97^{0}C, which may be due to the release of chemically bonded water and is associated with the kaolinite de-hydroxylation. Mass loss during the

process is 2.5%. Afterwards, an exothermic process can be found with a maximum temperature of 514.19^{0}C, which is a result of the decomposition of other small fragments of materials which have less biodegradability. The mass loss of 1.9% can be observed with the process. The reason as to why an exothermic peak can be seen in the latter part of the graph at 943.840C, can be traced to the characteristic of the kaolinite dissociation. Biyagama sludge melts at the 1047.24^{0}C showing the peak of the endothermic process [28].

Comparing the Thermo gravimetric Analysis of Kadana and Biyagama: weight loss is quiet high in Kadana water treatment sludge. The reason may be the higher amount organic matter content present in the Kadana sludge. As Biyagama and Kadana treatment plants are being fed from the Kelani river and Kalu Ganga respectively, it can be concluded that a high amount of organic matters are mixed in Kelani river water. Apart from that both DTA curves are slightly similar and shows similar thermal behaviour at about the same temperature ranges.

4.1.3 Bloating coefficient

As some kind of clay types show bloating property, the test was conducted to observe the behaviour of Kadana and Biyagama sludge.

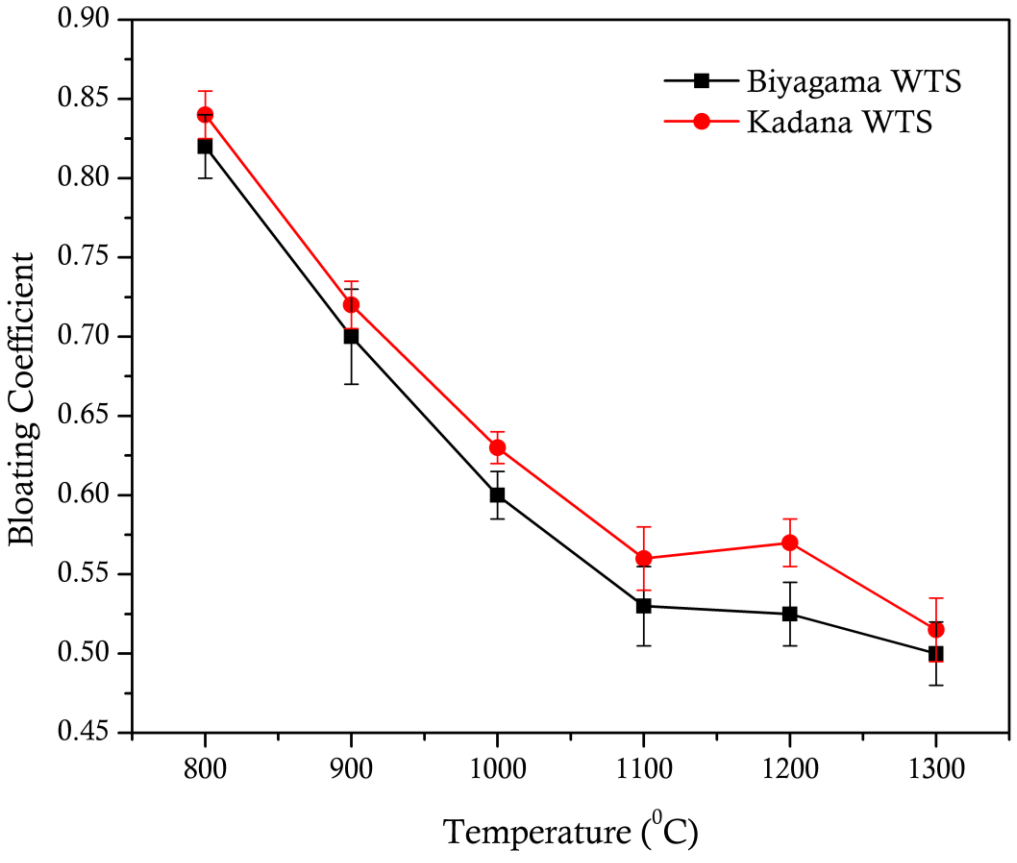

Figure 4.4; Graph-Bloating coefficient versus Temperature

Bloating coefficient of sludge was calculated using *Equation 4.1.*

$$K_P = V_F / V_I \quad (4.1)$$

Where,

K_P = Bloating coefficient

V_F = Final volume after sintering process

V_I = Initial volume of pre heated sludge cylinders

According to the bloating coefficient, clay types are categorized into 4 types.

K_p > 7-8; good bloat ability,

K_p = 4-5; medium bloat ability,

K_p = 2-2.5; poorly self-bloating,

K_p < 2; non-self-bloating clay.

Figure 4.4 shows the bloating coefficient (K_P) variation with temperature, and it can be deduced that the bloating coefficient measured at various temperatures ranging from 800 - 1300^{0}C are less than 1. The bloating coefficient reduces as the temperature increases for both types of sludge. Rapid change of gradient can be seen between the temperatures of 800-1100^{0}C because of the burning of organic matters, removal of chemically and physically bound water and phase changes of clay. According to *Figure 4.4* it is clear that there is no considerable amount of volume change after the temperature of 1100^{0}C. Considering the volume changes of clay at temperatures from 800^{0}C to 1300^{0}C, the two sludge types can be categorized under non-self-bloating clay.

4.1.4 Chemical composition of Water Treatment Sludge

Normally, WTS is categorized as a waste. Although, it is comprised of chemicals such as silicon, alumium, iron and etc. In accordance with the literature on the X-ray fluorescence test results of Kadana and Biyagama sludge *Table 4.2* is drawn

Table 4.2; Chemical Compositions of WTS

CHEMICAL COMPOSITIONS	**SIO_2**	**AL_2O_3**	**MGO**	**CAO**	**NA_2O**	**K_2O**	**P_2O_5**	**FE_2O_3**
KADANA (%)	34.66	29.38	0.62	1.67	0.06	0.94	0.27	10.12
BIYAGAMA (%)	36.54	28.56	0.52	2.3	0.05	0.78	0.25	15.11

When considering chemical compositions, a high amount of quartz(SiO_2) and metal oxides (Al_2O_3,MgO etc) can be seen in both sludge types, thus theycan be categorized as a clay material.

Presence of fluxing compounds such as Fe_2O_3, K_2O, MgO, Na_2O and CaO in the sludge will lower the temperature required to start the vitrification [29].

According to the past research papers, clays which have a 50% - 60% of SiO_2 and 15% -20% of Al_2O_3 are convinved of good bloating properties. A high amount of aluminium percentage can be seen due to the addition of a aluminium based coagulant for the purpose of flocculation of colloidal particles and suspension of impurities. Due to its chemical composition, authors selection is to use the waste for production of ceremic products as it shows the properties of clay. In addition, past researches have concluded that WTS increases strength properties, density, porosity and water impregnation [30].

4.2 Properties of developed internal curing fine aggregates

4.2.1 Water Absorption

The variation of water absorption capacity of ICFA with firing temperature is shown in *Figure 4.5*. According to the graph, water absorption decreases as the firing temperature increases. Water absoprtion capacity of the developed fine aggregates from Kadana sludge is slightly higher than that of Biyagama sludge. The water absorption capacity of aggregates which were fired up to 800^0C and higher temperatures is greater. It may be due to the high interconnectivity of pores and pore density at higher temperatures.

Considering Scanning electron micrographs of Kadana fine aggregates, there are no visible pores at 10,000 magnification. However, there should be an additional number of pores with lesser pore radius compared to the pores in aggregates with high firing temperature. To add to that point, according to *Figure.9*, a lesser density is shown in lower temperatures (800^0C, 900^0C). By considering that statement, it is clear that even though the size of pore radius is less, pore density should be high. Therefore, above two facts proves that water absorption should be high in aggregates which were fired to less temperatures.

After 1000^0C, the sludge starts to change its phase by melting. Even though there are visible pores at 10,000 magnification in SEM, reduction of pore interconnectivity may affect the reduction of the water absorption. With the phase change, sludge liquefies and flows into the pores and clogs the paths of pores, and reduces the interconnectivity between pores while large pores grow on the surface of the aggregate. These pores do not support the storage of water which is a requisite of internal curing.

Through all the above facts it can be concluded that water absorption should be reduced after a phase change.

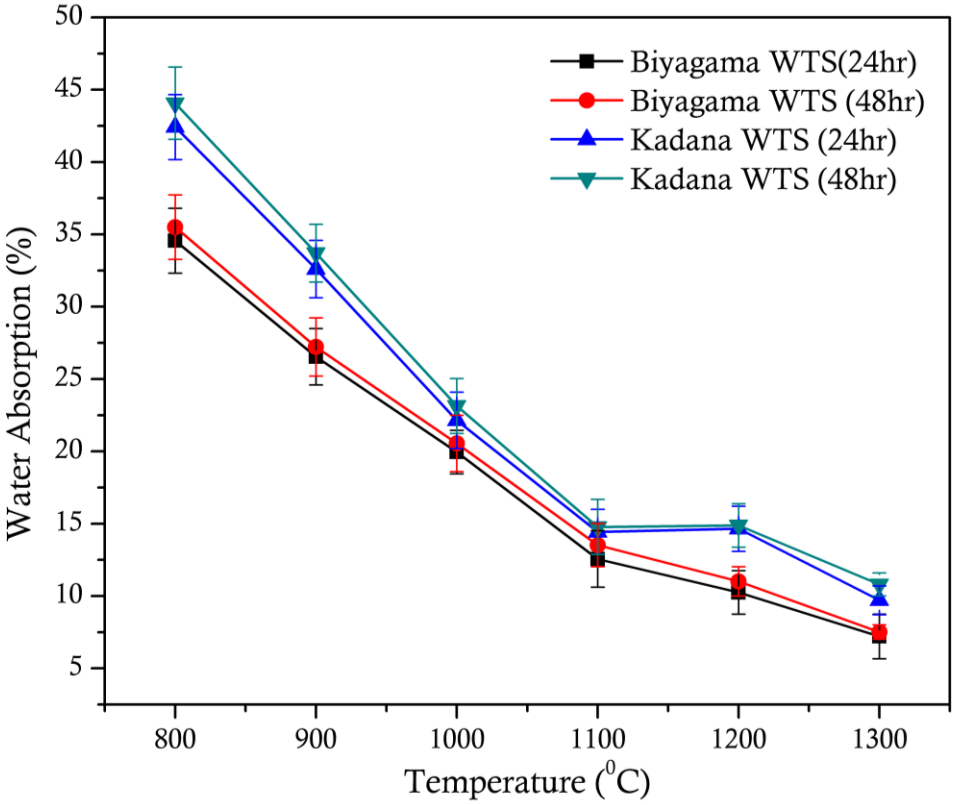

Figure 4.5; Graph- Water absorption versus Temperature

4.2.2 Water Desorption

The variation of water desorption of aggregates with firing temperature is shown in *Figure 4.6 and Figure 4.7* for Kadana sludge and Biyagama sludge respectively. As per the ASTM standards, 85% absorbed water should be released at 94% of internal relative humidity.

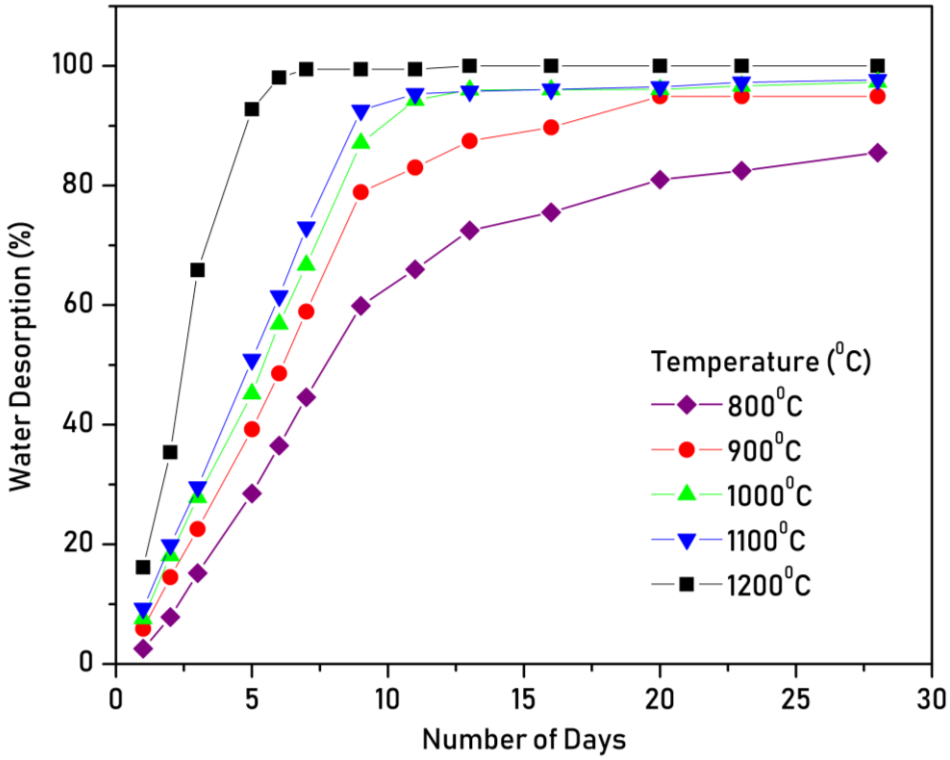

Figure 4.6; Graph-Water desorption versus Time (Kadana WTS)

According to the *Figure 4.6*, all the saturated aggregates made from Kadana sludge which was fired to 900^{0}C, 1000^{0}C, 1100^{0}C and 1200^{0}C except 800^{0}C desorbed more than 90% of stored water at 94% of relative humidity. As per the *Figure 4.6*, all curves are concave. It means, high water desorption rates are shown in the first 10 days and that with time gradually water absorption reduces. Even though aggregates for all temperatures achieve the limits which are needed to satisfy internal curing properties, the number of days spent to desorb 85% of water decreases with the temperature increment. The aggregate which was fired to 800^{0}C spent 28 days while the 900^{0}C aggregate spent 20 days to desorb 85% of absorbed water. All the other aggregates desorb 85% of its water within the first 10 days.

The SEM analysis unveils the fact that the aggregates produced by firing to 800^{0}C and 900^{0}C has no perfect pore structure for 10,000 magnifications. Furthermore, according to an early research, if the pore radius exceeds 0.02μm, that aggregate will desorb its absorbed water at 94% relative humidity [3]. Thus, the aggregates produced at 800^{0}C and 900^{0}C shows a lower rate and percentage of water desorption which should be a result of the pore sizes in the microstructure.

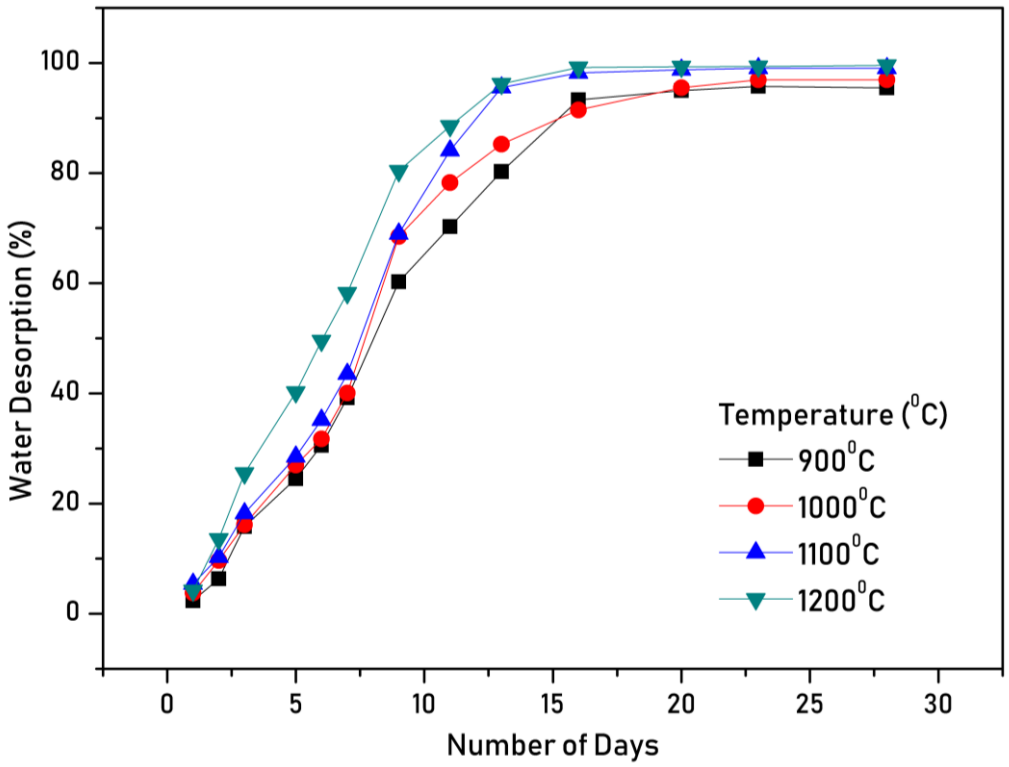

Figure 4.7; Graph-Water desorption versus Time (Biyagama WTS)

The fine aggregates developed using Biyagama sludge and fired to 900^{0}C, 1000^{0}C, 1100^{0}C and 1200^{0}C were tested to deduce the desorption capacity. Looking at *Figure 4.7*, it can be observed that all the saturated aggregates desorbed more than 90% of the absorbed water at 94% of relative humidity.

Figure 4.7, shows that all curves are concave. It means, a high water desorption rate is shown within the first 12 days which gradually decelerates during the last few days. Even though aggregates for all

temperatures achieve the limits which are crucial to qualify them as those that have internal curing properties, the number of days spent to desorb 85% of water decreases with the temperature increment. The aggregate which was fired to 900^0C and 1000^0C spent 16 days to desorb 85% of absorbed water. Other aggregates which were fired to 1100^0C and 1200^0C desorbed 85% of its water within the first 12days.

4.2.3 Relative Density

The relative density of the developed aggregates was measured since it is an indirect measurement of strength. The strength of the aggregate is usually proportional to the density. The variation of the relative densities of aggregates with temperatures are illustrated in *Figure 4.8.*

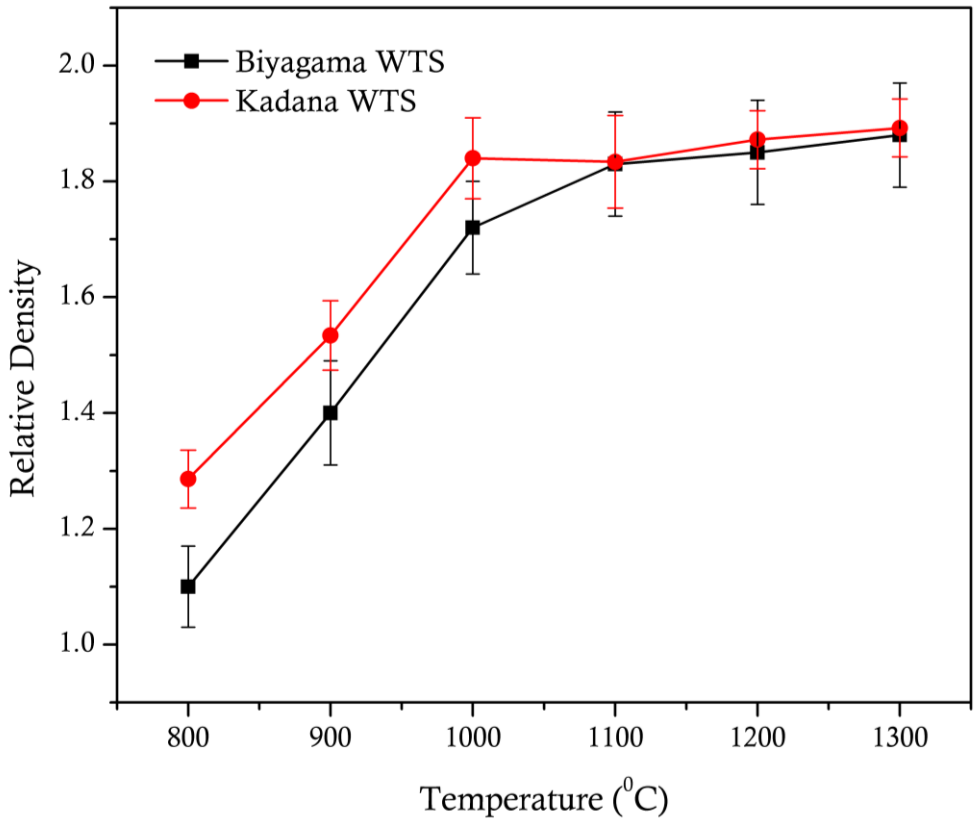

Figure 4.8; Graph-Relative density versus Temperature

According to *Figure 4.8*, both curves show similar shapes. However, the relative density of aggregates which were developed from biyagama sludge shows a slightly lower value than that of Kadana.

Relative density of Kadana fine aggregates increases with the temperature, and a maximum value of 1.89 was recorded at 1300^0C while a minimum value of 1.23 was recorded at 800^0C temperature. *Figure 4.8* shows a rapid increase in relative density untill 1000^0C, which then is at a constant level after passing that point. Relative density of Biyagama fine aggregates increases with the temperature while a maximum value of 1.88 and minimum value of 1.1 was recorded at 1300^0C and at 800^0C

temperature respectively. The graph shows a significant increment at lower temperatures up to 1000^0C which becomes slightly constant after 1050^0C.

In addition, a proper vitrification and sintering process makes the sludge denser and it shows a smoother surface on sludge samples which are fired to 1100^0C, 1200^0C and 1300^0C. This is true in regard to both sludge types.

The sludge samples of both Biyagama and Kadana which were fired to 800^0C and 900^0C display a low density which may be due to the incomplete vitrification and sintering process and tiny pores that remain invincible even at a magnification of 10,000. Furthermore, the low strength shown in aggregates which were fired to 800^0C and 900^0C can be crushed by fingers owing to their low density. The dust generation is high in those sludge specimens and a significant amount of sintered particles is also removed during crushing and sieving processes.

4.2.4 Microstructure of developed fine aggregates

The formation of pore structure in aggregates (developed using Kadana water treatment sludge) with the temperature is illustrated in *Figure. 4.* At the early stages of temperature levels at around 800^0C and 900^0C, sludge particles start combining through its contact point. However, according to the Scanning Electron Micrographs (SEM) images, the formation of pores and interconnected voids cannot be observed at the temperatures which range between 800^0C and 900^0C. These temperatures are found to be the initial temperatures which start the solid-state sintering step.

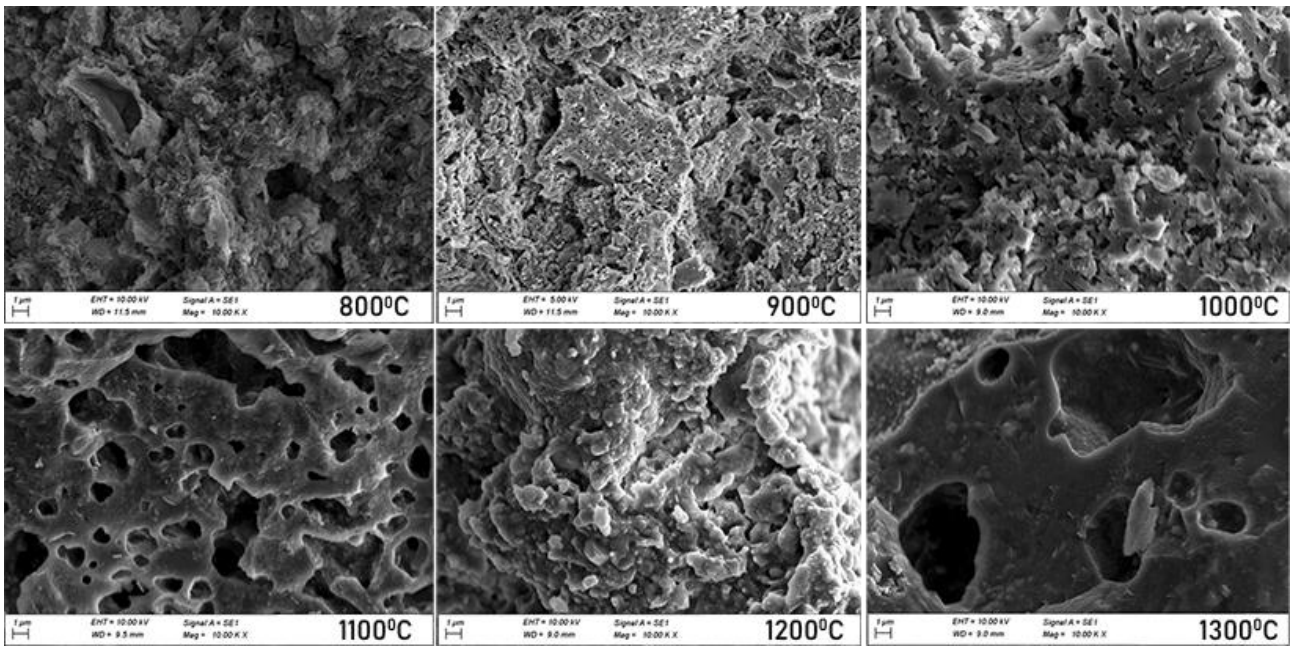

Figure 4.9; Scanning Electron Micrographs (Kadana WTS)

In accordance to SEM images shown in *Figure 4.9*, when the temperature increases to 1000^0C and 1100^0C, sludge particles combine with each other to generate pores and interconnected pore paths. These temperatures levels can be identified as the range in which the intermediate sintering process occurs. SEM images give a clear understanding on pore generation and shows that the peak occurs at 1100^0C temperature. As well as that, the sludge particles achieve enough plasticity, while the gas entrapped in the specimen escapes as it conduces to form interconnected pores at 1100^0C. Afterwards, sludge particles become less viscous material and start to discontinue the interconnected paths at around 1200^0C temperature due to fluidisation. Later, most of the pores become large while discontinuing the interconnected paths at around 1300^0C. However, it was observed that those pores could not store water needed for the cement hydration process.

4.3 Properties of Internal Curing Concrete

The physical and mechanical properties of normal concrete and internal curing concrete are described in this section. A comparison between the properties of normal concrete and internal curing concrete will give a clear idea on the performance of the newly developed internal curing aggregate.

The mix design for G30 normal concrete and internal curing concrete are illustrated in *Table 4.1*. Replacement of fine aggregates for the ICC was calculated as per the ASTM 1761M. ICFA quantity required is also depicted in the same table.

Table 4.3; Mix design values for normal and internal curing concrete

Type	*Water/cement ratio*	*Water (Kg/m^3)*	*Cement (Kg/m^3)*	*Coarse aggregates (Kg/m^3)*	*Fine aggregates (Kg/m^3)*	*Internal curing aggregates (Kg/m^3)*
NC	0.5	205	410	1060	770	-
ICC	0.5	205	410	1060	663.7	106.3

Replacement amount of internal curing fine aggregates were calculated as per the equation 3.1. According to the mix design for G30 concrete, 15 % of river sand is replaced with internal curing aggregates. And also, it need to be study the concrete properties by increasing the amount of internal curing fine aggregates as some past research papers have revealed that improved properties can be obtained by increasing the amount of ICFA used for the mix design [32, 33] Therefore, mechanical

and durability properties should be checked with different proportions of ICFA to analyze the excess replacement, and the optimum replacement should be identified.

Past literatures reveals that the addition of ICFA reduces the autogenous deformation and hence, mitigates the risk of early age cracking [33].

During the pre-wetting stage of developed fine aggregates, the particle size less than 150um were removed to avoid the material loss. As a result, gradation of the fine aggregate will be changed and it affects for the packing density of concrete. So, it is required to observe the properties carefully, in replacement of excess amount ICFA to concrete.

4.3.1 Workability

Slump values of internal curing concrete (ICC) and external curing concrete (ECC) were recorded as 155mm and 140mm respectively. The results depict that internal curing concrete shows a higher workability than normal concrete. This phenomenon is due to the fact that the newly added pre wetted internal curing aggregates cause an increase in the workability of concrete. As the surface of the internally cured aggregates has been saturated, moisture on the surface of those aggregates drive to increase the workability.

The workability of the ECC is enough to place the designed concrete. The extra workability of ICC is not useful in placing and handling. Furthermore, the amount of water can be reduced to achieve the same workability of ECC while increasing the strength of ICC by lowering the w/c ratio.

4.3.2 Compressive Strength of concrete

Compressive strength of concrete was measured in 3 sets: Not curing concrete (NCC), External curing concrete (ECC) and Internal Curing Concrete (ICC). Strength was recorded on the 7^{th} day, 14^{th} day and 28^{th} day. The graphical representation of the results is illustrated in a histogram in *Figure 4.10*.

The mean density of ECC was recorded as 2405 Kg/m^3 while ICC had a 2350Kg/m^3 density value. It may be due to the lower relative density of the internal curing aggregates than normal fine aggregates.

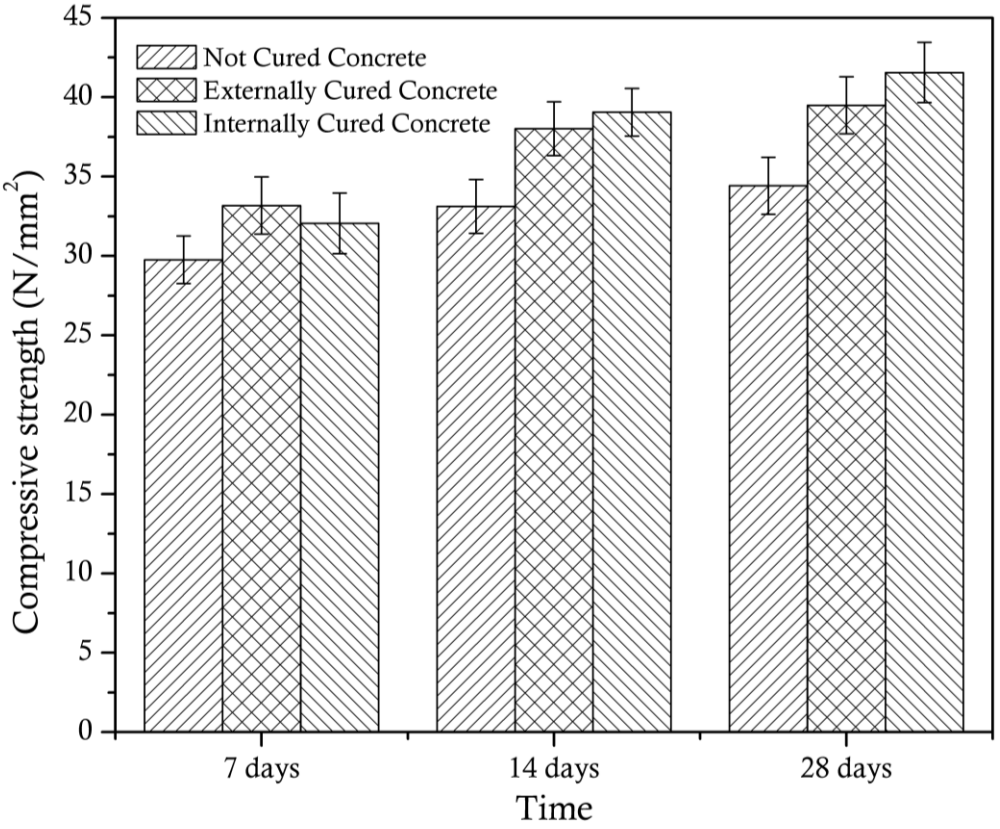

Figure 4.10; Compressive Strength of NCC, ECC and ICC

According to the histogram, ICC and ECC have achieved 60-70% of its target mean strength in 7 days. However, strength in the 7th day for ECC is higher than that of ICC, while not cured concrete showed a value lower than that of the cured samples. In the first 14 days, the rate of strength increment of ECC is higher. It may be happening due to the abundance of moisture in the concrete in early days. Even though that may be the case, with time the work expected by the external curing does not function. The reason behind this is to be traced to how the surface water maintains the evaporation of water inside the concrete, and how cured water can penetrate several millimetres in to the concrete as the permeability of concrete decreases with the cement hydration process.

In ICC, saturated internal curing fine aggregates start its process when the internal relative humidity starts to decrease. Complete and effective cement hydration process functions in ICC as a water pump, directing water to the cement system through the internal water reservoirs. Thus, strength of internal curing concrete will accelerate until the end of the cement hydration process.

Internal curing concrete has achieved a higher compressive strength with a higher workability than the other two types with same water/cement ratio, due its effective cement hydration. Thus, it is necessary to take into account the workability and compressive strength of ICC through the reduction of water/cement ratio, to achieve the workability limits of normal concrete.

4.3.3 Drying shrinkage of concrete

Drying shrinkage reduction is a predominant issue in internal curing concrete. Higher drying shrinkage affects the concrete strength, internal warping and external deflection as shrinkage cracks occur inside the concrete. The average drying shrinkage of external cured and internally cured concrete are illustrated in *Figure 4.11*.

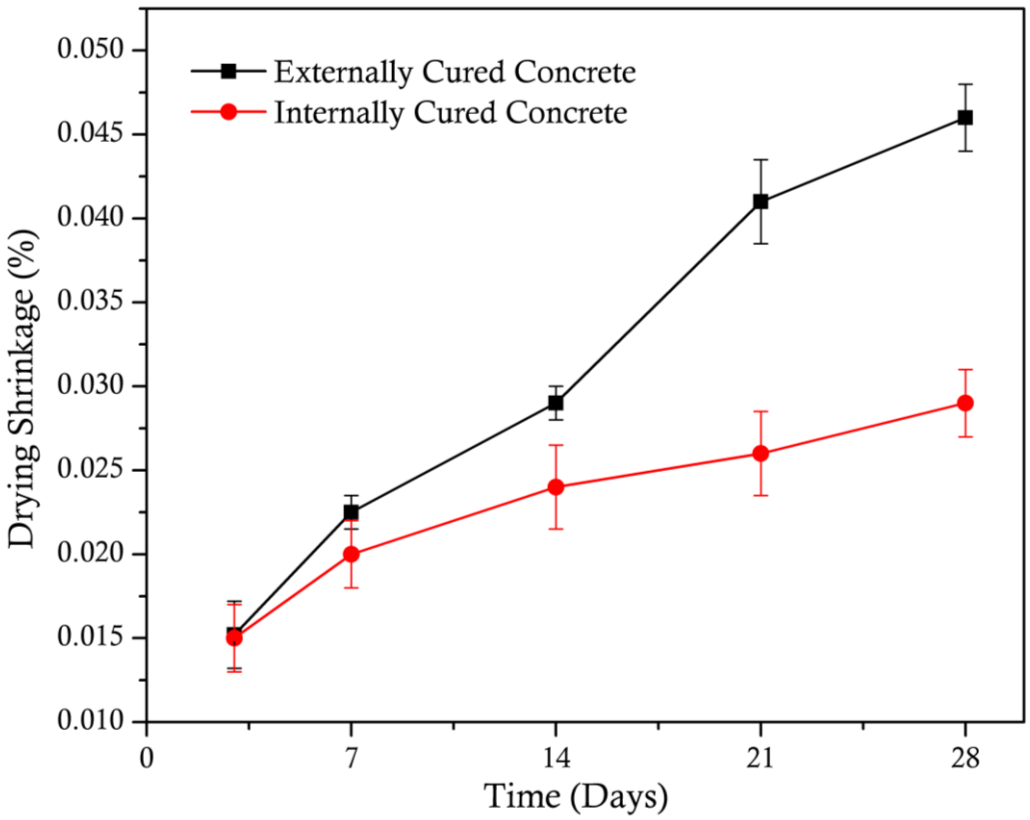

Figure 4.11; Graph-Drying Shrinkage versus Time of ECC & ICC

As the graph stipulates, it is clear that a considerable amount of shrinkage reduction is present in ICC. The early stage of the two curves shows some similarity in which the values rise up until day 14. That happens due to the internal moisture of the externally cured concrete. Yet, with time the concrete system dries since there are no internal reservoirs to supply the required moisture in ECC. Also, external curing water cannot supply water to the entire system of concrete. The latter part of the curves has highly deviated since high drying shrinkage is shown in ECC. The drying shrinkage is mainly due to the reduction of capillary water by evaporation, and the water in the cement system.

After the 14th day, drying shrinkage comes to a constant level in ICC. Internal reservoirs in ICC supply water to the concrete system to avoid concrete drying while maintaining internal relative humidity. Those developed aggregates have a desorption capacity of 95% to maintain internal relative humidity to minimize the drying shrinkage.

5 SUMMARY AND CONCLUSION

The main focal point of this research lies on the development of a fine aggregate using industrial waste with improved properties to be used as an alternative material for fine aggregates in the construction industry. A broad literature review on industrial wastes, development of fine aggregates, the internal curing concept, properties of internal curing aggregates and concrete have been brought under the scope covered by the topic: “Investigate the use of water treatment sludge as an internal curing fine aggregate for internal curing concrete.”

Also, among all factors, the most poignant factor to be considered is that the developed fine aggregate effectively functioned as an internal curing aggregate. Numerous tests were conducted to find whether water treatment sludge can be developed as a fine aggregate and later the tests conducted in furtherance of the major objective enabled the observation of the properties of fine aggregates which will facilitate internal curing.

Addressing the steps in the methodology which were followed to develop the fine aggregate, attention should be given to those that involved, drying the sludge under normal atmosphere or sunlight, crushing to powder form into a particle size less than 0.6mm, mixing with water (w/s; 0.05), moulding clay plates, leaving for few days to remove moisture, heating up to the specified temperature and last but not least, crushing to fine aggregate sizes.

The optimum heating temperature selected was 1150C. Notwithstanding, the temperature between 975C to 1200C can be used to sinter the casted clay plates. The temperature 1150C was used in developing aggregates in a laboratory scale. However, in large scale production, the temperature 980C was used to heat the casted sludge plates. Furthermore, the two main requirements which have to be fulfilled to indicate the existence of internal curing properties are the water absorption and water desorption capacity. The aggregates which were developed using water treatment sludge showed a water absorption capacity of 15%-25% which were also heated to levels of temperature which ranged from 975C to 1200C. The water desorption rate ranges between 90% to 95% while the relative density shows a value in the range between 1.7 to 2.0. It should also be noted that the aggregates prepared by water treatment sludge were not bloated, but the microstructure or pores that have to store water was already generated as the organic matters were burnt in specific temperatures. So, bloating coefficient of the sludge varied between 0.8 to 0.9.

The final stage of the research was to cast internal curing concrete with developed aggregates. The mix design was performed as per the codes ASTM C1761 and BS 1881-116;1983 for G35 concrete. Developed ICA was replaced with natural river sand in accordance with the code and the properties

were compared with those of conventional concrete. Workability of the ICC was higher than that of conventional concrete. Additionally, ICC achieved higher strength than ECC within 28 days. Among these observations Drying shrinkage was also present. The shrinkage results were shown to be lower in ICC than in ECC. Compressive strength and drying shrinkage results confirmed that the developed aggregate can be used for the concrete as an ICA with improved properties.

6 RECOMMENDATION

This experimental study was on developing an internal curing fine aggregate using water treatment sludge. Moreover, suitability of using those developed aggregates for G30 concrete was observed and satisfactory results were obtained. It is also suggested that this study be continued in developing high strength concrete using the developed aggregate and admixtures given that nowadays the construction industry uses high strength ready-mix concrete with different admixtures.

7 REFERENCES

[1] R. U. K. Piyadasa, "River sand mining and associated environmental problems in Sri Lanka," in *Sediment Problems and Sediment Management in Asian River Basins*, Hydrebad, 2011.

[2] The University of Memphis, Curing Concrete, Memphis: The University of Memphis, 2012.

[3] W. J. Weiss, Dale P. Bentz, "Internal Curing: A 2010 State-of-the-Art Review," National Institue of Standards and Technology, Gaithersburg, 2011.

[4] A. M. Neville, "Properties of Concrete," John Wiley & Sons, 1996.

[5] N. J. Carino, K. W. Meeks, "Curing of high-performance concrete: Report of the state-of-the-art," National Institue of Standards and Technology, Gaithersburg, 1999.

[6] M. Geiker, O. M. Jensen, D. P. Bentz, "On the Mitigation of Early Age Cracking," in *International Seminar on Self Desiccation III*, Lund, Sweden, June 2002.

[7] E. Mack, J. Clearly, N. Delatte, "Evaluation of Higfh Absorptive Materials to Improve Internal Curing of Low Permeability Concrete," FHWA/OH-2007/06, State Job Number 134227, Ohio, March 2007.

[8] D. Reeves, T. Friggle, "Internal Curing of Concrete Paving Laboratory and Field Experiences," American Concrete Institute, 2008.

[9] R. Henkensiefken, J. Castro, K. Roufi, J. Weiss, T. Nantung, J. Schlitter, "Development of Internally Cured Concrete for Increased Service Life," in *FHWA/IN/JTRP-2010/10, Joint Transportation Research Program, Indiana Department of Transportation and Purdue University* , West lafayette, India, 2010.

[10] C. Ozyildirim, "Laboratory Investigation of Lightweight Concrete Properties," Final Report VCTIR 11-R17, 2011.

[11] J. A. Humbell, P. Drumheller, "Densely crosslinked polymer networks of PEG in trimethylolpropane tracrylate for cell adhesion-resistant surfaces," *Biomedical Material Research,* vol. 29, pp. 201-205, 1995.

[12] P. F. Hensen, O. M. Jensen, "Water entrained cement based materials-II, Implementation and experimental results," *Ceement and Concrte Research,* vol. 6, pp. 973-978, 2002.

[13] H. W. Reinhardt, S. Weber, "A new generation of high performance concrete: Concrete with autogenous curing," *Advanced Cement Based Materials,* vol. 6, pp. 59-68, 1997.

[14] L. Keiser, M. Golios, J. Weiss, J. Castro, "Absorption and desorption properites of fine lightweight aggregate for application to internally cured concrete mixtures," *Cement & Concrete Composites,* vol. 33, pp. 1001-1008, 2011.

[15] J. Schlitter, D. Bentz, J. Weiss, K. Raoufi, "Parametric Assessment of Stress development and cracking in Internally-cured Restrained Mortars Experiencing Autogenous deformations and Thermal loading," *Cement and Concrete Composites,* vol. 22, pp. 199-205, 2001.

[16] M. Darter, C. Rao, "Evaluation of internally cured concrete for paving applications," in *Applied Research Associates*, Champaign, 2013.

[17] P. Briatka, T. Nantung, J. Weiss, R. Henkensiefken, “Plastic Shrinkage Cracking in Internally Cured Mixtures made with pre-wetted light weight aggregate,” *Concrete International* , vol. 32(2), pp. 49-54, 2010.

[18] S. Y. Hong, K. Y. Show, J. H. Tay, “Reuse of Industrial sludge as Pelletized Aggregate for concrete use,” *Journal of Environmental Engineering,* vol. 126, pp. 279-287, March-2000.

[19] C. L. Hwang, M. F. Hung, “Study of fine sediments for making lightweight aggregate,” *Waste Management & Research,* vol. 25, pp. 449-456, 2007.

[20] B. Standard, *Methods of test for Soils for civil engineering purposes, Part 2; Classification tests,* Giorgio Cavalieri, 1990.

[21] Selene M. A, Guelli U, Joze. A. B, Velle. A. A, U. Souza, Andressa R., V. Mendonca, “Thermogravimetric analysis and kinetic study of pyrolysis and combustion of residual textile sludge,” Hungary, 2015.

[22] C. E. Hori, M. H. M. Reis, N. D. Mora, C. R. G. Tavers, R. B. Luciana, C. S. Herek, “Characterization of ceremic bricks incorporated with Textile laudry sludge,” *Ceremics International,* vol. 38, pp. 951-959, 2012.

[23] A. International, *Standard Speification for Lightweight Aggregate for Internal Curing of Concrete,* 2017.

[24] B. Standard, *Testing Concrete - Part 02; Method for determination of slump,* 1983.

[25] B. Standard, *TEsting Concrete - Part 108; Method for making test cubes from fresh concrete,* 1983.

[26] B. Standard, *Testing concrete - Part 116; Method for determination of compressive strength of concrete cubes,* 1983.

[27] A. Standard, *Standard test method for Length Change of Hardened Hydraulic- Cement Mortar and Concrete,* 2003.

[28] J. Alexendre, J. I. Margem, R. Sanchez, C. M. F. Vieira, S. N. Monteiro, “Incorporation of sludge waste from water treatment plant in to red ceramic,” *Construction and Building Materials,* vol. 22, pp. 1281-1287, 2008.

[29] A. Idris, A. A. Samad, C. H. K. Wong, M. S. Jaafar, M. B. Amuniddin, A. G. Liew, “Incorporation of sewage sludge in clay bricks and its characterization,” *Waste Management Research Forum,* vol. 22, pp. 226-233, 2004.

[30] R. Surauskiene, V. Kizinievic. R. Zurauskas, O. Kizinievic, “Utilization of sludge waste from water treatment for ceramic products,” *Construction and Building Materials,* vol. 41, pp. 464-473, 2013.

[31] E. M. J. Clearly, N. Delatte, “Evaluation of High Absorptive Materials to Improve Internal Curing of Low permeabiltiy Concrete,” FHWA/OH-2007/06, State Job Number 134227, Ohio, March 2007

[32] L. Domagala, "The effect of lightweight aggregate water absorption on the reduction of water-cement ratio in fresh concrete", MATBUD'2015, *Procedia Engineering*, pp. 206-213, 2015.

[33] G.R. de Sensale, A.F. Goncalves, "Effects of fine LWA and SAP as internal water curing agents", *International Journal of Concrete structrues and materials*, Vol 8, No 3, pp. 229-238, 2014.