

The Study of Ladle Furnace Slag as Cementitious Material

by

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CERTIFICATION OF APPROVAL

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Approved by,

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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

(ATIRA BINTI ZULKARNIN)

ABSTRACT

Steel slag is a by-product of the steelmaking and steel refining processes. Different types of method of steel production resulted to different types of slag: blast furnace slag (BFS), basic oxygen furnace slag (BOFS), electric arc furnace (EAF) acid slag and ladle furnace (LF) basic slag. Southern Steel Malaysia produces about 120,000 tons per year of electric arc furnace slag (EAFS) and ladle furnace slag (LFS). These abundant wastes are initially stockpiled before been disposed to the landfill.

On the other hand, the use of Ordinary Portland Cement (OPC) in concrete had led to the high emission of Carbon dioxide (CO₂). With the aim to further reduce the amount of OPC used and to produce a green binder with a consistent high compressive strength and slower setting time, Ladle Furnace Slag (LFS) and Metakaolin were blended with OPC.

The raw materials in this research which are LFS and Metakaolin were characterized for their chemical, mineralogical and morphological properties. Four samples of mortars with different percentage of LFS composition were made following the standard procedure (ASTM C305-06). These mortars were then tested for initial and setting time test, soundness test and compressive strength test using the standard ASTM C191-08, IS: 4031-Part 3-1988 and ASTM C109 respectively. The sample with the highest compressive strength and shorter setting time will be determined as the best sample and the percentage of LFS used in that sample will be recorded as the optimum percentage of LFS needed to produce the green binder.

The findings of this research showed the sequence of compressive strength of the samples from the highest to lowest is $S1 > S3 > S4 > S2$. Thus, it can be concluded that the best percentage of LFS that can be used in binder is 10% which is from Sample 3. The initial and final setting times of Sample 3 are 6.66 hours and 7.42 hours respectively. The soundness of Sample 3 is as same as Sample 1 which is 10 mm. Further research is recommended with different percentage of LFS ranging from 11% to 19%.

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CHAPTER 1

INTRODUCTION

1.1 Background

Steel slag is a by-product of the steelmaking and steel refining processes (Yildirim & Prezzi, 2011). Total steel production in Spain from basic oxygen furnaces amounted to 16.5 million tons in 2004, leaving 2.5 million tons of residual slag as a by-product and current annual production continues at roughly similar levels (Setién, Hernández, & González, 2009). On the other hand, 10 to 15 million tons of steel slag are generated in the USA and approximately 14 to 40% of the steel slag output is initially stockpiled in the steel plants and, eventually, sent to slag disposal sites (Yildirim & Prezzi, 2011). According to Papayianni & Anastasiou (n.d.), the annual slag production in Greece is about 250,000 tons.

Different types of method of steel production resulted to different types of slag: blast furnace slag (BFS), basic oxygen furnace slag (BOFS), electric arc furnace (EAF) acid slag and ladle furnace (LF) basic slag. Among the metallurgical slags, the most abundant and better known is ground granulated blast furnace slag (GGBS) as an active addition to Portland cement (Setién et al., 2009). The combination limestone fluxes with coke ash and residues from the reduction of iron ore produced the GGBS as a by-product from the manufacture of pig iron (Ghosh, 1999). According to Ghosh (1999) also, GGBS is used extensively as a blending component due to its high latent hydraulic properties. Meanwhile, BOFS is formed during the conversion of hot metal from the blast furnace into steel in a basic oxygen furnace.

On the other hand, EAFS is produced in granulated form during the first stage of steel production (Ioanna Papayianni & Anastasiou, n.d.). Depending on the intended steel quality (carbon steel or stainless/ high alloy steel), two different slag types can be generated. They are electric arc furnace from carbon steel production and electric arc furnace from stainless steel production.

Setién et al (2009) stated that ladle furnace slag is produced in the final stages of steelmaking, when the steel is desulphured in the transport ladle, during what is generally known as the secondary metallurgy process. It is determined that LFS contains high amount of calcium and magnesium oxide and low content of ferrous oxides. Silicon and aluminium oxides constitute the other main oxides of LFS, which jointly make up less than 40% of the total weight.

Applications of LFS were vastly increasing and diverse. LFS is not only used for the correction of soil acidity, it also used in environmental engineering, aquiculture and cement industry as raw material for the Portland clinker fabrication or even inside the same steel factories (Setién et al., 2009). Although LFS is a weak cementing material (Ioanna Papayianni & Anastasiou, n.d.), further chemical composition and physical properties improvements are possible to be done unto the LFS to make it a good cement replacement material that can display strength, fire resistivity and durability and hence making the global consumption of this by-product to be potentially high.

1.2 Problem Statement

In this current situation, the amount of industrial and biomass waste increased from year to year. Southern Steel Malaysia for example produces about 120,000 tons per year of electric arc furnace slag (EAFS) and 20,000 to 25,000 tons per year of ladle furnace slag (LFS). In Korea, approximately 720,000 of LFS were buried and caused environmental pollution in the form of dust and contaminated leachate (Jin-young, 2015). According to the data generated by World Steel Association, approximately 400 kt of LFS is produced in 2011 (Montenegro, Celemín-Metachana, Cañizal, & Setién, 2013). Different studies done by Yildirim and Prezzi (2011) stated that 15-40% of 10-15 million tons of steel slag produced is wasted. These abundant wastes

are initially stockpiled before being disposed to the landfill. Due to the large amount of these wastes, the handling and disposal costs are high taking into account the transportation cost and workers' pays as well. Currently, most industrial slags are being used without taking full advantage of their properties or disposed rather than being used (Shi & Qian, 2000).

On the other hand, the use of Ordinary Portland Cement (OPC) in concrete had led to the high emission of Carbon dioxide (CO₂) although public knows that the main source of CO₂ comes from the burning of fossil fuels (Gartner, 2004). Fact shows that one ton of OPC produced will result in one ton of CO₂ emission and it also releases an estimated total of 5% of global anthropogenic CO₂. 50% of CO₂ emitted is from the process of heating the limestone while the remaining 50% is from indirect emissions such as burning fossil fuels to heat the kiln, electricity used to power the additional plant machinery and the final transportation of cement (Rubenstein, 2012). With the combination of the high emission of CO₂ and aggressive environment, the ozone layer became depleted quickly and hence will result in the global warming. These environmental problems of course should not be neglected and unattended. Thus, some changes need to be made especially in finding ways to prevent the causes of these problems.

Not only that, the mining and quarrying of limestone generates a lot of dust and hence pollutes the air. This is because fast development in the construction industry demands the cement industry to develop more cement products. This can lead to serious air pollution and quick reduction of raw resources. Furthermore, the blasting of limestone and the construction equipment caused noise pollution (Csanyi). In addition, quarrying and mining activities also caused visual intrusion, damage to landscapes, traffic, smoke, damage to caves, loss of land, and deterioration in water quality (Quarrying and the environment).

In order to tackle these problems, the use of ladle furnace slag is introduced as partial replacement of Ordinary Portland Cement. However, research showed that ladle furnace slag is a weak supplementary material that shows some hydraulic and pozzolanic properties (Ioanna Papayianni & Anastasiou, n.d.). However, the steel slag may cause volume expansion due to high free CaO content (Shi & Qian, 2000).

Theoretically, the utilization of LFS as partial cement replacement material will result in lower energy cost and higher later strength development (Shi & Qian, 2000). Thus, the chemical composition, physical properties, strength and other tests such as initial and final setting time and soundness test need to be done to make the standards specification of ladle furnace slag as cementitious material as comparable as or better than OPC.

The LFS will be blended with Metakaolin to enhance and control the compositional, structural and morphological properties of the raw material. The compressive strength of concrete must be enhanced with improvement of LFS blended with Metakaolin as a cement replacement material. Metakaolin is very useful as it contributes to the early strength of the concrete. The initial and final setting time of concrete is also expected to be decrease with the use of Metakaolin. It is hoped that this research can contribute to global sustainability by substituting the consumption of large amounts of quicklime or cement thus, reducing atmospheric greenhouse gas emissions (Ortega-López, Manso, Cuesta, & González, 2014).

1.3 Objective

The study of ladle furnace slag (LFS) as cementitious material is done to meet the following objectives:

- a) To determine the effect of LFS as a cementitious material for the binder in aspect of setting time and soundness.
- b) To determine the percentage of LFS used to increase the compressive strength of the binder.

1.4 Scope of Study

This research will involve a sample of ladle furnace slag (LFS) from Southern Steel Malaysia and Metakaolin from Chemical Engineering laboratory. The experiment work will be done in the Chemical, Civil and Mechanical Engineering laboratories in

Universiti Teknologi PETRONAS (UTP). The sample will be characterized to find out the chemical, mineralogical and morphological properties of the LFS. The time frame to complete the research is eight to nine months approximately. The test that will be conducted for the binder will include initial and final setting time, soundness test and compressive strength test. The correct or adequate percentage of the LFS used will be determined to increase the compressive strength of the binder using LFS as the cementitious material.

CHAPTER 2

LITERATURE REVIEW

2.1 CO₂ Emission From Manufacture of Portland Cement Clinker

Cement is a fine, soft, powdery-type substance that acts as an adhesive or glue, which set binds particles of fine aggregate together to produce mortar. Cement depends upon a reaction with water making it hydraulic materials. The most commonly used type of cement is Ordinary Portland Cement (OPC) which is made from limestone or chalk and shale or clay. Despite its massive uses in the construction industry, the manufacture of OPC emits large volume of Carbon dioxide (CO₂) to the environment (Gartner, 2004). Not only that, Gartner (2004) stated that some estimates put the cement industry as total as high as 5% of total global anthropogenic CO₂ emissions. Hence, in order to reduce the emission of CO₂ caused by the manufacture of OPC, the manufacture or production of OPC need to be reduced by introducing cement replacement materials. In this research paper, the raw materials involved are Ladle Furnace Slag (LFS) and Metakaolin as shown in Figure 1 and Figure 2 below.



FIGURE 1. Ladle Furnace Slag (Source: <http://www.cee.ntu.edu.sg/Research/Documents/2002/HTM/index.htm>)



FIGURE 2. Metakaolin (Source: <http://www.hpcbridgeviews.com/i67/Article3.asp>)

2.2 LFS As Cement Replacement Material

2.2.1 Chemical Composition

Chemical analysis was done to determine the chemical composition of LFS. According to Manso, Losañez, Polanco & Gonzalez (2005), the composition of CaO, SiO₂ and Al₂O₃ were 58.0%, 17.0% and 12.0% respectively whereas MgO, SO₃ and other chemical composition is 10.0%, 1.0% and 1.5% respectively. This chemical composition was similar to the research conducted by Setién, Hernández and González in 2009 which mentioned that 60% of LFS weight is made up of calcium and magnesium oxides. Other main acid oxides jointly make up to less than 40% of the total weight of the LFS (Setién et al., 2009). Another study found that the chemical composition of LFS were 32.41%, 50.65%, 1.36% and 2.66% for SiO₂, CaO, Al₂O₃ and Fe₂O₃ respectively (Anastasiou, Papayianni, & Papachristoforou, 2014).

2.2.2 Mineralogical Properties

To determine the mineralogical properties, a diffraction pattern and main peaks were identified. bregidite, olivine, larnite, belite and alite were the set of crystalline calcium silicates identified that account for 40% of the total material (Manso, Losañez, Polanco, & González, 2005). Not only that, calcium silicates under their various allotropic forms (diopside, merwinite, wollastonite, larnite, bredigite,

ingesonite or calcium olivine) are the major compounds of LFS (Setién et al., 2009). On the other hand, Montenegro et al. (2013) stated that the primary minerals found in LFS were periclase, portlandite, fluorite, calcite, mayenite, calcium-olivine, gehlenite, jasmundite, C3A, katotite, quartz and merwinite. The Rietveld analysis of LFS showed that mineral contents in LFS were mainly pleochroite/Q-phase ($\text{Ca}_{20}\text{Al}_{25}\text{Mg}_3\text{Si}_3\text{O}_{68}$), mayenite ($\text{Ca}_{12}\text{Al}_{14}\text{O}_{33}$), tricalcium aluminate ($\text{Ca}_3\text{Al}_2\text{O}_6$), wuestite (FeO), dicalcium silicate ($\beta\text{-Ca}_2\text{SiO}_4$), dolomite ($\text{CaMg}(\text{CO}_3)_2$), dicalcium ferrite ($\text{Ca}_2\text{Fe}_2\text{O}_5$) and akermanite ($\text{Ca}_2\text{MgSi}_2\text{O}_7$) (Adolfsson, Robinson, Engström, & Björkman, 2011). Not only that, Adolfsson et al. (2011) also suggested to consider the possibilities of increasing the content of calcium aluminates in the slag during processing, and to decrease the content of phases falling within the CaO-Al₂O₃-MgO-SiO₂ assemblage.

2.2.3 Morphological Properties

Based on Yildirim & Prezzi (2011), sand and silt size particles of LFS samples showed subrounded to subangular shapes. LFS sample also appeared as dusty and a few of aggregate particle sizes are observed (Setién et al., 2009). The most obvious particle size is 50-60 μm (Setién et al., 2009) and (Manso et al., 2005). It is proved that the fineness of ladle slag can increase the compressive strength thus showed that the potential cementitious property of LFS increased with their fineness regardless of some differences in their mineral composition (Shi, 2001). Figure 3 and Figure 4 show the SEM micrographs of LFS.

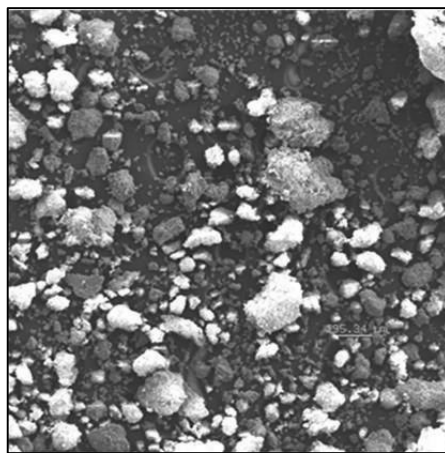


FIGURE 3. SEM micrographs of Ladle Furnace Slag; sand and silt size particle shapes (magnification = 50X). (Yildirim & Prezzi, 2011).

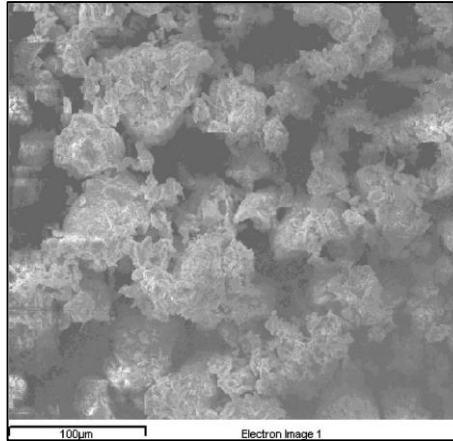


FIGURE 4. Scanning electron microscope micrography of ladle furnace reducing slag (Manso et al., 2005).

2.3 Current Application of LFS

Nowadays, LFS is used as a material for the stabilization of soils in embankment construction because it is feasible and useful (Montenegro et al., 2013). Application of LFS for soil stabilization also proven by the statement that the interaction of the most reactive compounds of LFS with the clayey fraction of soils is clearly a positive advantage in the preparation of well stabilized soils with good bearing capacity and low swelling (Ortega-López et al., 2014). Not only that, according to Ortega et al. (2014), the properties of soil-LFS mixes were useful for the preparation of embankments, road-beds, sub-bases and bases for civil works. LFS also can be used to increase powder content in fibre reinforced self-compacting concrete without compromising self-compactibility, strength or durability (Anastasiou et al., 2014). Figure 5 below shows the evenly distributed fibres at the cross section of a fractured concrete beam with 0.7% steel fibre content.



FIGURE 5. Evenly distributed fibres at the cross section of a fractured concrete beam with 0.7% steel fibre content (Anastasiou et al., 2014)

LFS is also seems to have a positive effect on strength development concerning 28-day and 120-day compressive strength as stated by Anastasiou et al (2014). Moreover, LFS is also can be used as a fine aggregate to correct the granulometric of thick sands, thereby obtaining a more compact and closed granulometric curve (Rodriguez, Manso, Aragón, & Gonzalez, 2009). LFS is also suitable to be used for paving roads as soil-cement mixture and in masonry mortars that provide mixtures with good properties such as binding, workability, shrinkage and durability (Manso et al., 2005). The application of LFS is suitable for safe, high-volume utilization of industrial by-products, providing a final product of improved performance (I. Papayianni & Anastasiou, 2010).

The incorporation of ladle slag in the plaster mortars reduces the setting time and also provide sufficient flexural and compressive strength (Rodríguez, Gutiérrez-González, Horgnies, & Calderón, 2013). Rodríguez et al. (2013) also stated that ladle furnace slag wastes are constantly viable and can be produced more sustainably by reducing the use of natural resources as well as by decreasing the amount of slag-based wastes.

Different studies showed that carbon dioxide activated ladle slag binder have a much reduced embodied energy in comparison to Portland cement products making it contribute to the reduce of natural consumption, landfill for disposal and also adds value to an industry waste (Mahoutian, Ghouleh, & Shao, 2014). Mahoutian et al.

(2014) also stated that slag-based building products at steel mill can reduce carbon dioxide emission from steel production.

2.4 Testing LFS as Cementitious Material

Research done by Papayianni & Anastasiou (n.d.) showed that LFS is a weak supplementary material that shows some hydraulic and pozzolanic properties. However, the properties can be improved by grinding or sieving it into finer material (Ioanna Papayianni & Anastasiou, n.d.). On the other hand, 70% LFS, 20% silica flour and 10% hydrated lime is the optimum composition system which gives a compressive strength around 70 MPa compared to other seven-batch factorial design (Shi & Hu, 2003). Another study conducted by the same researchers, Papayianni and Anastasiou in 2014 showed that a concrete that used LFS as 30% of total binder and EAF slag as coarse aggregates has a 28-d compressive strength levels as same as to those of conventional concrete. Not only that, the use of LFS as binder and EFS slag as aggregates improved the microstructure and porosity of an impermeable concrete (I. Papayianni & Anastasiou, 2010).

Although the best formulation is 50% Portland cement and 50% silica flour that gives the highest compressive strength (76.2 MPa), the use of 70% LFS in the earlier mentioned system showed that it has the potential to be used as cement replacement material (Shi & Hu, 2003). Figure 6 below shows the composition and compressive strength of ladle slag fines-hydrated lime-silica flour system cured at 175 °C for 4 h.

Composition and compressive strength of ladle slag fines–hydrated lime–silica flour system cured at 175 °C for 4 h				
No.	Composition (%)			Compressive strength (MPa)
	Ladle slag (–100 mesh) (X_1)	Hydrated lime (X_2)	Silica flour (X_3)	
22	80	0	20	5.3
23	80	20	0	4.8
24	0	40	60	41.5
25	0	20	80	38.4
26	33.33	33.3	33.33	43.4
27	50	20	30	57.1
28	65	10	25	72.4

FIGURE 6. Composition and compressive strength of ladle slag fines-hydrated lime-silica flour system cured at 175 °C for 4 h. (Shi & Hu, 2003).

According to Rodriguez et al. (2009), the use of LFS can save natural resources (sand) and manufactured products (cement and admixtures). This statement is supported by Manso et al. (2011) that conclude that the preparation of masonry mortars including ladle furnace slag as a significant component can reduce the use of sand and cement. Thus, the use of LFS produced a high quality product which satisfies the standard requirement (Manso, Rodriguez, Aragón, & Gonzalez, 2011). It also showed positive result for long term compression strength. Figure 7 below shows the compression test to breakage point done by Rodriguez et al. (2009).



FIGURE 7. Compression test to breakage point (Rodriguez et al., 2009).

The viability of converting steelmaking ladle slag into cementing binder through carbon dioxide activation was studied (Mahoutian et al., 2014). The study showed that stronger carbonation reactivity occurred by ladle slag with higher SiO_2 content and lower free lime whereas ladle slag with higher aluminate content or higher free lime content could not be activated by carbon dioxide to develop strength. The ultimate strength of the ladle slag activated by carbonation was attributed to the hybrid structure of carbonation and hydration products (Mahoutian et al., 2014).

Research showed that ladle slag and fly ash based alkali activated material (AAM) exhibited superior gains and better thermal stability than the ladle slag and metakaolin based AAMs believed to be due to unstable C-A-S-H phases formed in the latter group of samples (Natali Murri, Rickard, Bignozzi, & van Riessen, 2013). In addition, spherical morphology of fly ash improve workability of ladle slag and fly ash sample thus made it easier to be synthesized (Natali Murri et al., 2013).

In different study, stainless steel refining (SSR) slag mortars developed considerable compressive strength when activated by mixtures of 5 M NaOH and Na-silicate or 5 M KOH and K-silicate, followed by steam curing at 80°C (Salman et al., 2015). Hence, it is showed that SSR slag has the potential to be used as construction material.

2.5 Metakaolin

In the study of Evolution of binder structure in sodium silicate-activated slag Metakaolin blends, the addition of Metakaolin decreases the final strength slightly but does not change the relative strength development profile (Bernal, Provis, Rose, & Mejía de Gutierrez, 2011). Furthermore, the use of Metakaolin in the mixes extend setting time and provide workability which is of significant value in the application of alkali-activated slag binders (Bernal et al., 2011). However, different result was obtained in different studies when Metakaolin is added in the concrete containing GGBS. The presence of Metakaolin content in concrete containing GGBS causes an increase in strength during the early ages of hydration (Khatib & Hibbert, 2005).

In different study, higher Metakaolin content in concrete causes it to become denser, thus accelerating the setting (Khan, Nuruddin, Ayub, & Shafiq, 2014). Metakaolin has smaller particle size and higher specific area. These properties are favourable to produce highly dense and impermeable concrete (Khan et al., 2014). It is no doubt that Metakaolin can be used in mortar and concrete to improve their properties (Rashad, 2013). Rashad (2013) also added that Metakaolin can be used as a source of cementing materials in alkali activation of geopolymer.

CHAPTER 3

METHODOLOGY

3.1 Project Flowchart

The figure below shows the Project Flowchart:

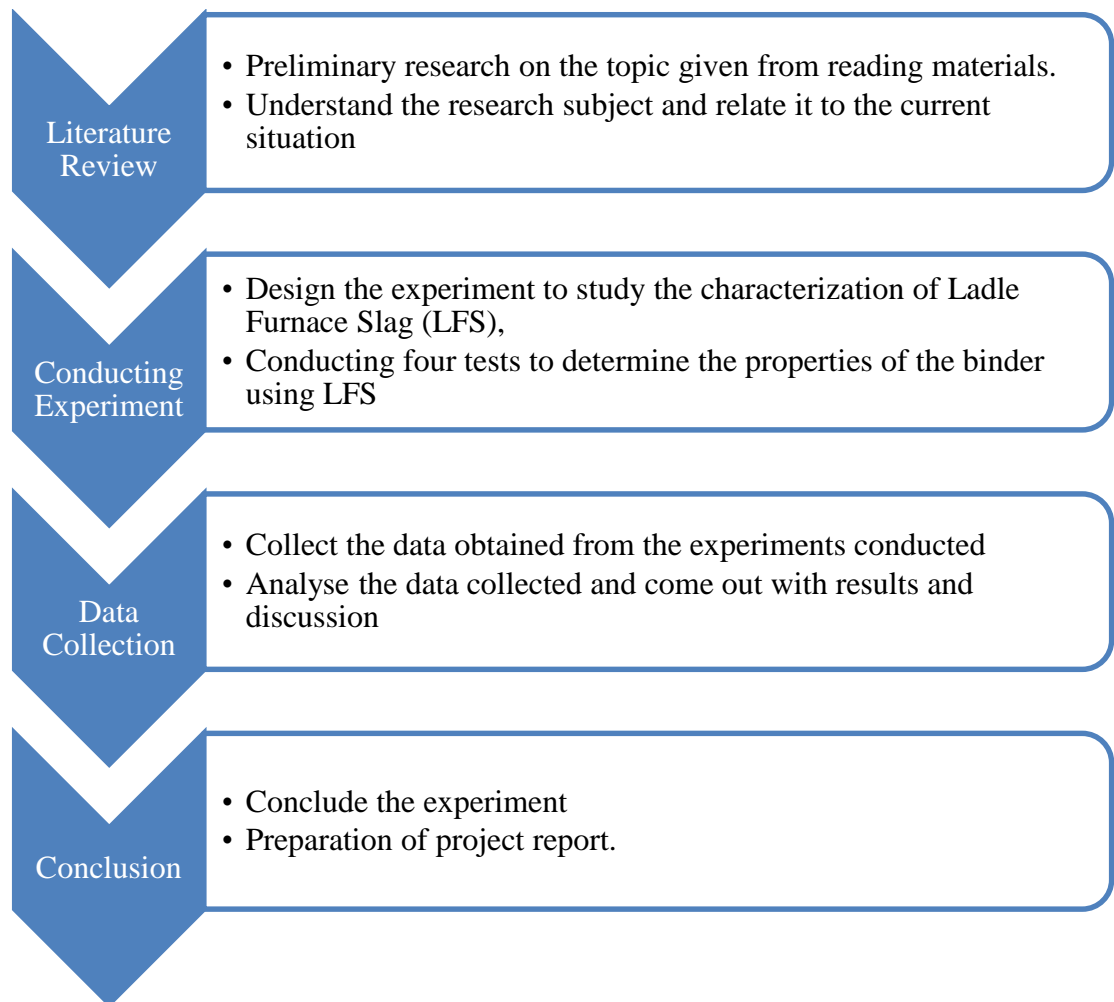


FIGURE 8. Process Flowchart

3.2 Materials and Method

3.2.1 Raw Material Preparation and Characterization of Ladle Furnace Slag (LFS) and Metakaolin.

The raw materials such as LFS and Metakaolin will be obtained from the local industry and representatively sampled for characterization work. The chemical, mineralogical and physical characteristics of these waste materials will be determined. Surface morphology and structural analysis of particles will also be conducted. Various equipments such as FESEM, FTIR, XRD, CHNO, Malvern Particle Size Analyzer, BET, etc. will be used for characterization.

The samples will be classified and graded accordingly to available standards and will also include additional information such as their source and processing methods in the production of these materials, yearly tonnage etc. to create a comprehensive database of these materials.

3.2.2 Preparation of Binder and Mortar

LFS with the different percentage will be blend with OPC and Metakaolin to produce the binder. Mortars will be produced following the standard procedure (ASTM C305-06). River sand will be used as fine aggregate. The sand to cement ratio used is two whereas the water to cement ration is 0.5. All sample mixes will be casted in a 50mm x 50mm x 50mm cubic mould and stored under controlled relative humidity and ambient temperature for 24 h. Demoulding will be done after 24 hours whereas curing will be done for 3, 7 and 28 days. Details of the mixes are given in Table 1 .

TABLE 1. Detail of mixes

Mix No.	Proportions (%) of CM			Content (g)				
	OPC	LFS	Metakaolin	OPC	LFS	Metakaolin	Sand	Water
S1	100	-	-	300	-	-	600	150
S2	85	5	10	255	15	30	600	150
S3	80	10	10	240	30	30	600	150
S4	70	20	10	210	60	30	600	150

3.2.3 Setting Time Test

The purpose of this test method is to establish whether or not cement or binder complies with a specification limit on time of setting. The initial and final setting time of fresh pastes will be determined using the Vicat apparatus (ASTM C191-08) as shown in Figure 9. The initial setting time is the time elapsed between initial contact of cement and water and the time when the penetration is measured or calculated to be 25 mm. The final setting time is the time elapsed between initial contact of cement and water and the time when the needle does not leave a complete circular impression in the paste surface.



FIGURE 9. Vicat Apparatus (Source: http://asianinstrument.com/civil_instruments.aspx)

3.2.4 Soundness Test

To do this test, a specimen of hardened cement paste is boiled for a fixed time so that any tendency to expand is speeded up and can be detected. Soundness means the ability to resist volume expansion. This test is primarily designed to detect the presence of any free lime which might be present inside grains of clinker. The test will be conducted according to IS: 4031-Part 3-1988 using the Le Chatelier Moulds consisting of a small brass cylinder containing a split, on each side of which are fixed a long pointer to magnify the movements as shown in the Figure 10 below:

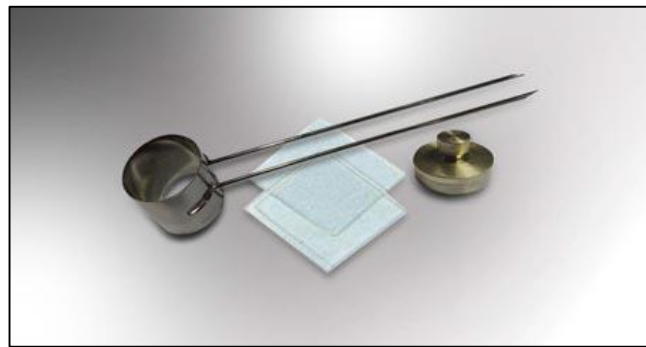


FIGURE 10. Le Chatelier Moulds and Flasks (Source: <http://civilblog.org/2013/05/04/specific-gravity-density-of-hydraulic-cement-is4031-part-11-1988/>)

3.2.5 Compressive Strength Test

The purpose of this test is to determine the compressive strength of mortars and results may be used to determine compliance with specifications. Compressive strength will be tested at 3, 7 and 28 days according to ASTM C109 using Universal Testing Machine as shown in Figure 11 below.



FIGURE 11. Universal Testing Machine (Source: http://www.eieinstruments.com/productdetail/Strength_of_Material_TestingUniversal_Testing_Machine)

3.3 KEY MILESTONE

The key milestone of this project is as shown in the FIGURE 12 below:

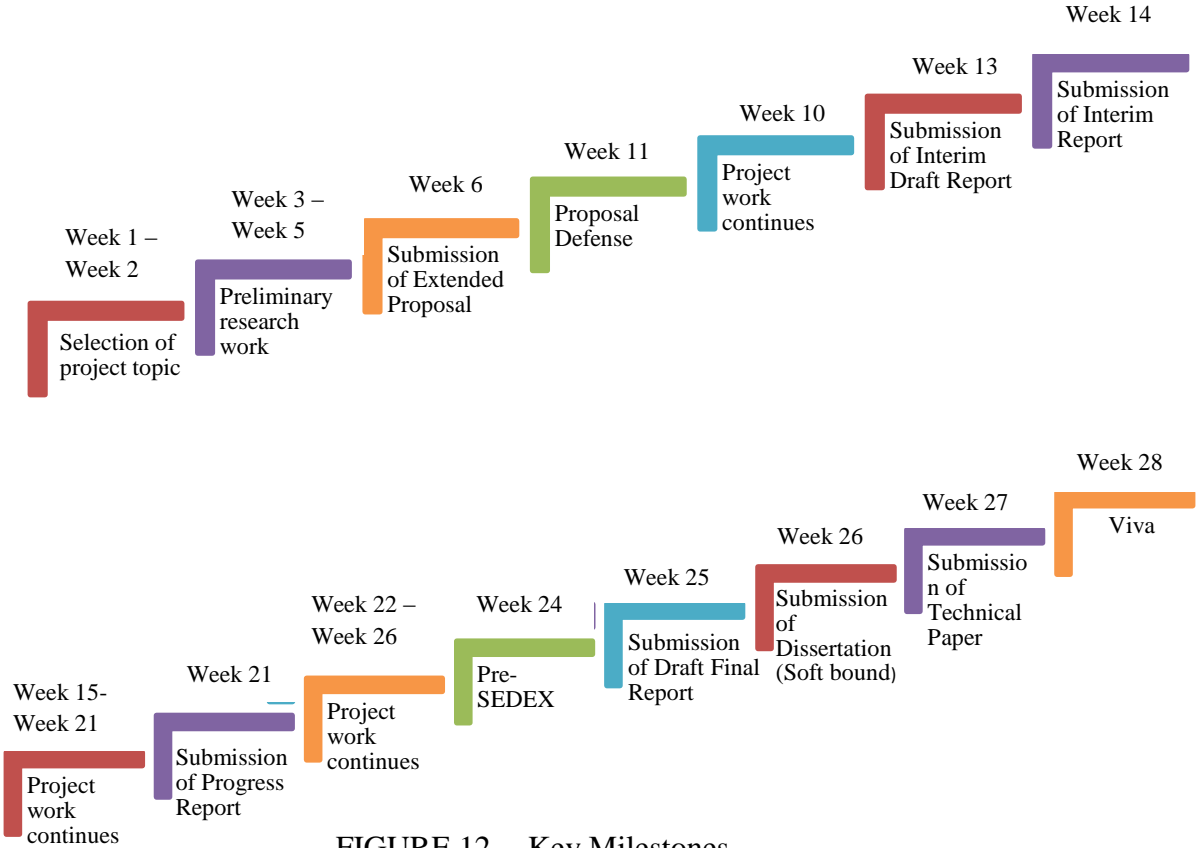


FIGURE 12. Key Milestones

3.4 GANTT CHART

3.4.1 FYPI

No	Activities / Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of Project Topic	█	█												
2	Discuss with Supervisor regarding process and planning of experiment			█											
3	Preliminary Research Work			█	█	█									
4	Preparation of Sample						█	█							
5	Submission of Extended Proposal						█								
6	Characterization of Ladle Furnace Slag and Metakaolin								█	█	█				
7	Proposal Defence										█				
8	Project Work Continues											█	█	█	█
9	Submission of Interim Report														█

3.4.2 FYP II

No	Activities / Week	15	16	17	18	19	20	21	22	23	24	25	26	27	28
10	Project Work Continues														
11	Mixing and casting mortars														
12	Compressive strength test														
13	Obtaining XRF Analysis Results														
14	Mixing and casting mortars (2 nd batch)														
15	Submission of Progress Report														
16	Project Work Continues														
17	Pre-SEDEX														
18	Submission of Draft Final Report														
19	Submission of Dissertation (Soft bound)														
20	Submission of Technical Paper														
21	Viva														

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Chemical Composition

X-ray Florescence (XRF) analysis was done to determine the element percentage of the materials. The result of the XRF analysis is as shown in TABLE 2 below:

TABLE 2. Element percentage of Ladle Furnace Slag

Elements	Percentage (%)		
	LFS	Metakaolin	Ordinary Portland Cement
Ca	78.4	2.08	82.5
Si	11.5	57.4	7.06
Fe	3.27	3.35	5.15
Al	1.76	28.5	1.32
Ti	0.506	3.46	0.229
P	0.391	3.07	0.336
Zn	0.209	0.0384	0.0324
K	0.0990	1.33	0.516
Sr	0.0709	0.0735	0.0496
Zr	0.0444	0.509	0.0300
Cu	0.0120	0.0224	0.0167
Mg	1.67	-	0.736
S	0.950	-	1.76
Mn	0.812	-	0.174
Cr	0.0527	-	0.0204
Cl	0.0521	-	0.0529
Ba	0.163	-	-
Rb	-	0.0113	0.0139
Ni	-	0.0175	-
As	-	0.0158	-
Ga	-	0.0395	-
Nb	45.865 PPM	0.0176	-
Y	-	89.666 PPM	-
Mo	-	-	40.558 PPM

The results shown in TABLE 2 show the major composition in LFS is Ca and Si whereas Metakaolin has a high content of Si and Al. OPC however, contain high amount of Ca, Si and Fe. Since Metakaolin has a high content of Si and Al, it became a good choice to add Metakaolin into the mixes to enhance the LFS and of course the binder propertis.

4.2 Morphological Properties

The appearance of LFS and Metakaolin in a scanning electron microscope (SEM) is presented in Figure 13 and Figure 14 respectively that are obtained under 15kV accelerating voltage, with the magnification up to 3000. The SEM micrography of LFS shows that it has subrounded to subangular shape whereas SEM micrography of Metakaolin show that it has particles of irregular shape having multiple layer of structure.

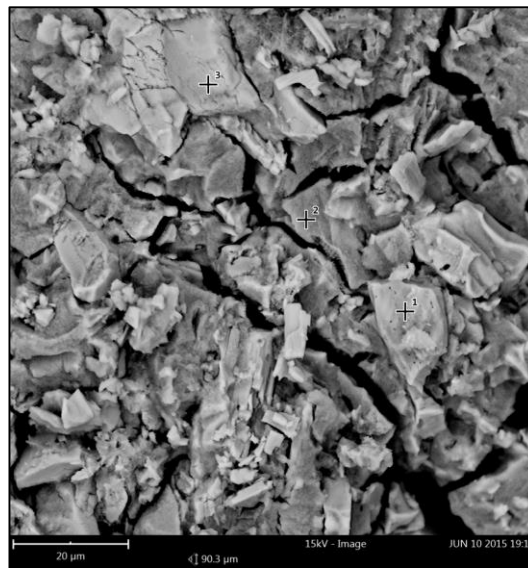


FIGURE 13. SEM micrography of LFS.

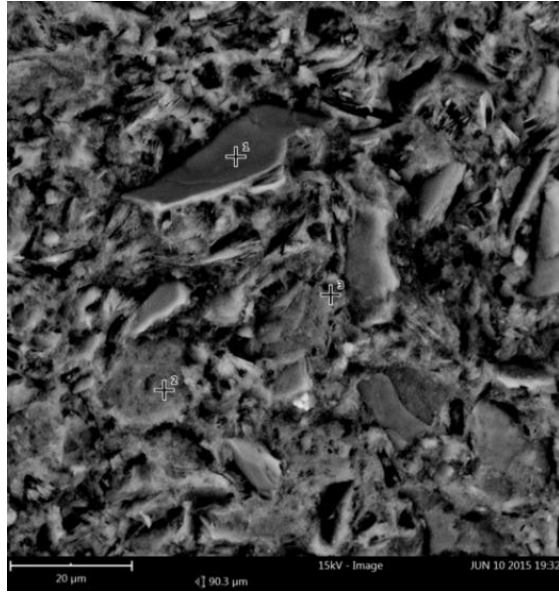


FIGURE 14. SEM micrography of Metakaolin.

4.3 Compressive Strength

The compressive strength of the samples is shown in the tables and figures below.

TABLE 3. Compressive strength of the samples after 3 days.

Cube Reference	Date Cast	Date Tested	Age (days)	Measured Size (mm)	Max Load (kN)	Compressive Strength (MPa)
S1 (i)	29/5/2015	1/6/2015	3	50 x 50 x 50	59.0	23.61
S1 (ii)	29/5/2015	1/6/2015	3	50 x 50 x 50	58.0	23.20
S1 (iii)	29/5/2015	1/6/2015	3	50 x 50 x 50	58.9	23.57
S2 (i)	29/5/2015	1/6/2015	3	50 x 50 x 50	72.4	28.97
S2 (ii)	29/5/2015	1/6/2015	3	50 x 50 x 50	75.7	30.29
S2 (iii)	29/5/2015	1/6/2015	3	50 x 50 x 50	73.7	29.48
S3 (i)	29/5/2015	1/6/2015	3	50 x 50 x 50	72.0	28.80
S3 (ii)	29/5/2015	1/6/2015	3	50 x 50 x 50	75.4	30.18
S3 (iii)	29/5/2015	1/6/2015	3	50 x 50 x 50	77.9	31.15
S4(i)	29/5/2015	1/6/2015	3	50 x 50 x 50	59.5	23.81
S4(ii)	29/5/2015	1/6/2015	3	51 x 50 x 50	46.6	18.66
S4(iii)	29/5/2015	1/6/2015	3	52 x 50 x 50	51.4	20.57

TABLE 4. Compressive strength of the samples after 7 days.

Cube Reference	Date Cast	Date Tested	Age (days)	Measured Size (mm)	Max Load (kN)	Compressive Strength (MPa)
S1 (i)	4/6/2015	11/6/2015	7	50 x 50 x 50	92.1	36.48
S1 (ii)	4/6/2015	11/6/2015	7	50 x 50 x 50	85.5	34.21
S1 (iii)	4/6/2015	11/6/2015	7	50 x 50 x 50	95.6	38.22
S2 (i)	4/6/2015	11/6/2015	7	50 x 50 x 50	79.9	31.96
S2 (ii)	4/6/2015	11/6/2015	7	50 x 50 x 50	49.2	19.60
S2 (iii)	4/6/2015	11/6/2015	7	50 x 50 x 50	60.3	24.10
S3 (i)	4/6/2015	11/6/2015	7	50 x 50 x 50	80.5	32.20
S3 (ii)	4/6/2015	11/6/2015	7	50 x 50 x 50	67.8	27.14
S3 (iii)	4/6/2015	11/6/2015	7	50 x 50 x 50	86.8	34.71
S4 (i)	4/6/2015	11/6/2015	7	50 x 50 x 50	76.5	30.60
S4 (ii)	4/6/2015	11/6/2015	7	50 x 50 x 50	76.7	30.66
S4 (iii)	4/6/2015	11/6/2015	7	50 x 50 x 50	74.6	29.86

TABLE 5. Compressive strength of the samples after 28 days.

Cube Reference	Date Cast	Date Tested	Age (days)	Measured Size (mm)	Max Load (kN)	Compressive Strength (MPa)
S1 (i)	27/6/2015	25/7/2015	28	50 x 50 x 50	69.6	39.84
S1 (ii)	27/6/2015	25/7/2015	28	50 x 50 x 50	94.3	37.74
S1 (iii)	27/6/2015	25/7/2015	28	50 x 50 x 50	108.0	43.20
S2 (i)	27/6/2015	25/7/2015	28	50 x 50 x 50	83.3	33.32
S2 (ii)	27/6/2015	25/7/2015	28	50 x 50 x 50	79.6	31.84
S2 (iii)	27/6/2015	25/7/2015	28	50 x 50 x 50	98.0	39.21
S3 (i)	27/6/2015	25/7/2015	28	50 x 50 x 50	64.4	34.75
S3 (ii)	27/6/2015	25/7/2015	28	50 x 50 x 50	101.6	40.65
S3 (iii)	27/6/2015	25/7/2015	28	50 x 50 x 50	89.8	35.92
S4 (i)	27/6/2015	25/7/2015	28	50 x 50 x 50	72.4	28.96
S4 (ii)	27/6/2015	25/7/2015	28	50 x 50 x 50	84.1	33.62
S4 (iii)	27/6/2015	25/7/2015	28	50 x 50 x 50	81.9	32.74

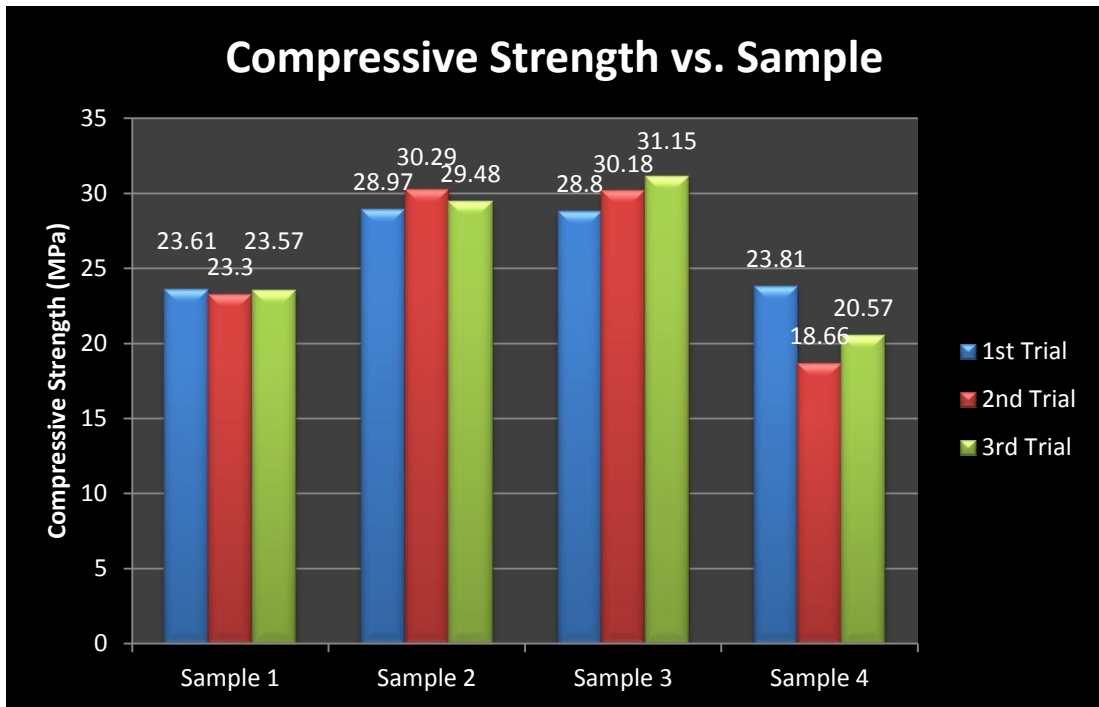


FIGURE 15. Compressive strength of the samples after 3 days

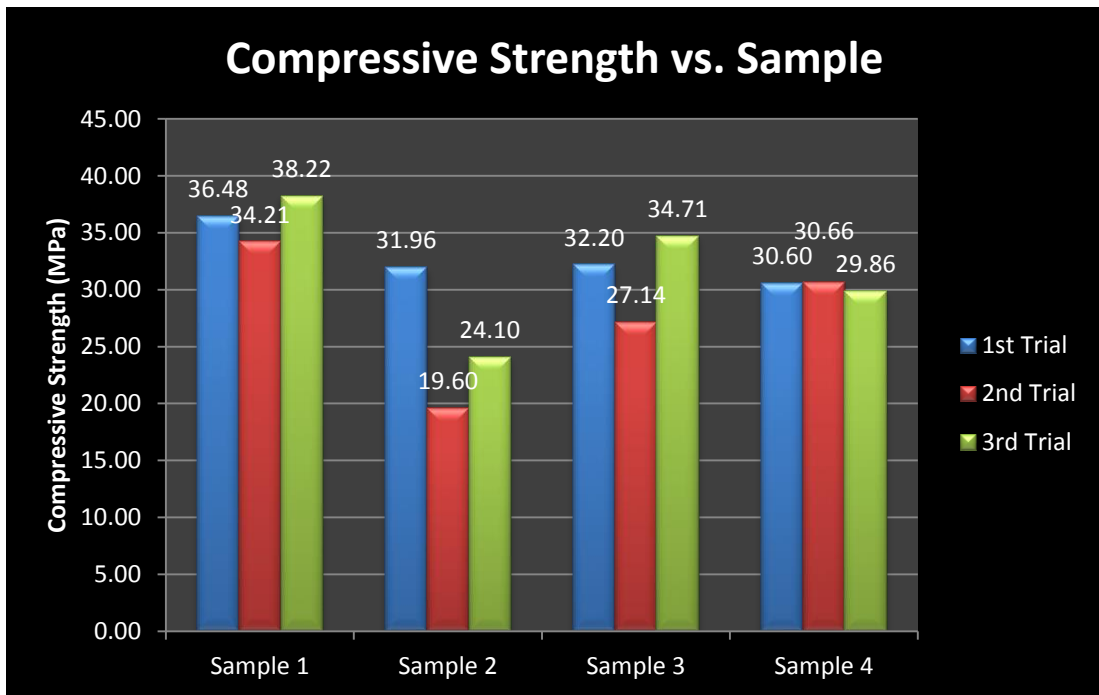


FIGURE 16. Compressive strength of the samples after 7 days.

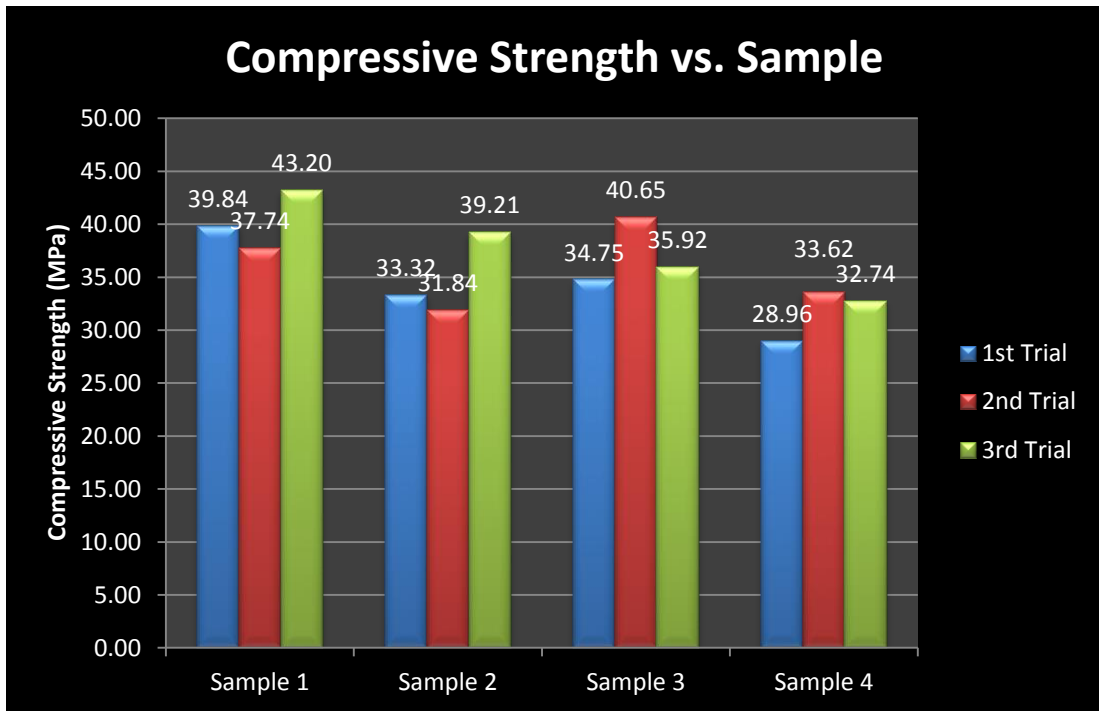


FIGURE 17. Compressive strength of the samples after 28 days.

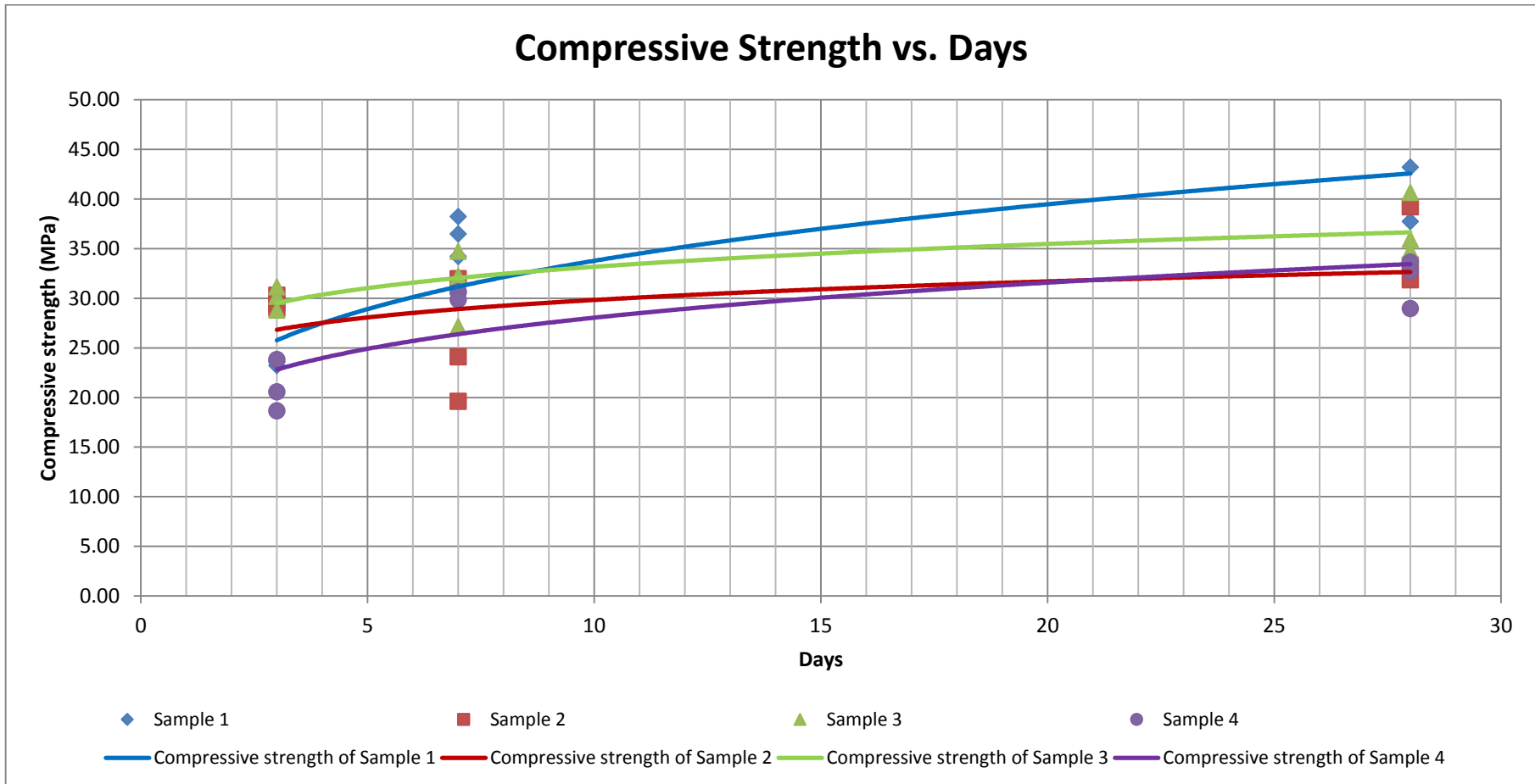


FIGURE 18. Compressive strength vs. Days.

As shown in the tables and figures above, the highest compressive strength achieved after 3 days is from sample S3 whereas the lowest compressive strength achieved is from sample S1 which used 100% OPC as the binder. From these results, it can be stated that OPC has lower early strength as compared to the samples which include LFS and Metakaolin in the binder composition.

However, different findings are obtained after 7 days. From Figure 16, the compressive strength of OPC is the highest whereas S2 has the lowest compressive strength. From Figure 12, the sequence of compressive strength from the highest to lowest can be shown as $S1 > S3 > S4 > S2$. From the comparison between samples that include LFS and Metakaolin in the binder composition, sample S3 has the highest compressive strength. Sample S3 which contains 10% of LFS, 10% of Metakaolin and 80% of OPC showed the best result at the early age and also after 28 days.

4.4 Setting Time

Among the samples containing LFS and Metakaolin, Sample 3 shows the best result in compressive strength after 28 days. Hence, setting time test was done for Sample 1 and Sample 3 to find the comparison between sample without LFS and sample with LFS. The calculations and results of the setting time for both samples are shown as below:

Sample 1 (100% OPC)

$$\begin{aligned}
 \text{Initial Setting Time} &= \left(\left(\frac{(H - E)}{(C - D)} \right) \times (C - 25) \right) + E \\
 &= \left(\left(\frac{(358 - 349)}{(24 - 27)} \right) \times (27 - 25) \right) + 349 \\
 &= 343 \text{ min} \\
 &= 5.72 \text{ hr}
 \end{aligned}$$

where:

E = time in minutes of last penetration greater than 25 mm,

H = time in minutes of first penetration less than 25 mm,

C = penetration reading at time E, and

D = penetration reading at time H.

Final Setting Time = 387 min

$$= 6.45 \text{ hr}$$

Sample 3 (10% LFS, 10% MK)

$$\begin{aligned} \text{Initial Setting Time} &= \left(\left(\frac{(H - E)}{(C - D)} \right) \times (C - 25) \right) + E \\ &= \left(\left(\frac{(428 - 409)}{(23 - 27)} \right) \times (23 - 25) \right) + 409 \\ &= 399.5 \text{ min} \\ &= 6.66 \text{ hr} \end{aligned}$$

where:

E = time in minutes of last penetration greater than 25 mm,

H = time in minutes of first penetration less than 25 mm,

C = penetration reading at time E, and

D = penetration reading at time H.

Final Setting Time = 445 min

$$= 7.42 \text{ hr}$$

From the calculations above, the setting time of Sample 3 is longer than the setting time of Sample 1. It shows that the partial replacement of OPC with LFS increases the setting time. Although 10 percent of Metakaolin added to the sample was meant to decrease the setting time, the results clearly show the opposite. Thus, as a recommendation, 20 percent of Metakaolin need to be added into the sample instead of 10 percent.

4.5 Soundness

Soundness test was done for Sample 1 and Sample 3. The calculations below show the soundness of the samples.

Sample 1 (100% OPC)

$$\text{Soundness} = d_2 - d_1$$

$$= 1.5 - 1.4$$

$$= 0.1 \text{ cm}$$

$$= 10 \text{ mm}$$

where:

d_2 = final distance between the indicator point

d_1 = initial distance between the indicator point

Sample 3 (10% LFS, 10% MK)

$$\begin{aligned}\text{Soundness} &= d_2 - d_1 \\ &= 1.1 - 1.0 \\ &= 0.1 \text{ cm} \\ &= 10 \text{ mm}\end{aligned}$$

where:

d_2 = final distance between the indicator point

d_1 = initial distance between the indicator point

The results above show that the soundness of the two samples is the same which is 10 mm. Sample 1 and Sample 3 have the same ability to retain its volume after setting without delayed destructive expansion.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

This research was done to determine the effect and percentage of LFS used as a cementitious material for the concrete. Not only that, this project aims to produce a green binder with a consistent high compressive strength and shorter setting time.

Based on the results, sample S3 with 10% LFS and 10% Metakaolin has the best results compared to S2 and S4 in compressive strength after 28 days. Sample S1 which acts a control shows highest compressive strength after 7 and 28 days. Although the strength of S3 is not as high as the strength of S1, the potential of implementing LFS as the cementitious material is possible since the difference between the results is not so significant. The implementation of LFS in the binder increases the setting time but has the same soundness with the sample without LFS.

Further research is recommended to be done by testing different percentages of LFS. In this case the percentage of LFS that is suitable for further research is 11% to 19% to find the most accurate percentage of LFS that can increase the compressive strength of the binder. Not only that, an increasing of percentage of Metakaolin is also suggested to decrease the setting time.

Since the sequence of compressive strength of the samples from the highest to lowest is $S1 > S3 > S4 > S2$, it can be concluded that the best percentage of LFS that can be used in binder is 10% which is from Sample 3.

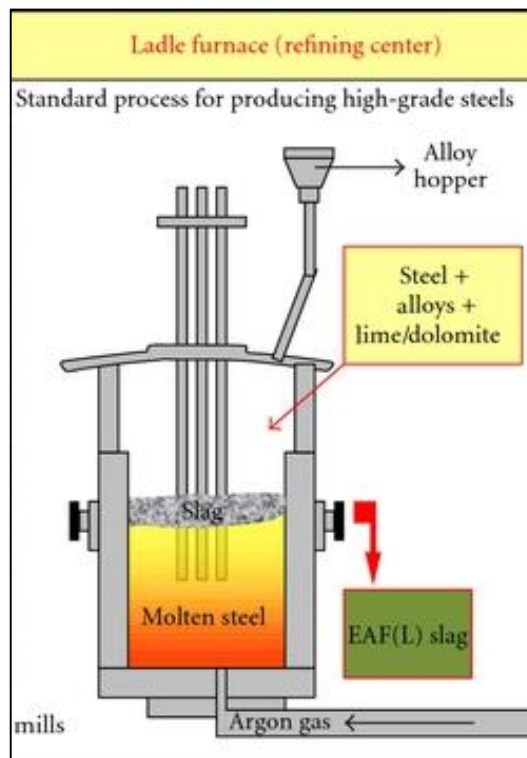
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APPENDICES



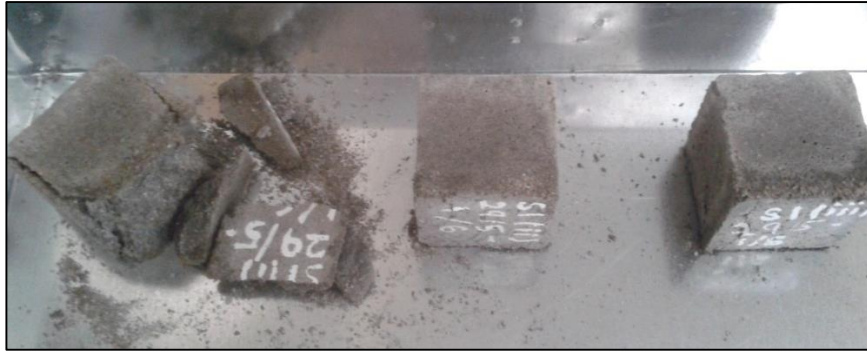
APPENDIX 1 . Schematic representation of the ladle refining process (Yildirim & Prezzi, 2011)



APPENFIX 2. Preparation of sample



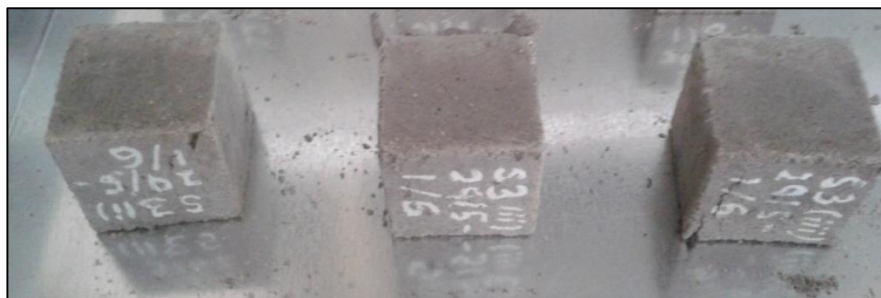
APPENDIX 3. Samples after demoulding.



APPENDIX 4. Sample 1 after compressive strength test for 3 days.



APPENDIX 5. Sample 2 after compressive strength test for 3 days.



APPENDIX 6. Sample 3 after compressive strength test for 3 days.



APPENDIX 7. Sample S4(i) after compressive strength test for 3 days



APPENDIX 8. Sample S4(ii) after compressive strength test for 3 days.



APPENDIX 9. Sample S4(iii) after compressive strength test for 3 days.



APPENDIX 10. Sample S1(i) after compressive strength test for 7 days.



APPENDIX 11. Sample S1(ii) after compressive strength test for 7 days.



APPENDIX 12. Sample S1(iii) after compressive strength test for 7 days.



APPENDIX 13. Sample S2(i) after compressive strength test for 7 days.



APPENDIX 14. Sample S2(ii) after compressive strength test for 7 days.



APPENDIX 15. Sample S2(iii) after compressive strength test for 7 days.



APPENDIX 16. Sample S3(i) after compressive strength test for 7 days.



APPENDIX 17. Sample S3(ii) after compressive strength test for 7 days.



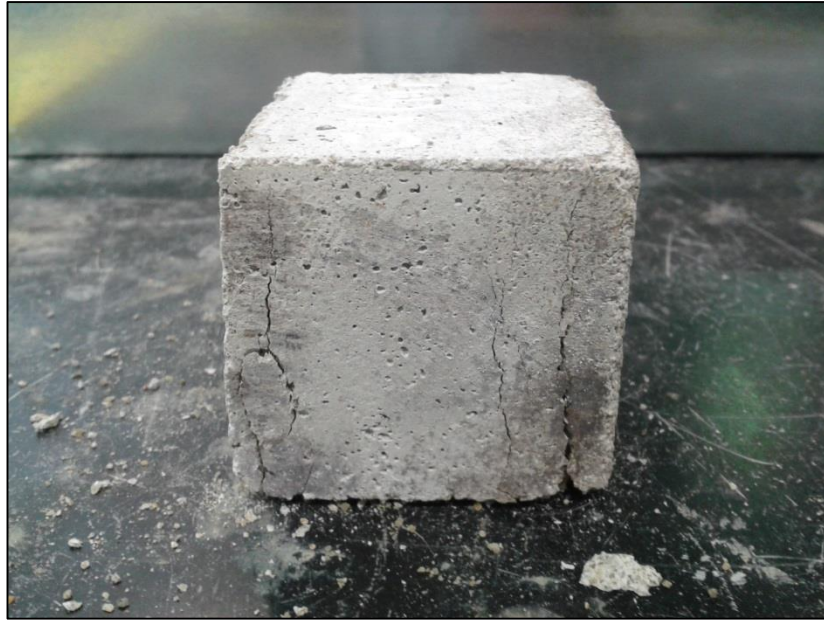
APPENDIX 18. Sample S3(iii) after compressive strength test for 7 days.



APPENDIX 19. Sample S4(i) after compressive strength test for 7 days.



APPENDIX 20. Sample S4(ii) after compressive strength test for 7 days.



APPENDIX 21. Sample S4(iii) after compressive strength test for 7 days.



APPENDIX 22. Sample S1(i) after compressive strength test for 28 days.



APPENDIX 23. Sample S1(ii) after compressive strength test for 28 days.



APPENDIX 24. Sample S1(iii) after compressive strength test for 28 days.



APPENDIX 25. Sample S2(i) after compressive strength test for 28 days.



APPENDIX 26. Sample S2(ii) after compressive strength test for 28 days.



APPENDIX 27. Sample S2(iii) after compressive strength test for 28 days.



APPENDIX 28. Sample S3(i) after compressive strength test for 28 days.



APPENDIX 29. Sample S3(ii) after compressive strength test for 28 days.



APPENDIX 30. Sample S3(iii) after compressive strength test for 28 days.



APPENDIX 31. Sample S4(i) after compressive strength test for 28 days.



APPENDIX 32. Sample S4(ii) after compressive strength test for 28 days



APPENDIX 33. Sample S4(iii) after compressive strength test for 28 days.