USE OF SECONDARY LEAD RECYCLING SLAG AS A CONSTRUCTION MATERIAL

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Submitted by

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DECLARATION

I, RAHUL SHARMA, hereby declare that the thesis entitled "**Use of Secondary Lead Recycling Slag as a Construction Material**" is an authentic record of my studies carried out as a requirement for the award of the degree of **Master of Engineering** in **Structural Engineering** under the supervision of **Dr Vivek Gupta,** Assistant Professor and **Dr Prem Pal Bansal,** Professor and Head, Department of Civil Engineering, Thapar Institute of Engineering and Technology, Patiala. The matter embodied in this report has not been submitted in part or full to any other institute or university for the award of any degree.

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CERTIFICATE

This is to certify that the above declaration made by the student concerned is correct according to the best of our knowledge and belief.

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ABSTRACT

Cement production emits anthropogenic CO_2 into atmosphere, leading to global warming. Also, cement manufacturing consumes abundant natural resources. So, it becomes essential to minimize the environmental impact, and atmospheric carbon dioxide as natural resources are getting declined and greenhouse emissions are becoming more evident.

Admixtures have opened a new era of mix design of required strength and durability to overcome these ill effects. In past decades, various techniques have been adopted to conserve natural resources by using sustainable engineering approaches. Various waste and SCMs have been used to replace Cement. Efforts have been made to save energy by promoting industrial waste like fly ash, silica fume, rice husk, meta-kaolin, GGBS, etc., which shows similar chemical properties as cement. The use of such pozzolanic material reduces the cost and helps reduce cement consumption.

Now a days, these by-products (waste) of the industries are been extensively used in the geopolymer mortar. Geopolymer is inorganic in nature and is typically ceramic. Geopolymer contains an extended range of Alumino-silicates networks held by a covalent bond. Geopolymer is formed by an alkaline solution and a solid Alumino-silicate material. And these by-products (waste) can be the source of silica and alumina in the geopolymer, and after polymerization leads to the production of molecular chains and large networks of units ultimately leading to the strong and hard binder. So the secondary lead slag may also be used in the generation of the geopolymer as it can be a source of silica and alumina.

This research investigates the feasibility analysis of secondary lead recycling slag powder in cementitious matrix as a construction material. Three types of secondary lead slag have been investigated in this study. This utilization of lead slag as construction material can help in solving the negative effects of secondary lead slag on environment. The entire study was divided into two parts.

In the first part, different mechanical properties of secondary lead slag-based cement mortar specimens with varying percentages (i.e., 0%, 5%, 10%, 15%, 20%, 25%, and 30%) of secondary lead slag have been studied. The results concluded that increase in the replacement of three types on Lead Slag after 5%, the results are not promising as compressive strength decreases rapidly. The strength development is better in Type 1 and 3 based cement mortar specimens as compared to Type 2 Lead Slag based mortar

specimens. The strength increases with the age of curing for all types of Lead Slag based cement mortar specimens, as the pozzolana reaction occurs at greater curing age. Type 1 and Type 3 lead slag is highly recommended upto 5% replacement.

In the second part, different mechanical properties of secondary lead slag-based geopolymer mortar specimens with varying percentages (i.e., 0%, 10%, 20%, and 30%) of secondary lead slag have been studied. The results concluded that The Lead Slag (Type 1, 2, 3) has good alkaline reactivity. It suggests the possibility of using the lead slag as geopolymer precursor. Type 3 lead slag is highly recommended as it has more Calcium as compared to Fly ash, increasing in hydration process and different hydration products like CASH ad C-S-H are formed in addition to the co-existing geopolymerization product (NaSH).

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CHAPTER 1

INTRODUCTION

1.1 Introduction

In recent decades, there is a growing demand for sustainable resource management and development has driven the investigation for using various industrial wastes or by-products as supplementary cementitious materials or admixtures for partially replacing the cement in mortars. The most common used binder in mix is ordinary Portland mix (OPC). The manufacturing of OPC contributes to anthropogenic Carbon dioxide emissions, which leads to global warming. Thus, it is essential to minimize environmental impact, and carbon dioxide intensity as resources decline and greenhouse emissions become more evident.

Use of sustainable engineering approaches in mix design can help in maximizing durability and conservation of materials. Various waste and supplementary cementitious materials such as blast furnace slag, fly ash, rice husk ash, silica fume, and meta-kaolin are used as substitution for Portland cement.

Lead slag is one of these materials which can be used as a substitution for cement and aggregates. Lead is a non-ferrous metal with large application in lead-acid batteries, medicine, and machine manufacturing (Chen *et al.,* 2009). Lead slag is a derivative (waste product) mainly formed by primary and secondary resources in lead manufacturing industries. Lead production process is mainly from the smelting of lead ore and the recovery of scrap lead based acid batteries. Primary lead production includes extracting Pb from the smelting of lead sulphide (PbS) concentrate. Secondary lead production includes extracting lead from scrap lead based acid batteries. (Sohn and Olivas-Martinez, 2014)

Figure 1.1 Secondary Lead Slag

Presently, disposing of lead slag is mostly in the landfills, which needs substantial amount of landfill area (Dong, 2001). Also, the lead slag have different heavy and hazardous elements in it like Pb, Cd, Zn, which are highly migratory (Seignez *et al.* 2008). There is a release of the toxic elements under different weathering and leaching environment (Ettler *et al.* 2009). The pH of leachates is mainly responsible for release of harmful components (Bernardez *et al.* 2013). The accumulation of these hazardous elements in landfill sites pollutes soil and the groundwater, which increase the risk of enhancement of these toxic elements in plants and animals, eventually creating a risk for human health (Chai *et al*. 2015).

Besides this, lead slag has other valuable metals like Si, Ca, Cu, and Fe. The recovery of these elements from lead slag can be used as a secondary resource (Yang *et al*. 2014).

Thus to solve these flaws in lead slag, various measures can be taken either by reducing the hazardous elements in lead slag or promoting harmless utilization of lead slag. The different researcher had determined the possibility of using the primary and secondary lead slag in various application like Road pavements, Cement concrete, mortar. Because of high Fe content in Lead Slag, it can be a good substitution for Iron ores in manufacturing cement clinker. Also, lead slag can be used in geopolymer where hazardous elements can be stabilized in a 3D structure of geopolymer network (D. Pan *et al*).

1.2 Role of Mineral and Chemical admixtures

Admixtures play a vital role in concrete or mortar by modifying the property of the cement in a fresh and hardened state, thereby improving the quality and the workability of the concrete. Different admixtures such as GGBS, slag, fly ash, RHA, silica fume, lead slag, meta-kaolin, etc., are used to control the adverse effects of $Ca(OH)_2$ formed during the cement hydration process. Comparing to OPC (Ordinary Portland Cement) these admixtures produce less amount of calcium hydroxide. During the cement hydration, $Ca(OH)_2$ and C-S-H (calcium silicate hydrate) are formed when dicalcium and tricalcium silicates react with water. There are great chances of leaching of calcium hydroxide which consequently make the concrete/ mortar weak, increase porosity and thereby decrease the durability of concrete. Also $Ca(OH)_2$ reacts with the sulphate present in the water and form calcium sulphate and thus increase the chances of reacting with tricalcium aluminate which leads to the deterioration. So these adverse effects of $Ca(OH)_2$ can be decreased by using the admixtures (pozzolana material). Pozzolana reactions help in improving the durability and desired strength by increasing the density of cement paste and thereby making it impervious.

Therefore the use of admixtures in the optimum quantity helps in improving the quality of the mortar/concrete as follows-

- Increasing strength of mortar/concrete.
- Improving the workability of concrete/mortar.
- Reducing rate of heat during hydration.
- Producing concrete of lightweight.
- Accelerating/ retarding initial setting time of cement concrete and mortar.
- Improving early strength and extensibility of concrete and mortar.
- Increasing the durability of concrete and mortar.
- Reducing the porosity of concrete and mortar.
- Controlling alkali-aggregate expansion.
- Enhancing resistance to sulphate attack.
- Increasing bond strength between concrete and steel rebars.
- Reducing segregation of concrete.
- Reducing bleeding of concrete.
- Improving resistance to corrosion.
- Enhancing chemical resistance.

1.3 Reaction mechanism of Lead Slag

The lead formation is mainly from smelting of lead ore and the recovery of scrap lead based acid batteries. Primary production of Lead involves extracting lead from the smelting of lead sulphide. Secondary production of Lead involves extracting lead from waste lead-acid batteries. (Sohn and Olivas-Martinez, 2014)

The Secondary Lead slag is produced mainly from expired scrap lead batteries during the transformation of lead sulphate and oxides of lead (PbO and PbO_2) into the metallic Pb in a rotary furnace. The flux used in extracting lead is Calcium carbonate ($CaCO_3$), and the reducing agents used are iron scraps and coal char. To compensate different impurities like iron, the sand is used, and CaO–FeO–SiO_2 is formed. This formation of matrix makes this type of slag different.It can be a ternary compound $CaO.(FeO)_2.(SiO_2)_2$ with respect to phase diagram, or can be a pseudo-binary compound, $2CaO.SiO_2$–FeO, as a result, has different properties such as magnetic, hardness as of rock, and resistance to acid and base.

As the oxides present in the lead slag are similar to those in the Portland cement, therefore the adoption of lead slag in the construction practices is very appealing. Also, the slag is also known for the substitution of aggregates. Use of slags in the aggregate form will be helpful where the excellent quality aggregates are not readily available.

The mechanism of hydration reaction between pozzolana and Portland cement is more complex than that of Portland cement. Typically, the rate of development of strength in OPC is faster than that of pozzolana. The chemistry in the hydration mechanism of Portland cement is due to the main compound, which is about 50% of the Portland cement and is composed of the primary mineral, tri-calcium silicate. This compound on hydration generates calcium silicate hydrate (C – S – H) and calcium hydroxide ($Ca(OH)_2$).

Now, in pozzolana, it can be represented as silica because the principal constituent in the pozzolana is non-crystalline silica glass. So, in the hydration reaction of pozzolana and the cement, the silica combines with the calcium hydroxide released on the hydration process of ordinary Portland cement. The Calcium hydroxide alone does not do anything for the strength; instead, it reacts with the reactive silica. Slowly and gradually, it leads to the formation of calcium silicate hydrate gel (C–S–H) which is a cementitious compound.

In the presence of water, the reaction mechanism between OPC, Pozzolana, and OPC - Pozzolana are below -

Hydration reaction of Ordinary Portland Cement -

$$\mathrm{OPC}\ (C_3S/C_2S) + H_2O = C - S - H + Ca\,(OH)_2 \qquad \text{Eqn (1)}$$

Hydration reaction of Pozzolana -

$$\mathrm{Pozzolana}\ (C_2AS) + H_2O = C - S - H + SiO_2 \qquad \text{Eqn (2)}$$

Hydration reaction of (Ordinary Portland Cement + Pozzolana) -

$$SiO_2 + Ca\,(OH)_2 + H_2O = (C - S - H)_4 \qquad \text{Eqn (3)}$$

The above equations depict a slow process of hydration when mineral admixture is used because the pozzolanic reactions occur only after the hydration reaction of cement. Another reason for the slow hydration process of Pozzolana is due to the silica and alumina, which form an impermeable coating around the particle of the pozzolana in an early stage of the hydration process.

The calcium silicate hydrate (C-S-H) gel produced is an amorphous material containing 60% of the paste volume. This gel forms a continuous layer that binds the particles of cement together and creates a strong and cohesive structure. The C-S-H gel is main compound of cement hydration structure that fills up the void and gives impermeability, leading to good strength and durability with pozzolana material.

On the other hand, calcium hydroxide also contributes to the development of strength by reducing the volume of pores, but the degree of strength development is more diminutive. The calcium hydroxide has a detrimental effect on durability because of its more soluble nature as compared to (C–S–H) gel. From the above equations, adding admixtures as partial replacement to cement provides strength by utilizing the weaker C-H, thereby creating more potent (C–S–H) gel. This removal of C-H from hydration cement system also improves durability, thus leading to both strength development and chemical resistance.

1.4 Geopolymer Terminology

Joseph Davidovits was the french scientist famous for his invention of geopolymer chemistry technology. Geopolymer is basically inorganic in nature and is typically ceramic. Geopolymer contains an extended range of Alumino-silicates networks held by a covalent bond. Geopolymer is amorphous (non-crystalline) in nature.

T.F. Yen *et al.* 2006 classified the geopolymer into two parts: organic containing and inorganic containing geopolymers. He defined geopolymer a chemical compound or a mixture of different compounds containing repeating chains and networks like -Si-O-Si-O- (Silico Oxide), -Si-O-Al-O- (Silico Aluminate), and -Fe-O-Si-O-Al-O- (Ferro-silico-aluminate) which are mainly formed by the reaction mechanism of geopolymerization.

Feng *et al.* 2012 defined that geopolymer is a structure formed by the condensation of the Silico Aluminate tetrahedral units with the alkali metal ions, which balance the charge on the aluminium in the tetrahedral units. Geopolymers are generally produced by a mixture that contains an alkaline solution and a solid aluminosilicate material. The process of geopolymerization then occurs at ambient temperature or at a slight high temperature. After that, the leaching of the solid aluminosilicate material in the alkaline solution occurs and leads to the migration of the leached compound from the surface of the solid into a developing gel phase. This mechanism is followed by nucleation and condensation of the gel phase, which leads to the formation of the solid binder.

The various by-products (waste) like slags, fly ash, RHA, silica fume, and meta-kaolin can be used to produce the source of the silica and alumina, can be used and is dissolved in AAS, and polymerization takes place and leads to the production of molecular chains and large networks of units ultimately leading to the strong and hard binder.

The secondary lead slag may also be used in the generation of the geopolymer as it can be a source of silica and alumina. The process of geopolymerization can stabilize or immobilize the admixtures like lead slag effectively, and thereby there can be the use of a large quantity of lead slag as a construction material. The only problem is with the alkaline activating solution, which is mainly sodium hydroxide and sodium silicate, and these resources are limited, and their cost is comparatively high. Also, there can be a negative impact of the activating solution on the environment and thus affect its popularity to some extent (D. Pan. *et al*).

The mechanism of geopolymer includes the formation of binder by the process of the polymerization reaction, which generally occurs at high temperatures. The water in the geopolymers does not affect the polymerization mechanism; instead, it only provides essential workability. Whereas in mortar/concrete, the hydration occurs with the reaction of alkalis with the water, and the hydration products formed are Calcium silicate and calcium hydroxide. With time, the calcium hydroxide evaporates and leads to the formation of the voids in the structure. These voids ultimately lead to the percolation of external ions and may lead to deterioration. But in the case of the geopolymers, comparatively fewer voids are formed due to the geopolymerization process, which provides good mechanical properties compared to concrete/mortar. And also improves durability.

1.5 Aims and objectives of the study

The primary aim of the study are as follows:

1. To conduct an extensive study on the feasibility analysis of secondary lead slag as a construction material.

2. To reduce the negative impact of secondary lead slag on environment and promoting harmless utilization of secondary lead slag as a construction material.

The primary objectives to achieve above aim are as follows:

1. To study different mechanical properties of secondary lead slag based cement mortar specimens with varying percentages (i.e., 0%, 5%, 10%, 15%, 20%, 25%, and 30%) of secondary lead slag.

2. To study the mechanical property of secondary lead slag based geopolymer mortar specimens with varying percentages (i.e., 0%, 10%, 20%, and 30%) of secondary lead slag.
3. To study the feasibility of secondary lead slag in construction applications by finding its strength behaviour with age and gaining insight on the elements responsible for the same.

1.6 Layout of Thesis

Chapter 1 provides an introduction to the analysis of the research work. The significance of the research, aims and objective of the investigation and the layout of the thesis.

Chapter 2 provides a literature review which provides in depth knowledge of the existing field of research.

Chapter 3 describes the materials and methods

Chapter 4 summarizes the results and discussion on the experimental investigation

Chapter 5 presents detailed summary of the work, conclusions and scope for further research.

The references are presented at the end of the thesis.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

The use of pozzolana material is gaining significant importance in the cement industry due to its beneficial effect on cement property. In this chapter, a literature review related to lead slag and strength-durability behaviour with mortar and concrete is carried. A detailed literature review of Lead slag generation, it's different characteristics, harmful effects on environment, harmless utilization as a construction material is presented, and knowledge gap and summary is identified. And at the end of the chapter, the research methodology is established.

2.2 Lead Slag Generation

Pb is non-ferrous, soft, toxic, heavy chemical metal denser than other common metals and has enormous applications in acid batteries, manufacturing of machines, and medical purposes. It is used wisely because of its softness, heaviness and malleability. Also, lead is a bad conductor of heat. The most important use of lead in the industry involves its usage as a constituent of alloys. Lead is not abundantly free in the nature rather it occurs as a cubic sulphide. Galena (PbS) is the most common ore of lead. Figure 2.1 shows the lead production from year 1840 to 2020.

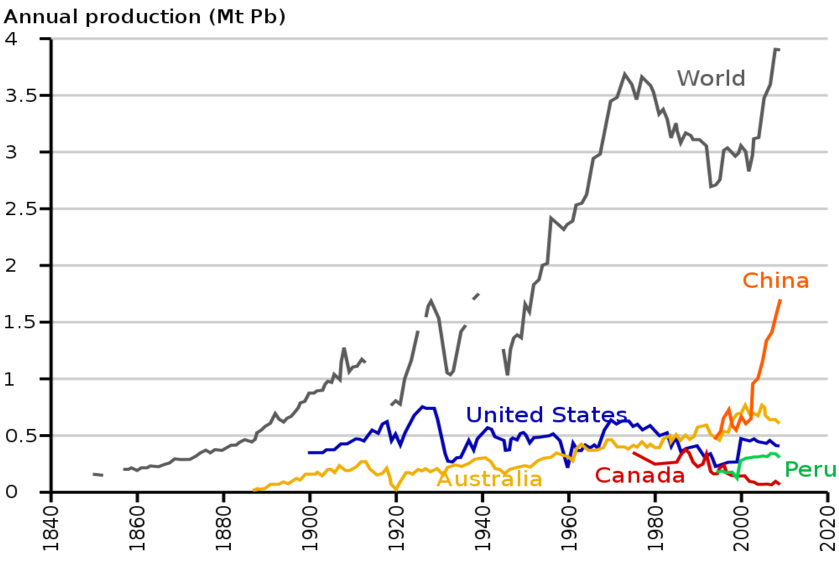


Figure 2.1 Lead Production from1840 to 2020 (Mudd G. M. 2013)

The lead slag is formed during lead production, which is typically a waste (derivative). The lead production includes two ways: Primary and secondary resources. The primary resources include the mined ores, and secondary resources include the scrap, mainly from lead-acid waste batteries. The main raw material in the primary resource is Galena (PbS) and in the secondary resource is waste lead-acid batteries. (Chen *et al.* 2009 and Ettler *et al.* 2014).

The different countries, the production of lead mainly involves recovery of secondary resources. In U.S., about 80% of Pb produce from secondary resources. And in Europe, 90% of the Pb produce from secondary resources. In countries like China, it is 42% of the total Pb production. The lead producing from secondary resources worldwide is about 60-66% (Zhang *et al.* 2016A, B). Primary lead recycling process considers smelting of lead concentrate. A total of 1 tonne production of Pb discharge about 7 tonne of Primary Lead Slag (Hou, 2011). And in recycling process of secondary Pb, 1 tonne of lead discharges 110–330 kg of secondary lead slag (Kreusch *et al*. 2007). Figure 2.2 shows the global lead and lead slag production, according to the Bureau of Metal Statistics, which is 11 million tonnes and 5.5 million tonnes, respectively (D. Pan, et al).

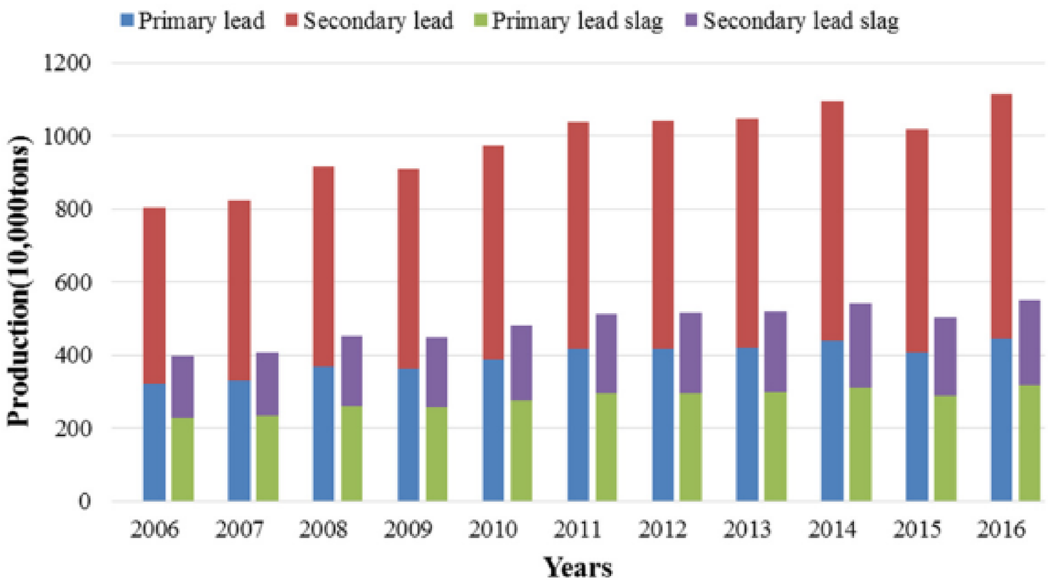


Figure 2.2 Lead and Lead Slag production from in the world (2006 to 2016) (D. Pan. *et al*)

The production of Lead slag from primary resources is from the process of smelting of PbS. The smelting process is done by two basic general processes. The first process involves sinister plant- blast furnace and the second process involves direct smelting reduction. In these two processes various reactions take place such as oxidation, reduction and refining of different compounds. Figure 2.3 shows the flow chart of Lead slag from primary resources from sinister plant- blast furnace (Sohn, 2014).

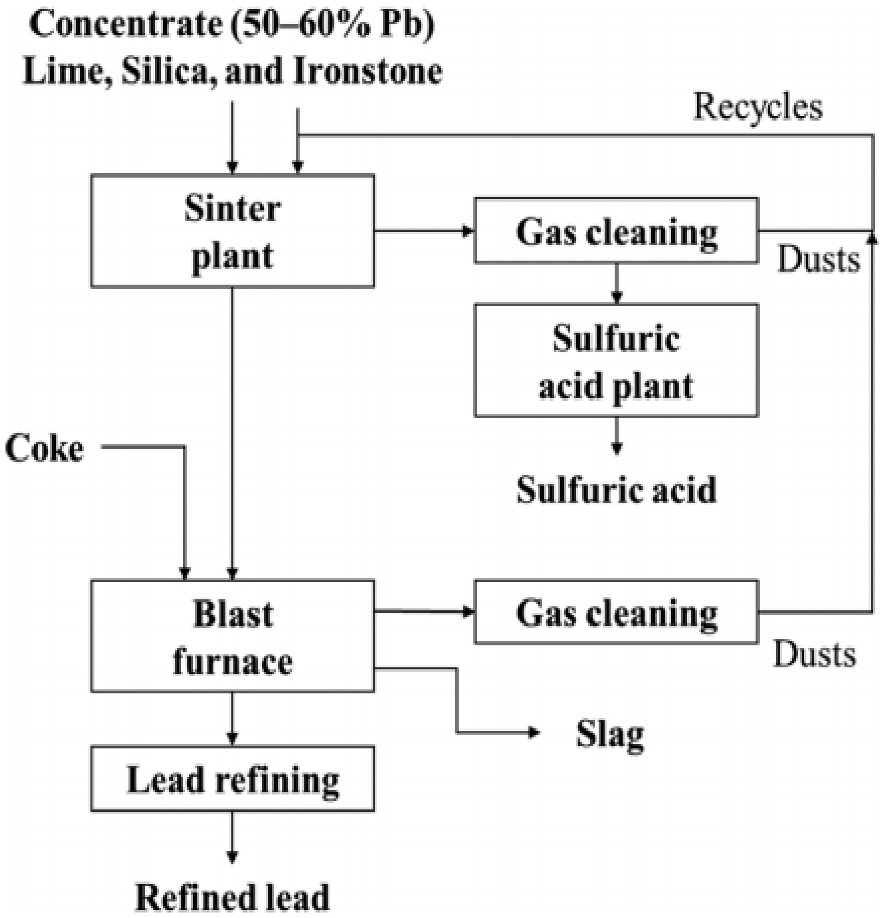


Figure 2.3 Flow chart of primary production of lead by sinister plant-blast furnace (Sohn, 2014)

The process of oxidation of Lead Sulphide takes place in a sinister blast furnace in the solid state, and sulphur gets removed, and the production of lead oxide takes place simultaneously. Then, lead oxide reduction occurs, and Pb is formed in a blast furnace. The temperature in blast furnace is decreased by adding flux, which contains lime, ironstone, and silica, and slag is formed in a molten form where the oxides of iron are removed.

In the other process of primary lead slag generation by direct smelting reduction, the reduction and oxidation reactions occur simultaneously. In this process, the lead and sulphides of the metal are left behind in the melted slag (Sobanska *et al.* 2016). At last, removal of slag occurs and is treated with water, and lead slag is formed. The lead slag produced by smelting reduction is the CaO-FeO-SiO_2 system. (Sohn, 2014)

Lead slag production from recycling of secondary lead is from the process of smelting which involves two methods: direct and indirect smelting (pyro-metallurgical process). (Sun *et al*. 2017). Main recycling source is from lead based acid scrap batteries, including 80% of the total secondary lead slag. (Smaniotto *et al.* 2009). In this method of recovery of secondary Pb, the reduction of $PbSO_4$ and lead oxides takes place and gets reduced to Pb. In smelting method, various reducing agents such as iron filling and coke are used and flux such as calcium carbonate is used to maintain the melting temperature are added. The temperature of paste of lead in the direct smelting process in scrap batteries is almost over 1000 °C. The eco-friendly efficiency of indirect process smelting is good because most of the sulfur is removed before the smelting. (Li *et al.* 2017a, b, and c). Figure 2.4 shows the flow chart of indirect smelting (Ellis, 2010).

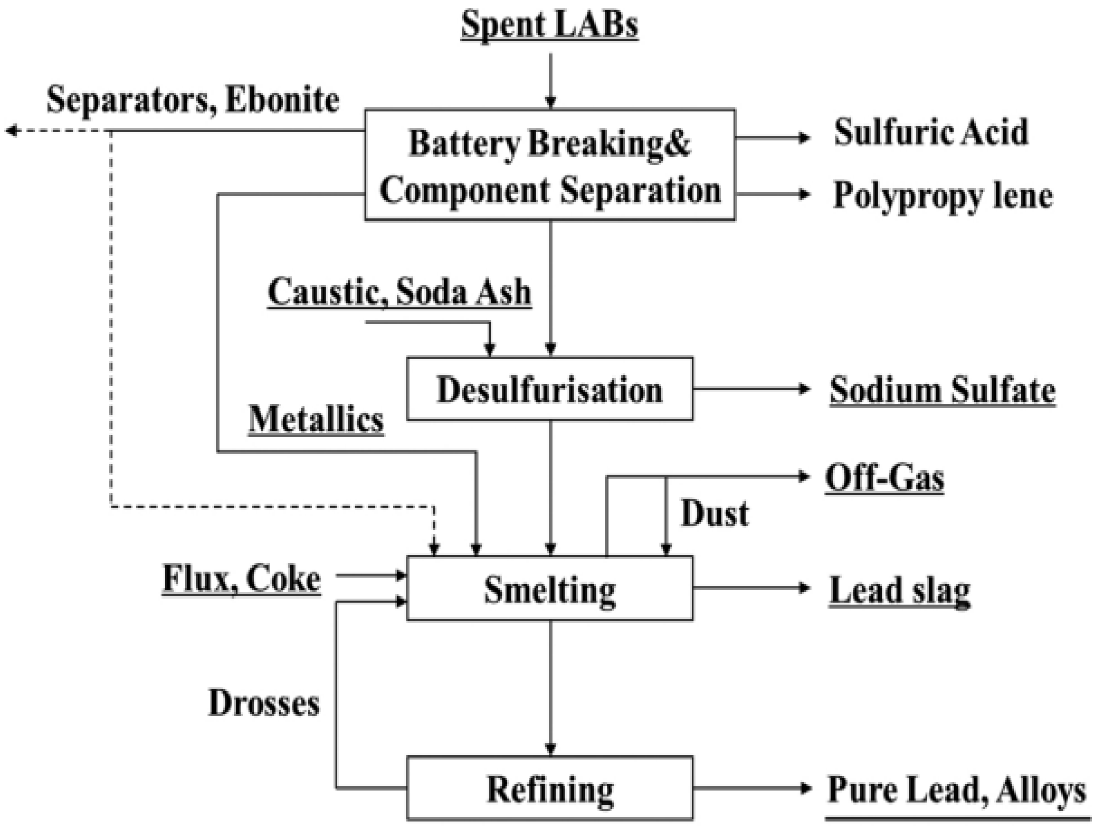


Figure 2.4 Flow Chart of secondary lead recycling by indirect smelting (Ellis, 2010).

Removing the sulfur is first step in refining Pb production done by hydro metallurgy which is an eco-friendly process. In this process, lead sulfate gets transformed into hydroxides (Morachevskii *et al.* 2001).

Solubility of lead carbonate and lead hydroxide is less as compared to lead sulfate. Due to their insoluble nature, they are collected as sludge and finally transferred to the smelter.

The following reaction concludes the less solubility of $PbCO_3$ and $Pb(OH)_2$ than with PbSO4.

$PbSO_4 + 2NaOH = Pb\ (OH)_2 + Na_2SO_4$ Eqn. (4)

$PbSO_4 + Na_2CO_3 = PbCO_3 + Na_2SO_4$ Eqn, (5)

At last, the lead is finally recovered with the reduction with iron. The following equations depict the formation of the Pb (Gomes *et al.* 2011).

$PbCO_3 = PbO + CO_2$ Eqn (6)

$2PbO + C = 2Pb + CO_2$ Eqn (7)

$PbO_2 + C = Pb + CO_2$ Eqn (8)

$PbSO_4 + 2C = PbS + 2CO_2$ Eqn (9)

$PbS + Fe = Pb + FeS$ Eqn (10)

When iron is added, it leads to the formation of impure metals like nickel, zinc, and copper. The copper and iron act as a sulfide. Lastly, the sand is added to minimize the non-necessary compounds such as Fe ion in calcium silicate matrix, which leading in formation of calcium–iron–silicate-based slag (Penpolcharoen, 2005).

2.3 Physical Characteristics of Lead Slag

The physical characteristics of primary and secondary lead slag are someway similar. The texture of both the slag is glassy and have black in colour. The highest content in the lead slag is of iron oxide (Fe_20_3). The lead slag density is between 3.5 to 3.8 g/cm^3. The particles size is between 100 microns to 4mm. (Barna *et al.* 2004). Specific gravity is between the range of 2.65 to 3.76 g/cm^3. Thus the conclusion can be made that lead slag has a similar density as of natural aggregates. (Penpolcharoen, 2005).

2.4 Chemical Characteristics of Lead Slag

Chemical characteristics of the lead slag generally depend on ores considered in the manufacturing of lead, the type of flux used to decrease the melting temperature, and the impurities present in iron and coke. If we talk about the normal range concerning different studies, the composition of primary slag includes 3.36% - 31.57% of ferric oxide, 9.49% – 28.90% of iron oxide, 4.50% - 23.05% of calcium oxide, 14.68% – 35.50 % of silicon dioxide, 2.46% - 6.2% of aluminium oxide, 0.15 % –5.4% of magnesium oxide, 2.8% – 11.1% of zinc oxide, 1.2% - 12.3% of lead oxide, 0.1% - 2.8% of copper oxide and 0.24% - 8.01% of sulphur content.

Secondly, the composition of secondary lead slag includes 20% – 55% Fe, 1.3% – 22% Ca, 3% – 21% Si, 0.75% - 16.3% Al, 0.26% - 2.5% Mg, 0.2% - 1% Zn, 1.14% – 22% Pb, 0.2 – 1.3% Cu, and 0.5%–21% S. (Shoaib *et al*. 2001)

2.5 Effects of Lead Slag on environment

The lead slag is a derivative formed in lead manufacturing process, which is mainly disposed off. The disposing of lead slag is basically done in landfills and stockpiling. The two methods consequently required a large amount of land and thereby leading in lot of environmental problems also. There are many key parameters affecting the environmental stability of lead slag, which are: pH change, interaction time of lead slag with water, and different mineral phases of lead slag. (Yin *et al.,* 2016). In an acidic environment, the lead slag has the ability to release zinc, iron, and calcium, and its weathering with water occurs quickly. The interaction with oxygen in open-air also increases the chance of oxidative weathering, which forms oxides and carbonate, which have higher chances of releasing the toxic elements. As the leaching time of the lead slag increases, the content of different poisonous elements in the lead slag also increases.

With time, these toxic elements like zinc and lead penetrate in the soil through the action of weathering and rainfall. This results in the pollution of the soil in the landfill, if there are no proper measures of anti-seepage mechanism in the soil then it leads in serious environmental problems. This is a serious problem because these toxic elements are not decomposed by bacteria's leading in migration to the plants bodies and consequently, affect the growth of plant and animals. With time, following food chain, these hazardous compounds also affect the health of humans by increasing the amount of lead content in human blood. (Tukker *et al.* 2006, and Hilts, 2003).

Also another severe problem can be due to entering of the toxic elements of lead slag in surface water and groundwater through the surface and sub-surface runoff leading to health problems for the local people because of the impurities in the drinking water.

De Andrade et al. examined the chemical content of Pb, Zn, Cu, and Cd from water near lead slag landfill site. The values of lead was 0.15 mg/l, zinc was 0.06 mg/l, copper was 0.05 mg/l, and cadmium was 0.002 mg/l (Lima and Bernardez, 2013). This can also pollute the atmosphere and lead to air pollution as size of the slag particles is tiny and can easily become dust in open conditions, leading to the release of toxic elements in the air. This can also cause serious health issues in humans (Da Silva *et al.* 2017).

2.6 Lead Slag as Construction Material

2.6.1 Use of Lead Slag as Construction Material in mortar/concrete

Different researchers have investigated the use of primary and secondary lead slag in cement mortar/concrete to substitute aggregates or OPC. Also, some researchers investigated the effects of the lead slag on different mechanical properties of the concrete and the mortar.

Seyed Hossein *et al*. 2011 examined effect of Lead Slag as a substitution of sand (F.A.) in different mechanical properties of concrete. Different tests like compressive strength, flexural strength, and water absorption are examined on 14, 28, and 56 days. Cement used was PPC (Pozzolanic Portland Cement), confirming ASTM standards. The lead slag used was the by-product of the lead blast furnace. Table 2.1 shows the chemical composition of PPC and Lead slag used.

Table 2.1 Chemical composition of different materials (Seyed Hossein *et al*. 2011)

Constituent (%)	Pozzolanic Portland Cement	Lead Slag	Constituent (%)	Pozzolanic Portland Cement	Lead Slag
SiO_2	26.85	12.5	**SO_3**	0.8	-
Al_2O_3	6.5	-	**Cl**	4	-
Fe_2O_3	3.2	-	**Pb**	-	3
CaO	56.5	19	**Zinc**	-	11
MgO	0.4	2	**Iron**	-	22.5
K_2O	1.1	-	**As**	-	0.55
Na_2O	0.44	-	**Chromium**	-	0.3

The replacement of sand with respect to lead slag was 30%. To study the effect in substitution of slag, they casted a total of 40 concrete specimens. Table 2.2 shows the mix design of concrete mixtures.

Table 2.2 Mixture of Cement Concrete (Seyed Hossein *et al*. 2011)

Mixture	Type of mix	Cement (kg/m³)	Lead Slag (kg/m³)	Sand (kg/m³)	Coarse aggregate (kg/m³)	water/ cement %
Std. Mix	100 % Sand	400	0	768	1152	0.4
Lead Slag	70 % Sand + 30% L.S.	230.4	230.4	537.6	1152	0.4

The cubical mould of 100 mm x 100mm x 100mm was considered to determine the compressive strength. For each mix, eight specimens were considered. The testing was done on three different days, and two samples each were chosen and tested. For determining the transverse strength, 1 cm x 1 cm x 5 cm specimens were casted. For each type of mix, four beam samples were considered and were tested for transverse strength at 28 and 56 days.

The compressive strength was demonstrated as per BS 1881: part 116. The rate of loading in testing was 2.5 kN/sec. The transverse strength was demonstrated as per ASTM C78-94. The three point loading test was performed, and the rate of loading kept was 0.2 kN/sec.

The result concluded that, the lead slag does not significantly affect the rate of compressive strength at 30% substitution of sand. But strength increase with age in all concrete mixtures. Also, the presence of lead slag for examining the flexural strength, the results concluded the decrease in flexural strength of concrete specimens. The flexural strength is the highest of the control mix only. Thus conclusion can be drawn that lead slag is not appropriate for providing strength in the case of flexural members. Also, the water absorption of the specimens decreases with increase of replacement % of lead slag but dry density increases.

G.De Angelis *et al.* 2002 analyzed the possibility of the lead slag from recycling of the waste lead based batteries in the cement matrix and also analyzed the possibility of using the lead slag as a filler material as a substitution for sand in mortar. This analysis helps stabilizing the disposal of waste and also producing a recycling material in order to get environmental and economic benefits. As a result, raw slags have been characterized thoroughly, and various lead slag based mortar specimens are casted with varying & of lead slag have been investigated. Study focuses on feasibility analysis of the lead slag and the 42.5 Grade PPC Cement. Table 2.3 shows the typical composition of the lead slag sample used in the research.

Table 2.3 Composition of Lead Slag (G.De Angelis *et al.* 2002)

Components	Percentage (By weight)	Components	Percentage (By weight)	Components	Percentage (By weight)
Fe (Iron)	40.8	**As**	0.10	**Ca**	1.3
Pb (Lead)	16.7	**Mn**	0.25	**Sr**	0.01
Si	3	**Ni**	0.11	**Ba**	0.13
Zn	1	**Cr**	0.07	**Cl**	0.72
Sn	0.31	**Cd**	0.02	S_{total}	11.2
Al	0.75	**Na**	0.37	$S_{Sulphide}$	10.0
Sb	0.27	**K**	0.09	$S_{Sulphate}$	0.85
Cu	1.2	**Mg**	0.26	$S_{Sulphur}$	0.33

Different mixtures are established consisting of sand or without sand by varying lead slag percentages. Table 2.4 shows the cement-slag ratio considered in the research, which is characterized as 3/1, 2/1, 1/1, 1/2, and Control mix. The replacement of cement is done with respect to lead slag.

Table 2.4 Mixture of Cement Paste with varying amount of Lead Slag (G.De Angelis *et al.* 2002)

Parameters	Control Mix	3:1	2:1	1:1	1:2
Water used (g)	340	480	400	320	200
Portland Cement (PC) (g)	850	1200	1000	800	500
Lead Slag (g)	-	400	500	1000	1000
W / c ratio	0.4				
Lead Slag (%)	-	19.2	26.3	41.7	58.8

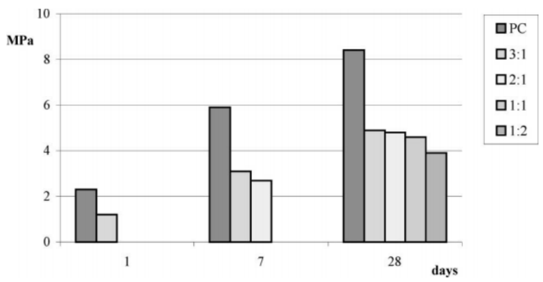
Table 2.5 shows the replacement level of lead slag with respect to sand as a filler. The characterization is done based on the cement-slag ratio, which is A, B, C, and D. Also, the ratio of cement and (sand + lead slag) is kept same throughout which is 1/3.

Table 2.5 Mixture of Cement Mortar with varying amount of Lead Slag (G.De Angelis *et al.* 2002)

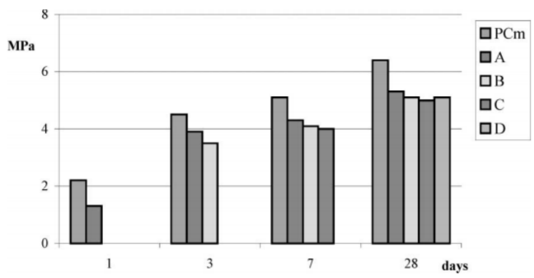
Parameters	Control Mix	Type A	Type B	Type C	Type D
Water used (g)	72.6	72.6	72.6	72.6	72.6
Portland Cement (g)	121	121	121	121	121
Lead Slag (g)	-	25	50	94.5	165
Sand (g)	363	337.9	312.3	268.4	196
W / c ratio	0.60				
Lead Slag (%)	-	4.5	9.1	17	30
Cement/(Lead Slag + Cement)	1/3	1/3	1/3	1/3	1/3

Different mechanical tests like compressive, flexural, and tensile properties were examined at multiple curing ages of one, three, seven, and 28 days. To determine flexural strength, 40mm x 40mm x 160mm prism specimens were casted. The broken samples by the flexural test has been used in determining compressive strength. Flexural strength Is demonstrated as per UNI 6133-72; the compressive strength Is demonstrated as per UNI 6134-72, and the flexural strength was demonstrated as per UNI 6135-72.

The result concluded that in the case of all the three mechanical strengths, the values decrease with the increase of replacement of lead slag with cement or sand. Figure 2.5, 2.6, and 2.7 shows the results of different mechanical properties.

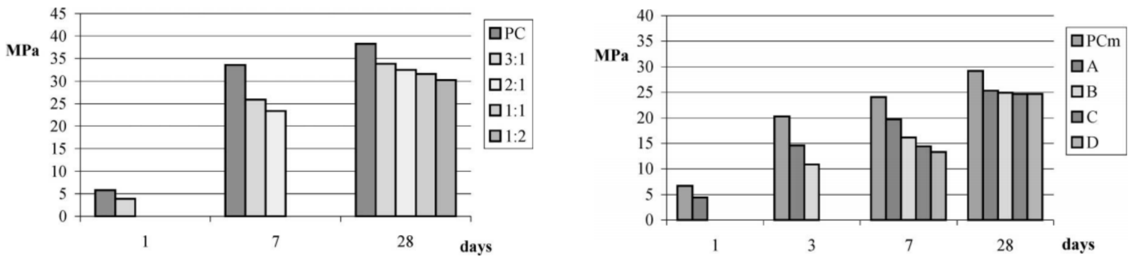


(a)



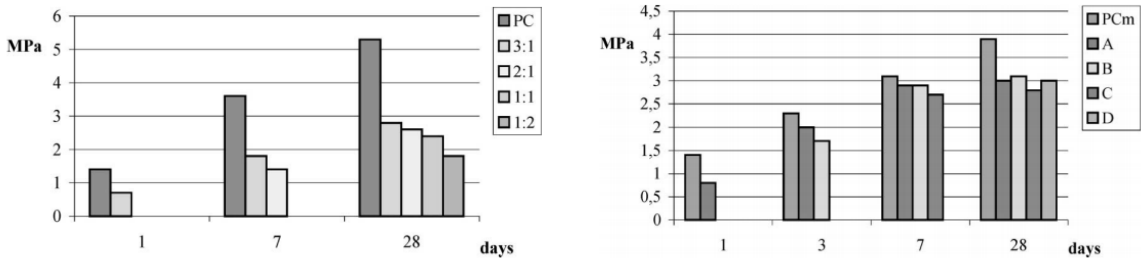
(b)

Figure 2.5 Flexural Strength of (a). Lead Slag-Cement Specimen (b) Lead Slag-Mortar Specimen (G.De Angelis *et al.* 2002)



(a) **(b)**

Figure 2.6 Compressive Strength of (a) Lead Slag-Cement Specimen (b) Lead Slag-Mortar Specimen (G.De Angelis *et al.* 2002)



(a) **(b)**

Figure 2.7 Tensile Strength of (a) Lead Slag-Cement Specimen (b) Lead Slag-Mortar Specimen (G.De Angelis *et al.* 2002)

A leaching assessment was also done on lead slag before and after cementation on type B mixture and 1:1-type mixture. The leaching was done with the deionized water under continuous stirring conditions for 6 h and 24 h. The leaching procedure results are compiled on Figure 2.8. The Pb is leached in great amount, more in cement specimens. In leaching procedure for 6 hours, more Pb was extracted.

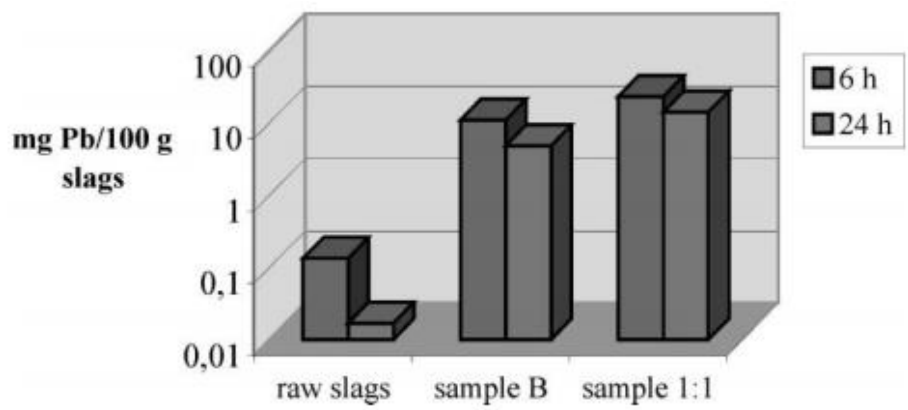


Figure 2.8 Leaching of Pb from Lead Slag before and after cementation (G.De Angelis *et al.* 2002)

Saikia *et al*. 2008 investigated the lead slag and used it to replace the filler aggregates (fine) in the cement mortar- Lead Slag specimens. The study of chemical and mineralogical characteristics of the Lead slag with cement was also demonstrated. The lime reactivity and the hydration chemistry of the cement and the lead slag was demonstrated. Various properties like compressive strength of mortar samples are monitored in order to determine mechanical behavior of the mortar samples containing partial replacement of Lead slag with the fine aggregates. Lastly, the leaching assessment procedures were analyzed by three different procedures.

The Lead Slag used in the research is metallurgical slag which is a black color fine material, a derivative of the Blast Furnace. Residue from the furnace is being cooled down and is broken into a fine derivative. Size of particles used in research was between 0 mm to 4 mm. The typical composition of Lead Slag is shown in Table 2.6.

Table 2.6 Composition of Lead Slag (Saikia *et al*. 2008)

Component (Oxides)	Percentage (% by dry basis)	Component (Oxides)	Percentage (% by dry basis)	Components (Elements) (mg/kg)	Percentage (% by dry basis)
Al_20_3	5.41	PbO	2.47	As	4200
BaO	0.84	SiO_2	25.7	Cd	5.81
CaO	18.9	SnO_2	0.503	Cr	1001
CuO	0.42	ZnO	5.01	Mo	279
Fe_2O_3	33.95	PO_4^{3-}	1.19	Ni	416
K_20	0.308	SO_4^{2-}	1.17	Sb	3077
MgO	1.41	As, Sb	1.19	Se	292
MnO_2	0.656	L.O.D.	2.50	V	805
Na_2O	0.872	L.O.I.	3.95		

Different mortar samples were casted for examining compressive behaviour having varying lead slag percentages as a partial substitution of fine aggregate. Hobart mixing machine was used, and the cement to fine aggregate ratio is kept 1/2. A mixture containing 25% Lead Slag and 5% fine aggregates was prepared. And a control mix containing 100% of the natural aggregate was casted. The consistency of all the mortar specimens was kept constant. The w/c ratio were kept as 0.50, 0.48, 0.58, and 0.60. 4cm x 4cm x 16cm prism specimens were casted to determine the mechanical properties. The vibration was done to release the volume captured by the entrapped air. The compressive strength was demonstrated after three curing ages of 3, 7, and 28 days. The prism samples were broken into the cubical specimen of 40 mm cubes in order to get compressive strength. A total of 3 samples were casted for different mixtures. The average of the three values was considered.

The Leaching behavior of the Lead slag-based cement mortar specimens was done to examine the compatibility with the environment so to find the suitability of the lead slag in construction in mortar or concrete. The leaching assessment of the mortar specimens was examined by three standards. First was the European standard test EN 12457-2, in which a powder of the broken samples was used. The second method adopted was the Leaching test by diffusion, and the last test was NEN 7345, in which the cube of 40 mm was used.

The result concluded that Lead Slag could be utilized in the mortar or concrete as a 25% of substitution of the fine natural aggregates. Compounds formed after the hydration of the lead slag do not involve any of the hazardous compounds that could affect the properties of the mortar or concrete. Also, the high content of Zinc and Lead Oxide does not alter the hydration process. The compressive strength of the specimens that contained 25% of Lead Slag are comparable with the control mix specimens containing fine aggregates only.

Saikia *et al.* 2012 studied technical aspects of lead slag from blast furnace as a replacement with cement. This study was the further research of his previous research Saikia et al. (2008). Various properties of cement based mortar were demonstrated.

The Lead Slag used in the research is metallurgical slag which is a black color fine material, a derivative of Blast Furnace. The residue from furnace is being cooled down and is broken into a finer crushed sample. The size of the particles used in the study is between 0 mm to 4 mm. The typical composition of Lead Slag is shown in Table 2.7.

Table 2.7 Composition of Lead Slag (Saikia *et al.* 2012)

Oxides	**Amount**	**Oxides**	**Amount**	**Oxides**	**Amount**
Al_2O_3	5.4	SiO_2	25.7	MgO	1.4
BaO	0.84	SnO_2	0.50	MnO_2	0.66
CaO	18.9	PO_4^{3-}	1.2	Na_2O	0.87
Fe_2O_3	34.0	SO_4^{2-}	1.2	LOD	2.5
K_2O	0.31	Others	1.2	LOI	4.0

The mortar preparations includes selection of water-cement ratio, that changes with water content of Lead Slag and thus results in the flow-ability of the mortar mix. Specific gravity was another point of concern because when lead slag is used as substitution to cement, this led to segregation after the compaction. Thus the 100% replacement of Lead slag was not possible. A previous study concludes that up to 35% of the replacement of the lead slag with sand provides good strength results.

Table 2.8 shows the mortar composition, which contains a control mix with no replacement and a 25% and 35% replacement of lead slag with sand. The ratio of cement to sand was considered 1:3 considering EN 196-1. The water to cement ratio was taken constant. The flow-ability of mortar mix was also constant(163mm). The procedure of the flow table test was followed from 1015-3:1998. The Vicat apparatus was followed to determine the setting times from EN 196-3:1987.

Table 2.8 Composition of Mortar Mix (Saikia *et al.* 2012)

Parameters	**Control Mix**	**At Constant water-cement ratio**		**At Constant Flow**	
		25% Lead Slag	**35% Lead Slag**	**25% Lead Slag**	**35% Lead Slag**
Cement (g)	450				
Water(ml)	225	220	217	207	198
Standard Sand (g)	1350	1012.5	877.5	1012.5	877.5
Lead Slag (g)	0	337.5	472.5	337.5	472.5
Water/Cement	0.50	0.49	0.48	0.46	0.44

For determining mechanical properties, mortar prism of dimensions 40mm x 40mm x 160mm were casted according to EN 196-1. The specimens were cured up to five different stages of 3, 7, 28, 60, and 90 days. Compressive and flexural strength were determined in accordance to mechanical properties. Also, the M.0.E (E) was found at different ages of specimens by using U.P.V. Water absorption of specimens was demonstrated in accordance with ASTM C642. The prism samples were broken into the cubical specimen of 40 mm cubes in order to get water absorption of 28 days curing age. The solution used in determining water absorption was lime water. Weights of specimens were taken after 24 hours of dipping was specimens in the lime water. Total of 3 samples were prepared for different mixtures. The average of the three values was considered for each composition of mortar mix.

Figure 2.9 shows the graph of variation in content of lead slag used and flow at a constant w/c ratio. The addition of the lead slag increased the flowability of the mortar specimens. Inclusion of the lead slag, there is a reduction in mechanism of rate of hydration of cement particles leading in amount of free water in the mixes, thereby increasing the mortar mixes' flowability.

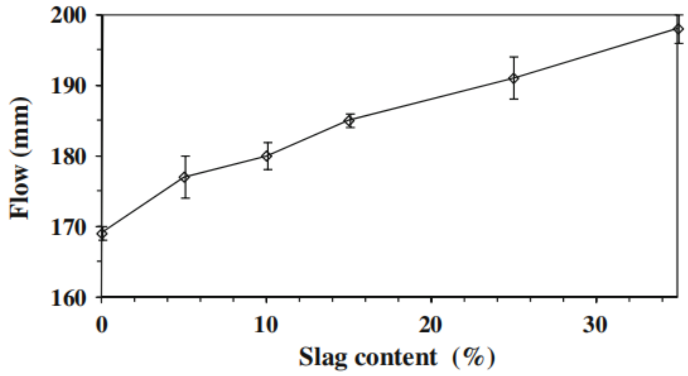


Figure 2.9 Variation of Flow and Lead Slag Content (Saikia *et al.* 2012)

Figure 2.10 shows the graph of variation in flowability and the w/c ratio. The addition of the lead slag as substitution with fine aggregate decreases a demand for water in the mixture because of the presence of large cube particles in the lead slag as compared to natural sand. So this was concluded that consistency of the mixes having 25% and 35% lead slag having a water-cement ratio of 0.46 and 0.44, are comparable with the flowability of the control mix of the water-cement ratio of 0.5

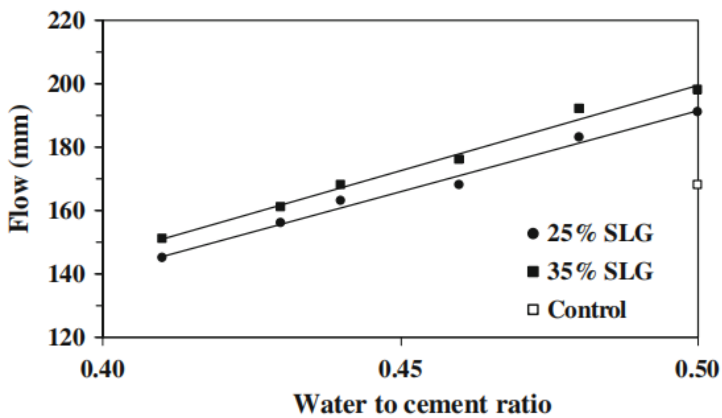


Figure 2.10 Variation of Flowability and Water-cement ratio of 25% and 35% Lead Slag specimens (Saikia *et al.* 2012)

Table 2.9 shows the initial and final setting time of different mix having 25%, 35%, and control mix. There is retardation in the setting time in the case of lead slag based samples when compared with control samples, and a further increase in the setting time as the replacement increases. This delay is due to the presence of Pb and Zn present in lead slag. High level of pH may dissolve some contents of the lead slag and also the amorphous glassy phases. So lead, and zinc can form insoluble components and lead to the formation of the layer on the dehydrated particles of cement and prevent the hydration process.

Table 2.9 Initial and Final Setting Time of Mortar mix (Saikia *et al.* 2012)

Mixtures	**I.S.T (minutes)**	**F.ST (minutes)**
Control Mix	323	405
5% Lead Slag	78	448
10% Lead Slag	394	486
15% Lead Slag	435	586
35% Lead Slag	650	856

Figure 2.11 shows the different mechanical properties of the mortar specimens having 25% and 35% lead slag and control mix at a constant w/c ratio of 0.5. Compressive, flexural strength, and MOE (*E*) of the specimens containing lead slag were less than the control specimens. Increasing the percentage of the lead slag leads to reducing the strengths. So there is a decrease in the water demand and high water absorption tendency and thereby porosity of the specimens having lead slag as compared to control specimens.

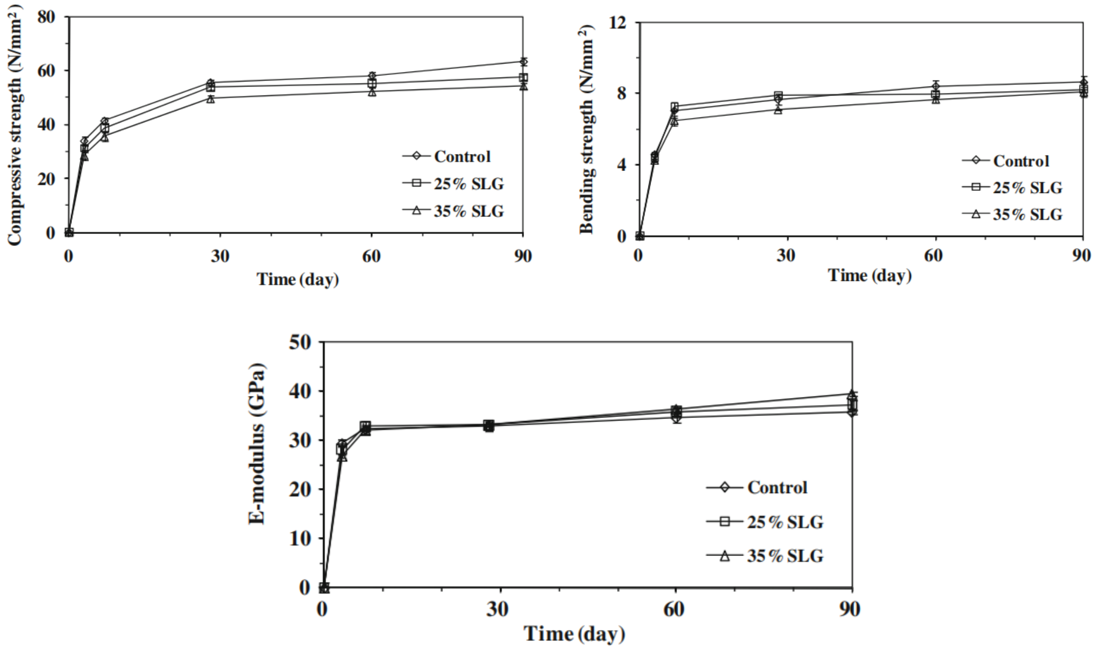


Figure 2.11 Compressive, flexural, and modulus of elasticity of mortar at w/c of 0.5 (Saikia *et al*. 2012)

Figure 2.12 depicts different mechanical properties of mortar specimens having 25% and 35% lead slag and control mix at a constant flow for up to 90 days. The compressive, flexural, and modulus of elasticity of specimens containing lead slag was greater than that of standard specimens, and strength of specimens having 35% of the lead slag was greater than specimens having 25% of the lead slag. This is because there is a decrease in w/c ratio value in specimens containing lead slag. Thus, the reduction of the water in the mortar specimens because of inclusion of the lead slag imparts strength property to specimens.

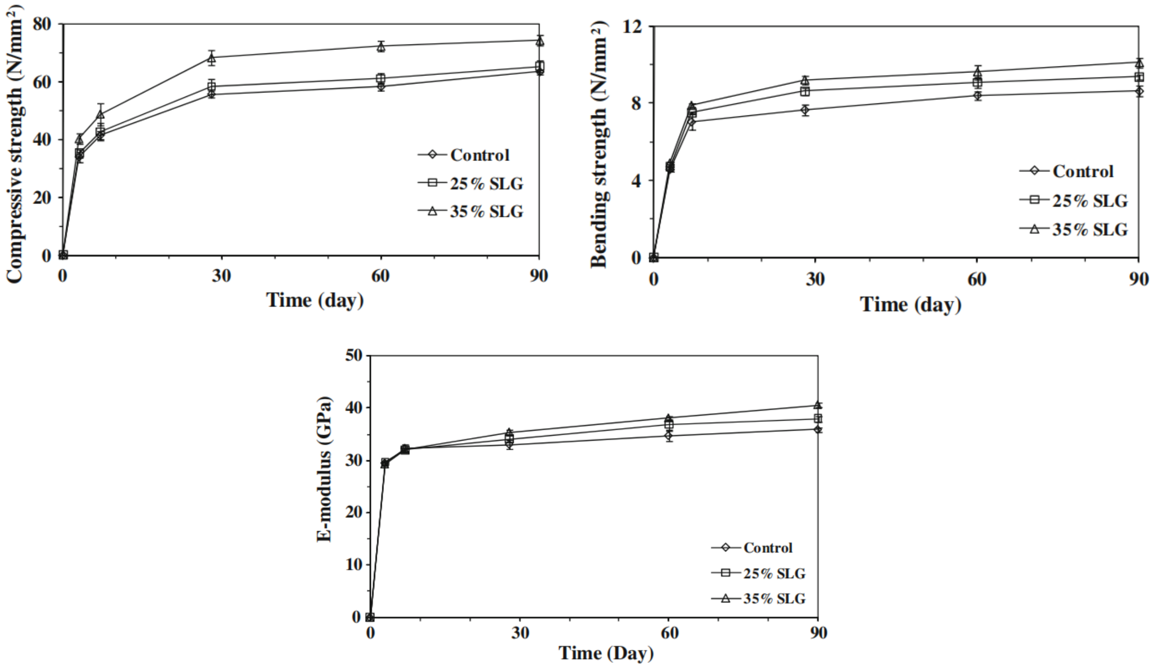


Figure 2.12 Compressive, flexural, and modulus of elasticity of mortar at constant flow (Saikia *et al.* 2012)

The result concluded that adding the slag as a substitution for the natural sand leads in increase of flowability of fresh mortar mix. The addition of lead slag in the mortar mix increases the initial and final setting time. Strengths of mortar specimens decreased at a fixed w/c ratio and increased at a fixed flow value with an increase in the content of lead slag. The reduction of water content in mortar mix because of inclusion of the lead slag helped improve strength properties.

Cioffi *et al.* 2000 investigated effect of secondary lead slag in cement stabilization, solidification process in the manufacturing of concrete specimens The effect of the secondary lead slag in the hydration mechanism has been studied by the casting different mixtures having lead slag in the powdered form.

The secondary lead recycling slag used in the research is the smelter slag produced in automotive batteries in the process of extracting lead. Lead slag is a hazardous waste produced at the rate of 10,000 tons/year in Italy. The slag contains different metals in it which are responsible for the negative impact on the hydration mechanism as the metals are responsible for early setting and hardening. The typical elemental composition of Secondary Lead Slag is shown in Table 2.10.

Table 2.10 Composition of Lead Slag (Cioffi *et al.* 2000)

Compounds	Percentage (By weight)	Compounds	Percentage (By weight)
Al_2O_3	2.17	SiO_2	6.28
Fe_2O_3	68.20	CaO	4.09
PbO	13.79	CuO	1.83

The specimens were casted by replacing the pozzolana cement with secondary lead slag at different percentages of 0, 15, 30, and 65 and were cured for the time interval between 2 hours to 14 days. The lead slag used in the mix was finer than a 500-micron sieve. The lead slag was also partially replaced with the aggregates in the casting of concrete specimens. The aggregate size was divided into three classes of fine, medium, and coarse aggregate. In this study, the lead slag was replaced with all three aggregate classes. The four different groups were made consisting of W, F, M, and C. The study aimed to study the specific surface property of lead slag. In W, the lead slag was partially replaced with aggregates. And in F, M, and C, the lead slag was partially replaced with all three classes of aggregates. Table 2.11 shows the composition of the different mixtures established in the study.

Table 2.11 Composition of Mixtures (Cioffi *et al.* 2000)

Groups	Binder (%weight)	Aggregate (%weight)	Secondary Lead Slag (%weight)
W	15	66	19
F, M and, C	15	75	10

The above table shows that the total slag content in every mix is 85% and 15% cement. To determine the mechanical properties, cylindrical and cubical specimens were casted. Cylindrical specimens of size 100mm x 150mm were casted, and cubical samples of size

150 mm were casted. According to the Italy standards, nine groups of concrete specimens were casted of strength ranging from C12/15 to C50/60. For each class of mixtures, two cubical samples were casted and cured for 28 days at 25 degrees Celcius and 100% relative humidity. The first specimen was for determining compressive strength and the second was for determining the leaching behavior

Table 2.12 Compressive Strength of Concrete Specimen (Cioffi *et al.* 2000)

Groups	Compressive Strength
W	23 MPa
F	No hardening (after 28 day curing period)
M	23.9 MPa
C	25.6 MPa

Table 2.12 shows the result of the compressive strength on 150 mm cube. Each group data was average of the eight cubes casted. No hardening after 28 day curing of F group as not absorbed. F group had the highest slag content with a high specific surface area. The compressive strength of other groups was remarkably better as compared to the F group. The cube of group C belongs to C20/25, and of group W and M was less as compared to the C group. This can subsequently increase by adding additives like soluble silicates, which can precipitate the heavy metals in the lead slag like metallic lead. The metals in the mixture lead to partial solubilization in the medium alkaline solution in cement and lead to a negative impact on the hydration mechanism. The solubilization of the lead slag depends on the surface chemistry and is high in group F, where the lead slag replaces the fine aggregates class. Thus, the specific surface area is high in this case, and hardening occurs. Also, the cement used in the mixture was 15% which is the important binder element in the production of the concrete class of C20/25. Hence in this research work, the results almost match with the conventional concrete.

The result concluded that the presence of secondary lead slag in the mix of up to 30% does not affect the hydration mechanism negatively. And also, the aim of the research work to achieve the C20/25 class of strength can be achieved by partially replacing the lead slag.

Penpolcharoen, 2005 studied the secondary lead slag from the lead smelting industry, which uses Calcium Carbonate as its flux and is investigated to use as pozzolana or aggregate replacement in concrete specimens. Research's main aim was to investigate the utilization of the secondary lead slag in the powder form to partially replace the OPC I to examine the reactivity of the slag. As the lead slag is the by-product and is dumped in the landfill, so it was also examined for the partial replacement of aggregate. The different mechanical properties were studied and a leaching procedure was performed to determine its present toxicity. The magnetic behavior of the lead slag was also studied.

The Lead Slag used in the research was a secondary type and was produced by smelting with calcium carbonate as a flux. The slag was developed into two different sizes: F and A. F denotes the fine secondary slag passing from a 200-micron sieve which was further replaced from Ordinary Portland cement, and A depends the coarse slag passing from sieve number four and was further replaced with natural aggregates. The OPC was locally processed from Thailand. The typical elemental composition of Secondary Lead Slag and ordinary Portland cement are shown in Table 2.13.

Table 2.13 Chemical Composition of Secondary Lead Slag and OPC (Penpolcharoen, 2005)

Compounds	Secondary Lead Slag (wt. %)	OPC (Wt. %)	Compounds	Secondary Lead Slag (wt. %)	OPC (Wt. %)
CaO	10.53	64.26	Na_2O	4.12	4.12
SiO_2	24.40	20.39	SO_3	6.64	6.64
Al_2O_3	3.44	5.16	TiO_2	0.19	0.19
Iron Oxide	46.81 (FeO)	3.10 (Fe_2O_3)	PbO	1.37	1.37
MgO	1.28	1.28	Others	0.74	0.74
K_2O	0.48	0.48	LOI	+7.48	1.12

The preparation of the specimens includes the casting of concrete block specimens of size 70 mm x 190mm x 390 mm. The fine F and coarse A were partially replaced with OPC and aggregates. The water to cement ratio of specimens was taken constant at 0.63 by weight, and the required workability was kept. The ratio of the binder, sand, and coarse aggregate was kept at 1:4.3:5. The specimens were demoulded after 24 hours. Different mechanical properties like compressive behavior were determined after the curing of 28, 60, and 90 days. The determination of water absorption was done within 24 hours by the process of oven drying. The results of the above properties were compared with the standard specimens containing 0% secondary lead slag. To examine the magnetic property in the lead slag, the mortar specimens were casted of 50mm cubes. Also, various tests like compressive and water absorption were investigated in the mortar specimens. The leaching behavior was also determined according to US-EPA SW846 of the lead in the specimens. The broken specimens were kept in the acidic medium at a pH of 5 for 18 hours, and an atomic absorption spectrometer determined the lead content.Table 2.14 shows the Component proportion of Secondary Lead Slag.

Table 2.14 Component proportion of Secondary Lead Slag (Penpolcharoen, 2005)

Specimen ID	Binders (%)		Agg. (%)	
	O.P.C. Cement	Fine aggregate	Crush Stone	Coarse Aggregate
Standard	100	-	100	-
F-20	80	20	100	-
F-40	60	40	100	-
A-20	100	-	80	20
A-40	100	-	60	40
A-60	100	-	40	60
A-80	100	-	20	80
A-100	100	-	-	100
F-20A-50	80	20	50	50
F-30A-50	70	30	50	50
F-20A-100	80	20	-	100
F-30A-100	70	30	-	100

The leaching test was only performed in the specimens containing high content of secondary lead slag: A100, F20A100, and F30A100. The lead content determined by the process was around 0.06 for all three specimens, which is below the acceptable standards of the Thailand hazardous waste characterization.

Figure 2.13 shows graph of compressive strength results when Lead Slag was replaced with OPC only. The standard specimen with no lead slag has completed its whole development of strength in 28 days, whereas, the specimens containing lead slag are developing strength up to 90days. The development of strength of the specimens in the first 28 days was comparatively slower for the specimens containing slag compared to the standard specimen. And after the 90 days curing period all the specimens containing lead slag. Previous research shows that replacing OPC with pozzolana like Fly Ash having lower calcium content lowers the early compressive strength. The rate of strength increase will occur at a greater curing period because of the pozzolana reaction. The calcium hydroxide produced is very important in breaking the glassy phase of the lead slag, which leads to the pozzolana reaction.

Also, the lead slag used in the research has less calcium oxide content than OPC; the high replacement of lead slag with OPC will decrease the strength development of the specimens. But in this research work, the compressive strength increases in increase of lead slag. The reason believed is other than calcium oxide content, which is assumed to be due to magnetic effect, size, and shape.

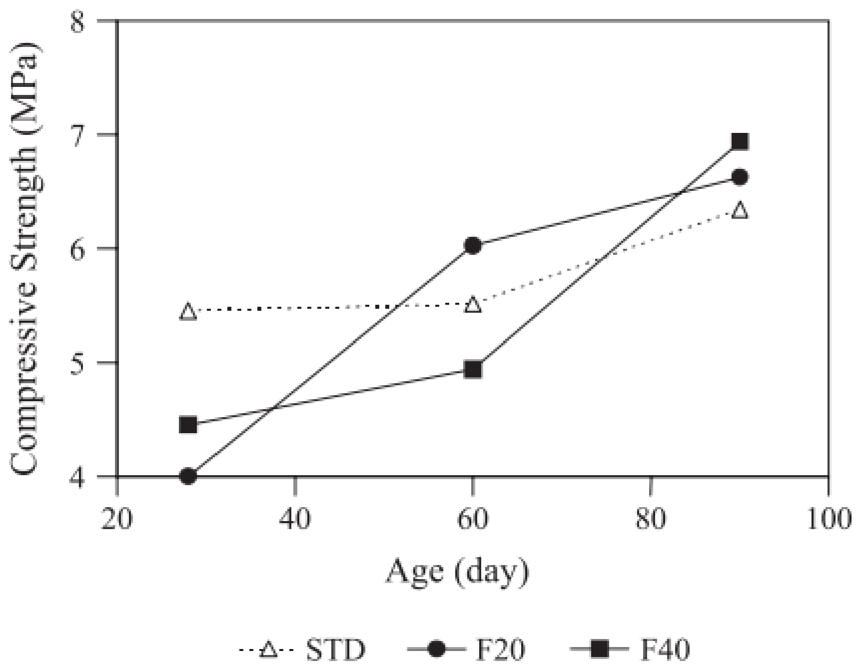


Figure 2.13 Compressive strength of specimens at different ages (Lead Slag replaced with OPC) (Penpolcharoen, 2005)

When the lead slag is used as a filler material as the replacement of the stone aggregates, the lead slag acts as a filler and helps in reducing the porosity. This imparts the filler effect and imparts strength to the specimen. A previous study concludes that the filler effect and the pozzolana effect both increase as mineral content increases. But the filler effect dominates as compared with the pozzolana effect. The magnetic effect in the lead slag is considered another reason for imparting greater strength to the specimens due to the magnetic forces in the microparticles of the paste and also induces the absorbed water into the magnetic water.

Figure 2.14 shows the graph of compressive strength results when Lead Slag was replaced with stone aggregates only. The graph results show that the strength development of the standard specimen from 28 days to 90 days is less as compared to other specimens. As the addition of lead slag was done, the strength achieved was greater because the cement in all the specimens was the same. Also, in these types of specimens, the hardness of lead slag type aggregates was more as compared to natural limestone stone aggregates. This was also confirmed by performing the abrasion and the impact test.

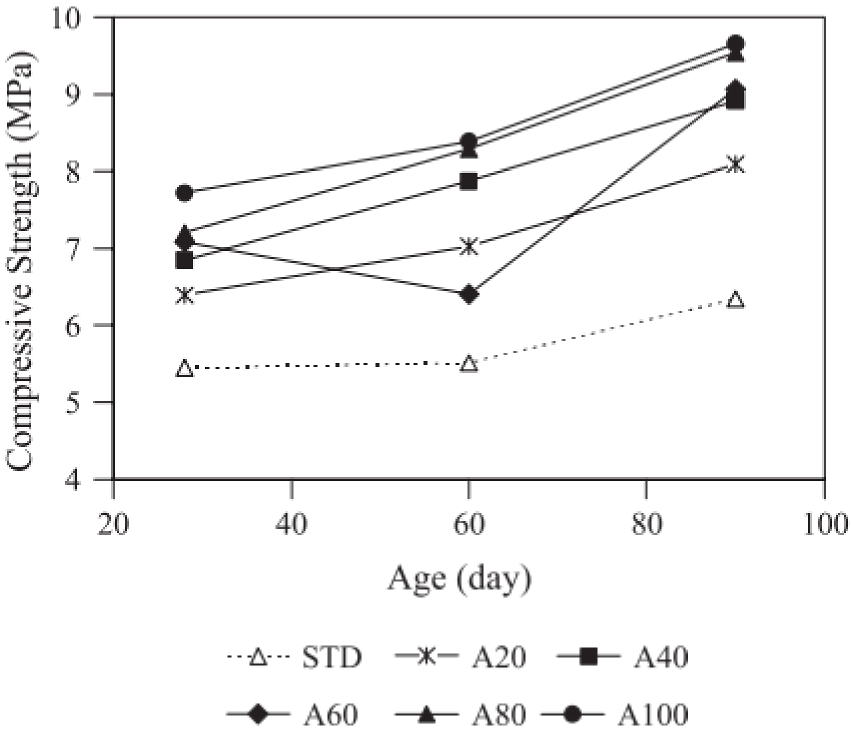


Figure 2.14 Compressive strength of specimens at different ages (Lead Slag replaced with Stone Aggregates) (Penpolcharoen, 2005)

Figure 2.15 shows the graph of compressive strength results when Lead Slag was replaced with Lead slag and stone aggregates both. The results depict that all the specimens exhibit larger strength than that of the standard specimen. At constant fine content, the strength is more of the specimens containing more aggregate slag. And when the same quantity of lead slag aggregate is used, the specimens containing 20% F shows more strength than that of specimens containing 30% F. It can be shown by specimen code F20A100, which exhibits larger compressive strength compared to the standard specimen. The reason behind this is believed to have a combined effect of hydration mechanism, pozzolana reaction, filler effect, and magnetic effect.

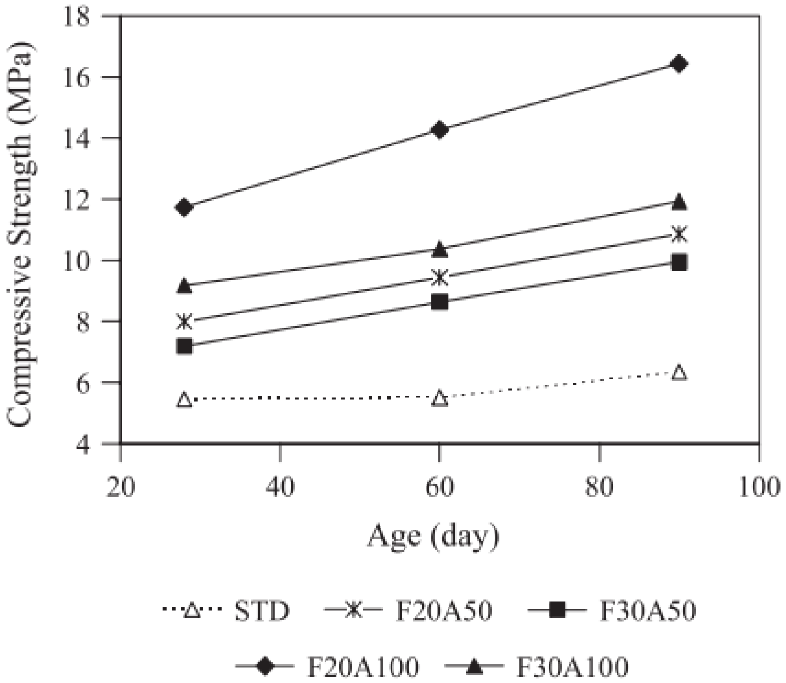


Figure 2.15 Compressive strength of specimens at different ages (Lead Slag replaced with both Stone Aggregates and OPC) (Penpolcharoen, 2005)

The result concluded that lead slag can be used in the concrete and the mortar mixtures. The secondary lead slag possesses a magnetic property because of its CaO-FeO-SiO_2 element. The magnetic property also have a important role in the mechanical properties of specimens. The high concentration of the Iron oxide and sulphur trioxide in the lead slag, the durability, and chemical resistance aspects need to be reviewed further.

Atzeni *et al.* (1996) studied chances of using the granulated slag produced from the smelting of Pb and Zn as a substitution of the sand in the mortar and concrete. The compounds in the granulated slag are Calcium, Silicon, Iron, and aluminium, including Lead concentrations in small amounts. The slag particles size was sufficient to substitute the sand in mortar and concrete. And also, results concluded in this research work lead to good mechanical properties. But the limitation of the work is that the concrete waste specimens, when kept in acid for the extraction of Pb, created some issues.

The research work's main aim was to utilize the granulated slag in two different ways. The first was as a substitution to cement as a pozzolana material after proper grinding into the fine particles, and the second was the substitution of the sand because the advantage, in this case, is that the particle size of lead slag is identical to the aggregates used in the concrete. Different micro-structural and mechanical behavior of the specimens with replacing with sand has been examined in the mortar and the concrete.

The lead slag used in the research was produced in Italy by two processes. The First was Lead Smelting (K), and the second was imperial smelting (I.S.). The manufacturing was done in Sardinia in Italy. The typical elemental composition of Lead Slag is shown in Table 2.15. The chemical composition was determined from AAS and plasma techniques.

Table 2.15 Chemical Composition of Lead Slag (Atzeni *et al.* 1996)

Elements (% by weight)	Lead Slag (K)	Lead Slag (I.S.)	Limiting Value	Elements (% by weight)	Lead Slag (K)	Lead Slag (I.S.)	Limiting Value
SiO_2	18.3	11.3	-	Pb	3.6	1.4	0.5
Al_2O_3	5.5	5.5	-	Cu	0.2	0.6	0.5
FeO	26.1	52.9	-	Cd	0.008	0.001	0.01
CaO	15.6	3.3	-	Hg	0.009	0.009	0.01
Zn	14	9.8	-	As	0.15	0.18	0.01

The preparation of the mortar includes the water-cement ratio of 0.50 by mass. The aggregate to cement ratio was kept at 3. The replacement of lead slag was done with sand(K and I.S.). Also, in later cases, the equivalent volume substitution (Kv and I.S.v) was done by considering the specific gravity. To determine the mechanical properties, a mortar prism of dimensions 40mm x 40mm x 160mm was casted and kept for 12 months. Also, concrete specimens of 160 mm side were casted, and the mechanical test was conducted after 28 days. Table 2.16 shows the typical composition of concrete mix design.

Table 2.16 Composition of concrete mix (kg/m3) (Atzeni *et al.* 1996)

Parameter	Calcareous type gravel (I)		Calcareous type gravel (II)		Siliceous type gravel	
	Control specimen	Lead-slag specimen	Control specimen	Lead slag specimen	Control specimen	Lead slag specimen
Cement	350	350	200	200	350	350
Fly Ash	-	-	-	50	50	50
Water	150	150	160	180	180	180
Sand	1000	750	1000	950	650	650
Lead Slag	-	250	250	-	250	250
Gravel	950	950	950	950	950	950

At last, the elution test was performed in order to get the eluted metal content. The crushed sample of concrete up to 10 mm was considered. A total of 100 gm of crushed sample from concrete was taken and added in the 1600 cm^3 of distilled water or in the acetic acid. The pH in the test was kept constant up to 5. Or the calcium hydroxide can also be held in the agitator (200 mm height and 120 mm diameter with two propellers having 120 revolutions/minute) for 24 hours. The metal concentration was calculated using the plasma technique by ICP-OES.

The mechanical properties of mortar included compressive and flexural strength. All the mortar specimens were replaced with lead slag by equivalent weight and equivalent volume. The flexural strength test was done by three-point bending, and the result concluded that the flexural strength was almost constant after the month's aging, and a very little variation was noticed in the values ranging between 8 MPa to 10MPa. The compressive strength results with age are shown in Figure 2.16.

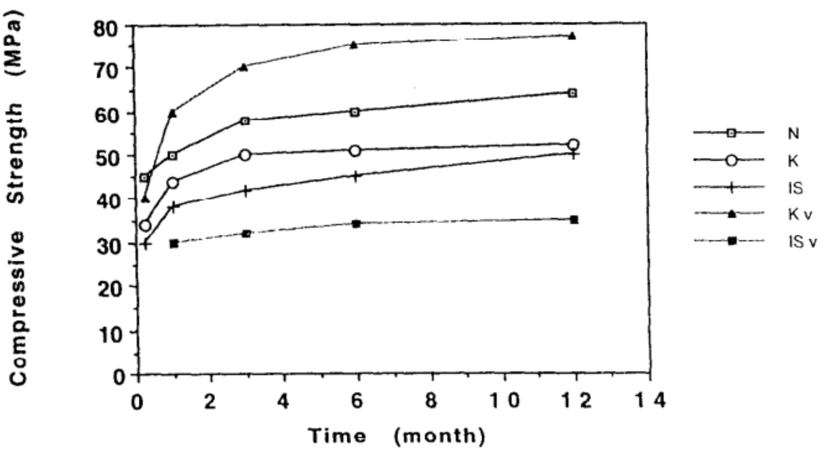


Figure 2.16 Compressive strength of Lead slag based mortar specimens over time (Atzeni *et al.* 1996)

The mortar specimens with sand achieved 50 MPa of strength after 28 day and 60MPa after 12 months period, and the specimens having an equivalent weight of lead slag achieved 40 MPa and 44 MPa in 28 days and 49 MPa and 51 MPa in 12 months (I.S. and K respectively). The compressive strength of mortar samples containing lead slag by an equivalent volume decreased drastically, which was not more than 35 MPa in 12 months of I.S.-type Lead slag. And that of K -type lead slag was 60 MPa after one month and 80 MPa after 12 months.

Table 2.17 shows the compressive strength of the different concrete specimens at 28 days. The results concluded that the substitution of sand with lead slag do not change in mechanical property of concrete.

Table 2.17 Compression strength of Lead slag based concrete specimens (Atzeni *et al.* 1996)

Parameters	**Calcareous type gravel (I)**		**Calcareous type gravel (II)**		**Siliceous type gravel**	
	Control specimen	Lead slag specimen	Control specimen	Lead slag specimen	Control specimen	Lead slag specimen
Compressive Strength	55 MPa	58 MPa	20 MPa	18 MPa	43 MPa	42 MPa

The elution test was done in water and the acetic acid solution at a pH of 5 because the water circulating in the landfill is slightly acidic in nature. Table 2.18 shows the Concentration of elements eluted from concrete specimens.

Table 2.18 Concentration of elements eluted from the concrete specimens (Atzeni *et al.* 1996)

Elements Eluted	**Calcareous type gravel (I)**		**Calcareous type gravel (II)**		**Siliceous type gravel**	
In water	**Control specimen**	**Lead slag specimen**	**Control specimen**	**Lead slag specimen**	**Control specimens**	**Lead slag specimens**
Lead	< 0.10	0.15	< 0.10	< 0.01	< 0.10	< 0.10
Zinc	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Copper	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10
Cr	< 0.10	< 0.10	< 0.10	< 0.01	< 0.10	< 0.10
In Acetic Acid	**Control specimens**	**Lead slag specimens**	**Control specimens**	**Lead slag specimens**	**Control specimens**	**Lead slag specimens**
Lead	0.30	9.0	1.1	4.5	< 0.15	6.60
Zinc	0.80	60	7.80	90	< 0.20	45
Copper	< 0.10	0.25	< 0.10	0.25	< 0.10	< 0.10
Cr	< 0.10	< 0.10	0.12	0.12	< 0.10	< 0.10

The research concluded that the partial or the total substitution of the sand with the granulated lead slag from the kivcet smelting process and imperial smelting process is feasible, provided that the design mix of the mortar or concrete does not impair the mechanical properties of the specimens. Also, using these types of slag in a construction material does not need any preliminary treatment, and this is the primary benefit in the concrete science, which is in the vicinity of the plants, ultimately leading in the relieving the problem of the waste disposal.

M.Alwaeli, 2021 investigated the effect of replacing the fine aggregates with granulated lead/zinc waste on mechanical behavior of the concrete. The fine aggregates were substituted by Granulated zinc lead slag in four percentages of replacement of 25%, 50%, 75%, and 100%. The substitution was done by equivalent mass. The primary objective of study is to study the effect of GZLS on compressive behaviour of concrete. The Lead Slag used in the research is granulated lead-zinc slag. The typical chemical composition of the Lead Slag is shown in Table 2.19.

Table 2.19 Chemical composition of the different components (M.Alwaeli, 2021)

Elements	Portland Cement	Sand	Granulated lead-zinc slag
Oxygen	52.5	50.48	59.07
Aluminium	2.44	6	6.16
Silica	11.75	33.22	8.18
Magnesium	2.43	1.91	3.78
Iron	0.66	3.34	11.11
Sodium	-	3.01	-
Carbon	-	0.36	-
Sulphur	1.23	-	1.57
Calcium	24.55	2.34	8.80
Lead	-	-	0.65
Others	5.25	0	0.89

The preparation of the specimens includes the preparation of the four mixes of replacing fine aggregate with slag (25%, 50%, 75%, 100%), and one control mix was prepared. And no additive was used in the research. Table 2.20 show mix proportion of Concrete mix.

Table 2.20 Mix proportions of Concrete mix (M.Alwaeli, 2021)

Mixture	Cement (kg/m^3)	Fine Aggregate (kg/m^3)	Granulated lead-zinc slag (kg/m^3)	Water (kg/m^3)	Gravel (2-4)	Gravel (4-8)
Control	413	1112	-	213	318	162

C-25%	4346	8840	2946	2124	334	170
C-50%	5082	6892	6892	2335	390	199
C-75%	4572	3166	9507	2228	361	183
C-100%	4707	00	13038	2375	369	190

To examine compressive behavior, cubical specimen of 10 cm were casted to measure compressive strength according to the European Standard. Figure 2.17 shows the compressive strength of concrete mixture after 28 days.

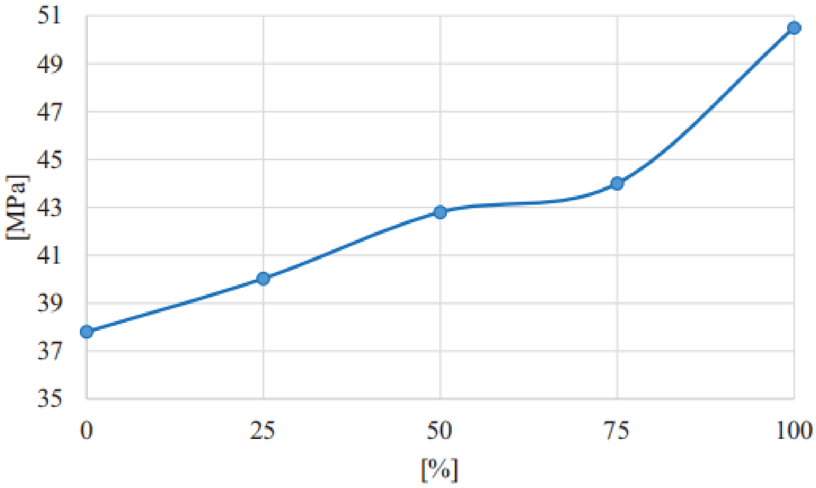


Figure 2.17 Compressive strength of concrete mixture (M.Alwaeli, 2021)

Table 2.21 show compressive strength of concrete specimens. Result acquired in the table is the average of the three measurements**.** The substitution of the fine aggregates with the lead-zinc slag increase strength.

Table 2.21 Compressive Strength of Concrete specimens (M.Alwaeli, 2021)

Mixture	Compressive Strength	Mixture	Compressive Strength
Control	37.80	**C-75%**	44
C-25%	40.03	**C-100%**	50.50
C-50%	42.80	**Standard Deviation**	4.8

The results show that the 25% replacement of the lead-zinc slag with the fine aggregates has minimal effects on compressive behavior of concrete specimens. The compressive strength of 25% replacement increases by 6% as compared with control specimens. Increase after 25% in lead-zinc slag has a high effect on compressive behavior. Compared with the control specimen with no slag replacement, compressive strength of 50% of replacement specimens was 42.80 MPa, the compressive strength of 75% of replacement samples was 44 MPa, and that of 100% of replacement specimens was 50.50 MPa.The results of the research concludes that compressive strength of the concrete having 100% substitution of slag is the highest.

2.6.2 Use of Lead Slag as Construction Material in Geopolymer

Many other researchers have investigated and used the primary and secondary lead slag in geopolymer to replace fly ash. Also, some researchers examined about the effects of the lead slag on the mechanical properties geopolymer based mortar and concrete.

Geopolymer is basically inorganic in nature and is typically ceramic. Geopolymer contains an extended range of Alumino-silicates networks held by a covalent bond. Geopolymer is amorphous (non-crystalline) in nature. T.F. Yen *et al.* 2006 classified the geopolymer into two parts: organic containing and inorganic containing geopolymers. He defined geopolymer as a chemically mineral compound or a mixture of different compounds containing repeating chains and networks like -Si-O-Si-O- (Silico Oxide), -Si-O-Al-O- (Silico Aluminate), and -Fe-O-Si-O-Al-O- (Ferro-silico-aluminate) which are mainly formed by the reaction mechanism of geopolymerization.

Yousef *et al.* 2009 concluded that the geopolymer could be used for stabilisation and the immobilisation of different types of by-products from different industries in the 3D units of the geopolymer structure. Also, Guo *et al.* (2017) conveyed that use of lead slag in the geopolymer was solved in the NaOH, which was further dispersed in the 3D network of the geopolymer.

S.Onisei *et al,* 2011 used the Lead Slag used in the research was manufactured by IMNR-INCDMNR in Romania. The lead slag in the industry was produced by smelting the lead concentrate after the autoclave. The fly ash used was produced from the combustion from the PPC Plant. The grinding of both the materials was done in a mill in Germany below 125 micrometers. The activating solution was formed by the NaOH pallets by mixing it with distilled water, and then it was cooled at room temperature for 24 hours. After 24 hours, the Sodium silicate solution was added. The typical chemical compositions of the Lead Slag and fly ash is shown in Table 2.22.

Table 2.22 Chemical composition of the Lead Slag (S.Onisei et al, 2011)

Oxides	Fly Ash	Lead Slag	Oxides	Fly Ash	Lead Slag
SiO_2	51.4	21.57	**PbO**	< 0.01	12.29
Al_2O_3	23.5	1.74	**ZnO**	< 0.01	6.19

Fe_2O_3	7.83	31.56	**As_2O_3**	-	0.20
CaO	9.22	3.04	**CoO**	-	0.02
MgO	1.70	0.16	**Cr_2O_3**	-	0.35
Na_2O	0.87	13.01	**CuO**	-	1.64
K_2O	1.46	0.25	**MnO**	-	0.10
SO_3	2.65	8.02	**NiO**	-	< 0.01

The chemical components of lead slag and fly ash were determined by XRF. In the lead slag, the element concentration of Cobalt, copper, chromium, and manganese were determined by ICP-AES, and that of arsenic was determined by a direct-coupled plasma spectrophotometer. And lastly, the chemical analysis of the leachates was determined by the ICP-MS.Table 2.23 shows the composition of concrete mixture.

Table 2.23 Composition of Concrete mix (S.Onisei *et al*, 2011)

Mixture ID	Fly Ash (wt%)	Lead Slag (wt%)	Sodium Silicate (wt%)	Sodium Hydroxide (wt%)
GLS-0	100	0	29.4	26.9
GLS-28	71.8	28.2	25.4	20.8
GLS-51	48.9	51.2	22.0	15.7
GLS-70	29.8	70.2	19.4	11.5
GLS-86	13.8	86.2	17.2	8.0
GLS-100	0	100	15.3	5.0

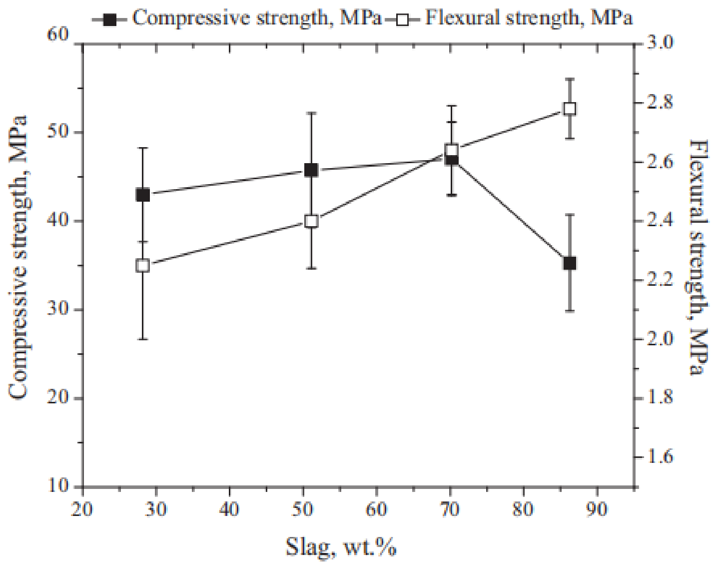


Figure 2.18 Flexural and compressive strength with variation in lead slag content (S.Onisei et al, 2011)

Figure 2.22 shows the graphical representation of Compressive and flexural strength.The results concluded that the flexural strength of the specimens is comparable in each case, whose range is typically from 2.2 to 2.8 MPa, Whereas the compressive strength clearly from the figure there is an increase of strength up to 70% addition of lead slag. Further, an increase in the lead slag content decrease in strength development.

2.7 Various Leaching Procedure and Tests

There are many procedures of leaching tests adopted to measure the natural weathering and conditions of landfills containing lead slag. USEPA (United states environmental protection agency), 2008, and ECFS, 2002 established the leaching tests procedures, which are TCLP and the EN 12457–2 leaching test. The aim of procedure mainly is to demonstrate whether the lead slag is hazardous or not so that accurate information on the characteristic of lead slag could be known before disposing in the landfill or stockpiling. The tests also help in providing the information if any special treatment is required in lead slag before disposal. Bernardez, 2011 performed a TCLP test of the lead smelting slag. The results shows that Lead, zinc, and cadmium have a less tendency of leaching in the weak acidic exposure for the short-term slag water interaction. The TCLP test has a short-term procedure of 18 hours, and the EN 12457-2 test has a 24 hours interaction time with water. These two tests concluded that content of severe hazardous elements in the lead slag were less as compared with the limiting value (Saikia *et al.* 2012).

If there is a long-term interaction between the lead slag and water, there are greater chances of an increase in content of hazardous elements in the water as compared with that of the leaching procedure. The zinc was released in the maximum amount during a leaching test conducted for 12 years, which exceeds the limiting values of toxic waste. (Ettler and Johan, 2014).

There is one more leaching test (SPLP) established by the USEPA (United States Environment Protection Agency) for predicting the leaching of the lead slag on the surface and sub-surface water by simulation of the interaction between lead slag and precipitation. (USEPA, 2008). The leaching test results conducted by the SPLP test were compared with US EPA standards. The results of the SPLP concluded that content of the hazardous compounds in the lead slag exceeded the US Standards of drinking water. (De Andrade Lima & Bernardez, 2011, 2013).

Saikia *et al.*, 2012 analyzed leaching assessment by three procedure The Leaching behavior of the Lead slag-based cement mortar specimens was done as per EN 12457-2 and NEN 7345. The leaching of different metals from mortar specimens having lead slag was determined. The procedure includes the mixing of finely powdered lead slag with the deionized water containing the ratio of deionized water to lead slag of 10:1. The leaching procedure was also established by the diffusion method (NEN 7345). The procedure's aim was also to calculate the release rate of chemical components from the specimen. The leaching of the chemical elements from the specimen's body is comparatively less than the crushed specimens. Thus we can say that the crushed specimens are not valid in predicting the actual leaching rate of different components. Therefore, the NEN 7345 is more precise and accurate test.

L.R. P. de Andrade Lima. (2012) investigated the stability of lead slag by leaching in a wide range of pH and water. The TCLP test was conducted for the same in order to measure the toxic contents in lead slag.

The stability of Lead Slag was firstly demonstrated over an extensive pH range. The samples were finer than 2 mm, placed in a beaker (Teflon) of 250 ml capacity, and finally sealed by parafilm. This was kept in different fluids for 24 hours to 28 hours. Fluids were hydrochloric acid, acetic acid, nitric acid, or sodium hydroxide at room temperature without any shaking. The powder to liquid ratio was 10:1. (10 g of the slag powder in 100 ml of fluid. Finally, the liquid was separated from the powder by the method of decantation. Whereas in the fluid containing HCl and NaOH, the samples of about 60 ml were evaporated at 70 degrees Celcius and the dissolved elements were weighed in the balance. The various elemental concentration was then determined from ICP-OES.

Secondly, to demonstrate the stability of slag powder in water, the fine powder passing from 1 mm was made and then put in a Teflon beaker sealed with parafilm. This was kept in the presence of water for 1 day to 30 days. The whole procedure was performed at room temp. And without any shaking. The powder to liquid ratio was 10:1. (10 grams of slag powder in 100 ml fluid. Finally, the liquid was separated from the powder by the method of decantation. Fnally the pH was measured, and various elemental concentration was then determined from ICP-OES. Table 2.24 shows the result of leaching.

Table 2.24 Leaching analysis (in mg/L) (L.R. P. de Andrade Lima, 2012)

Elements	TCLP	Threshold value (US PEA - Std.)	Elements	TCLP	Threshold value (US PEA - Std.)
Ag	< 0.1	5	**Hg**	< 0.01	0.2
As	< 0.1	5	**Pb**	0.4	5
B	0.1	-	**Sb**	-	-
Ba	0.3	100	**Se**	< 0.1	1
Cd	< 0.01	1	**U**	< 1.0	-
Cr	< 0.1	5	**Zn**	-	-

The results concluded that in the weak pH range, the detection was less than the threshold values (US EPA - Standards). In TCLP, the different contents of different toxic elements were significantly less than the threshold limits.

2.8 Research Significance (Need of present Investigation)

The main dispose of Secondary Lead Slag is generally in the landfill, which needs a great amount of land, and this discharge causes serious environmental problems. This improper disposal of the secondary-lead slag also causes an extensive waste of different resources. So reducing the adverse toxicity of secondary lead slag and promoting the utilization of secondary lead slag as a construction material should be the measures considered so to decrease the effect of secondary lead slag in environment. Therefore, this research focuses on utilizing the secondary recycling lead slag as a construction material.

Although many researchers are working towards the harmless utilization of lead slag as a construction material, relatively minor work has been done on the structural performance of the mortar and concrete containing secondary lead slag as an pozzolana admixture. The strength and durability improvement of the mortar/concrete are the crucial factors in concrete science. Hence, the objective of the research is to provide information and to gain insight into the behavior of secondary lead slag as substitution of cement and fly-ash and to conduct an extensive study on the feasibility analysis of secondary lead slag as a construction material.

In addition to that, the information on the different mechanical and leaching behaviour is found in the past literature but is comparatively less. And yet this information is crucial for the use of secondary lead slag in for structural applications. Therefore the investigation is carried in order to study the leaching behaviour of the toxic elements to test the environmental suitability of the secondary lead slag by TCLP test. The test main aim is to predict whether the secondary lead slag is hazardous or non-hazardous so that accurate information on its characteristics can be known.

2.9 Closing Remarks

A brief literature review on the Utilization of Lead Slag as the construction material has been carried out in a detailed manner and on the based of this the significance of study have been recognized.

CHAPTER 3

MATERIALS AND METHODS

3.1 Introduction

This chapter represents brief details of different material used and methods carried considering the different material properties, different mix design proportions sample preparations, set-up for different tests carried out for measuring different properties, and different procedure for testing.

3.2 Material Properties

Cement, Standard sand, fine aggregates, secondary lead slag, fly ash, NaOH, Na_2SiO_3 and water has been used in this study. The physical and chemical properties of these materials have been discussed in the below sections.

3.2.1 Cement

Cement is fine material having Grey appearance formed by the calcination of lime and clay. As the concrete/ mortar hardens, the cement works as a binder and form a paste with water which acts like a binder. Normal Cement mainly consists two primary compounds, including argillaceous minerals and calcareous minerals. Argillaceous minerals have clay majorly, and calcareous minerals have $CaCO_3$. The present research used OPC grade - 43 (Ultra tech cement) in accordance to IS:1489-1991. The various test results on cement properties are given in Table 3.1 and were tested according to the standards provided in the code IS:4031 (Part 4, Part 5, Part 6 and Part 11) 1988.

Table 3.1 Physical properties of OPC- 43 Grade

S. No.	Physical Property	Results	Std. values as per code (IS: 1489-1991)	IS Code Std. referred for Test
1.	Normal Consistency (%)	28.5%	-	IS: 4031 (Part 4)
2.	Initial setting time	49 minutes	Not lesser than half hour	IS: 4031 (Part 5)
3.	Final Setting time	5 hours 40 minutes	Not greater than six hours	
4.	Fineness	3.5%	<10%	IS:4031 (Part 1)

5.	**Specific gravity**	3.0438	-	IS:4031 (Part 11)
6.	**Compressive Strength**	3 Day = 18.5 N/mm^2	15 N/mm^2	IS:4031 (Part 5)
		7 Day = 28.97 N/mm^2	22 N/mm^2	
		28 Day = 36 N/mm^2	31 N/mm^2	

Chemical Composition of OPC of grade - 43 is shown in below table.

Table 3.2 Chemical composition of OPC

Elements	Composition	Elements	Composition
SiO_2	22.5	MgO	2.21
Al_2O_3	6.71	Na_2O	0.31
Fe_2O_3	4.50	SO_3	2.1
CaO	60.7	K_2O	0.30

3.2.2 Standard Sand

The Standard sand is quartz, Grey light or white in colour and must be free from silt content. The grain of sand particles must be angular, and the shape of the grain particles must be in spherical in shape. There shall be elongated or flattened particles of grains only in the small or minute quantity. The chemical characteristics of the standard sand includes that it must be free from the organic impurities. The present study used the Standard Sand of all three grades conforming to Indian standard IS:650:1991. The standard sand is 100% passing from 2 mm sieve and is 100% retaining on 90 micron sieve and particle size distribution of standard sand is shown in below table.

Table 3.3 Particle Size Distribution (IS:650:1991)

Sr. No.	Particle Size	Percent used
1.	Less than 2 mm and larger than 1 mm	33.333
2.	Less than 1 mm and larger than 500 microns	33.333
3.	Less than 500 microns and larger than 90 microns	33.333

3.2.3 Secondary Lead Slag

The secondary lead recycling slag was used in the research, which is basically produced while extracting Lead from the waste lead based acid battery in a rotary furnace. Secondary lead slag for the present study was provided by local recycling plant. The company is mainly dedicated to producing Lead and lead products considering an environment-friendly process. A total of three secondary lead slag were provided by the company and were used in this research. The first was mix slag, the second was iron-rich lead slag, and the third was silica-rich lead slag.

Table 3.4 Three type of Secondary Lead Slag used in Study

Lead Slag (Type 1)	Mix Slag
Lead Slag (Type 2)	Iron rich lead slag
Lead Slag (Type 3)	Silica rich lead slag

Figure 3.1, 3.2 and 3.3 shows the images of three type of secondary lead slag respectively.

Figure 3.1 Lead Slag (Type 1)

Figure 3.2 Lead Slag (Type 2)

Figure 3.3 Lead Slag (Type 3)

The typical production of the secondary lead slag is basically by recycling of waste lead based acid battery in the recycling plant. Figure 3.4 show waste lead-acid battery recycling plant.

Figure 3.4 Waste Lead-acid batteries recycling plant (Recycling Plant)

The Basic steps in extracting lead from the scrap in the plant includes the following operations-

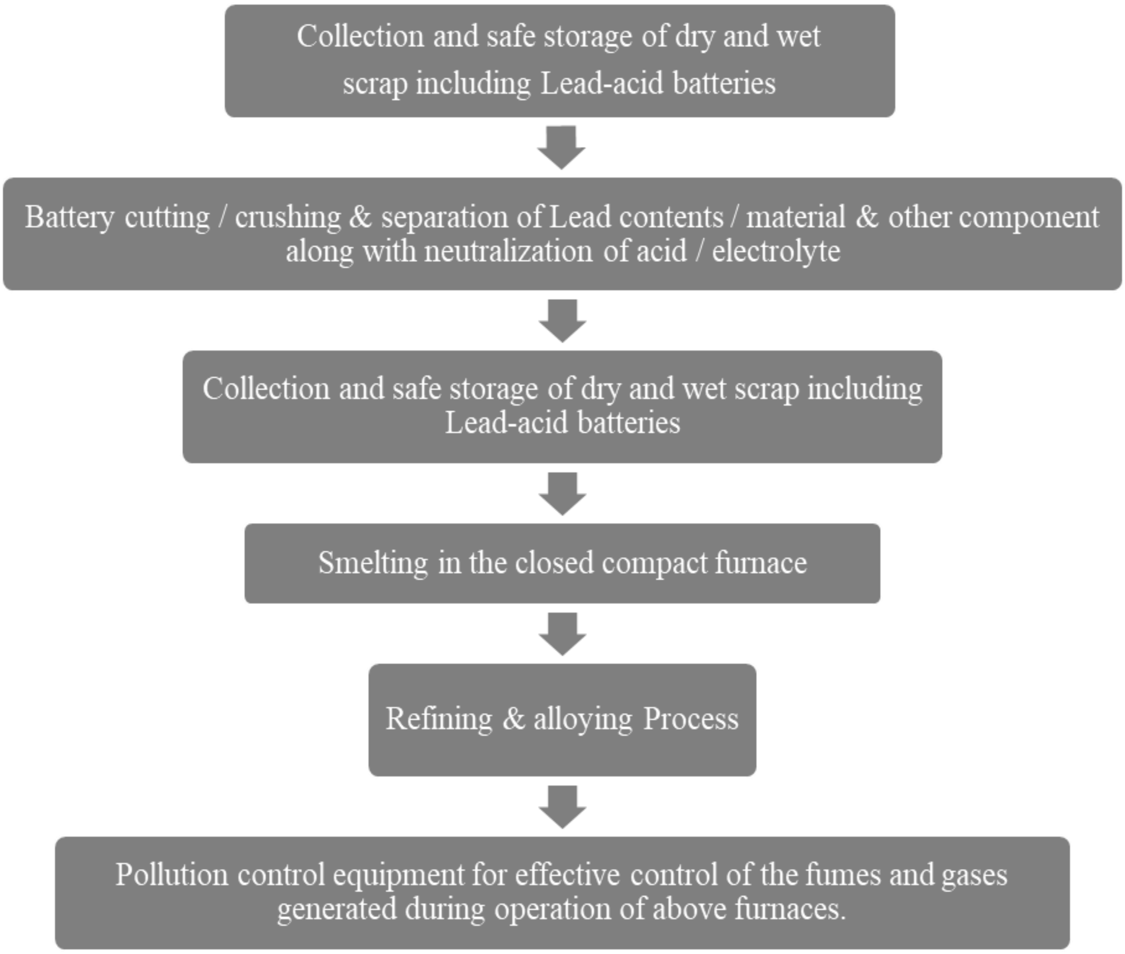


Figure 3.5 Flowchart showing basic operations in Lead Battery Recycling process

The rotary furnace is used to treat and recover the metallic lead from the waste lead-acid batteries. Also, for the reduction of the Galena, oxides of lead and different lead concentrate. The rotary furnace in plant works at 1000-1200 degree C. Also, the temperature indicators are provided inside in electric control panel and the Thermo-couple probe at the exhaust duct of the rotary furnace for maintaining the temperature of the furnace to a certain level. The raw ingredients requirement in the rotary furnace includes lead-acid waste scrap, lead scrap, lead ash, lead concentrate, and other lead-bearing scrap.

The smelting process then occurs in the rotary furnace, where the raw ingredients, along with the soda ash and the iron tuning, are put in the furnace. After smelting, melted lead is formed and converted to the lead blocks. The secondary lead slag is then separated and treated for further recovery. Figure 3.6 shows the Rotary furnace and Secondary Lead Smelting

Figure 3.6 Rotary Furnace for Lead Battery Recycling (Recycling Plant)

Figure 3.7 shows the aadvantages of using Rotary furnace in recycling plant.

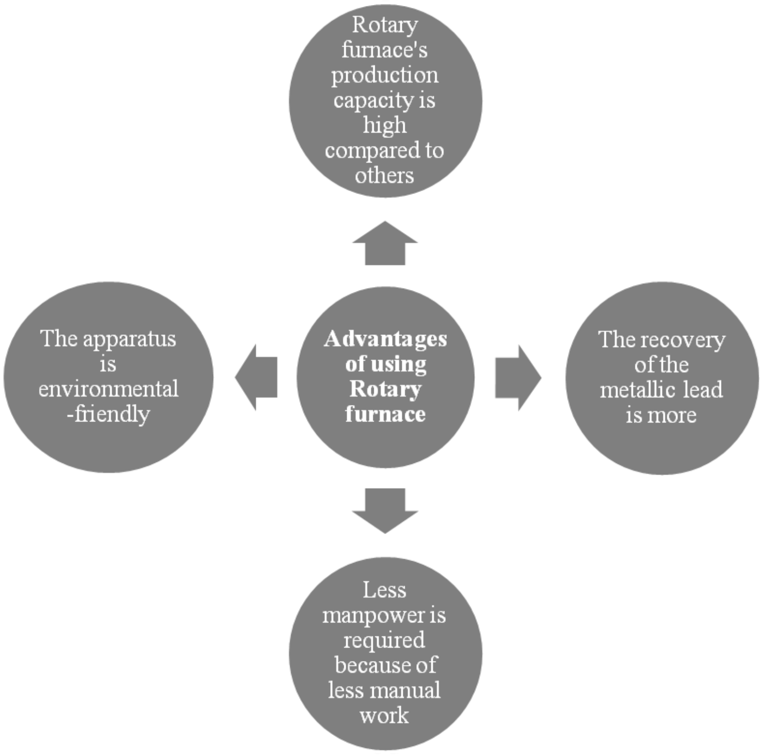


Figure 3.7 Advantages of using Rotary furnace

3.2.3.1 Chemical Characteristics of Lead Slag

The quantitative determination of chemical elements of all three type lead slag was done by EDS (Energy-dispersive X-ray Spectroscopy). The typical chemical composition of all three lead slag are shown in Table 3.5. A total of 16 elements were predicted by EDS.

Table 3.5 Typical Chemical Elements of Lead slag by EDS

Elements	% (Type 1 Lead Slag)	% (Type 2 Lead Slag)	% (Type 3 Lead Slag)
Fe	30.8	32.50	21.18
O	19.97	17.39	33.46
S	13.77	16.83	2.5
Pb	11.90	17.12	2.26
C	8.53	4.19	7.12
Na	7.90	9.35	9.56
Si	2.23	0.26	13.83
Br	1.40	0.495	1.75
Ca	1.40	0.183	4.45
Mn	1.10	0.18	0.37
Mg	0.30	0.123	0.41

Cr	0.30	0.01	0.23
Al	0.17	0.83	2.31
As	0.17	0.01	0
K	0.03	0.35	0.61
Cu	0.03	0.36	0.2

The chemical composition was also demonstrated by AAS (Atomic Absorption Spectroscopy) technique. The typical chemical composition of all three lead slag are shown in Table 3.6. The AAS Spectroscopy was done on 12 elements.

Table 3.6 Typical Chemical Elements of Lead slag by AAS

Elements	Method of Test	% (Type 1 Lead Slag)	% (Type 2 Lead Slag)	% (Type 3 Lead Slag)
Fe	Digestion followed by AAS	22.62	33.4	28.6
S	CHNSO Analyzer	5.76	23.32	6.54
Pb	Digestion followed by AAS	9.95	10.9	7.06
C	CHNSO Analyzer	1.14	0.47	0.40
H	CHNSO Analyzer	-	0.17	0.04
Na	Digestion followed by flame photometer	5.88	6.48	15.0
Si	Gravimetric Method	16.83	13.5	25.1
Ca	Digestion followed by AAS	2.60	0.49	5.94
Mg	Digestion followed by AAS	0.35	0.50	0.93
Al	Digestion followed by AAS	-	3.54	3.36
K	IS: 10158-1982 (2003)	-	0.43	0.77
Cr	Digestion followed by ALPHA 23rd edition 3111D	0.024	-	-
Mn	Digestion followed by ALPHA 23rd edition 3111B	1.36	-	-

3.2.3.2 Physical Characteristics of Lead Slag

The Lead slag used in the study is black in colour and have a glassy appearance. The particle size analysis is done to get the distribution of various particles range. Analysis is done by manual sieving and in HORIBA Laser Scattering Particle size Distribution Analyzer LA-950. This apparatus works on the principle of scattering of light. The refractive index adopted in the procedure is of Fe_2O_3. The dry sieve analysis data is compiled in Table 3.6, 3.7 and 3.8 for three lead slags respectively.

Table 3.6 Sieve Analysis of Type 1 (Lead Slag)

Sieve Number	Mass of Lead Slag retained (grams)	% of Lead slag retained	% of Lead slag passing	Cumulative % retained
600 μ	0.02	0.002	99.998	0.002
425 μ	0.08	0.008	99.992	0.01
300 μ	9.15	0.913	99.087	0.923
150 μ	423	42.205	57.795	43.128
90 μ	452	45.099	54.901	88.227
75 μ	30	2.993	97.007	91.22
Pan	88	8.78	91.22	100
Total	1002.00		Summation (F) =	323.51
			Fineness Modulus =	3.235

Fineness Modulus of Lead Slag (Type 1) = ΣF/100 = 3.235

Table 3.7 Sieve Analysis of Type 2 (Lead Slag)

Sieve Number	Mass of Lead Slag retained (grams)	% of Lead slag retained	% of Lead slag passing	Cumulative % retained
600 μ	0.5	0.05	99.95	0.05
425 μ	1.55	0.155	99.845	0.205
300 μ	3.65	0.365	99.635	0.57
150 μ	284	28.38	71.62	28.95
90 μ	650	64.955	35.045	93.905
75 μ	25	2.498	97.502	96.403

Pan	36	3.597	96.403	100
Total	1000.7		Summation (F) =	320
			Fineness Modulus =	3.2

Fineness Modulus of Lead Slag (Type 2) = ΣF/100 = 3.2

Table 3.8 Sieve Analysis of Type 3 (Lead Slag)

Sieve Number	Mass of Lead Slag retained (grams)	% of Lead slag retained	% of Lead slag passing	Cumulative % retained
600 µ	0.9	0.09	99.91	0.09
425 µ	2.75	0.275	99.725	0.365
300 µ	6.1	0.61	99.39	0.975
150 µ	734	73.349	26.651	74.324
90 µ	237	23.683	76.317	98.007
75 µ	8	0.799	99.201	98.806
Pan	12	1.199	98.801	100.000
Total	1000.75		Summation (F) =	372.572
			Fineness Modulus =	3.725

Fineness Modulus of Lead Slag (Type 3) = ΣF/100 = 3.725

Table 3.9 shows the different physical characteristics of lead slag. Specific gravity of Lead slag raw samples were calculated in lab by Le Chaterlier flask as per IS:4031(Part 11) 1988. The fineness of the lead slag was calculated by dry sieving through 90 micron sieve as per IS:4031 (Part 1) 1996.

Table 3.9 Physical Properties of Lead slag

Characteristics	Lead slag (Type 1)	Lead slag (Type 2)	Lead slag (Type 3)	IS Code Standards referred for Test
Property	Mix Slag	Iron Rich	Silica Rich	-
Specific Gravity	3.56	3.44	3.21	IS:4031 (Part 11) 1988
Fineness Modulus	3.235	3.2	3.725	-

The particle size distribution results by HORIBA Analyzer LA-950 is shown in Figure 3.9, 3.10, and 3.11 for three slag (Type 1, Type 2, and Type 3) respectively. Figure 3.8 shows the apparatus.

Figure 3.8 HORIBA Laser Scattering Particle size Distribution Analyzer LA-950

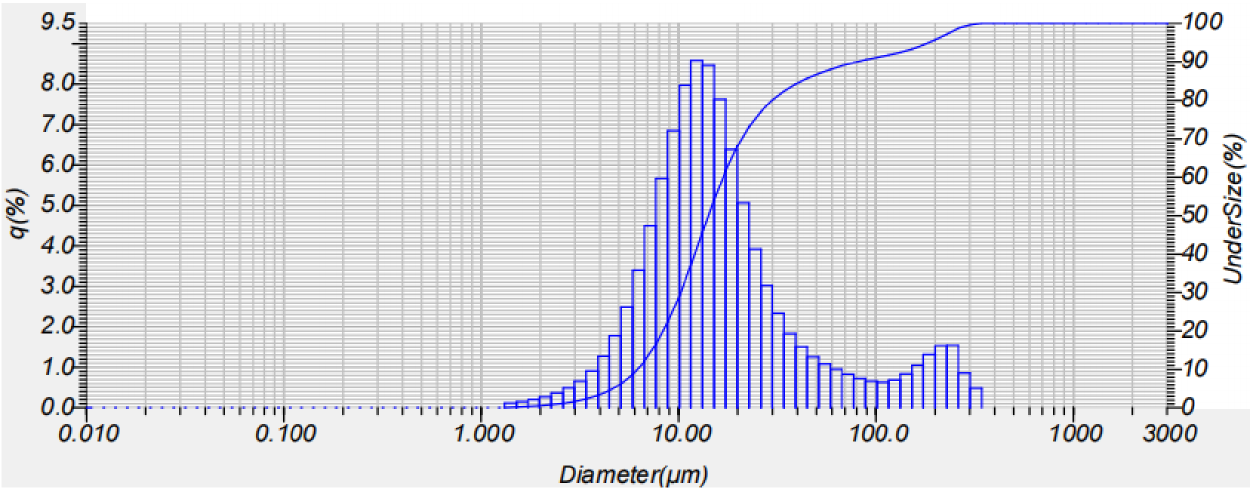


Figure 3.9 Particle size distribution curve of Lead Slag (Type 1)

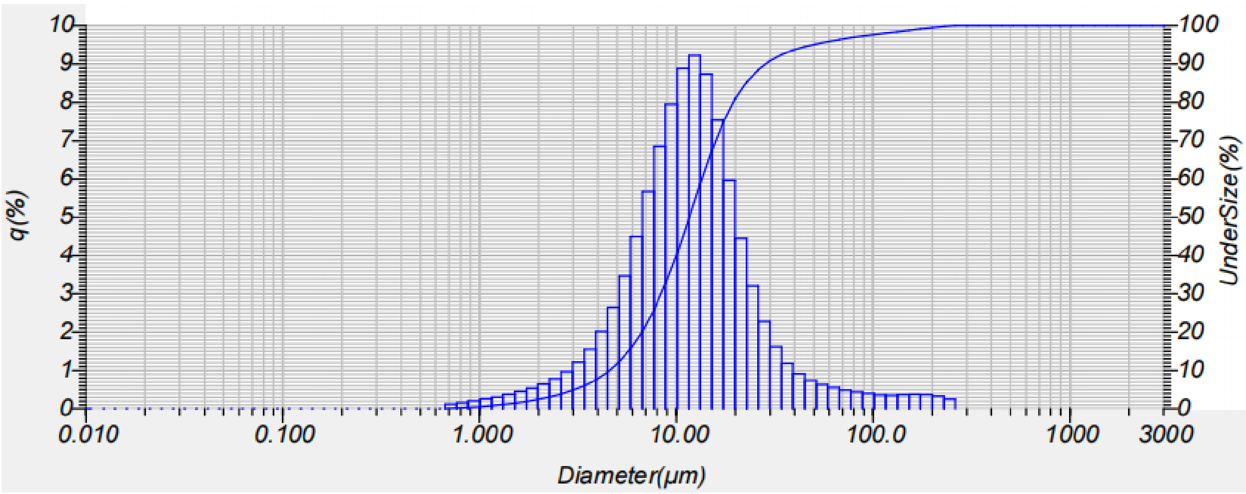


Figure 3.10 Particle size distribution curve of Lead Slag (Type 2)

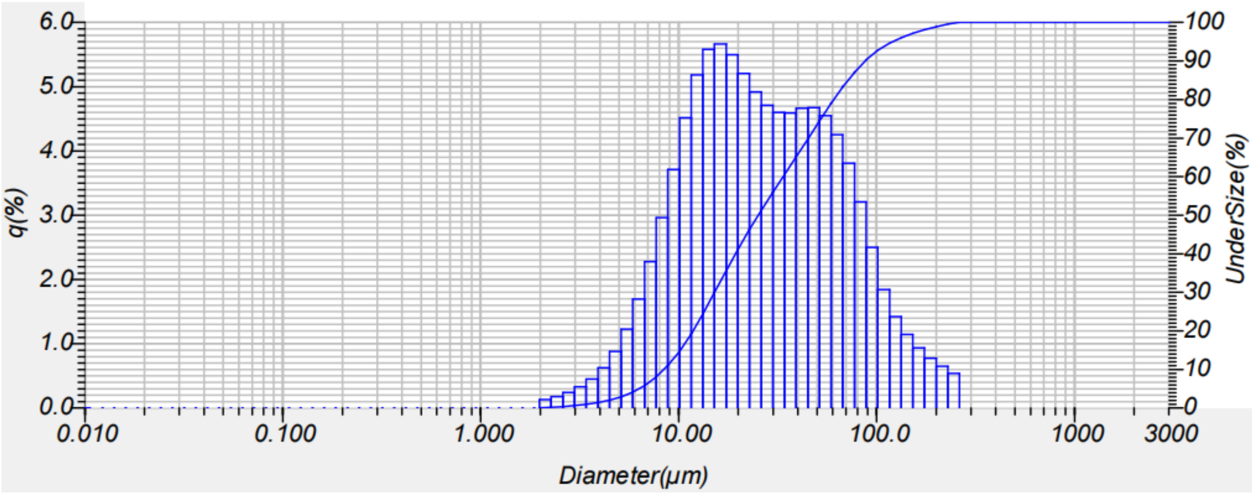


Figure 3.11 Particle size distribution curve of Lead Slag (Type 3)

3.2.3.3 Micro-structure of Lead Slag

SEM (Scanning Electron Microscopy) was performed on raw lead slag samples. EDS (Energy Dispersive X-ray Spectroscopy) was also conducted to get the different elemental concentration of the lead slag raw samples. The test is based on the idea that each element has a distinct atomic structure, which means that the peaks absorbed after excitation are also distinct. A high-energy X-ray beam was used to emit the distinctive X-rays from the raw slag samples. When a beam contacts the raw sample, the atoms in the ground state are stimulated, and the electrons in the inner shells is ejected from shells developing a electron hole. Figure 3.12 shows the apparatus.

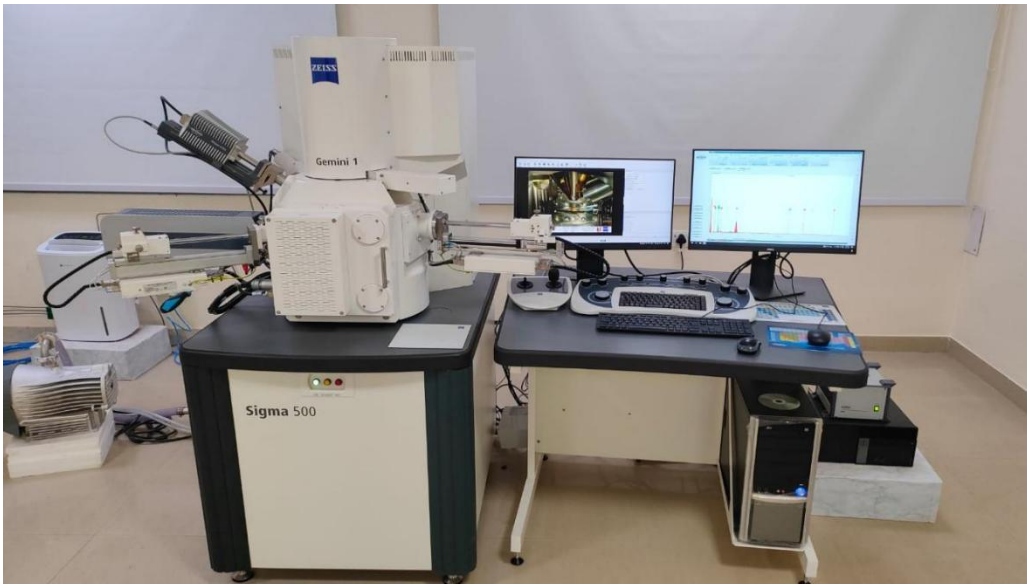


Figure 3.12 EDS (Energy Dispersive X-ray Spectroscopy) SAI Lab, Thapar University

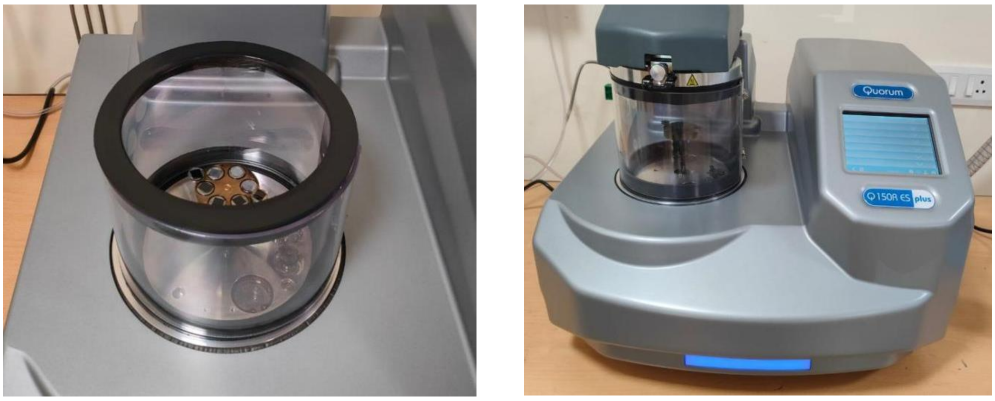


Figure 3.13 Gold plating on the Lead slag (SAI Lab, Thapar University)

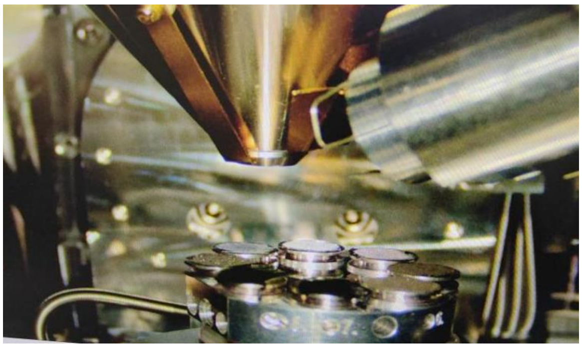


Figure 3.14 Placement of Sample in SIGMA 500

Figure 3.13 shows the procedure of the gold plating which was done on the raw samples of lead slag whose SEM and EDS was to be demonstrated, and Figure 3.14 shows the placement of the sample in the SIGMA 500.

Figure 3.15, 3.16, 3.17 shows SEM Images of Lead Slag (Type 1), (Type 2), and (Type 3) respectively. Figure 3.18, 3.19, 3.20 shows peaks observed in the Sample of Lead Slag (Type 1), (Type 2), and (Type 3) respectively.

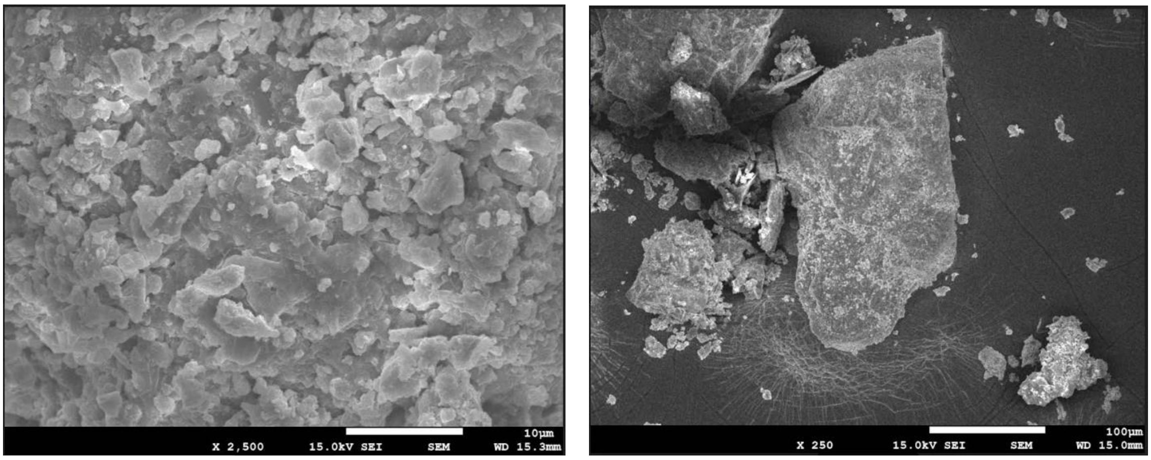


Figure 3.15 SEM Images of Lead Slag (Type 1)

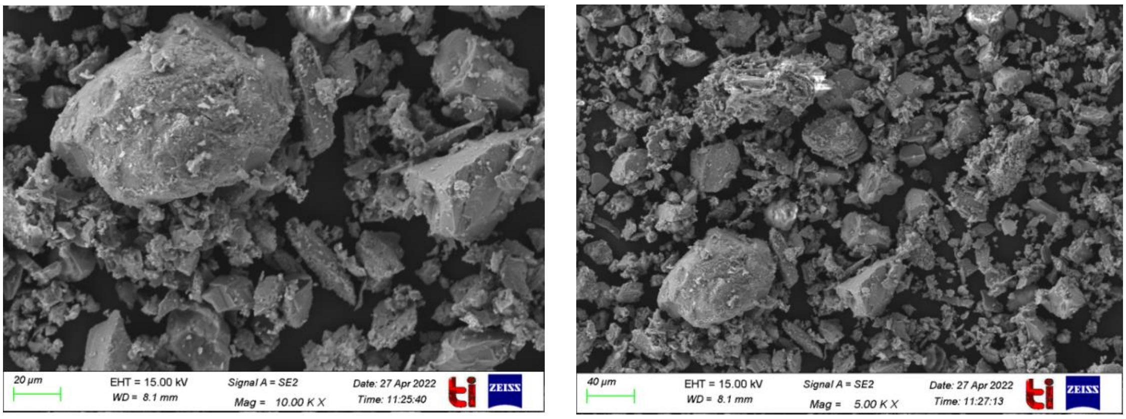


Figure 3.16 SEM Images of Lead Slag (Type 2)

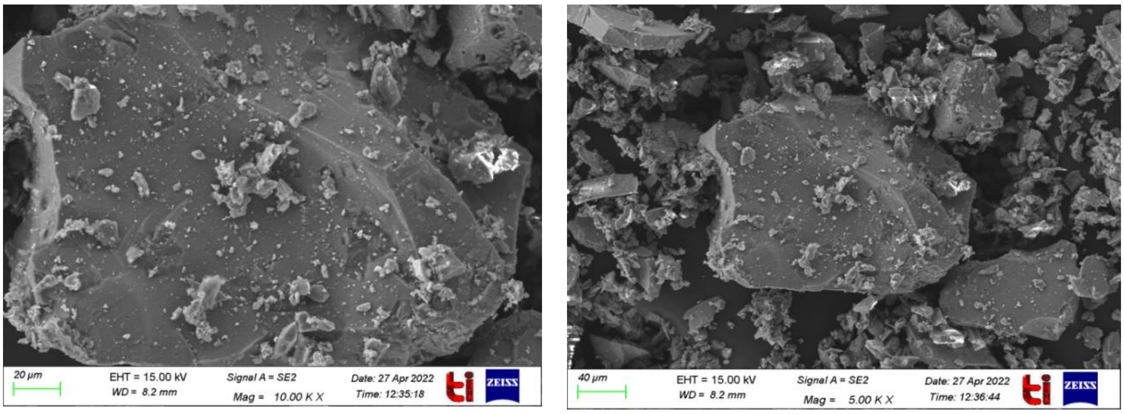


Figure 3.17 SEM Images of Lead Slag (Type 3)

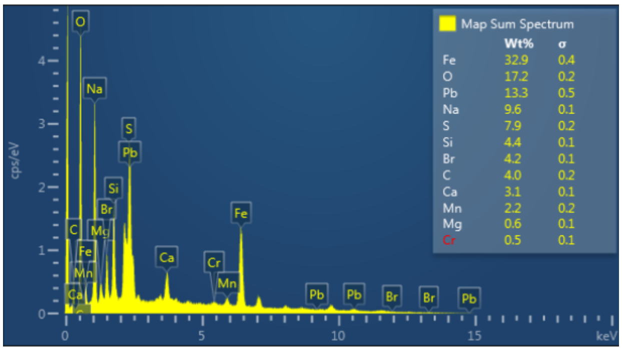


Figure 3.18 Peaks observed in the Sample of Lead Slag (Type 1) in EDS

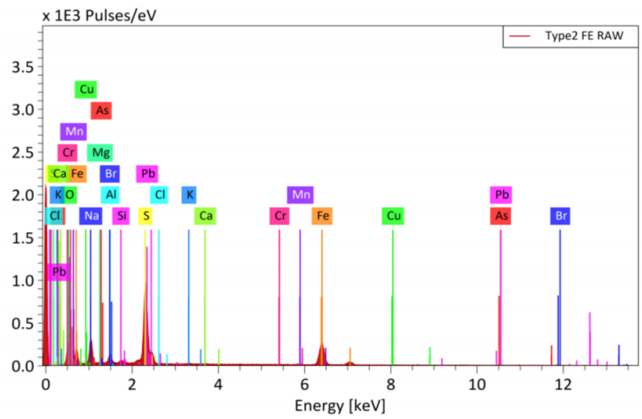


Figure 3.19 Peaks observed in the Sample of Lead Slag (Type 2) in EDS

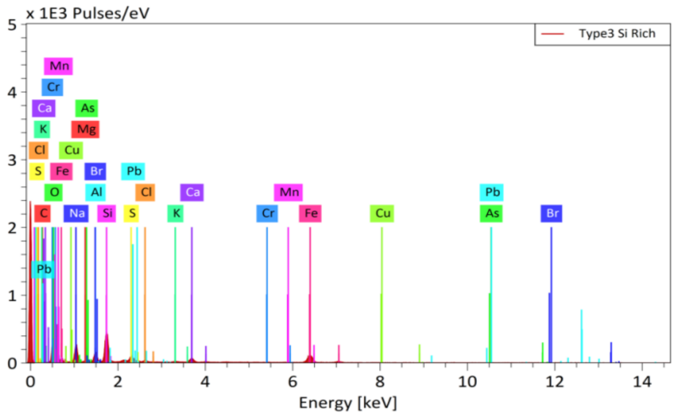


Figure 3.20 Peaks observed in the Sample of Lead Slag (Type 3) in EDS

3.2.4 Fly Ash

In present study, Class F fly ash is used throughout the inquiry. Fly- ash used was lump-free and had a greyish look. Different laboratory examinations revealed the diverse physic al properties, which are compiled in Table 3.10. Table 3.11 shows different compositions of fly-ash.

Table 3.10 Physical characteristics of Fly-ash

Properties	Value
Specific Gravity	2.87
Bulk Density (kg/m^3)	375
Colour	Grey

Table 3.11 Chemical composition Of Fly-ash

Elements	%	Elements	%	Elements	%
Al_2O_3	27.08	**S**	1.25	**Fe_2O_3**	4.11
SiO_2	53.54	**CaO**	3.90	**K_2O**	2.36
MgO	0.32	**Na_2O**	0.33		

3.2.5 Sodium Hydroxide (NaOH) and Sodium Silicate (Na_2SiO_3)

Pallets (hemispherical shape) of sodium hydroxide with diameters of 5 mm to 7 mm were employed in the investigation. It's also known as lye or caustic soda. NaOH pallets are generally crystalline solids with a high water solubility. When it is neutralised with acid or introduced to water, it reacts with the moisture in the air and releases heat. The sodium silicate used in the investigation was in liquid form. The activating solution for the preparation of geopolymer has been made with NaOH, Na_2SiO_3, and distilled water.

10 Molarity concentration of Sodium Hydroxide (NaOH) was prepared. 400 grams (Molarity x molecular weight) of NaOH pellets was dissolved in the distilled water and make up to a total of 1-liter solution. The solution was kept for cooling for 2 to 3 hours. After cooling, the sodium silicate solution of same volume was added to the solution and was kept for 24 hours and was then used in casting of specimens.

Figure 3.22 Sodium Hydroxide

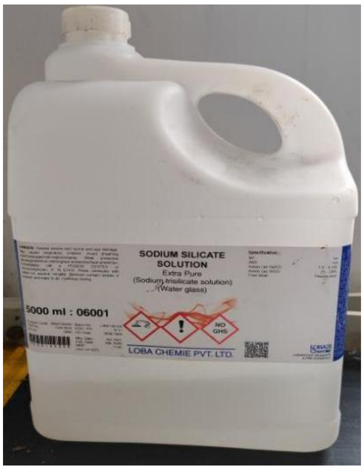


Figure 3.23 Sodium Silicate

Figure 3.24 Sodium Hydroxide Pallets

3.2.6 Water

Water is an important component of concrete/mortar because it plays an active role in the chemical reaction in cement and water. The quantity and quality of water must be carefully considered because it aids in the cement gel formation which provides strength. Potable water is generally regarded as adequate. Tap water is used in this study with a pH of 7 $\pm$1. Geopolymer mortar specimens were prepared with distilled water. The water in the mortar specimens was added considering the consistency of the cement obtained. Total water added = (p/4 +3) of the total lead slag + standard sand quantity. Here P = Percentage required to make a paste with a standard consistency.

3.3 Mix Design Proportions

3.3.1 Mix design Proportions of Lead Slag based cement mortar specimens

The mortar specimens were designed as per IS Code 1727:1967 (Methods of test for pozzolanic materials). The ratio of the binding material and the standard sand was kept at 1:3. The replacement percentages of lead slag with cement were varied from 5% to 30% (i.e., 0%, 5%, 10%, 15%, 20%, 25%, and 30%). A total of three lead slag samples were studied (Type 1, Type 2, and Type 3) and were replaced with cement. The specimens for each mixture with a variable amount of lead slags were then casted. The replacement of the lead slag with cement was done by equivalent volume substitution. Equivalent volume substitution was done by considering the Specific gravity of the pozzolana material (i.e., Lead Slag) and the Specific Gravity of the OPC 43 Grade 1 Cement. The pozzolana-cement mortar specimens shall contain Pozzolana (Lead Slag): Cement (OPC Grade 43): Standard Sand in the proportion of xN: 1: 3 confirming to IS Code. (x = Percentage replacement of lead slag with cement).

$$N = \frac{\text{Specific Gravity of Lead Slag}}{\text{Specific Gravity of Cement}}$$

Specific Gravity of Ordinary Portland cement (Grade 43) = 3.0438.

Specific Gravity of Lead Slag (Type 1) = 3.56.

Specific Gravity of Lead Slag (Type 2) = 3.44.

Specific Gravity of Lead Slag (Type 3) = 3.21.

N for Lead Slag (Type 1) = N_1 = 1.1695.

N for Lead Slag (Type 2) = N_2 = 1.13.

N for Lead Slag (Type 3) = N_3 = 1.055.

The water in the mortar specimens was added considering the consistency of the cement obtained, which was 28.5%. Total water added = (p/4 + 3) of the total binder + standard sand quantity.

P = Percentage required to make a paste with a standard consistency.

Total water = $\frac{P}{4} + 3 = \frac{28.5}{4} + 3$ = 10.125% of (Lead Slag + Standard Sand). Different Specimens ID's for all three types of lead slag based mortar specimens were designated for different mixes for simplicity. Table 3.12 shows the designed different Mix ID's for three types of lead slag and the different proportions of Lead Slag: Cement: Standard Sand.

Table 3.12 Name and proportions for designed Specimen ID's

%replacemnt of Cement	Specimen ID (Type 1)	Lead Slag : Cement : Standard Sand	Specimen ID (Type 2)	Lead Slag : Cement : Standard Sand	Specimen ID (Type 3)	Lead Slag : Cement : Standard Sand
0%	Control	$0N_1$: 1: 3	Control	$0N_2$: 1: 3	Control	$0N_3$: 1: 3
5%	T1-LS5	$0.05N_1$:0.95:3	T2-LS5	$0.05N_2$:0.95:3	T3-LS5	$0.05N_3$:0.95:3
10%	T1-LS10	$0.1N_1$:0.90:3	T2-LS10	$0.1N_2$:0.90:3	T3-LS10	$0.1N_3$:0.90:3
15%	T1-LS15	$0.15N_1$:0.85:3	T2-LS15	$0.15N_2$:0.85:3	T3-LS15	$0.15N_3$:0.85:3
20%	T1-LS20	$0.2N_1$:0.80: 3	T2-LS20	$0.2N_2$:0.80: 3	T3-LS20	$0.2N_3$:0.80: 3
25%	T1-LS25	$0.25N_1$:0.75:3	T2-LS25	$0.25N_2$:0.75:3	T3-LS25	$0.25N_3$:0.75:3
30%	T1-LS30	$0.30N_1$:0.70:3	T2-LS30	$0.30N_2$:0.70:3	T3-LS30	$0.30N_3$:0.70:3

In the above table T1, T2, T3 depicts the three types of lead slag (TI for Type 1, T2 for Type 2, and T3 for Type 3). LS denotes the Lead Slag replacement percentage (i.e., 0%, 5%, 10%, 15%, 20%, 25%, and 30%). Table 3.13 shows the mix proportions of mortar specimens containing Lead Slag (Type 1).

Table 3.13 Mix Proportions of Mortar Specimens containing Lead Slag (Type 1)

Lead Slag (Type 1) % Replacement of Cement	Specimen ID (Type 1)	Ordinary Portland Cement - 43 Grade (g)	Lead Slag (Type 1) (g)	Standard Sand (g)			Added Water (g)
				Grade 1	Grade 2	Grade 3	
0%	Control	500	0	500	500	500	202.5
5%	T1-LS5	475	29.23	500	500	500	202.93
10%	T1-LS10	450	58.48	500	500	500	203.36
15%	T1-LS15	425	87.72	500	500	500	203.79
20%	T1-LS20	400	116.96	500	500	500	204.22
25%	T1-LS25	375	146.19	500	500	500	204.65
30%	T1-LS30	350	175.44	500	500	500	205.08

Table 3.14 shows the mix proportions of mortar specimens containing Lead Slag (Type 2).

Table 3.14 Mix Proportions of Mortar Specimens containing Lead Slag (Type 2)

Lead Slag (Type 2) % Replacement of Cement	Specimen ID (Type 2)	Ordinary Portland Cement - 43 Grade (g)	Lead Slag (Type 2) (g)	Standard Sand (g)			Added Water (g)
				Grade 1	Grade 2	Grade 3	
0%	Control	500	0	500	500	500	202.5
5%	T2-LS5	475	28.25	500	500	500	202.83
10%	T2-LS10	450	56.51	500	500	500	203.16
15%	T2-LS15	425	84.76	500	500	500	203.49
20%	T2-LS20	400	113.02	500	500	500	203.82
25%	T2-LS25	375	141.27	500	500	500	204.15
30%	T2-LS30	350	169.52	500	500	500	204.48

Table 3.15 shows the mix proportions of mortar specimens containing Lead Slag (Type 3).

Table 3.15 Mix Proportions of Mortar Specimens containing Lead Slag (Type 3)

Lead Slag (Type 3) % Replacement of Cement	Specimen ID (Type 3)	Ordinary Portland Cement - 43 Grade (g)	Lead Slag (Type 3) (g)	Standard Sand (g)			Added Water (g)
				Grade 1	Grade 2	Grade 3	
0%	Control	500	0	500	500	500	202.5
5%	T3-LS5	475	26.36	500	500	500	202.64
10%	T3-LS10	450	52.73	500	500	500	202.78
15%	T3-LS15	425	79.09	500	500	500	202.91
20%	T3-LS20	400	105.46	500	500	500	203.05
25%	T3-LS25	375	131.83	500	500	500	203.19
30%	T3-LS30	350	158.19	500	500	500	203.33

3.3.2 Mix design Proportions of Lead Slag based geopolymer mortar specimens

Unlike pozzolana-cement mortar, geopolymer mortar has no standard mix design approaches. The geopolymer mortar specimens were designed after preparing various trial mixes. The ratio of fly-ash and the standard sand was kept at 1:3. The replacement percentages of lead slag with fly-ash were varied from 10% to 30% (i.e., 0%, 10%, 20%, and 30%). A total of three lead slag samples were studied (Type 1, Type 2, and Type 3) and were replaced with fly-ash. The specimens for each mixture with a variable amount of lead slags were then casted. The replacement of the lead slag with cement was done by equivalent volume substitution. Equivalent volume substitution was done by considering the Specific gravity of Lead Slag and the Specific Gravity of Fly-ash. The geopolymer mortar specimens contain Lead Slag: Fly-ash: Standard Sand in the proportion of xM: 1: 3 confirming to IS Code. (x = Percentage replacement of lead slag with fly-ash).

$$M = \frac{\text{Specific Gravity of Lead Slag}}{\text{Specific Gravity of Fly Ash}}$$

Specific Gravity of Fly Ash = 2.87.

Specific Gravity of Lead Slag (Type 1) = 3.56.

Specific Gravity of Lead Slag (Type 2) = 3.44.

Specific Gravity of Lead Slag (Type 3) = 3.21.

M for Lead Slag (Type 1) = M_1 = 1.24.

M for Lead Slag (Type 2) = M_2 = 1.1986.

M for Lead Slag (Type 3) = M_3 = 1.1185.

The activating solution for the preparation of geopolymer mortar specimens was made by Sodium hydroxide pallets, Sodium silicate, and distilled water.

10 Molarity concentration of NaOH was prepared. 400 grams (Molarity x molecular weight) of NaOH pellets were dissolved in the distilled water and make up to a total of 1-liter solution. The solution was kept for cooling for 2 to 3 hours. After cooling, the sodium silicate solution of same weight was added to the solution and was kept for 24 hours and was then used in casting of specimens. Different Specimens ID's for all three types of lead slag based mortar specimens were designated for different mixes for simplicity.

Table 3.16 shows the designed different Mix ID's for three types of lead slag and the different proportions of Lead Slag : Fly-Ash : Standard Sand. In the table GP denotes Geopolymer, and T1, T2, T3 depicts the three types of lead slag (TI for Type 1, T2 for

Type 2, and T3 for Type 3). LS denotes the Lead Slag replacement percentage (i.e., 0%, 10%, 20%, and 30%).

Table 3.16 Name and proportions for designed Specimen ID's

%replacemnt of Fly Ash	**Specimen ID (Type 1)**	**Lead Slag : Fly-Ash : Standard Sand**	**Specimen ID (Type 2)**	**Lead Slag : Fly-Ash : Standard Sand**	**Specimen ID (Type 3)**	**Lead Slag : Fly-Ash : Standard Sand**
0%	GP-Control	$0M_1$: 1: 3	GP-Control	$0M_2$: 1: 3	GP-Control	$0M_3$: 1: 3
10%	GP-T1-LS10	$0.1M_1$:0.90:3	GP-T1-LS10	$0.1M_2$:0.90:3	GP-T3-LS10	$0.1M_3$:0.90 3
20%	GP-T1-LS20	$0.2M_1$:0.80: 3	GP-T2-LS20	$0.2M_2$:0.80: 3	GP-T3-LS20	$0.2M_3$:0.80:3
30%	GP-T1-LS30	$0.3M_1$:0.70: 3	GP-T2-LS30	$0.3M_2$:0.70: 3	GP-T3-LS30	$0.3M_3$:0.70:3

Table 3.17 shows the mix proportions of mortar specimens containing Lead Slag (Type 1).

Table 3.17 Mix Proportions of Geopolymer Mortar Specimens containing Lead Slag (Type 1)

Lead Slag (Type 1) % Replacement of Fly Ash	**Specimen ID (Type 1)**	**Fly-Ash (g)**	**Lead Slag (Type 1) (g)**	**Standard Sand (g)**			**Activating Solution (g)**
				Grade 1	**Grade 2**	**Grade 3**	
0%	GP- Control	450	0	450	450	450	225
10%	GP-T1-LS10	405	51.3	450	450	450	225
20%	GP-T1-LS20	360	102.6	450	450	450	225
30%	GP-T1-LS30	315	153.9	450	450	450	225

Table 3.18 shows the mix proportions of mortar specimens containing Lead Slag (Type 2).

Table 3.18 Mix Proportions of Geopolymer Mortar Specimens containing Lead Slag (Type 2)

Lead Slag (Type 2) % Replacement of Fly Ash	Specimen ID (Type 2)	Fly-Ash (g)	Lead Slag (Type 2) (g)	Standard Sand (g)			Activating Solution (g)
				Grade 1	Grade 2	Grade 3	
0%	GP- Control	450	0	450	450	450	225
10%	GP-T2-LS10	405	53.94	450	450	450	225
20%	GP-T2-LS20	360	107.87	450	450	450	225
30%	GP-T2-LS30	315	161.81	450	450	450	225

Table 3.19 shows the mix proportions of mortar specimens containing Lead Slag (Type 1).

Table 3.19 Mix Proportions of Geopolymer Mortar Specimens containing Lead Slag (Type 3)

Lead Slag (Type 3) % Replacement of Fly Ash	Specimen ID (Type 3)	Fly-Ash (g)	Lead Slag (Type 3) (g)	Standard Sand (g)			Activating Solution (g)
				Grade 1	Grade 2	Grade 3	
0%	GP- Control	450	0	450	450	450	225
10%	GP-T3-LS10	405	50.33	450	450	450	225
20%	GP-T3-LS20	360	100.67	450	450	450	225
30%	GP-T3-LS30	315	150.99	450	450	450	225

3.4 Specimen Preparations

3.4.1 Lead Slag based mortar specimens

The lead slag-based cement mortar specimens were prepared using the mix proportion in section 3.3.1. Two eighty five lead slag based cement mortar specimens were casted to investigate the different mechanical properties. Out of which, 171 were cubical specimens of 50 mm to determine the compressive strength at 7, 28 and 56 days, and 114 prism specimens of 40 mm x 40 mm x 160 mm to determine the flexural strength at 7 and 28 days . Seven lead slag replacement percentage (i.e., 0%, 5%, 10%, 15%, 20%, 25%, 30%) are considered in the research work.

To determine the compressive strength, mortar cubes were casted according to IS: 1727. For each set of a mixture, nine specimens were casted. A total of three samples has been tested for compressive strength for different curing period. An extra 10% of the material was considered for the preparation of cubical specimens so to avoid any complications in casting.

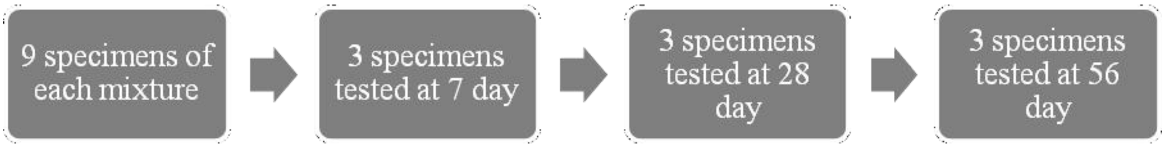


Figure 3.25 Compressive strength assessment chart of mortar cubical specimens

To determine the flexural strength, 40mm x 40mm x 160mm prism specimens were casted according to IS: 1727. For each set of a mixture, six specimens were casted. The testing was done at two different curing ages (i.e., 7 days and 28 days). A total of three specimens were tested for flexural strength for each curing period. An extra 5% of the material was considered for the preparation of the prism specimens so to avoid any complications in casting.

Figure 3.26 Flexural strength assessment chart of mortar prism specimens

The procedure of preparation of specimens was as follows-

1. The raw materials were kept at room temperature. All the materials were weighed with great accuracy and precision according to the requirement of mix design. The three grades of standard sand, cement, lead slag, and water were the key ingredients of the mortar specimens.

Figure 3.27 Three Grades of Standard Sand

2. The cubical and prism moulds were cleaned properly It was ensured that no lumps or traces of mortar or any other material were there on the mould. It was made ensure that the interior faces are smooth are cleaning.

3. A thin film of the oil was provided on the interior faces of the mould. After this, the screws were tightened in order to get the perfect dimensions of the cube or prism moulds. And it was also ensured that no leakage of mortar occur.

Figure 3.28 Cubical and Prism Moulds

4. Each set of mortar mix was mixed in the mechanical mortar mixing machine (Digi mortar mixer). Firstly only the raw materials were added, and the mixing was done in a dry state. After dry mixing, the water was then added, and the digi mixer was started at low speed and was mixed for 30 seconds.

Figure 3.29 Digi-mortar mixer

5. Then the mixer was stopped, and adhered mortar along the walls of the bowl was removed and, with the help of trowel, was collected at the bottom of the bowl and was placed in the middle. After this, the mixture was set at high speed, and mixing was done for 1 minute.
6. After mixing, the process of casting was started. The cubical and prism moulds were filled. The excess of the mortar was removed from the mould after filling. The vibration of the mould was done on a compaction machine for one minute.

Figure 3.30 Casting of Cubical and Prism Moulds

7. After the vibration, the mould were kept for drying for a period of 24 hours in the room temperature. At last, the samples were demoulded, and were kept in curing tank for different ages of curing (7, 28, and 56 days).

Figure 3.31 Demoulding

3.4.2 Lead Slag based geopolymer mortar specimens

The lead slag-based geopolymer mortar specimens were prepared using the mix proportion in section 3.3.2. Sixty lead slag based geopolymer mortar specimens were casted to investigate the compressive strength at elevated temperature at 3 and 7 days curing. To determine the compressive strength, mortar cubes of 50 mm cube were casted. For each set of a mixture, six specimens were casted. The testing was done at two different ages. A total of three samples has been tested for compressive strength for different ages. An extra 10% of the material was considered for the preparation of cubical specimens so to avoid any complications in casting.

Figure 3.32 Compressive strength assessment chart of geopolymer mortar cubical specimens

Procedure of preparation of specimens was as follows-

1. The raw materials were kept at room temperature. All the materials were weighed with great accuracy and precision according to the requirement of mix design.
2. The cubical moulds were appropriately cleaned. It was ensured that no lumps or traces of geopolymer mortar or any other material were there on the mould. It was made to ensure that the interior faces are smooth are clean.
3. A thin film of the oil was provided on the interior faces of the mould. After this, the screws were tightened in order to get the perfect dimensions of the cube or prism moulds. And it was also ensured that no leakage of mortar occur.
4. Each set of geopolymer mortar mixes was mixed in the mechanical mortar mixing machine (Digi mortar mixer). Firstly only the raw materials were added, and the mixing was done in a dry state. After dry mixing, the activating solution was then added, and the mixer was started at low speed and was mixed for 30 seconds.
5. Then, the mechanical mixer was stopped, and adhered geopolymer mortar along the walls of the bowl was removed and, with the help of a trowel, was collected at the bottom of the bowl and was placed in the middle. After this, the mixture was set at high speed, and mixing was done for 1 minute.
6. After mixing, the process of casting was started. The cubical moulds were filled. The excess of the mortar was removed from the mould after filling. The vibration of the mould was done on a compaction machine for one minute.

Figure 3.33 Casting of Cubical Moulds

Figure 3.34 Mould wrapped with a thin film of plastic polythene

7. After the vibration, the mould was wrapped with a thin film of plastic polythene and were kept in the oven at 70^0 Celsius for 24 hours.

Figure 3.35 Moulds kept in oven for 24 hours at 70^0 C (Elevated Temperature Curing)

8. At last, the specimens were demoulded from the mould and were kept for different ages of testing (i.e., 3 and 7 days) at room temperature.

Figure 3.36 Demoulding

3.5 Tests Conducted

This section shows different types of tests conducted on lead slag-based cement mortar specimens and geopolymer mortar specimens to demonstrate the mechanical properties. TCLP test was also demonstrated to measure toxicity level.

3.5.1 Compressive Strength [IS: 1727 (Part 10):1967]

Compressive strength was determined for the mortar cubical specimens. Compressive strength of lead slag-based cement mortar specimens and geopolymer-based mortar specimens were demonstrated as per IS: 1727 (Part 10):1967. Cubical samples of size 50mm x 50mm x 50mm were tested. The compressive strength of lead slag-based cement mortar specimens were tested for 7, 28, and 56 days. The compressive strength of geopolymer based mortar specimens were tested for 3 and 7 days. Test was conducted in ACTM (Automatic Compression Testing Machine), which had total capacity of 5000 kN. At the time of testing of the specimens, the loading rate was 0.1 kN/sec. The side face of the cubical sample was put in an upward direction under direct load from CTM. The reason for keeping the side face at the top is because the surface is smooth because at time of casting it was touched by the face of the moulds. And the top face of the specimen was kept at the front face. Figure 3.37 shows the apparatus of the Compressive Testing Machine.

Figure 3.37 CTM for Compression Strength Test

The compressive strength was calculated by the formula below-

$$\text{Compressive Strength (MPa)} = \frac{\text{Load}}{\text{Area}}$$

$$= \frac{\text{P x 1000}}{\text{50 x 50}} \text{ MPa}$$

P = Load at breakage in CTM

3.5.2 Flexural Strength [IS: 1727 (Part 11):1967]

The flexural strength test was performed on the mortar prism specimens. The flexural strength of the lead slag-based cement mortar specimens were demonstrated as per IS: 1727 (Part 11):1967. Prism samples of size 40mm x 40mm x 160mm were tested. The flexural strength of lead slag-based cement mortar specimens were tested for 7 and 28 days. Test was conducted in Flexural Machine, having a total capacity of 100 kN. At the time of testing of the specimens, the loading rate was 0.03 kN/sec. The three-point load test has been done to determine flexural strength. Figure 3.36 shows the apparatus of the Flexural Testing Machine by three point load test.

Figure 3.38 Flexural Testing Machine for determining Flexural Strength

Figure 3.39 shows the prism setup for three-point loading.

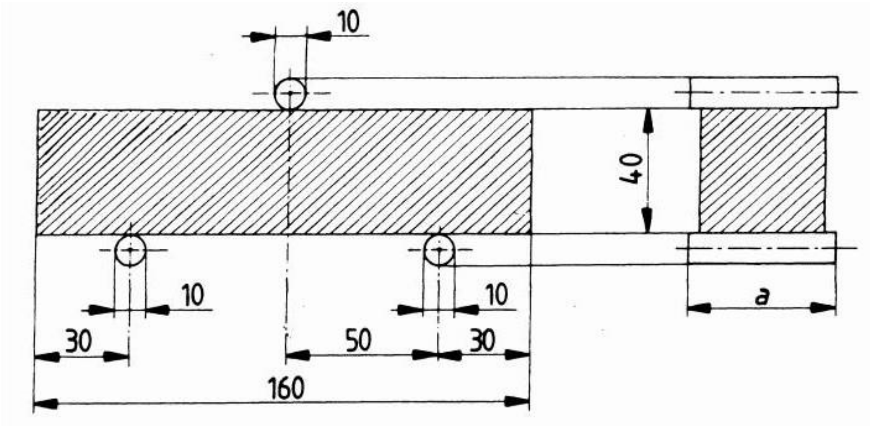


Figure 3.39 Dimensions of Prism setup for three point load test

Flexural strength was calculated by the formula below-

Flexural Strength (MPa) = $\frac{M}{Z}$

M = Maximum Bending Moment (N.mm) = $\frac{P \text{ x } 160}{4}$

Z = Section Modulus (mm^3) = $\frac{a^3}{6}$

P = Load in which prism break (N)

a = Length of prism = Breadth of prism

Figure 3.40 Specimens after 3 point load test

3.6 Closing Remarks

The materials and methods explained in this chapter includes the different physical and chemical properties of materials used, the mix designing proportions, the sample preparations, and procedure of the different test carried.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

The present contains detailed description of the result, a complete examination of experimental program was carried out by replacing cement with different types of lead slag in lead slag-based cement mortar specimens. Seven lead slag replacement percentage (i.e., 0%, 5%, 10%, 15%, 20%, 25%, and 30%) were taken. The different types of lead slag was placed with fly-ash in lead slag-based geopolymer mortar specimens. Four lead slag replacement percentage (i.e., 0%, 10%, 20%, and 30%) were taken. The different mechanical properties discussed in this chapter are compressive and flexural strength.

4.2 Compressive Strength of Lead Slag based cement Mortar Specimens

After sample preparation, specimens were kept in the curing tank and tested in uni-axial CTM. The lead slag-based cement mortar specimens are tested at different curing ages of 7, 28, and 56 days. The total of three specimens has been casted for each mix, and the average of the three was considered the compressive strength of sample.

4.2.1 Compressive Strength of Type 1 Lead Slag based cement mortar specimens

Table 4.1 shows data set of the Compressive Strength of Lead Slag (Type 1) based cement mortar Specimen at different ages of curing.

Table 4.1 Compressive Strength of Lead Slag (Type 1) based cement mortar Specimen at different ages

Specimen ID	No. of specimens	7 days (MPa)	28 days (MPa)	56 days (MPa)
Control	1	31.12	34	38.28
	2	28.72	34.76	38.52
	3	27.08	34.8	37.12
	Average	**28.97**	**34.52**	**37.97**
	S.D.	2.0301	0.4508	0.7487
T1-LS5-CS	1	31.46	32.64	36.08
	2	28.6	32.88	34.96
	3	26.36	32.39	34

	Average	**28.81**	**32.64**	**35.01**
	S.D.	2.5583	0.244	1.041
T1-LS10-CS	**1**	25.61	31	32.72
	2	23.48	28.56	31.6
	3	26.45	29.93	31.92
	Average	**25.18**	**29.83**	**32.08**
	S.D.	1.5309	1.2252	0.5769
T1-LS15-CS	**1**	21.22	24.16	30.24
	2	22.5	26.65	29.24
	3	23.92	26.8	30.6
	Average	**22.54**	**25.87**	**30.03**
	S.D.	1.3486	1.4833	0.7047
T1-LS20-CS	**1**	20.88	23.2	28.96
	2	17.64	21.6	27.8
	3	19.19	21.62	27.48
	Average	**19.24**	**22.14**	**28.08**
	S.D.	1.6165	0.9169	0.7787
T1-LS25-CS	**1**	17.74	21.1	28.28
	2	15.2	19.24	25.84
	3	15.71	19.87	26.12
	Average	**16.22**	**20.07**	**26.75**
	S.D.	1.3441	0.9423	1.3352
T1-LS30-CS	**1**	13.7	17.81	27.28
	2	15.46	16.79	20.12
	3	13.47	16.94	21.2
	Average	**14.21**	**17.18**	**22.87**
	S.D.	1.0867	0.55171	3.86

Table 4.2 shows the percentage variation in compressive Strength of Lead Slag (Type 1) based cement mortar Specimens at different ages of curing.

Table 4.2 % variation in compressive Strength of Lead Slag (Type 1) based cement mortar Specimen at different ages

Specimen ID	C.S. of cubical mortar specimens (MPa)					
	7 days	% var	28 days	% var	56 days	% var
Control	28.97	Reference	34.52	Reference	37.97	Reference
T1-LS5-CS	28.81	-0.56	32.64	-5.45	35.01	-7.8
T1-LS10-CS	25.18	-13.09	29.83	-13.59	32.08	-15.52
T1-LS15-CS	22.54	-22.18	25.87	-25.06	30.03	-20.93
T1-LS20-CS	19.24	-33.6	22.14	-35.86	28.08	-26.05
T1-LS25-CS	16.22	-44.01	20.07	-41.86	26.75	-29.56
T1-LS30-CS	14.21	-50.96	17.18	-50.23	22.87	-39.78

(Note: (+) positive sign indicates increase in C.S., and (–) negative indicates decrease of C.S.)

Figure 4.1 shows the graphical representation of the Compressive Strength of Lead Slag (Type 1) based cement mortar Specimens with the varying lead slag percentages of 0%, 5%, 10%, 15%, 20%, 25%, and 30%.

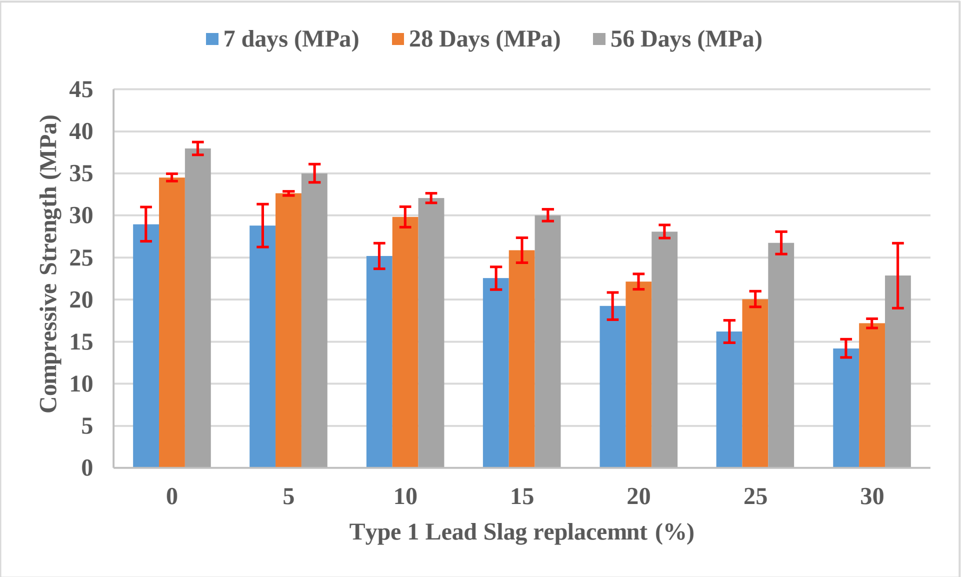


Figure 4.1 Compressive Strength vs Type 1 Lead Slag replacement (%)

Figure 4.2 shows graphical representation of Compressive Strength of Lead Slag (Type 1) based cement mortar Specimens with the different curing ages of 7, 28, and 56 days.

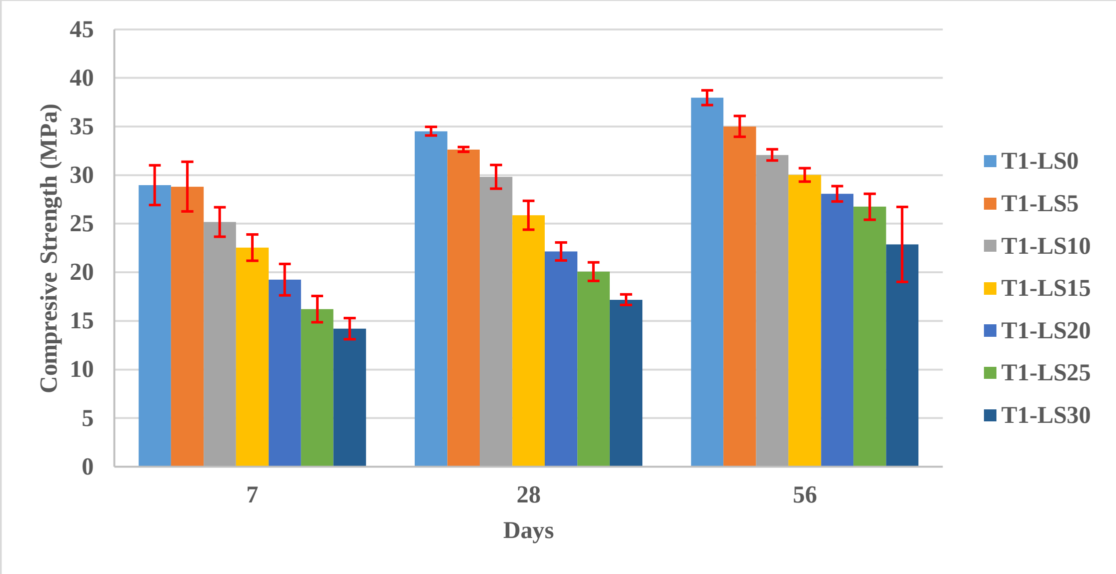


Figure 4.2 Compressive Strength vs Age of mortar specimens

The following observations can been made from Table 4.1 & 4.2, and Figure 4.1 & 4.2.

- Clearly, compressive strength of mortar specimens decreases with an increase in lead slag replacement (%) at all ages and increases with the age of curing.
- Compressive strength of mortar specimens consisting of 5% (T1-LS5) lead Slag (Type1) is comparable with the control specimen having 0% of lead slag (Control) at all ages of testing, particularly at 7 days as the strength decreases only by 0.56%. At 28 days, a decrease of 5.45% is observed, and at 56 days, a decrease of 7.8% is observed.
- Further increase in the replacement of Lead Slag after 5%, the results are not promising as compressive strength decreases rapidly.
- At 7 days of testing, with 10% replacement with OPC, compressive strength decreases by 13.09% (T1-LS10). With 15% replacement, the strength decreases by 22.18% (T1-LS15). With further replacement of 20%, 25%, and 30%, the decrease in compressive strength is 33.65% (T1-LS20), 44.01% (T1-LS25), and 50.96% (T1-LS30) respectively.
- At 28 days of testing, with 10% replacement of lead slag with OPC, compressive strength decreases by 13.59%. With 15% replacement with OPC, compressive strength decreases by 25.06%. And beyond this compressive strength decreases tremendously by 35.86%, 41.86%, and 50.23% for 20%, 25%, and 30% replacement of lead slag respectively.
- At 56 days of age, with 10% replacement with OPC, compressive strength decreases by 15.52%. With 15% replacement, the compressive strength decreases by 20.93%. With further replacement of 20%, 25%, and 30%, the decrease in compressive strength is

26.05%, 29.56%, and 39.78% respectively. The strength development at 56 days was better as compared at 28 days curing age.

- In contrast to other specimen ID's, the strength development in T1-LS5 is mainly due to more uniform calcium silicate hydrate (C-S-H) gel which is generally about 60% of the paste volume resulting in the less voids and denser configuration (impermeability), leading to good strength. This gel forms a continuous layer that binds the particles of cement together and creates a strong and cohesive structure. As a result of excessive amount of lead slag in the cement mortar specimens, there is an increase in the void and lack of C-S-H gel is evident in specimens beyond 5% substitution of Lead Slag (Type 1).
- The compressive strength increase with the age of curing, as the pozzolana reaction occurs at greater curing age. The calcium hydroxide produced is very important in breaking the glassy phase of the lead slag, which leads to the pozzolana reaction. This reason is also reported by Penpolcharoen (2005).
- Also the Lead Slag (Type 1) contains different metals in it which are responsible for the negative impact on the hydration mechanism. This is due to the presence of metals (Pb, Zn) present in lead slag leading in formation of the layer on dehydrated cement particles preventing hydration process.This reason is also reported by Cioffi *et al.* (2000).
- Also, the slow hydration of Lead Slag (Type 1) is due to presence of high silica and alumina, which can form an impermeable coating around the particle of the lead slag in the hydration process and ultimately decreasing compressive strength.

4.2.2 Compressive Strength of Type 2 Lead Slag based cement mortar specimens

Table 4.3 shows the data set of the Compressive Strength of Lead Slag (Type 2) based cement mortar Specimens at different ages of curing.

Table 4.3 Compressive Strength of Lead Slag (Type 2) based cement mortar Specimens at different age of curing

Specimen ID	No. of specimens	7 days (MPa)	28 days (MPa)	56 days (MPa)
Control	**1**	31.12	34	38.28
	2	28.72	34.76	38.52
	3	27.08	34.8	37.12

	Average	**28.97**	**34.52**	**37.97**
	S.D.	2.0301	0.4508	0.7487
T2-LS5-CS	**1**	27.28	31.48	33.7
	2	24.75	29.92	33.8
	3	25.12	32	32.94
	Average	**25.72**	**31.13**	**33.48**
	S.D.	1.3671	1.0824	0.4726
T2-LS10-CS	**1**	24.16	26.08	29.92
	2	22.52	27.47	28.92
	3	22.24	28.32	30.36
	Average	**22.97**	**27.29**	**29.73**
	S.D.	1.0371	1.1306	0.7379
T2-LS15-CS	**1**	19.36	21.32	26.16
	2	16.84	23.08	25.28
	3	18.65	22.04	26.24
	Average	**18.28**	**22.15**	**25.89**
	S.D.	1.2991	0.8848	0.5326
T2-LS20-CS	**1**	15.04	20.4	22.64
	2	13.52	18.88	21.56
	3	13.74	18.6	23.08
	Average	**14.1**	**19.29**	**22.43**
	S.D.	0.8223	0.9685	0.7821
T2-LS25-CS	**1**	11.76	18	20.92
	2	12.52	16.56	21.78
	3	13.02	15.84	19.68
	Average	**12.43**	**16.8**	**20.79**
	S.D.	0.6326	1.0998	1.0557
T2-LS30-CS	**1**	12.2	15.2	18.28
	2	11.52	13.64	17.68
	3	10.56	14.6	16.36
	Average	**11.43**	**14.48**	**17.44**
	S.D.	0.8242	0.7868	0.9822

Table 4.4 shows the percentage variation in compressive Strength of Lead Slag (Type 2) based cement mortar Specimens at different ages of curing.

Table 4.4 % variation in compressive Strength of Lead Slag (Type 2) based cement mortar Specimens at different age of curing

Specimen ID	C.S. of cubical mortar specimens (MPa)					
	7 days	% var	28 days	% var	56 days	% var
Control	28.97	Reference	34.52	Reference	37.97	Reference
T2-LS5-CS	25.72	-11.24	31.13	-9.81	33.48	-11.84
T2-LS10-CS	22.97	-20.7	27.29	-20.95	29.73	-21.7
T2-LS15-CS	18.28	-36.89	22.15	-35.84	25.89	-31.82
T2-LS20-CS	14.09	-51.33	19.29	-44.11	22.43	-40.94
T2-LS25-CS	12.43	-57.09	16.8	-51.33	20.79	-45.24
T2-LS30-CS	11.43	-60.55	14.48	-58.05	17.44	-54.07

(Note: (+) positive sign indicates increase in C.S., and (–) negative indicates decrease of C.S.)

Figure 4.3 shows the graphical representation of the Compressive Strength of Lead Slag (Type 2) based cement mortar Specimens with the varying lead slag percentages of 0%, 5%, 10%, 15%, 20%, 25%, and 30%.

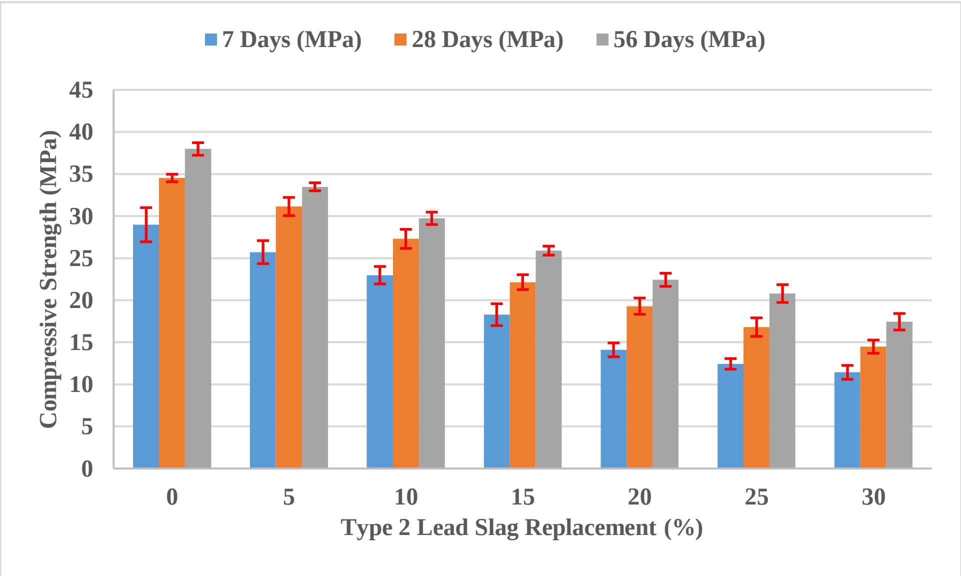


Figure 4.3 Compressive Strength vs Type 2 Lead Slag replacement (%)

Figure 4.4 shows graphical representation of Compressive Strength of Lead Slag (Type 2) based cement mortar Specimens with the different curing ages of 7, 28, and 56 days.

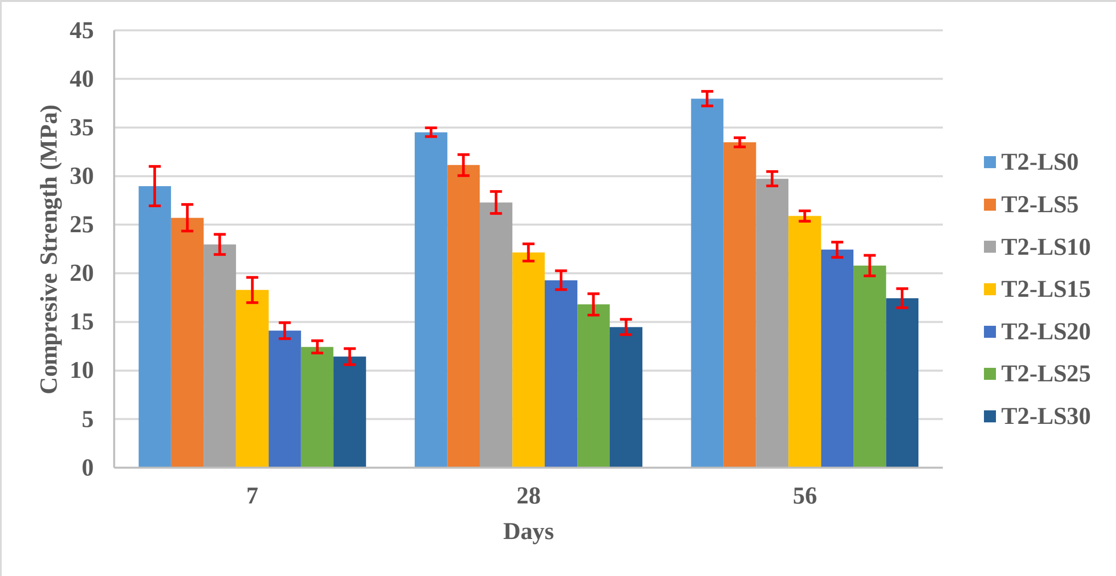


Figure 4.4 Compressive Strength vs Age of mortar specimens

The following observations can been made from Table 4.3 & 4.4, and Figure 4.3 & 4.4.

- Clearly, compressive strength of mortar specimens decreases with an increase in lead slag replacement (%) at all ages and increases with the age of curing.
- Compressive strength of mortar specimens consisting of 5% (T2-LS5) lead Slag (Type2) is not comparable with the control specimen having 0% of lead slag (Control) at all ages of testing, which is comparable in Lead Slag (Type 1) based mortar specimens. Here at 7 days, the strength decreases by 11.24%. At 28 days, a decrease of 9.81% is observed, and at 56 days, a decrease of 11.84% is observed.
- Further increase in the replacement of Lead Slag after 5%, the results are not promising as compressive strength decreases drastically in this type of slag.
- At 7 days of testing, with 10% replacement with OPC, compressive strength decreases by 20.7% (T2-LS10). With 15% replacement, the strength decreases by 36.89% (T2-LS15). With further replacement of 20%, 25%, and 30%, the decrease in compressive strength is 51.33% (T2-LS20), 57.09% (T2-LS25), and 60.55% (T2-LS30) respectively.
- At 28 days of testing, with 10% replacement of lead slag with OPC, compressive strength decreases by 20.95%. With 15% replacement with OPC, compressive strength decreases by 35.84%. And beyond this compressive strength decreases tremendously by 44.11%, 51.33%, and 58.05% for 20%, 25%, and 30% replacement of lead slag respectively.
- At 56 days of age, with 10% replacement with OPC, compressive strength decreases by 21.7%. With 15% replacement, the compressive strength decreases by 31.82%. With further replacement of 20%, 25%, and 30%, the decrease in compressive strength is

40.94%, 45.24%, and 54.07% respectively. The strength development at 56 days were better as compared at 28 days curing age, and strength development of 28 days were better than 7 days.

- The strength development in 5% replacement or beyond replacement of Lead Slag (Type 2) is less as compared to control specimen due to non-uniform calcium silicate hydrate (C-S-H) gel which is generally about 60% of the paste volume resulting in the more voids and less denser configuration (permeability), leading to lesser strength. As a result of excessive amount of lead slag in the cement mortar specimens, there is an increase in the void and lack of C-S-H gel is evident in specimens containing substitution of Lead Slag (Type 2).
- The compressive strength increase with the age of curing, as the pozzolana reaction occurs at greater curing age. The calcium hydroxide produced is very important in breaking the glassy phase of the lead slag, which leads to the pozzolana reaction. This reason is also reported by Penpolcharoen (2005).
- Also the Lead Slag (Type 2) contains different metals in it which are responsible for the negative impact on the hydration mechanism. This is due to the presence of metals (Pb, Zn) present in lead slag leading in formation of the layer on dehydrated cement particles preventing hydration process.This reason is also reported by Cioffi *et al.* (2000).
- Also, the lead slag (Type 2) used in the research has less calcium oxide than OPC 43. So when equal volume of OPC is replaced by Calcium oxide, the net content of CaO decreases leading in the slow hydration process resulting is less strength.

4.2.3 Compressive Strength of Type 3 Lead Slag based cement mortar specimens

Table 4.5 shows the data set of the Compressive Strength of Lead Slag (Type 3) based cement mortar Specimens at different ages of curing.

Table 4.5 Compressive Strength of Lead Slag (Type 3) based cement mortar Specimens at different age of curing

Specimen ID	No. of specimens	7 days (MPa)	28 days (MPa)	56 days (MPa)
Control	**1**	31.12	34	38.28
	2	28.72	34.76	38.52
	3	27.08	34.8	37.12

	Average	**28.97**	**34.52**	**37.97**
	S.D.	2.03	0.4508	0.7487
T3-LS5-CS	**1**	28.65	30.8	34.5
	2	26.35	31.8	34.6
	3	27.04	32.4	33.74
	Average	**27.35**	**31.67**	**34.28**
	S.D.	1.17832	0.8083	0.4726
T3-LS10-CS	**1**	25	30.76	30.72
	2	24.59	28.44	31.72
	3	25.56	29.6	32.36
	Average	**25.05**	**29.6**	**31.6**
	S.D.	0.4859	1.16	0.8265
T3-LS15-CS	**1**	24.32	26.52	28.96
	2	22.16	27.32	28.48
	3	23.4	26	27.44
	Average	**23.29**	**26.61**	**28.29**
	S.D.	1.0839	0.664	0.777
T3-LS20-CS	**1**	20.52	25.52	27.44
	2	21.24	23.4	25.56
	3	20.28	24.14	25.08
	Average	**20.68**	**24.35**	**26.03**
	S.D.	0.499	1.0763	1.2472
T3-LS25-CS	**1**	15.32	20.24	21.88
	2	18.5	21.24	21.28
	3	17.6	21.2	23.16
	Average	**17.14**	**20.89**	**22.11**
	S.D.	1.6408	0.5662	0.9603
T3-LS30-CS	**1**	13	15.8	19.32
	2	14.56	18.16	17.38
	3	13.6	15.16	18.48
	Average	**13.72**	**16.37**	**18.39**
	S.D.	0.7851	1.58	0.9729

Table 4.6 shows the percentage variation in compressive Strength of Lead Slag (Type 3) based cement mortar Specimens at different ages of curing.

Table 4.6 % variation in compressive Strength of Lead Slag (Type 3) based cement mortar Specimens at different age of curing

Specimen ID	C.S. of cubical mortar specimens (MPa)					
	7 days	% var	28 days	% var	56 days	% var
Control	28.97	Reference	34.52	Reference	37.97	Reference
T3-LS5-CS	27.35	-5.61	31.67	-8.26	34.28	-9.73
T3-LS10-CS	25.05	-13.53	29.6	-14.25	31.6	-16.78
T3-LS15-CS	23.29	-19.6	26.61	-22.91	28.29	-25.49
T3-LS20-CS	20.68	-28.62	24.35	-29.46	26.03	-31.46
T3-LS25-CS	17.14	-40.83	20.89	-39.48	22.11	-41.78
T3-LS30-CS	13.72	-52.64	16.37	-52.57	18.39	-51.56

(Note: (+) positive sign indicates increase in C.S., and (–) negative indicates decrease of C.S.)

Figure 4.5 shows the graphical representation of the Compressive Strength of Lead Slag (Type 3) based cement mortar Specimens with the varying lead slag percentages of 0%, 5%, 10%, 15%, 20%, 25%, and 30%.

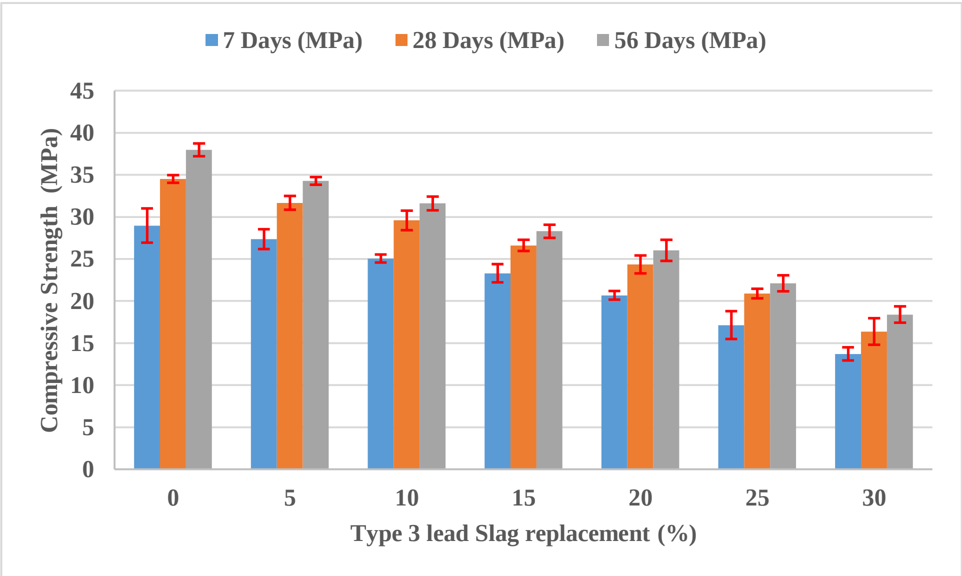


Figure 4.5 Compressive Strength vs Type 3 Lead Slag replacement (%)

Figure 4.6 shows graphical representation of Compressive Strength of Lead Slag (Type 3) based cement mortar Specimens with the different curing ages of 7, 28, and 56 days.

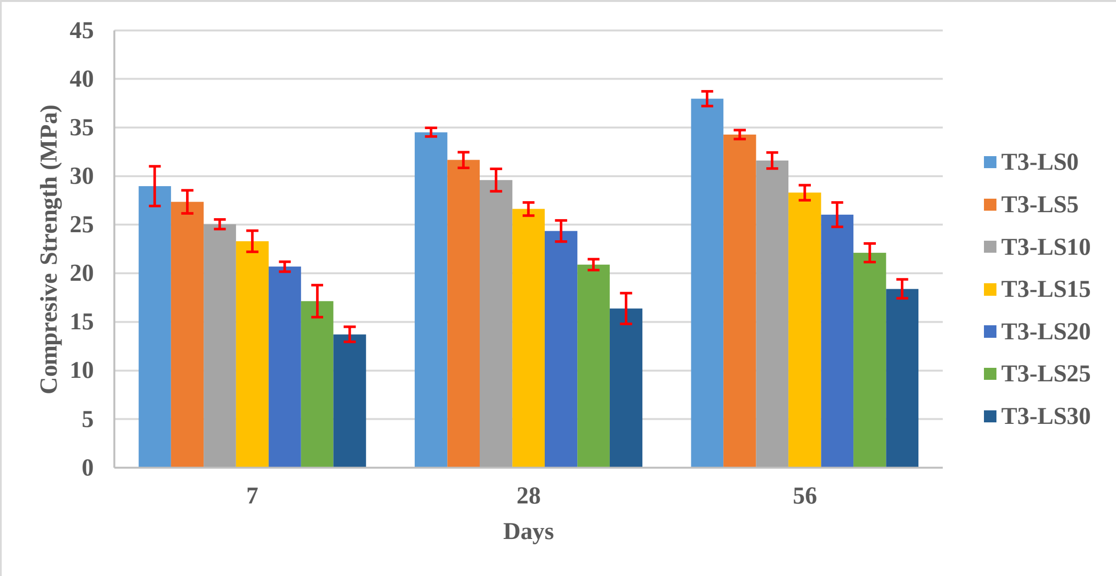


Figure 4.6 Compressive Strength vs Age of mortar specimens

The following observations can been made from Table 4.5 & 4.6, and Figure 4.5 & 4.6.

- Clearly, compressive strength of mortar specimens decreases with an increase in lead slag replacement (%) at all ages and increases with the age of curing.
- Compressive strength of mortar specimens consisting of 5% (T3-LS5) lead Slag (Type1) is somewhat comparable with the control specimen having 0% of lead slag (Control) at all ages of testing, particularly at 7 days testing where the strength decreases by 5.61%. At 28 days, a decrease of 8.26% is noticed, and at 56 days, a decrease of 9.73% is noticed. Further increase in the replacement of Lead Slag after 5%, the results are not promising as compressive strength decreases vulnerably.
- At 7 days of testing, with 10% replacement with OPC, compressive strength decreases by 13.53% (T3-LS10). With 15% replacement, the strength decreases by 19.6% (T3-LS15). With further replacement of 20%, 25%, and 30%, the decrease in compressive strength is 28.62% (T3-LS20), 40.83% (T3-LS25), and 52.64% (T3-LS30) respectively.
- At 28 days of testing, with 10% replacement of lead slag with OPC, compressive strength decreases by 14.25%. With 15% replacement with OPC, compressive strength decreases by 22.91%. And beyond this compressive strength decreases tremendously by 29.46%, 39.48%, and 52.57% for 20%, 25%, and 30% replacement of lead slag respectively.
- At 56 days of age, with 10% replacement with OPC, compressive strength decreases by 16.78%. With 15% replacement, the compressive strength decreases by 25.49%. With further replacement of 20%, 25%, and 30%, the decrease in compressive strength is

31.46%, 41.78%, and 51.56% respectively. The strength development of 28 days were better as compared at 56 days curing age and that of 7 days are better than 28 days.

- The strength development in 5% replacement or beyond replacement of Lead Slag (Type 3) is less as compared to control specimen due to non-uniform calcium silicate hydrate (C-S-H) gel which is generally about 60% of the paste volume resulting in the more voids and less denser configuration (permeability), leading to lesser strength. As a result of excessive amount of lead slag in the cement mortar specimens, there is an increase in the void and lack of C-S-H gel is evident in specimens containing substitution of Lead Slag (Type 3).
- The compressive strength increase with the age of curing, as the pozzolana reaction occurs at greater curing age. The calcium hydroxide produced is very important in breaking the glassy phase of the lead slag (Type 3), which leads to the pozzolana reaction. The same reason is also reported by Penpolcharoen (2005).
- Also the Lead Slag (Type 3) contains different metals in it which are responsible for the negative impact on the hydration mechanism. This is due to the presence of metals (Pb, Zn) present in lead slag leading in formation of the layer on dehydrated cement particles preventing hydration process.This reason is also reported by Cioffi *et al.* (2000).
- Also, the slow hydration of Lead Slag (Type 3) is due to presence of high silica and alumina, which can form an impermeable coating around the particle of the lead slag in the hydration process and ultimately decreasing compressive strength.

4.2.4 Comparison between the Compressive behavior of three types of Lead Slag

In this section the comparison in the compressive behavior of the three types lead slag based cement mortar specimens is done in order to determine the best among the three. Figure 4.7 shows the variation of 7 days compressive strength of all types.

Clearly the 7 days compressive strength of mortar specimens containing 5% lead Slag is highest of Type 1 Lead Slag and is somewhat comparable with the control specimen. The strength development is far better than Type 2 and Type3 lead Slag is mainly due to more uniform calcium silicate hydrate (C-S-H) gel resulting in the less voids and denser configuration, leading to good strength. Further replacement in Lead slag decreases the compressive strength drastically in all three slags. All types of Lead Slag contains various metals in it which are responsible for the negative impact on the hydration mechanism as the metals are responsible for early setting and hardening. But the 7 days strength development is better in Type 1 and Type 3 based cement mortar specimens as compared to Type 2 Lead Slag based mortar specimens.

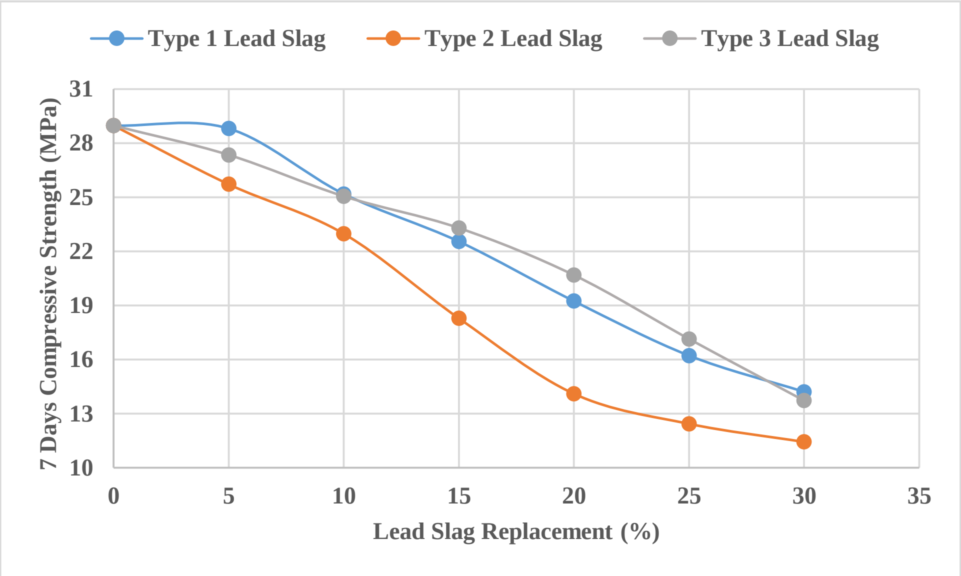


Figure 4.7 Variation of 7 Days Compressive Strength of three types of lead slag

Figure 4.8 shows the variation of 28 days compressive strength of all types. Clearly the 28 days compressive strength of Type 1 and Type 3 Lead Slag based cement mortar specimens is far better than Type 2 Lead Slag based cement mortar specimens at all levels of replacement. Type 1 and Type 3 based specimens have almost similar strength development at 28 days. The possible reason for the slow hydration in Type 2 is mainly due to less CaO content as compared to Type 1 and Type 2 lead slag resulting in slow hydration mechanism.

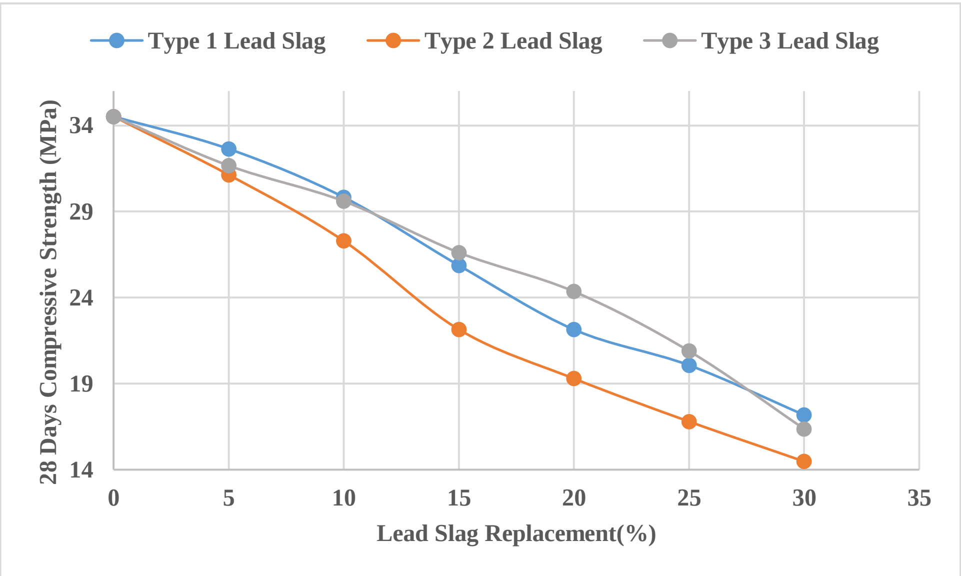


Figure 4.8 Variation of 28 Days Compressive Strength of three types of lead slag

Figure 4.9 shows the variation of 56 days compressive strength of all types. Clearly the 56 days compressive strength of Type 1 lead slag based mortar specimens is better than Type 2 and Type 3 lead slag based mortar specimens at all levels of replacement. The pozzolanic reaction in Type 1 lead slag based specimens is much better than the other two types. The calcium hydroxide formed is very important in breaking the glassy phase of the lead slag, which leads to the pozzolana reaction. So the pozzolana reaction is best in Type 1 and is least in Type 2. So the Type 1 Lead Slag is highly recommended

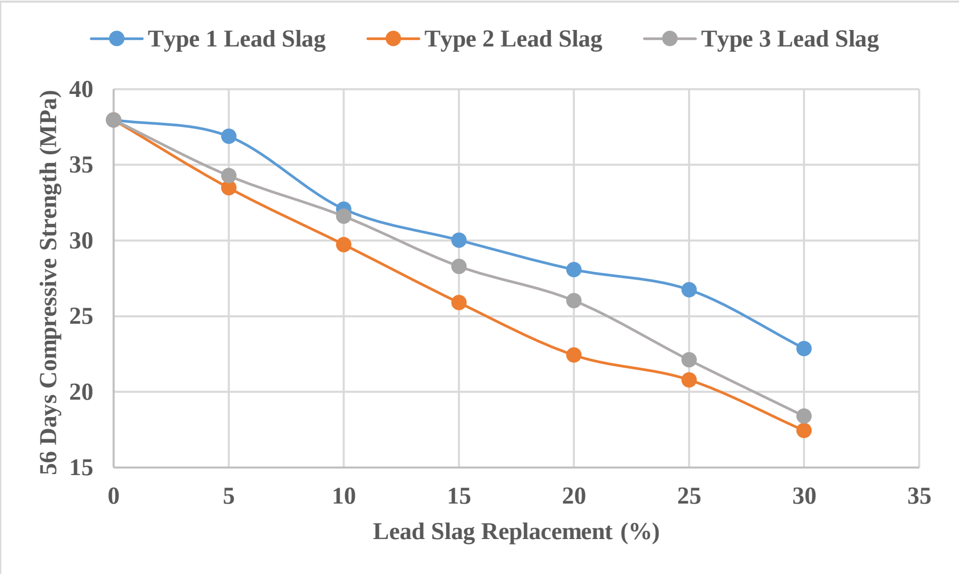


Figure 4.9 Variation of 56 Days Compressive Strength of three types of lead slag

4.3 Flexural Strength of Lead Slag based cement Mortar Specimens

After sample preparation, specimens were kept in the curing tank and were tested in a flexural testing machine. The lead slag-based cement mortar specimens were tested at 7 and 28 days. The three specimens were casted for each mix, and the average of the three was considered the flexural strength of the specimen.

4.3.1 Flexural Strength of Type 1 Lead Slag based cement Mortar Specimens

Table 4.7 shows the data set of the Flexural Strength and percentage variation of Lead Slag (Type 1) based cement mortar Specimens at different ages of curing.

Table 4.7 Flexural Strength of Lead Slag (Type 1) based cement mortar Specimens at different age of curing

Specimen ID	7 days (MPa)	7 days (MPa) Avg.	S.D.	% var	28 days (MPa)	28 days (MPa) Avg.	S.D.	% var
Control	9.94	9.81	0.1317	Refe-rence	11.93	12.39	0.4346	Refe-rence
	9.68				12.45			
	9.83				12.79			
T1-LS5-FS	8.18	8.91	0.8389	-9.17	11.18	10.98	0.7697	-11.4
	8.74				11.63			
	9.83				10.13			
T1-LS10-FS	7.54	8.25	0.9008	-15.93	10.05	9.64	0.9509	-22.2
	9.26				10.31			
	7.95				8.55			
T1-LS15-FS	7.88	7.5	0.3437	-23.57	9.49	9.05	0.4009	-26.94
	7.43				8.96			
	7.20				8.7			
T1-LS20-FS	6.94	7.29	0.3564	-25.73	8.13	8.53	0.3402	-31.18
	7.28				8.66			
	7.65				8.78			
T1-LS25-FS	5.96	6.74	0.6969	-31.34	8.25	7.71	0.4836	-37.74
	6.94				7.58			
	7.31				7.31			
T1-LS30-FS	7.20	6.6	0.5064	-31.85	7.76	7.44	0.3189	-39.96
	6.68				7.43			
	6.19				7.13			

(Note: (+) positive sign indicates increase in C.S., and (–) negative indicates decrease of C.S.)

Figure 4.10 shows the graphical representation of the Flexural Strength of Lead Slag (Type 1) based cement mortar Specimens with the varying lead slag percentages of 0%, 5%, 10%, 15%, 20%, 25%, and 30%.

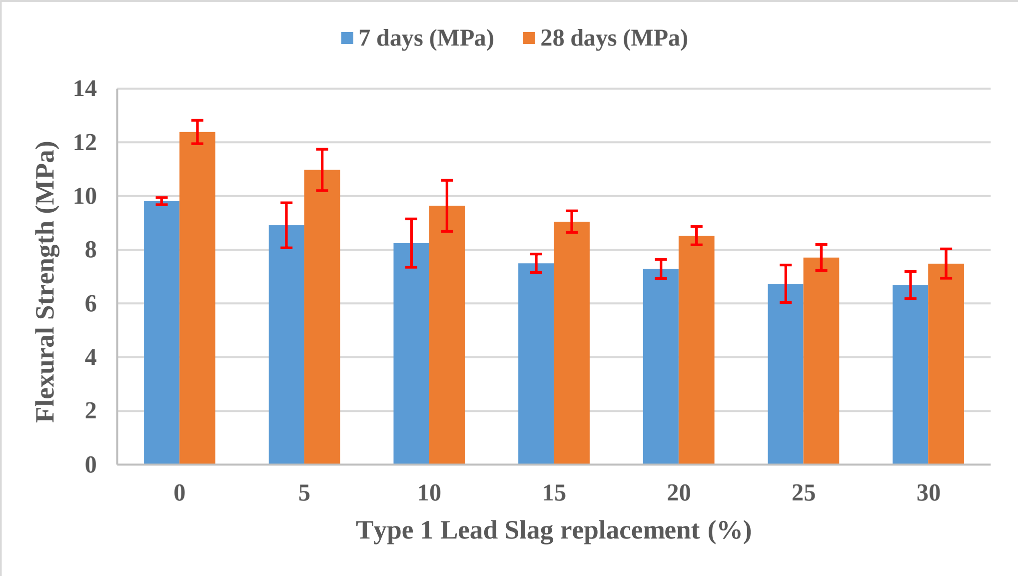


Figure 4.10 Flexural Strength vs Type 1 Lead Slag replacement (%)

Figure 4.11 shows the graphical representation of the Flexural Strength of Lead Slag (Type 1) based cement mortar Specimens with the different curing ages of 7 and 28 days.

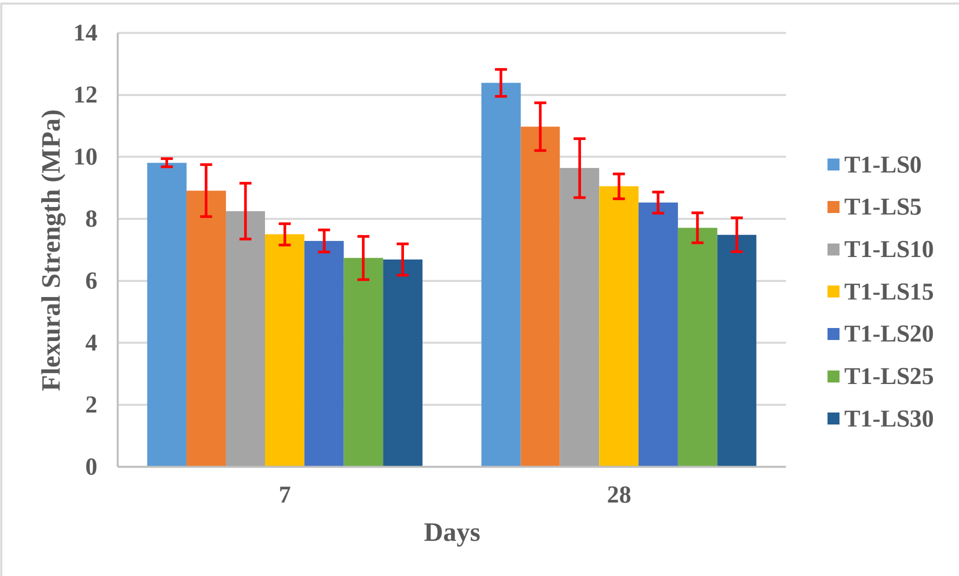


Figure 4.11 Flexural Strength vs Age of mortar specimens

Following observations can be made from Table 4.7 and Figure 4.10 & 4.11:

- Clearly, flexural strength of lead slag based mortar specimens decreases with an increase in the lead slag replacement (%) at all ages, and increase with the different age of curing.
- It is found that the flexural strength of mortar specimens consisting of all replacement of Lead slag (Type 1) is not comparable with control specimen (Control). The results of the flexural strength are not promising as it decreases rapidly.

- At 7 days of testing, with 5% replacement with OPC, flexural strength decreases by 9.17% (T1-LS5). With 10% replacement, the strength decreases by 15.93% (T1-LS15), and with 15% replacement of lead slag the decrease in strength is noticed as 23.57% (T1-LS15). With further replacement of 20%, 25%, and 30%, the decrease in flexural strength is 25.73% (T1-LS20), 31.34% (T1-LS25), and 31.85% (T1-LS30) respectively.
- At 28 days of testing, with 5% replacement with OPC, flexural strength decreases by 11.4% (T1-LS5). With 10% replacement, the strength decreases by 22.2% (T1-LS15), and with 15% replacement of lead slag the decrease in strength is noticed as 26.94% (T1-LS15). And beyond this flexural strength decreases tremendously by 31.18% (T1-LS20), 37.74% (T1-LS25), and 39.96% (T1-LS30) for 20%, 25%, and 30% replacement of lead slag respectively. The flexural strength development at 7 days was better as compared at 28 days curing age but strength decreases drastically.
- The strength development in 5% replacement or beyond replacement of Lead Slag (Type 1) is less as compared to control specimen due to non-uniform calcium silicate hydrate (C-S-H) gel which is generally about 60% of the paste volume resulting in the more voids and less denser configuration (permeability), leading to lesser strength. As a result of excessive amount of lead slag in the cement mortar specimens, there is an increase in the voids and lack of C-S-H is evident in the specimens containing replacement of Lead Slag (Type 1).
- The flexural strength increase with the age of curing, as the pozzolana reaction occurs at greater curing age. The calcium hydroxide produced is very important in breaking the glassy phase of the lead slag, which leads to the pozzolana reaction. This reason is also reported by Penpolcharoen (2005).
- Also the Lead Slag (Type 1) contains different metals in it which are responsible for the negative impact on the hydration mechanism. This is due to the presence of metals (Pb, Zn) present in lead slag leading in formation of the layer on dehydrated cement particles preventing hydration process.This reason is also reported by Cioffi *et al.* (2000).
- Also, the slow hydration of Lead Slag (Type 1) is due to presence of high silica and alumina, which can form an impermeable coating around the particle of the lead slag in the hydration process and ultimately decreasing flexural strength.

4.3.2 Flexural Strength of Type 2 Lead Slag based cement Mortar Specimens

Table 4.8 shows the data set of the Flexural Strength and percentage variation of Lead Slag (Type 2) based cement mortar Specimens at different ages of curing.

Table 4.8 Flexural Strength of Lead Slag (Type 2) based cement mortar Specimens at different age of curing

Specimen ID	7 days (MPa)	7 days (MPa) Avg.	S.D.	% var	28 days (MPa)	28 days (MPa) Avg.	S.D.	% var
Control	9.94	9.81	0.1317	Reference	11.93	12.388	0.4346	Reference
	9.68				12.45			
	9.83				12.79			
T2-LS5-FS	7.61	7.34	0.2634	-25.22	11.10	10.46	0.6571	-15.54
	7.31				9.79			
	7.09				10.50			
T2-LS10-FS	6.23	6.06	0.1924	-38.22	8.48	8.56	0.5862	-30.87
	5.85				8.03			
	6.11				9.19			
T2-LS15-FS	5.93	5.81	0.1635	-40.76	7.99	7.88	0.2625	-36.43
	5.89				8.06			
	5.63				7.58			
T2-LS20-FS	5.81	5.6	0.1924	-42.93	7.13	6.9	0.2976	-44.3
	5.55				6.56			
	5.44				7.01			
T2-LS25-FS	4.76	4.9	0.1516	-50.07	6.86	6.7	0.1691	-45.91
	4.88				6.53			
	5.06				6.71			
T2-LS30-FS	4.43	4.2	0.1984	-57.2	6.11	5.93	0.2625	-52.17
	4.13				5.63			
	4.05				6.04			

(Note: (+) positive sign indicates increase in F.S., and (–) negative indicates decrease of F.S.)

Figure 4.12 shows the graphical representation of the Flexural Strength of Lead Slag (Type 2) based cement mortar Specimens with the varying lead slag percentages of 0%, 5%, 10%, 15%, 20%, 25%, and 30%.

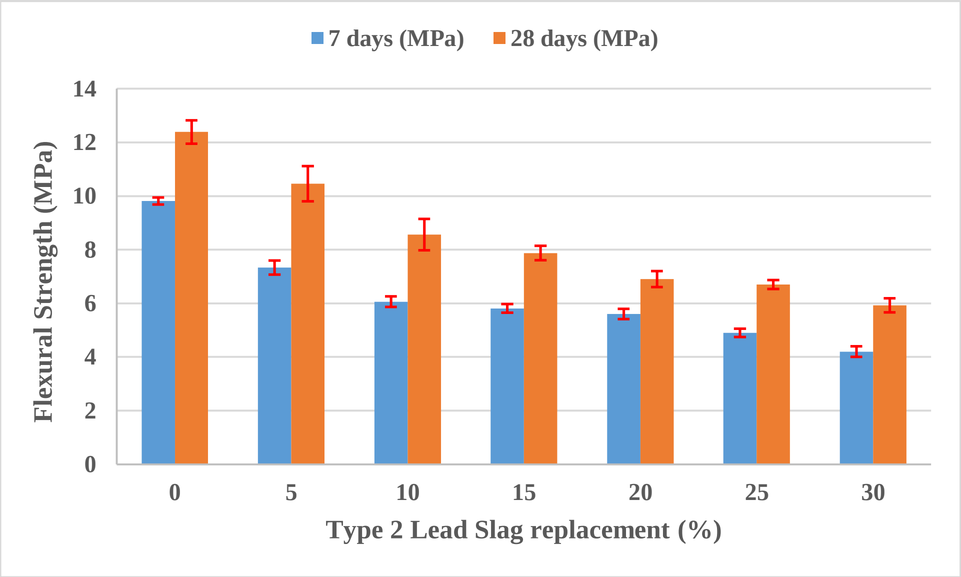


Figure 4.12 Flexural Strength vs Type 2 Lead Slag replacement (%)

Figure 4.13 shows the graphical representation of the Flexural Strength of Lead Slag (Type 2) based cement mortar Specimens with the different curing ages of 7 and 28 days.

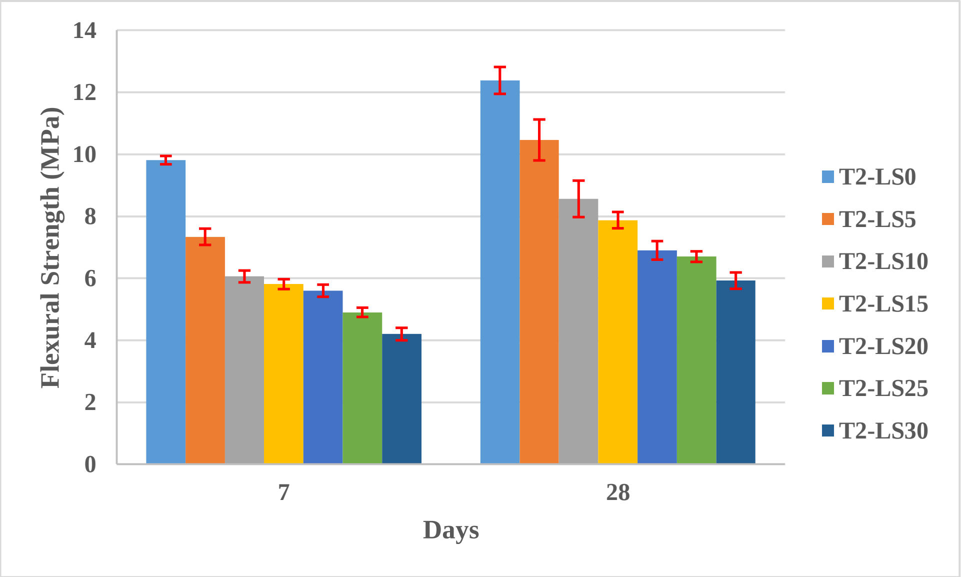


Figure 4.13 Flexural Strength vs Age of mortar specimens

Following observations can be made from Table 4.8 and Figure 4.12 & 4.13:

- Clearly, flexural strength of lead slag based mortar specimen's decreases with an increase in the lead slag replacement (%) at all ages, and increase with the different age of curing.
- It is found that the flexural strength of mortar specimens consisting of all replacement of Lead slag (Type 2) is not comparable with control specimen (Control). The results of the flexural strength are not promising as it decreases rapidly. The flexural strength observed in Type 2 Lead slag based mortar specimens is very less as compared to Type 1 Lead slag based mortar specimens.
- At 7 days of testing, with 5% replacement with OPC, flexural strength decreases by 25.22% (T2-LS5). With 10% replacement, the strength decreases by 38.22% (T2-LS15), and with 15% replacement of lead slag the decrease in strength is noticed as 40.76% (T2-LS15). With further replacement of 20%, 25%, and 30%, the decrease in flexural strength is 42.93% (T2-LS20), 50.066% (T2-LS25), and 57.2% (T2-LS30) respectively.
- At 28 days of testing, with 5% replacement with OPC, flexural strength decreases by 15.54% (T2-LS5). With 10% replacement, the strength decreases by 30.87% (T2-LS10), and with 15% replacement of lead slag the decrease in strength is noticed as 36.43% (T2-LS15). And beyond this flexural strength decreases tremendously by 44.3% (T2-LS20), 45.91% (T2-LS25), and 52.17% (T1-LS30) for 20%, 25%, and 30% replacement of lead slag respectively. The flexural strength development at 28 days was better as compared at 7 days curing age but strength decreases drastically.
- The strength development in 5% replacement or beyond replacement of Lead Slag (Type 2) is less as compared to control specimen due to non-uniform calcium silicate hydrate (C-S-H) gel which is generally about 60% of the paste volume resulting in the more voids and less denser configuration (permeability), leading to lesser strength. As a result of excessive amount of lead slag in the cement mortar specimens, there is an increase in the voids and lack of C-S-H is evident in the specimens containing replacement of Lead Slag (Type 2).
- The flexural strength increase with the age of curing, as the pozzolana reaction occurs at greater curing age. The calcium hydroxide produced is very important in breaking the glassy phase of the lead slag, which leads to the pozzolana reaction. This reason is also reported by Penpolcharoen (2005).
- Also the Lead Slag (Type 2) contains different metals in it which are responsible for the negative impact on the hydration mechanism. This is due to the presence of metals (Pb, Zn) present in lead slag leading in formation of the layer on dehydrated cement

particles preventing hydration process.This reason is also reported by Cioffi *et al.* (2000).

- Also, the lead slag (Type 2) used in the research has less calcium oxide than OPC 43. So when equal volume of OPC is replaced by Calcium oxide, the net content of CaO decreases leading in the slow hydration process resulting is less strength.

4.3.3 Flexural Strength of Type 3 Lead Slag based cement Mortar Specimens

Table 4.9 shows the data set of the Flexural Strength and percentage variation of Lead Slag (Type 3) based cement mortar Specimens at different ages of curing.

Table 4.9 Flexural Strength of Lead Slag (Type 3) based cement mortar Specimens at different age of curing

Specimen ID	7 days (MPa)	7 days (MPa) Avg.	S.D.	% var	28 days (MPa)	28 days (MPa) Avg.	S.D.	% var
Control	9.94	9.96	0.3008	Refe-rence	11.93	12.39	0.4346	Refe-rence
	9.68				12.45			
	10.28				12.79			
T3-LS5-FS	9.11	9.46	0.574	-3.57	10.8	10.14	0.9003	-18.16
	9.15				10.5			
	10.13				9.11			
T3-LS10-FS	8.48	7.81	0.7409	-20.38	8.89	8.91	0.1516	-28.05
	7.95				8.78			
	7.01				9.08			
T3-LS15-FS	7.31	7.38	0.3233	-24.85	8.29	8.33	0.3577	-32.8
	7.09				7.99			
	7.73				8.7			
T3-LS20-FS	6.60	6.94	0.3375	-29.3	7.88	7.6	0.2411	-38.65
	7.28				7.5			
	6.94				7.43			
T3-LS25-FS	7.28	6.68	0.5408	-31.98	7.09	7.34	0.2197	-40.77
	6.53				7.43			
	6.23				7.5			
	6.04	5.563	0.5091	-43.31	7.46	7.25	0.2764	-41.47

T3-LS30-FS	5.03				7.35			
	5.63				6.94			

(Note: (+) positive sign indicates increase in F.S., and (–) negative indicates decrease of F.S.)

Figure 4.14 shows the graphical representation of the Flexural Strength of Lead Slag (Type 3) based cement mortar Specimens with the varying lead slag percentages of 0%, 5%, 10%, 15%, 20%, 25%, and 30%.

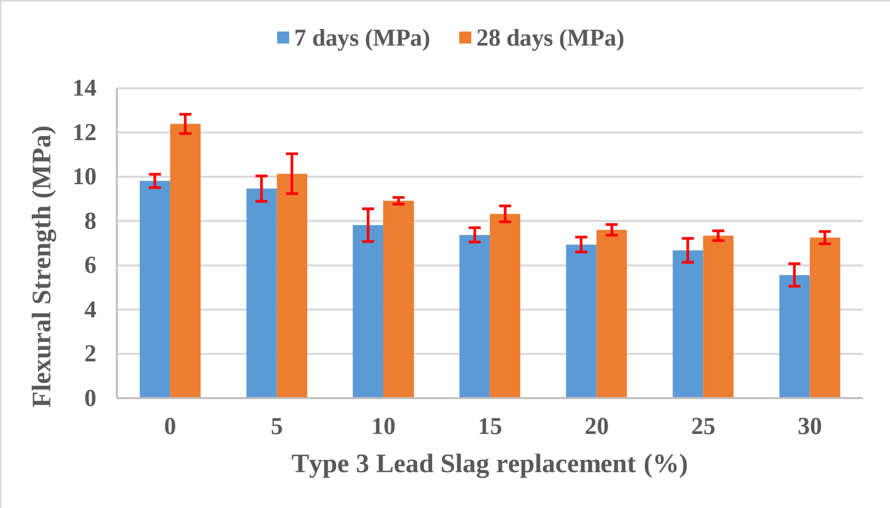


Figure 4.14 Flexural Strength vs Type 3 Lead Slag replacement (%)

Figure 4.15 shows the graphical representation of the Flexural Strength of Lead Slag (Type 3) based cement mortar Specimens with the different curing ages of 7 and 28 days.

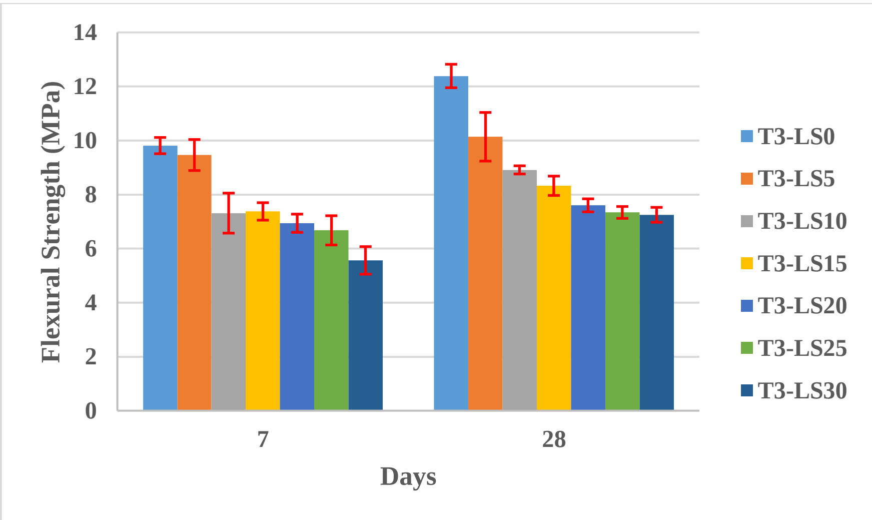


Figure 4.15 Flexural Strength vs Age of mortar specimens

Following observations can be made from Table 4.9 and Figure 4.14 & 4.15:

- Clearly, flexural strength of lead slag (Type 3) based mortar specimen decreases with an increase in lead slag replacement (%) at all ages, and increase with the different age of curing.
- It is found that the flexural strength of mortar specimens consisting of 5% (T3-LS0) of lead slag (Type 3) is comparable with control specimen (Control) at 7 days of curing age. The flexural strength of 7 days decreases only by 3.56% (T3-LS5). At 28 days, a decrease of 18.16% is noticed which is comparatively high as compared to 7 days curing age specimens.
- The results of the flexural strength are not promising as it decreases rapidly. The flexural strength observed in Type 3 Lead slag based mortar specimens greater as compared to Type 2 Lead slag based mortar specimens, and is almost comparable with Type 1 lead Slag based mortar specimens.
- At 7 days of testing, with 5% replacement with OPC, flexural strength decreases by 20.38% (T3-LS5), and with 15% replacement of lead slag the decrease in strength is noticed as 24.85% (T3-LS15). With further replacement of 20%, 25%, and 30%, the decrease in flexural strength is 29.3% (T3-LS20), 31.98% (T3-LS25), and 43.31% (T3-LS30) respectively.
- At 28 days of testing, with 5% replacement with OPC, flexural strength decreases by 28.05% (T3-LS5), and with 15% replacement of lead slag the decrease in strength is noticed as 32.8% (T3-LS15). And beyond this flexural strength decreases tremendously by 38.65% (T3-LS20), 40.77% (T3-LS25), and 41.47% (T3-LS30) for 20%, 25%, and 30% replacement of lead slag respectively. The flexural strength development at 7 days was better as compared at 28 days curing age.
- The strength development in 5% replacement or beyond replacement of Lead Slag (Type 3) is less as compared to control specimen due to non-uniform calcium silicate hydrate (C-S-H) gel which is generally about 60% of the paste volume resulting in the more voids and less denser configuration (permeability), leading to lesser strength. As a result of excessive amount of lead slag in the cement mortar specimens, there is an increase in the voids and lack of C-S-H is evident in the specimens containing replacement of Lead Slag (Type 3).
- The flexural strength increase with the age of curing, as the pozzolana reaction occurs at greater curing age. The calcium hydroxide produced is very important in breaking the glassy phase of the lead slag (Type 3), which leads to the pozzolana reaction. The same reason is also reported by Penpolcharoen (2005).

- Also the Lead Slag (Type 3) contains different metals in it which are responsible for the negative impact on the hydration mechanism. This is due to the presence of metals (Pb, Zn) present in lead slag leading in formation of the layer on dehydrated cement particles preventing hydration process.This reason is also reported by Cioffi *et al.* (2000).
- Also, the slow hydration of Lead Slag (Type 3) is due to presence of high silica and alumina, which can form an impermeable coating around the particle of the lead slag in the hydration process and ultimately decreasing flexural strength.

4.3.4 Comparison between the Flexural behavior of three types of Lead Slag

In this section the comparison in the flexural behavior of the three types lead slag based cement mortar specimens is done in order to determine the best among the three. Figure 4.16 shows the variation of 7 days flexural strength of all types.

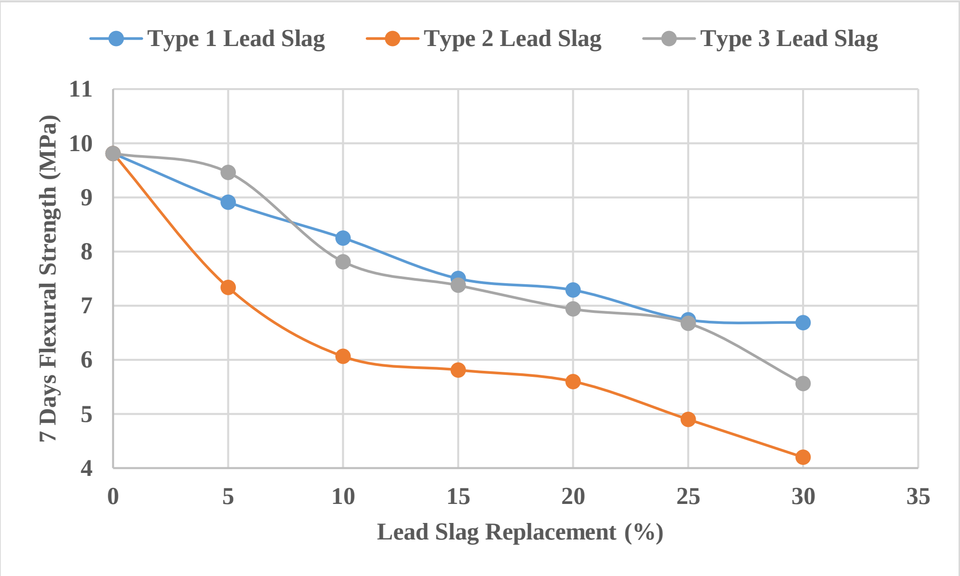


Figure 4.16 Variation of 7 Days Flexural Strength of three types of lead slag

Clearly the 7 days flexural strength of mortar specimens containing 5% lead Slag of Type 3 Lead Slag based specimens is somewhat comparable with the control specimen. The strength development is far better than Type 1 and Type 2 lead Slag based specimens, which is mainly due to more uniform calcium silicate hydrate (C-S-H) gel resulting in the less voids and denser configuration, leading to good strength. Further replacement in Lead slag decreases the flexural strength drastically in all three slags. All types of Lead Slag contains various metals in it which are responsible for the negative impact on the hydration mechanism as the metals are responsible for early setting and hardening. But the 7 days strength development is better in Type 3 and Type 1 based cement mortar specimens as compared to Type 2 Lead Slag based mortar specimens.

Figure 4.17 shows the variation of 28 days flexural strength of all types.

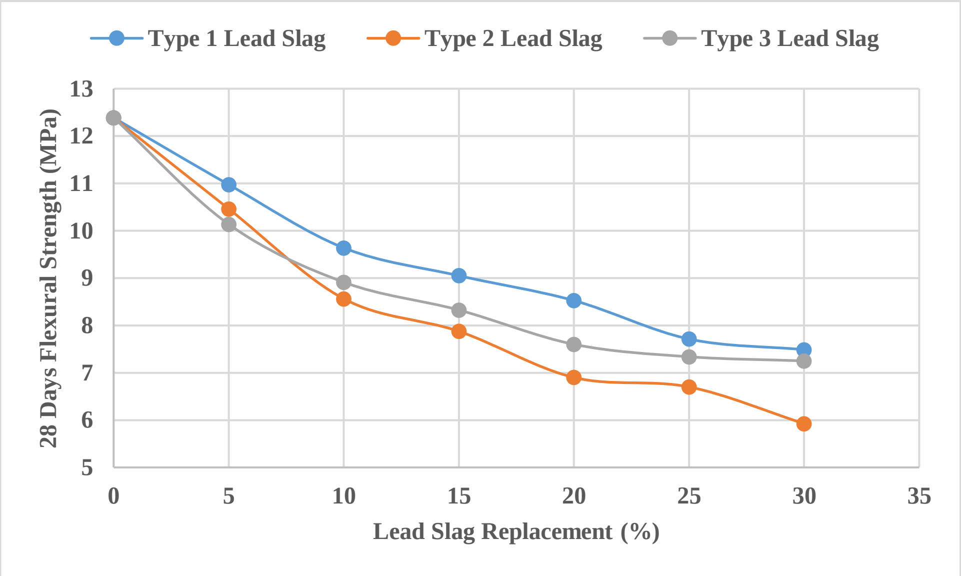


Figure 4.17 Variation of 28 Days Flexural Strength of three types of lead slag

Clearly the 28 days flexural strength of Type 3 and Type 1 lead slag based cement mortar specimens is better than Type 2 lead slag based mortar specimens at all levels of replacement. The pozzolanic reaction in Type 3 lead slag based specimens is much better than the other two types. The calcium hydroxide formed is very important in breaking the glassy phase of the lead slag, which leads to the pozzolana reaction. So the pozzolana reaction is best in Type 1 and Type 3 and is least in Type 2. So the Type 3 and Type 1 Lead Slag is highly recommended.

4.4 Compressive Strength of Geopolymer Mortar Specimens with varying Lead Slag

After casting of the cubical moulds, the mould was wrapped with a thin film of plastic polythene and were kept in the oven at 70^0 Celsius for 24 hours. At last, the specimens were demoulded from the mould and were kept for different ages of testing (i.e., 3 and 7 days) at room temperature.

After sample preparation, they were tested in a uni-axial compression testing machine. The lead slag-based geopolymer mortar specimens were tested at 3 and 7 days. The three specimens were casted for each mix, and the average of the three was considered the compressive strength of the specimen.

4.4.1 Compressive Strength of Type 1 Lead Slag based Geopolymer Mortar Specimens

Table 4.10 shows the data set of the Compressive Strength and percentage variation of Lead Slag (Type 1) based geopolymer mortar Specimens at different ages.

Table 4.10 Compressive Strength of Lead Slag (Type 1) based geopolymer mortar specimens at different age

Specimen ID	3 days (MPa)	3 days (MPa) Avg.	S.D.	% var	7 days (MPa)	7 days (MPa) Avg.	S.D.	% var
GP-Control	26.78	27.82	0.9007	Refer-ence	29.55	28.92	0.54483	Refer-ence
	28.35				28.56			
	28.34				28.66			
GP-T1-LS10	28.17	28.48	0.5894	+2.37	31.68	31.66	0.2448	+9.46
	29.16				31.4			
	28.12				31.89			
GP-T1-LS20	30.25	29.75	0.4847	+6.92	31.36	32.12	0.74117	+11.07
	29.28				32.17			
	29.72				32.84			
GP-T1-LS30	30.46	31.04	0.6886	+11.56	33.92	35.26	1.08254	+21.93
	31.8				35.98			
	30.87				35.89			

(Note: (+) positive sign indicates increase in C.S., and (–) negative indicates decrease of C.S.)

Figure 4.18 shows the graphical representation of the Compressive Strength of Lead Slag (Type 1) based geopolymer mortar Specimens with the varying lead slag percentages.

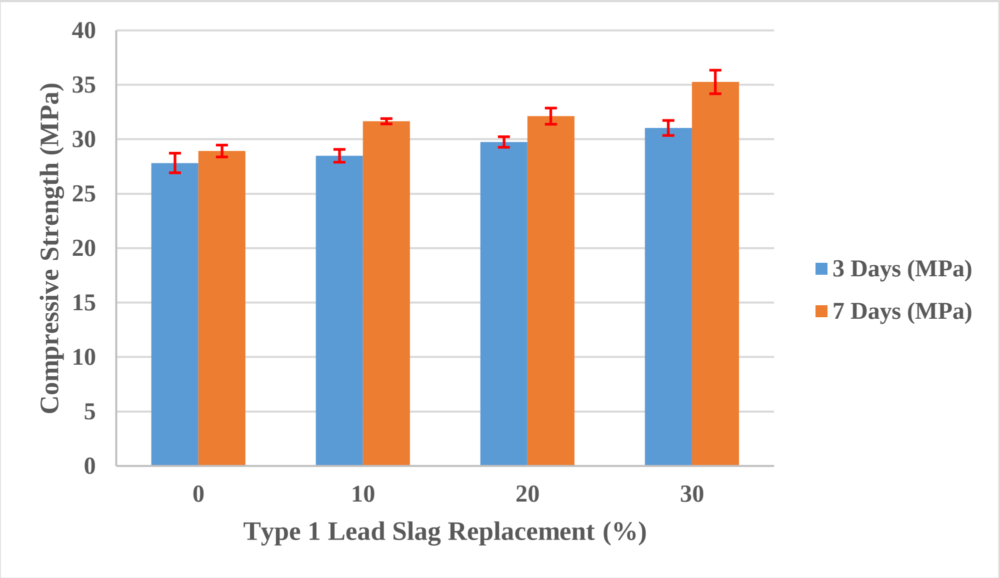


Figure 4.18 Compressive Strength vs Type 1 Lead Slag replacement (%)

Figure 4.10 shows the graphical representation of the Compressive strength of Lead Slag (Type 1) based geopolymer mortar Specimens with the different curing ages of 3 and 7 days.

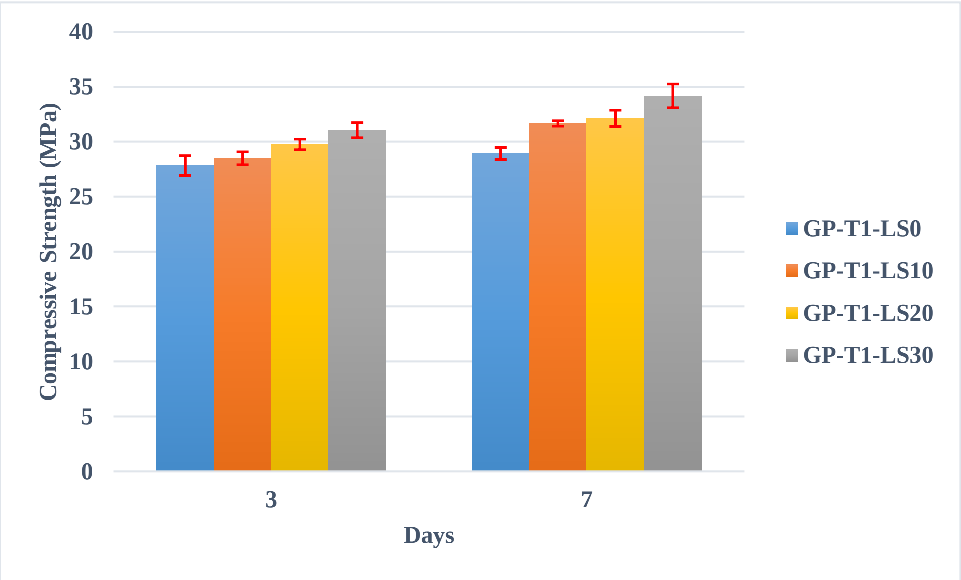


Figure 4.19 Compressive Strength vs Age of geopolymer mortar specimens

Following observations can be made from Table 4.10 and Figure 4.18 & 4.19:

- Clearly, compressive strength of geopolymer mortar specimens increases with an increase in lead slag replacement up to 30% at all ages, and also increase with the age.
- Compressive strength of geopolymer mortar specimens containing 10% (GP-T1-LS10) lead Slag (Type1) is comparable with the control specimen having 0% of lead slag (GP-Control), particularly at 3 days as the strength increases only by 2.37 %. Whereas at 7 days, an increase of 9.46 % is noticed.

- The results are very promising as compressive strength increases rapidly. The Lead Slag (Type 1) has good alkaline reactivity. It suggests the possibility of using the lead slag as geopolymer precursor.
- At 3 days of testing, with 20% replacement with Fly-ash, compressive strength increases by 6.92 % (GP-T1-LS20). With further increase in replacement of 30%, the increase in compressive strength of 11.56 % (GP-T1-LS30) is noticed.
- At 7 days of testing, with 20% replacement of lead slag with Fly-ash, compressive strength increases by 11.07 %. And increases by 21.93% for 30% replacement of lead slag.
- The high compressive strength in control specimen (GP-Control) is due to the process of geopolymerization that includes polymerization mechanism of Al and Si obtained from the fly-ash which reacted with alkali activating solutions, at elevated temperature curing. In the polymerization mechanism, strong Na-Al-Si bonds are formed which leads in high compressive strength by maintaining the homogeneity in the matrix.
- With replacement of Fly-Ash with Lead slag (Type 1), the compressive strength increases up to 30% replacement at all ages. Due to inclusion of Lead slag, the increase in compressive strength is due to additional Al and Si that reacted with alkali activating solution and polymerization occurs. At 7 days the strength development noticed is high as compared to 3 days.
- The geopolymer act as a matrix for the solidification/stabilization and immobilization of different toxic elements present in lead slag (Type 1) like Pb, Zn, and Cd which can be fixed in 3D network of geopolymer structures. Toniolo & Boccaccini, 2017 also report this reason. Guo *et al*. 2017 also explored the immobilization mechanism of lead slag in geopolymers. Moreover, Zheng *et al.* (2015) confirmed the immobilization of Pb^{2+}, Zn^{2+}, and Cd^{2+} in geopolymers.

4.4.2 Compressive Strength of Type 2 Lead Slag based Geopolymer Mortar Specimens

Table 4.11 shows the data set of the Compressive Strength and percentage variation of Lead Slag (Type 2) based geopolymer mortar Specimens at different ages.

Table 4.11 Compressive Strength of Lead Slag (Type 2) based geopolymer mortar specimens at at different age

Specimen ID	3 days (MPa)	3 days (MPa) Avg.	S.D.	% var	7 days (MPa)	7 days (MPa) Avg.	S.D.	% var
GP-Control	26.78	27.82	0.9007	Refer-ence	29.55	28.92	0.5448	Refer-ence
	28.35				28.56			
	28.34				28.66			
GP-T2-LS10	27.77	27.55	0.7036	+0.98	30.24	30.83	0.8470	+6.6
	26.76				31.8			
	28.12				30.45			
GP-T2-LS20	29.05	28.82	0.2979	+3.57	31.36	31.99	0.5624	+10.61
	28.48				32.17			
	28.92				32.44			
GP-T2-LS30	30.46	30.71	0.6023	+10.38	32.92	33.09	0.5288	+14.44
	31.4				32.68			
	30.28				33.69			

(Note: (+) positive sign indicates increase in C.S., and (–) negative indicates decrease of C.S.)

Figure 4.20 shows the graphical representation of the Compressive Strength of Lead Slag (Type 2) based geopolymer mortar Specimens with the varying lead slag percentages of 0%, 10%, 20%, and 30%.

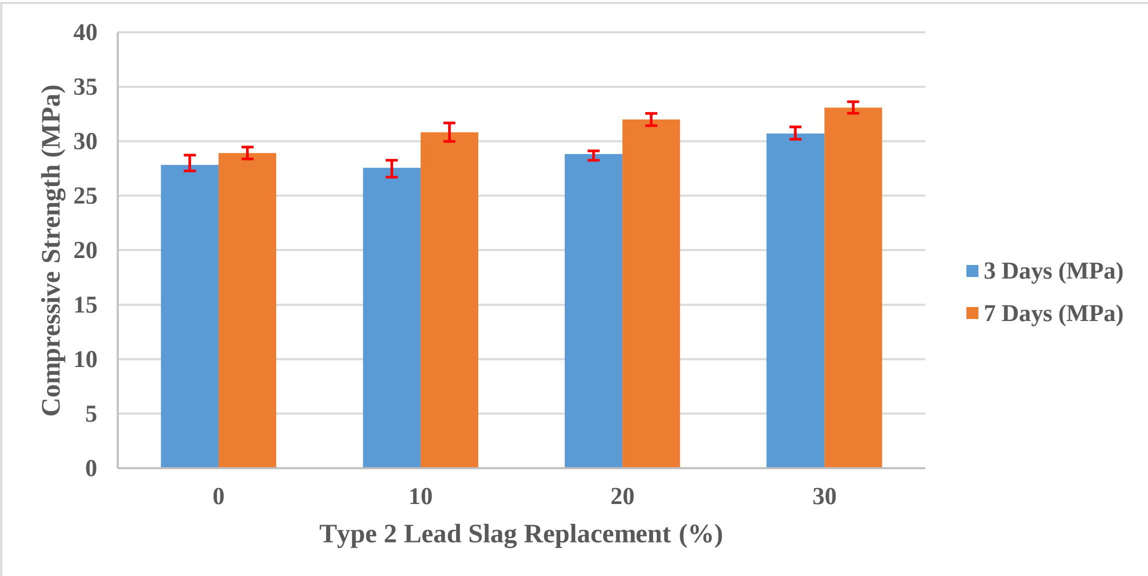


Figure 4.20 Compressive Strength vs Type 2 Lead Slag replacement (%)

Figure 4.11 shows the graphical representation of the Compressive strength of Lead Slag (Type 2) based geopolymer mortar Specimens with the different curing ages of 3 and 7 days.

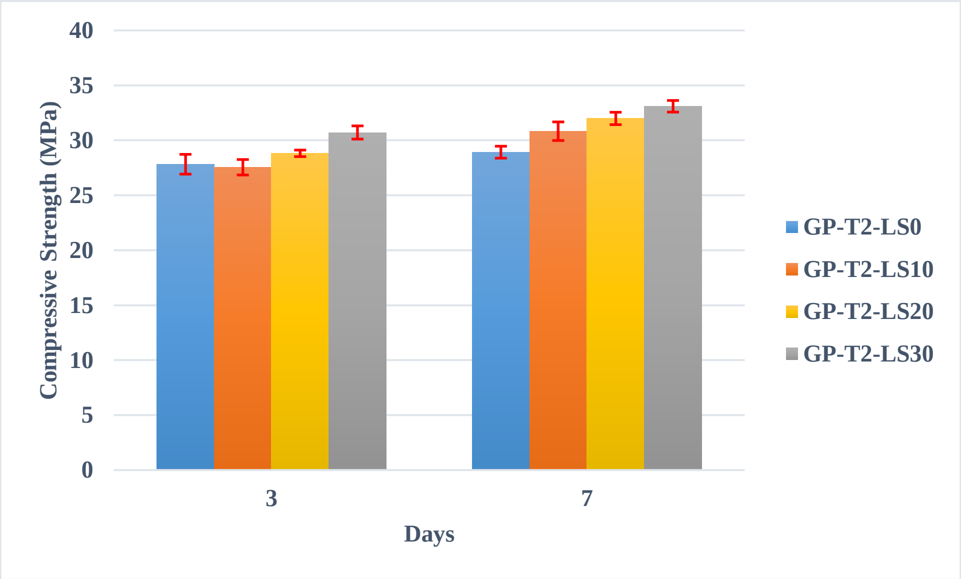


Figure 4.21 Compressive Strength vs Age of geopolymer mortar specimens

Following observations can be made from Table 4.11 and Figure 4.20 & 4.21:

- Clearly, compressive strength of geopolymer mortar specimens increases with an increase in the lead slag replacement up to 30% at all ages, and also increase with the age.
- Compressive strength of geopolymer mortar specimens containing 10% (GP-T2-LS10) lead Slag (Type 2) is comparable with the control specimen having 0% of lead slag (GP-Control), particularly at 3 days as the strength increases only by 0.98 %. Whereas at 7 days, an increase of 6.6 % is noticed.
- The results are very promising as compressive strength increases rapidly. The Lead Slag (Type 2) has good alkaline reactivity but is less as compared with Type 1 Lead Slag as strength increases more rapidly there. It suggests the possibility of using the lead slag as geopolymer precursor.
- At 3 days of testing, with 20% replacement with Fly-ash, compressive strength increases by 3.57 % (GP-T2-LS20). With further increase in replacement of 30%, the increase in compressive strength of 10.38 % (GP-T2-LS30) is noticed.
- At 7 days of testing, with 20% replacement of lead slag with Fly-ash, compressive strength increases by 10.61%. And increases by 14.44% for 30% replacement of lead slag.

- The high compressive strength in control specimen (GP-Control) is due to the process of geopolymerization that includes polymerization mechanism of Al and Si obtained from the fly-ash which reacted with alkali activating solutions, at elevated temperature curing. In the polymerization mechanism, strong Na-Al-Si bonds are formed which leads in high compressive strength by maintaining the homogeneity in the matrix.
- With replacement of Fly-Ash with Lead slag (Type 2), the compressive strength increases up to 30% replacement at all ages. Due to inclusion of Lead slag, the increase in compressive strength is due to additional Al and Si that reacted with alkali activating solution and polymerization occurs. At 7 days the strength development noticed is high as compared to 3 days.
- The geopolymer act as a matrix for the solidification/stabilization and immobilization of different toxic elements present in lead slag (Type 2) like Pb, Zn, and Cd which can be fixed in 3D network of geopolymer structures. Toniolo & Boccaccini, 2017 also report this reason. Guo *et al*. 2017 also explored the immobilization mechanism of lead slag in geopolymers. Moreover, Zheng *et al.* (2015) confirmed the immobilization of Pb^{2+}, Zn^{2+}, and Cd^{2+} in geopolymers.

4.4.3 Compressive Strength of Type 3 Lead Slag based Geopolymer Mortar Specimens

Table 4.12 shows the data set of the Compressive Strength and percentage variation of Lead Slag (Type 3) based geopolymer mortar Specimens at different ages.

Table 4.12 Compressive Strength of Lead Slag (Type 3) based geopolymer mortar specimens at different age

Specimen ID	3 days (MPa)	3 days (MPa) Avg.	S.D.	% var	7 days (MPa)	7 days (MPa) Avg.	S.D.	% var
GP-Control	26.78	27.82	0.9007	Reference	29.55	28.92	0.5448	Reference
	28.35				28.56			
	28.34				28.66			
GP-T3-LS10	24.4	23.83	0.4933	-14.34	31.48	32.27	1.097	+11.57
	23.6				31.8			
	23.5				33.52			
GP-T3-	28.2			+4.48	35.28			+19.27

LS20	30.12	29.07	0.9724		34.56	34.49	0.8239	
	28.89				33.64			
GP-T3-LS30	31	31.49	0.522	+13.19	36.6	37.02	1.0502	+27.99
	32.04				38.21			
	31.44				36.24			

(Note: (+) positive sign indicates increase in C.S., and (–) negative indicates decrease)

Figure 4.22 shows the graphical representation of the Compressive Strength of Lead Slag (Type 3) based geopolymer mortar Specimens with the varying lead slag percentages of 0%, 10%, 20%, and 30%.

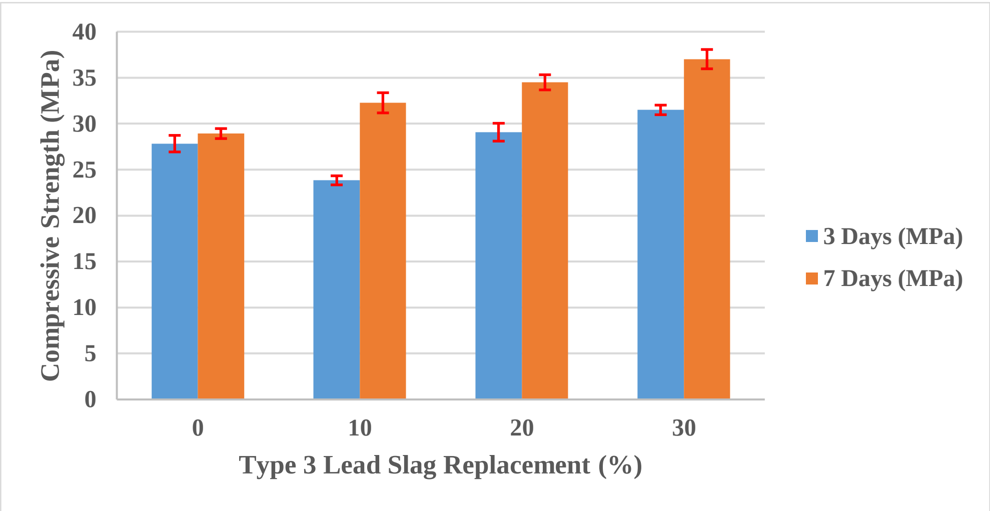


Figure 2.22 Compressive Strength vs Type 3 Lead Slag replacement (%)

Figure 4.23 shows the graphical representation of the Compressive strength of Lead Slag (Type 3) based geopolymer mortar Specimens with the different curing ages of 3 and 7 days.

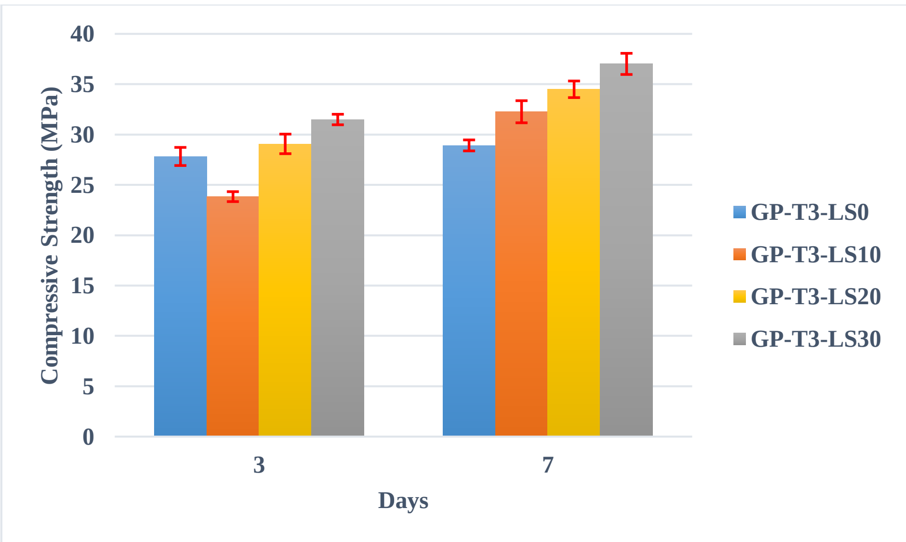


Figure 2.23 Compressive Strength vs Age of geopolymer mortar specimens

Following observations can be made from Table 4.12 and Figure 4.22 & 4.23:

- Clearly, compressive strength of the geopolymer mortar specimens increases with an increase in the lead slag replacement up to 30% at all ages.
- Compressive strength of geopolymer mortar specimens containing 10% (GP-T3-LS10) lead Slag (Type 3) is not comparable with the control specimen having 0% of lead slag (GP-Control), particularly at 3 days as the strength decrease by 14.34 %. Whereas at 7 days, an increase of 11.57 % is noticed.
- The results are very promising as compressive strength increases rapidly except for 10% replacement at 3 days. The strength development after 7 days is very better as compared with 3 days. The Lead Slag (Type 3) has good alkaline reactivity. It suggests the possibility of using the lead slag as geopolymer precursor. The results concluded that the strength development is better than Type1 lead Slag, and is very better than Type 2 lead Slag.
- At 3 days of testing, with 20% replacement with Fly-ash, compressive strength increases by 4.48% (GP-T3-LS20). With further increase in replacement of 30%, the increase in compressive strength of 13.19 % (GP-T3-LS30) is noticed.
- At 7 days of testing, with 20% replacement of lead slag with Fly-ash, compressive strength increases by 19.27%. And increases by 27.99% for 30% replacement of lead slag.
- The high compressive strength in control specimen (GP-Control) is due to the process of geopolymerization that includes polymerization mechanism of Al and Si obtained from the fly-ash which reacted with alkali activating solutions, at elevated temperature curing. In the polymerization mechanism, strong Na-Al-Si bonds are formed which leads in high compressive strength by maintaining the homogeneity in the matrix.
- With replacement of Fly-Ash with Lead slag (Type 3) at 3 day, the compressive strength decreases in GP-T3-LS10 (with 10% replacement) due to weak polymerization compounds formed and non- homogeneity in the matrix. But further replacement of Lead Slag (Type 3) up to 30% increases compressive strength. Due to inclusion of Lead slag, the increase in compressive strength is due to additional Al and Si that reacted with alkali activating solution and polymerization occurs.
- With replacement of Fly-Ash with Lead slag (Type 3), the compressive strength increases upto 30% replacement at 7 days. Due to inclusion of Lead slag, the increase in compressive strength is due to additional Al and Si that reacted with alkali activating

solution and polymerization occurs. At 7 days the strength development noticed is high as compared to 3 days.

- Also Type 3 Lead Slag contains more Calcium as compared to Fly ash. So when replaced, the percentage Calcium increases and hydration process occurs and different hydration products like CASH ad C-S-H are formed in addition to the co-existing geopolymerization product NASH.
- The geopolymer act as a matrix for the solidification/stabilization and immobilization of different toxic elements present in lead slag (Type 3) like Pb, Zn, and Cd which can be fixed in 3D network of geopolymer structures. Toniolo & Boccaccini, 2017 also report this reason. Guo *et al*. 2017 also explored the immobilization mechanism of lead slag in geopolymers. Moreover, Zheng *et al.* (2015) confirmed the immobilization of Pb^{2+}, Zn^{2+}, and Cd^{2+} in geopolymers.

4.4.4 Comparison between the Compressive behavior of three types of Lead Slag

In this section the comparison in the compressive behavior of the three types lead slag based geopolymer specimens is done in order to determine the best among the three. Figure 4.24 shows the variation of 3 days compressive strength of all types.

Clearly the strength development in Type 1 Lead Slag based specimens is better than Type 2 and Type 3 at 10% and 20% replacement of lead slag. But at 30% replacement, the Type 3 Lead Slag based specimens dominates. Possible reason for this the polymerization mechanism which yielded strong Na-Al-Si bonds in Type 1 and Type 2 based specimens and thereby increase the compressive strength.

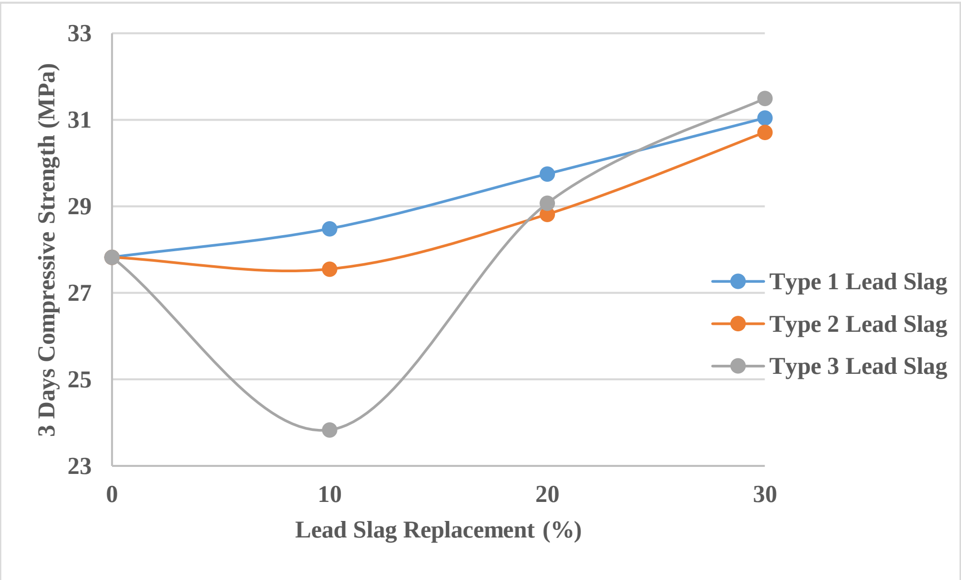


Figure 2.24 Variation of 3 Days Compressive Strength of 3 types of lead slag

Based on the additional Al and Si content in Type 3 Lead Slag based specimens the compressive strength should increase at 10% replacement but it is decreasing. The possible reason for this is non- homogeneity in the matrix which leads in less compressive strength. But further increase in replacement upto 30%, the results are very promising in Type 3 lead slag based specimens and it has good alkaline reactivity. Figure 4.25 shows the variation of 7 days compressive strength of all types.

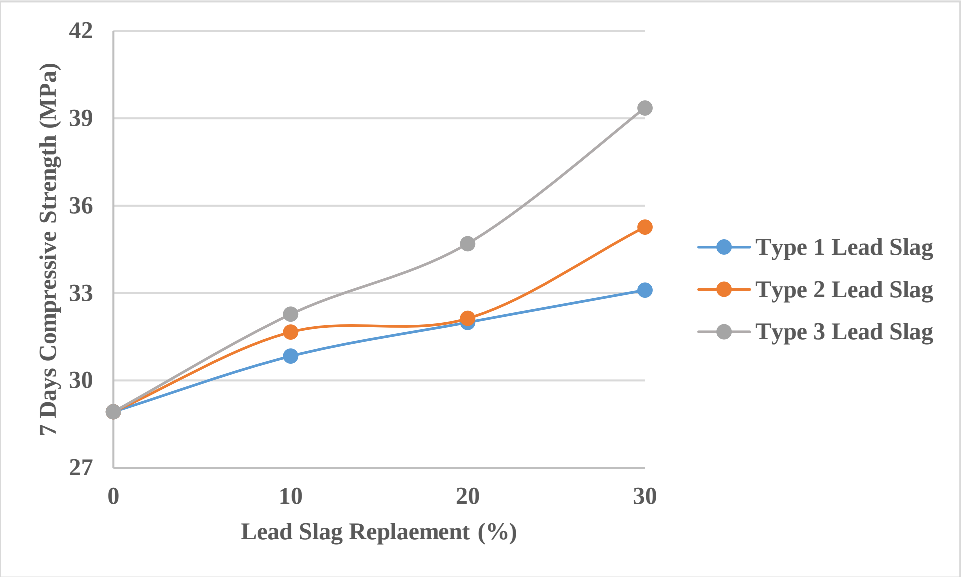


Figure 2.25 Variation of 7 Days Compressive Strength of 3 types of lead slag

Clearly when compared with Type 1 lead Slag, and Type 2 Lead Slag based geopolymer specimens, the strength development is very high in Type 3 Lead Slag based geopolymer specimens. This is due to the addition of extra Al and Si content in geopolymer relatively (Due to more Si and Al in Type 3 Lead Slag that Type 1 Lead Slag) which increases the NaSH (Polymerization Product). Thus Type 3 Lead Slag is highly recommended as results are very convincing. Also Type 3 Lead Slag contains more Calcium as compared to Fly ash. So when replaced, the percentage Calcium increases and hydration process occurs and different hydration products like CASH ad C-S-H are formed in addition to the co-existing geopolymerization product.

CHAPTER 5

CONCLUSION

5.1 Introduction

This research investigates the feasibility analysis of secondary lead recycling slag powder in cementitious matrix as a construction material. This utilization of lead slag in construction can help in solving the negative impact of lead slag on environment. The entire study was divided into two parts. In the first part, different mechanical properties of secondary lead slag-based cement mortar specimens with varying percentages (i.e., 0%, 5%, 10%, 15%, 20%, 25%, and 30%) of secondary lead slag. In the second part, different mechanical properties of secondary lead slag-based geopolymer mortar specimens with varying percentages (i.e., 0%, 10%, 20%, and 30%) of secondary lead slag.

5.2 Main conclusion of the study

On the basis of the aim and objectives of the study, different material properties, methods and techniques adopted, and all other parameters included with this study, the below are the various conclusion that can be made.

- There is retardation in the setting time in the case of lead slag based mortar mix, and setting time increases as the replacement increases. This delay is due to the presence of metals (Pb, Zn) present in lead slag leading in formation of the layer on dehydrated cement particles preventing hydration process.
- The inclusion of lead slag resulted in increase in flowability of the mortar specimens. This is due to reduction in rate of hydration of cement particles leading in amount of free water in the mixes, thereby increasing the mortar mixes flowability.
- Compressive and flexural strength of lead slag (Type 1, 2 ,3) based cement mortar specimens decreases with an increase in lead slag replacement (%) at all ages and increases with the age of curing for all types of Lead Slag based cement mortar specimens, as the pozzolana reaction occurs at greater curing age.
- Compressive strength of mortar specimens containing 5% (T1-LS5) lead Slag (Type1) is comparable with the control specimen having 0% of lead slag (Control) at all ages of testing, particularly at 7 days as the strength decreases only by 0.56%.

- It is found that the flexural strength of mortar specimens consisting of 5% (T3-LS5-FS) of lead slag (Type 3) is comparable with control specimen (Control) at 7 days of curing age. The flexural strength of 7 days decreases only by 3.56%.
- Further increase in the replacement of Lead Slag after 5%, the results are not promising as compressive strength decreases rapidly. The strength development is better in Type 1 and Type 3 based cement mortar specimens as compared to Type 2 Lead Slag based mortar specimens. Type 1 and Type 3 lead slag is highly recommended upto 5% replacement.
- Compressive strength of lead slag (Type 1, 2) based geopolymer mortar specimens increases with an increase in lead slag replacement upto 30% at all ages, and also increase with the age. Due to inclusion of Lead slag, the increase in compressive strength is due to additional Al and Si that reacted with alkali activating solution and polymerization occurs.
- Compressive strength of geopolymer mortar specimens containing 10% (GP-T3-LS10) lead Slag (Type3) is not comparable with the control specimen having 0% of lead slag (GP-Control), particularly at 3 days as the strength decrease by 14.34 %. Whereas at 7 days, an increase of 11.57 % is noticed.
- Clearly the early strength development (3day) in Type 1 Lead Slag based geopolymer specimens is better than Type 2 and Type 3 at 10% and 20% replacement of lead slag. But at 30% replacement, the Type 3 Lead Slag based specimens dominates.
- The 7 days compressive strength of Type 3 lead slag based geopolymer mortar specimens is better than Type 1 and Type 2 based lead slag based geopolymer mortar specimens.
- Type 3 Lead Slag is highly recommended for geopolymer as results are very convincing. Also Type 3 Lead Slag contains more Calcium as compared to Fly ash. So when replaced, the % of Calcium increases and hydration process occurs and different hydration products like CASH ad C-S-H are formed in addition to the co-existing geopolymerization product.
- The geopolymer act as a matrix for stabilization of different toxic elements present in lead slag (Type 1, 2, 3) like Pb, Zn, and Cd which can be fixed in 3-D network of geopolymer structures.
- The Lead Slag (Type 1, 2, 3) has good alkaline reactivity. It suggests the possibility of using the lead slag as geopolymer precursor.

5.3 Limitation of the Study

This study has some flaws as the research topic is vast, and more research vulnerability is required in order to explore different properties and characteristics of secondary lead slag. The limitations of the present study are as follows:

- Concrete is more widely used in construction instead of mortar.
- Replacement of Fly-ash is only done up to 30% in this study.
- The elevated temperature curing was used in geopolymer specimens which need to be optimized to ambient conditions.
- The present study does not use any super-plasticizer, which helps improve mechanical properties by decreasing the water requirement of cement.
- Durability aspects of the mortar are missing in the study, which is also essential.
- Non-destructive tests are also not carried that tells a furthermore realistic perspective.
- The only problem is with the alkaline activating solution, which is mainly sodium hydroxide and sodium silicate, and these resources are limited, and their cost is comparatively high.
- Also, there can be a negative impact of the activating solution on the environment and thus affect its popularity to some extent.

5.4 Future scope of the study

- After studying all aspects, it is comprehended that up to 5% of Lead Slag (Type 1) as a partial cement replacement have good strength results. The study can be done on cement concrete as it is widely used as a construction material. Type 1 Lead Slag is highly recommended for cement concrete.
- Also, 30% replacement of Fly Ash with Lead slag of all three types is very promising as strength increases rapidly. So extensive study can be done on lead slag-based geopolymer concrete up to 50% replacement of fly ash and for longer curing ages.
- Type 3 lead slag is highly recommended for geopolymer concrete. Also both types of curing: at elevated temperature and at ambient condition should be done and compared.
- Durability aspects of the lead slag-based concrete should also be studied.
- The present study does not use any super-plasticizer and is limited. So in further research, the use of super-plasticizer can be done, leading in lesser water requirement of cement ultimately increasing in strength.

- The leaching behavior of the toxic elements must be examined in order to test the environmental suitability of the secondary lead slag by the TCLP test. The test main aim is to predict whether the leaching of lead slag occurs or not while incorporating as a construction material.
- Mineralogical characteristics of Lead Slag is also need to be acknowledged.

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