ABSTRACT

The abstract provides a brief summary of the study conducted on the utilization of industrial wastes as supplementary cementitious materials (SCMs) in cement production. The study aims to investigate the feasibility of using coal fly ash and metal slag as SCMs, which can reduce environmental pollution and promote sustainable development. The study used various tests including compressive strength, consistency, fineness, and settling time to evaluate the feasibility of blending the SCMs with cement at different proportions ranging from 10% to 100%. The results of the study showed that the samples containing up to 40% slag exhibited good compressive strength and consistency, which is comparable to samples containing no SCM. However, it is important to note that the long-term performance and durability of the material should also be considered in addition to the compressive strength and consistency. Further investigations are recommended to evaluate the performance of the material in terms of its durability and long-term behavior. Overall, the findings of the study suggest that utilizing industrial wastes as SCMs in cement production is a promising solution for sustainable development and environmental protection. It can also contribute to reducing the carbon footprint associated with cement production, as it reduces the need for virgin raw materials and the associated energy consumption and emissions.

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CHAPTER -1

INTRODUCTION

1.1 Introduction

The cement industry is one of the most resource-intensive industries, with high energy consumption and high greenhouse gas emissions. Industrial wastes, such as fly ash, slag, and silica fumes, have been used as supplementary cementitious materials (SCMs) in cement production for several decades. India is the second largest cement producer in the world and accounted for over 7% of the global installed capacity. As India has a high quantity and quality of limestone deposits through-out the country, the cement industry promises huge potential for growth. In 2019, India was the second largest producer of cement in the world. The country had about eight percent of the global installed capacity that year. Consumption of cement at this time stood at around 328 million metric tons. The Indian cement industry is the second largest in the world, as measured by production (502 million tons per annum in 2018), with over 8% (502 million tons per annum in 2018) of the global installed capacity and supporting over 1 million jobs. [17]

The domestic production of cement has reached at 356 million tons in FY 2022, up from 296 million tons in FY 2021. The consumption of cement has been steadily increasing, reaching 355.46 million tons in FY 2022, and is projected to hit 450.78 million tons by the end of FY 2027. The rise in demand for cement is largely attributed to the construction of residential, commercial, and industrial buildings. Additionally, the government's recent infrastructure projects such as airports and roads have also contributed to the growth of the cement industry.

In addition to climate and species protection, the conservation of natural resources is one of the major ecological challenges of our time. In this context, the focus is also on cement and concrete production. The conservation of limestone is an important challenge for the cement industry in the coming years. Limestone is the principal raw material for production of cement, and the demand for cement is expected to continue to grow as the global population increases and urbanization expands.[2]

utilizing slag and fly ash can be an effective way to conserve limestone in the cement industry. Both slag and fly ash are byproducts of other industrial processes, and they can be used as partial substitutes for limestone in cement production. Slag is a byproduct of steel production, and it is rich in silica, alumina, and calcium [1]. When used in cement production, it can replace some of the limestone and reduce the need for virgin materials. Fly ash, on the other hand, is a byproduct of coal-fired power plants, and it is rich in silica, alumina, and calcium oxide. Like slag, it can be used as a partial substitute for limestone in cement production.

By using slag and fly ash, the cement industry can reduce its demand for limestone and conserve this important natural resource. Additionally, the use of byproducts like slag and fly ash can help to reduce waste and promote a more sustainable industrial economy. Using slag and fly ash in cement production can also reduce greenhouse gas emissions and have several environmental benefits.[2]

In order to reduce CO2 emissions and meet its target of a 33-35% reduction in emissions by 2030, India is focusing on the cement and concrete industries, which are responsible for up to 7% of total world CO2 emissions [3]. These industries are facing challenges related to the use of supplementary cementing materials, designing of concrete mixtures, and augmentation of concrete durability towards sustainability.

To address these challenges, India is developing Portland-limestone cement (PLC), which involves introducing limestone into cement. By doing so, the amount of clinker needed for production of a specific amount of cement would decrease, resulting in significant fuel and energy savings and conservation of natural resources.

In this graphical review, the benefits, and challenges of mixing limestone in cement are discussed, including the effect of limestone on various physiochemical properties of cement and the contribution of PLC development towards a green economy. Overall, the development of PLC in India has the potential to significantly reduce CO2 emissions and improve the sustainability of the cement and concrete industries.[3]

The utilization of industrial wastes in cement production not only conserve the resources but also provides a sustainable solution for managing industrial wastes. This project aims to investigate the feasibility of using industrial wastes as SCMs in cement production. The experimental analysis will be conducted to evaluate the physical and chemical properties of industrial wastes, including their compressive strength, setting time, fineness, and chemical composition. The industrial wastes that will be studied include fly ash and blast furnace slag.

The experimental analysis will be conducted in three phases. The first phase will involve the characterization of industrial wastes, including their physical and chemical properties. The second phase will involve the production of cement with different proportions of industrial wastes and the evaluation of their compressive strength, setting time, fineness, and chemical composition. The third phase will involve the feasibility study of the utilization of industrial wastes in cement production by comparing the performance of cement produced with industrial wastes with that of conventional cement. The results of this study will provide valuable insights into the feasibility of using industrial wastes as SCMs in cement production. The study will also contribute to the sustainable management of industrial wastes and the reduction of the environmental impact of cement production.

As supplementary cementitious materials (SCMs) in cement production can reduce the environmental impact of the industry while also providing a sustainable solution for managing industrial wastes [3]. The study also includes a feasibility analysis of the utilization of industrial wastes in cement production by comparing the performance of cement produced with industrial wastes with that of conventional cement. The results of this study can contribute to the sustainable management of industrial wastes, the reduction of the environmental impact of cement production, and the improvement of the performance of cement.

India is one of the largest producers of fly ash in the world due to its heavy reliance on coal-based power generation. According to the Central Electricity Authority (CEA) of India, in 2019-20, the country generated about 217 million metric tons of fly ash from thermal power plants. Around the world, electricity is generated using both fossil fuels (such as coal and oil) and renewable sources (such as solar and wind power). However, the reliance on fossil fuels for energy has negative consequences, such as the depletion of non-renewable resources and pollution [4].

One of the major issues related to energy production is the generation of coal fly ash (CFA) in thermal power plants. Every year, millions of tons of CFA are generated globally, and while nearly half of it is utilized in various ways, the other half is left unused. This can lead to different forms of pollution, such as air and water pollution, as CFA contains toxic elements that can be released into the environment. Overall, the large production of CFA and the improper handling of its waste can lead to various negative environmental impacts. Therefore, there is a need to find alternative and sustainable solutions to address this problem and reduce the dependency on fossil fuels for energy production.

The Indian government has implemented various policies and initiatives to promote the utilization of fly ash in different industries such as cement, construction, and agriculture. As per the Ministry of Power, in 2020-21, about 68% of the fly ash generated in India was utilized, primarily in the cement and construction industries.

The use of fly ash in the Indian cement industry has been particularly significant. According to the Cement Manufacturers' Association, the cement industry in India consumed about 44.3 million metric tons of fly ash in 2019-20, accounting for about 20% of the total fly ash generated in the country. Additionally, the Indian government has set a target to increase the utilization of fly ash in the cement industry to 100% by 2030 [4].

Overall, the production of fly ash in India is expected to continue to grow in the coming years due to the country's increasing demand for power and its efforts to promote the sustainable use of industrial waste materials.

Blast slag is a by-product of steel industry, which comes from pig iron refining processing. Because of its physical, chemical, and mineralogical properties, it can be used as a substitute for aggregates in civil engineering projects. Blast furnace slag has the useful components like Cao, MgO with high basicity's (Cao/SiO2). It has high fluxing capacity and is being charged in the blast furnace due to easy melt and better utilization of calcium values. In the European countries, 30% of such slags are recycled into the blast furnace. However, the most harmful components in the LD slag are P&S which are to be removed before use either in sintering plant or blast furnace.

India is one of the largest producers of steel in the world and is also a significant producer of blast furnace slag. According to the Ministry of Steel of India, the country produced about 109.2 million metric tons of crude steel in 2020-21. Based on this figure, it can be estimated that the production of blast furnace slag in India in the same year would be around 35-40 million metric tons. The utilization of blast furnace slag in India is primarily in the cement and construction industries. The Indian government has implemented various policies and initiatives to promote the utilization of blast furnace slag in different industries. In the construction industry, blast furnace slag is commonly used as a partial replacement for cement in concrete and other building materials. According to a report by Markets and Markets, the Indian ground granulated blast furnace slag (GGBFS) market size was valued at USD 243.4 million in 2020, and it is projected to reach USD 348.3 million by 2025, with a compound annual growth rate (CAGR) of 7.4% during the forecast period. Clinker is a crucial component in the production of cement and is obtained by heating a mixture of limestone, clay, and other materials at high temperatures. However, the production of clinker is energy-intensive and contributes to a significant portion of carbon emissions.

In some cases, slags from the iron and steel industries can be used as a replacement for clinker in the production of cement. Slags are the by-products of the iron and steel industries and are usually produced during the process of smelting ores to extract the metal. These slags have cementitious properties and can be used as a partial replacement for clinker in cement production. The use of slag as a replacement for clinker reduces the energy consumption and carbon emissions associated with the production of cement. Additionally, the use of slag in cement production can also help in reducing the waste generated by the iron and steel industries.

Overall, the use of slag as a replacement for clinker in cement production is an environmentally friendly and sustainable solution that can help reduce the carbon footprint of the cement industry while also addressing the waste generated by the iron and steel industries. The production of blast furnace slag in India is expected to continue to grow in the coming years due to the increasing demand for steel and the growing awareness of the benefits of using industrial waste materials in construction and other applications.

The generation of fly ash and blast furnace slag can have the following negative environmental effects. Here are some of the key environmental effects associated with their generation:

Negative Environmental Effects:

Air pollution: The generation of fly ash and blast furnace slag is often associated with industrial processes that emit air pollutants, such as sulfur dioxide and nitrogen oxides. These pollutants can contribute to air pollution and have adverse health effects on nearby communities and ecosystems.

Water pollution: The generation of fly ash and blast furnace slag can lead to the release of heavy metals and other pollutants into nearby water sources if these materials are not managed properly. This can harm aquatic life and human health if the water is used for drinking or irrigation purposes.

Energy and water use: The generation of fly ash and blast furnace slag requires energy and water inputs. Depending on the source of the materials and the production process, this can contribute to greenhouse gas emissions and water stress in some regions.

Land use impacts: The mining and extraction of raw materials required for the generation of fly ash and blast furnace slag can have impacts on land use, biodiversity, and soil quality. For example, the extraction of coal for the generation of fly ash can contribute to deforestation, habitat loss, and soil degradation.

Cost of Handling the wastes: These industrial wastes need large areas of dumps with many numbers of machinery requires to handle the wastes. The cost of handling fly ash and slag waste can vary depending on several factors such as the location, quantity, quality, and the availability of markets for their reuse. Here are some factors that can affect the cost of handling these wastes:

I. Transportation: The cost of transporting fly ash and slag from the source to the cement production plant can be a significant factor. The distance between the source and the plant, as well as the mode of transportation, can affect the cost.

II. Storage: Storing fly ash and slag requires space and infrastructure, such as silos and warehouses. The cost of building, maintaining, and operating storage facilities can affect the overall cost.

III. Quality control: The quality of fly ash and slag can vary, and their properties can affect the performance of the resulting cement products. Ensuring the quality of these materials requires testing and quality control measures, which can increase the overall cost.

IV. Market demand: The availability of markets for fly ash and slag reuse can affect the cost. If there is high demand for these materials in other industries, such as construction or road building, the cost of handling and transporting them may be lower.

V. Environmental regulations: Depending on the location, environmental regulations may require specific handling and disposal methods for fly ash and slag. Compliance with these regulations can add to the cost of handling this waste.

To minimize their negative impacts, it is important to implement appropriate regulatory frameworks, management practices, and technologies to ensure that these materials are utilized as supplementary materials in various industries.

However, the benefits of using these materials in cement production can outweigh the costs, including reducing greenhouse gas emissions, conserving natural resources, and improving the properties of cement products.

The utilization of industrial wastes, such as fly ash and blast furnace slag has several benefits including:

- **Reducing the environmental impact:** Industrial waste is produced in large quantities and their disposal can cause environmental pollution. By utilizing industrial wastes in cement production, the environmental impact of their disposal is reduced.
- **Conserving natural resources:** The utilization of industrial wastes in cement production reduces the demand for natural resources, such as limestone and clay.
- **Improving the performance of cement:** Fly ash and blast furnace slag are supplementary cementitious materials (SCMs) that can improve the performance of cement. They improve the workability of concrete, increase its durability, and reduce the heat of hydration.
- **Cost savings:** The use of industrial wastes in cement production can reduce the cost of cement production by reducing the demand for raw materials and energy.
- **Carbon footprint reduction:** The use of industrial wastes in cement production can reduce the carbon footprint of cement production by reducing the amount of carbon dioxide emissions associated with the production of cement.
- **Reducing the need for virgin raw materials:** Both slag and fly ash are waste products generated by other industries such as steel and power generation. Using these materials in cement production can reduce the need for virgin raw materials such as limestone and clay, conserving natural resources.
- **Lowering greenhouse gas emissions:** The production of cement is energy-intensive and releases a significant amount of carbon dioxide into the atmosphere. However, the use of slag and fly ash can reduce the amount of cement clinker needed to produce a certain amount of cement, lowering greenhouse gas emissions associated with cement production.
- **Improving waste handling:** The use of slag and fly ash in cement production can help reduce the amount of waste generated by other industries. This not only conserves natural resources but also reduces the amount of waste that would otherwise be sent to landfills.
- **Enhancing the properties of cement:** Both slag and fly ash contain minerals that can improve the properties of cement. For example, slag can improve the durability of concrete

by reducing permeability and increasing resistance to sulfate attack. Fly ash can enhance the workability and durability of concrete, while also reducing heat of hydration.

Overall, the utilization of industrial wastes in cement production is a sustainable solution that provides benefits to the environment, the cement industry, and the economy.

So, the main purpose of this experimental analysis is to study the feasibility of using these waste products as recycling materials in the cement production and to learn the basic methodology involved in the cement manufacturing.

1.2 Problem Statement

The ratio of clinker to these industrial wastes in cement manufacturing is an important parameter that affects the quality and environmental impact of cement production. However, finding the optimal ratio can be challenging due to several factors, including the properties of the raw materials, the desired properties of the resulting cement, and the environmental impact of the production process. Optimizing the ratio of clinker to recycled materials in cement manufacturing to reduce the environmental impact and improve the quality of cement products. The challenge is to find the optimal ratio that balances the need for reducing greenhouse gas emissions, conserving natural resources, enhancing the properties of cement products, and minimizing production costs.

This problem statement highlights the need to balance several factors in cement production and find a solution that is environmentally sustainable, economically feasible, and meets the desired performance standards for cement products.

1.3 Objectives

- To collect the by-product samples from the respective industries and analyze.
- To make various proportions of cement samples by blending with raw materials properly for satisfying the relevant industrial standards.
- To characterize the cement samples prepared
- To determine the optimal ratio of cement sample by considering strength and cost of the prepared samples.

CHAPTER -2

LITERATURE REVIEW

2.1 Literature Review

- **Osman Gencel, et. al. (2021)** This review examines the usage of steel slag in the cement and concrete industry and its environmental effects [7]. The physical and chemical structure of steel slag, its impact on the characteristics of concrete, and its various applications in different usage areas are also discussed. The review is based on a thorough analysis of existing literature on steel slag usage in the cement and concrete industry. The review emphasizes the importance of using steel slag as a by-product of the steel industry, which would otherwise be inconvenient to store and release into the environment. The benefits of reusing steel slag in the cement and concrete industry are discussed, including economic and ecological balance. The review also highlights the challenges in managing steel slag and recommends effective strategies for its utilization. The authors present a comprehensive overview of the impact of steel slag on the properties of concrete, including its effects on workability, compressive strength, durability, and other important factors. The review also presents case studies of steel slag usage in the cement and concrete industry in different parts of the world and assesses their effectiveness. Finally, the review provides recommendations for the optimal utilization of steel slag in the cement and concrete industry. The authors suggest that further research is needed to fully understand the potential of steel slag as a construction material and to develop effective strategies for its utilization. They also recommend the implementation of sustainable practices for the management and utilization of steel slag to minimize its impact on the environment.
- **Singh S.K. et. al**. in their article, introduced the possibility of adding LD slag with the cement raw materials for production of Portland cement clinker by the various proportions of replacement of clinker with raw materials. The raw mix samples were burnt in muffle furnace at 1400° C and determined the free lime content by the ethylene glycol method to compare the burnability of raw materials. The chemical and mineralogical composition of prepared clinker samples was done by XRF and XRD techniques, which tells the addition of LD slag in the raw mix did not alter the mineralogical composition of the clinker. Also found that iron content present in the slag acts as a fluxing agent. This study showed that LD slag can be used as a replacement of raw materials in the production of clinker.[18]

- **Tsakiridis P.E, et. al. (2008)** The aim of this research work was to investigate the feasibility of using steel slag as an additive in the production of Portland cement clinker. The researchers prepared two samples of raw meals, one with ordinary raw materials and another with 10.5% steel slag, and sintered them at 1450 °C. They conducted chemical and mineralogical analyses as well as microscopic examinations, which showed that the use of steel slag did not affect the mineralogical characteristics of the produced cement clinker. [5]

- **I.M. HELMY (2003)** According to this paper the main aim is to study the utilization of the several industrial wastes that are by-pass cement dust, phosphor gypsum and granulated slag as mineralizers in the formation of port land cement clinker. In this work, the author has found the effect of cement dust, phosphor gypsum and granulated slag when they are used in the formation of clinker also investigated the formation of clinker by XRD technique. These wastes mixed with limestone in various proportions and fired at 1200° C, 1300° C and 1400° C and then suddenly cooled at atmospheric air. And estimated the results of samples of free lime and insoluble residue contents, then concluded that addition of these industrial wastes increases the formation of alite in the clinker because of these wastes includes high Cao content and phosphor gypsum acts as mineralizer in the clinker formation.

- **MIFENG GOU et. al. (2019)** The main aim of this article is to Utilize the tailings in cement and concrete, this paper reviews the potential utilization of tailings as a replacement for fine aggregates, as supplementary cementitious materials (SCMs) in mortar or concrete, and in the production of cement clinker. Their review on this work says, the compressive strength of concrete decreased as the replacement content of the tailings as SCMs increased, even when tailings were ground into smaller particles, also in the cement. As a result, the utilization of tailings in cement and concrete will be good for the environment both in the solid waste processing and virgin materials used in the construction industry.

- **Mandal A.K, et al.** (Construction and Building Materials, 2017): This review examines the use of fly ash and slag as partial substitutes for cement in concrete production. The authors discuss the properties of these materials, their effects on concrete properties and durability, and their environmental benefits.

- **Paulo Ricardo de Matos, et. al. (2020)** The paper emphasizes the need for effective solid waste management practices to reduce the environmental impact of solid waste generated by the steel industry in India. The authors discuss various recycling and utilization techniques that can be employed to manage steel making slag, including its use in road construction, as an aggregate in concrete, and as a raw material to produce cement. The paper also highlights the efforts of the Indian government towards developing policies and regulations for solid waste management in the steel industry. The authors recommend the implementation of integrated solid waste management practices that include segregation, collection, transportation, and disposal of solid waste in a safe and sustainable manner. The paper provides valuable insights into the challenges of solid waste management in the steel industry in India and highlights the need for effective management practices to reduce the environmental impact of solid waste generated. The recommendations provided by the authors can be useful for policymakers, industry stakeholders, and researchers in developing effective strategies for sustainable solid waste management. [6]

- **Pitroda J, et. al. (2012)** This research study investigates the use of thermal industry waste as a partial replacement of cement in concrete production. Fly ash is used as a supplementary cementitious material to replace cement in concrete mixtures at varying levels (0%, 10%, 20%, 30%, and 40% by weight of cement) for M-25 and M-40 mix. The mechanical properties of the resulting concrete mixtures were evaluated and compared to conventional concrete in terms of compressive and split strength. The tests were conducted up to 28 days for compressive strength and 56 days for split strength. [8]

- **Papadakis V.G. (1999)** This study investigates the use of a low-calcium fly ash as an additive in mortar, replacing a portion of either Portland cement or aggregate. The development of strength, heat, porosity, bound water, and calcium hydroxide content was measured [9]. Results showed that higher strengths were observed after 14 days in aggregate replacement, while higher strengths were observed after 91 days in cement replacement. The final strength gain was found to be proportional to the content of active silica. Bound water content and porosity results showed that fly ash reacts with calcium hydroxide and binds small amounts of water. A simplified scheme describing the chemical reactions of the fly ash in hydrating cement is proposed, with quantitative expressions for estimating the composition of a fly ash concrete. Overall, the study provides insights into the use of fly ash as an additive in mortar and its effect on concrete properties.

- **Bouzoubaa N, et. al. (1999)** This paper discusses the impact of grinding fly ash on their physical and chemical properties, and their effect on mortars and concretes incorporating blended fly ash cement. It compares the production of blended fly ash cement by grinding Portland cement clinker and fly ash together versus separately followed by blending. The combined grinding process for producing high-volume fly ash blended cements is also examined. The paper concludes with recommendations for further research in this area, particularly regarding the effects of grinding on the quality of blended fly ash cement and their mechanical and durability properties in concrete. [10]

- **Caijun Shi, et. al. (2008)** This paper discusses the potential applications of copper slag, a by-product of copper smelting and refining, in the cement and concrete industry [11]. Common management options for copper slag are described, including recycling, and producing value-added products. However, a large amount of copper slag is still disposed of in dumps or stockpiles. The use of copper slag in cement and concrete production is investigated by many researchers, as it offers potential environmental and economic benefits. Copper slag can be used as raw material for clinker, cement replacement, and coarse and fine aggregates. The paper reviews the characteristics of copper slag and its impact on the engineering properties of cement, mortars, and concrete. Overall, the use of copper slag in cement and concrete production has the potential to provide significant benefits to the industry and the environment.

- **Hooton RD, et. al. (2009)** The study reviewed the effect of ground, granulated, blast-furnace slag and slag-blended cements on the drying shrinkage of concrete. The results showed that concretes containing slag had, on average, only a slightly higher drying shrinkage of 2.9% compared to concretes without slag. The study also found that the only mix design parameter that significantly influenced drying shrinkage was the total aggregate volume. Any increase in drying shrinkage of the slag concrete was typically reduced with increasing aggregate content. The level of slag replacement and the w/cm of the concrete mixture were not found to affect the relative drying shrinkage. The study also noted that the aggregate content of concrete made with slag was often lower than a comparable concrete made without slag, but a correction for this made the shrinkage of slag concretes the same as for Portland cement concretes. Finally, the study suggested that the increased relative shrinkage of slag-containing concretes may be partially due to the reduced gypsum content of the cementitious mixture, although this requires further investigation. [12]

- **Karasin A, et. al. (2014)** This study aims to investigate the effect of using fly ash as a replacement for cement in concrete structures exposed to high sulfate environments. The study conducted laboratory tests to determine the compressive strength and sulfate resisting capacity of concrete using 20% fly ash as a mineral additive instead of cement. Soil samples were tested for sulfate content according to standard parameters, the samples were then exposed to a Na2SO4 solution to simulate exposure to aggressive environments. The results of the study suggest that using fly ash as a replacement for cement does not significantly affect the compressive strength of concrete over time, and it may be a viable way to reduce cement usage in construction. The study is particularly relevant in areas where concrete structures are at risk of deterioration due to contact with underground water containing high sulfate levels.[13]

- **Kurama H, et. al. (2008)** The present study aims to determine and evaluate the applicability of an industrial bottom ash (CBA), supplied from Tunçbilek Power Station-Turkey, in concrete industry. In the laboratory experiments, the bottom ash was used up to 25% as a partial substitute for the Portland cement [14]. In order to be able to reduce the unburned carbon content, CBA was treated by three different processes (particle size classification, heavy medium separation, and electrostatic separation). Based on the obtained results, it was concluded that the addition of CBA up to 10% as a replacement material for Portland cement could improve the mechanical properties of concrete, and thus, could be used in the concrete industry. The effect of operating parameters on treatment processes has also been discussed in the paper.

- **Cao D, et. al. (2008)** The study discusses the potential utilization of fly ash from coal-fired power plants in China, focusing on aluminum recycling and using fly ash in concrete products. The chemical and physical analyses of Chinese fly ash samples are presented, including X-ray diffraction (XRD), ICP (Inductive Coupled Plasma), particle size analysis, and water requirement. The study reveals that reasonable amounts of aluminum can be found in fly ash samples, but the recovery process is currently sophisticated. Therefore, simpler techniques are suggested for the initial steps in the utilization of Chinese fly ash. The study also compares the situation in China and Germany regarding fly ash utilization.[15]

- **Abdel Rahman A, et. al. (2016)** This study investigated the use of active silica in improving the mechanical and physio-chemical properties of blended cement pastes made of Portland blast-furnace slag cement (PSC) containing cement kiln dust (CKD). Two sources of active silica, silica fume (SF) and rice husk ash (RHA), were used in this investigation. The study found that the partial substitution of PSC by CKD resulted in an increase in the rate of hydration and an improvement in the compressive strength of the hardened PSC-CKD pastes. The addition of active silica further improved the physio-mechanical characteristics of the hardened PSC-CKD pastes. X-ray diffraction (XRD) and differential thermal analysis (DTA) were used to analyze the phase composition of the formed hydration products, which helped to explain the observed improvements in the properties of the blended cement pastes. The investigation used a rigorous methodology, including the preparation of standardized cement pastes and the use of various testing techniques to analyze their properties. The study provides valuable insights into the potential of using active silica to enhance the performance of blended cement pastes, which could have important implications for the construction industry. The investigation emphasizes the importance of using appropriate testing techniques, such as XRD and DTA, to gain insight into the properties of cement pastes and the effects of active silica. The study contributes to the growing body of research on sustainable construction materials, by exploring the potential of using waste materials to improve the properties of blended cement pastes. The investigation highlights the potential of using waste materials, such as CKD and RHA, as sources of active silica in blended cement pastes, which could have important environmental benefits. The results of this investigation could be useful for practitioners and researchers in the construction industry, providing insights into ways of improving the performance of blended cement pastes. [16]

CHAPTER -3

MATERIALS & METHODOLOGY

3.1 Raw Material Collection

Limestone is the primary raw material used in cement production, which is typically sourced from quarries. In this project, the limestone used was sourced from a quarry located at coordinates 15.034790, 78.022685.

In this study, coal fly ash and metal slag were used as supplementary cementitious materials (SCMs) to replace some portion of Limestone clinker, thereby reducing the amount of clinker needed to produce concrete. Fly ash was collected from a power plant located at the given coordinates; metal slag was collected from Steel Plant Industry located at the given coordinates. The location of the fly ash and metal slag used in this study were at coordinates 17.591937, 83.125194 and 17.595572, 83.195940 respectively. The following fig 3.1 shows the location of raw materials collected from various locations

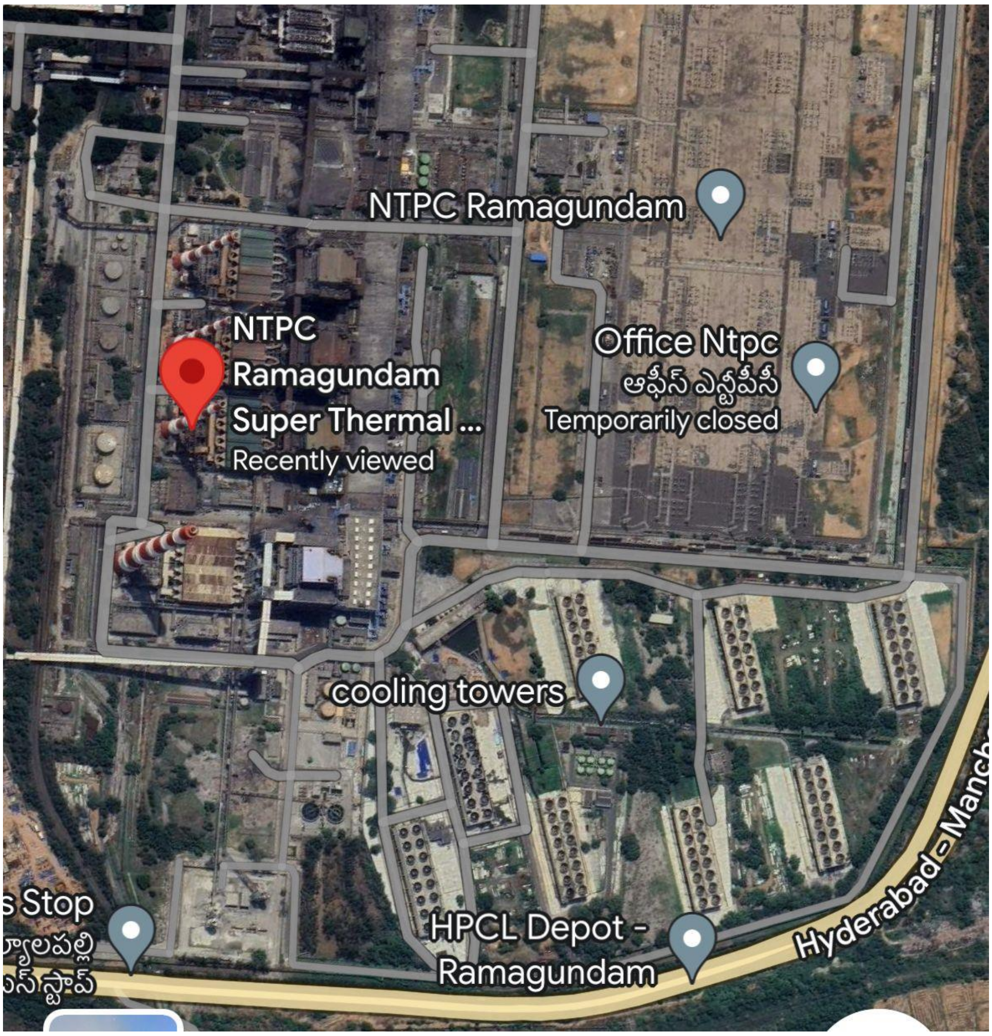


Figure 3.1: raw material collection locations

The following locations represent the coordinates of the raw materials:

Blast Furnace Slag at Dump site

3.2 Raw materials used in Cement Production

3.2.1 Limestone

Limestone is a major raw material in the production of cement which is the main source of the material lime commonly grey to white in colour and may also appears as yellow and brown. It is a Meta-sedimentary rock mainly consists of calcium carbonate ($CaCo_3$), carbonates of calcium and magnesium ($CaCo_3.MgCo_3$) also Known as calcite and dolomite. It is a soft rock with a Mohs hardness of 2 to 4 and can easily scratched. Limestone often contains some amount of silica (SiO_2), alumina oxide (Al_2O_3) and iron oxides (Fe_2O_3). The most common constituent of limestone is calcium carbonate ($CaCo_3$) whereas dolomite ($CaCo_3.MgCo_3$) is an uncommon mineral in limestone.

Limestone is an important industrial raw material that has high demand in steel and iron industries in the past nineteenth century. It is a raw material for the manufacture of quick lime (Cao), slaked lime ($Ca(OH)_2$), cement and mortar also used as a soil conditioner to neutralize acidic soils, reagent in flue gas desulfurization for air pollution control.

Meta-Sedimentary limestone deposits forms in thousands of square meters, and relatively uniform in quality and thickness. These quarries are large and long lived. In cement industry pure limestone should have lime percentage greater than 52% and Lime Saturation Factor (LSF) should be 105-110. The density of limestone ranges from 1.5 to 2.7 g/cm3.

It has a wide range of physical properties, depending on its composition and the conditions under which it was formed. Here are some of the key physical properties of limestone:

1. Colour: Limestone is typically light coloured, ranging from white to grey, yellow, brown, and even black.
2. Hardness: Limestone has a hardness of 3-4 on the Mohs scale, which is a measure of mineral hardness. It is softer than most igneous rocks, but harder than most clastic sedimentary rocks.

3. Porosity: Limestone is generally porous, with a range of porosity depending on the type of limestone and the conditions under which it was formed. Some types of limestone, such as chalk, are very porous and can hold large amounts of water.
4. Density: Limestone has a density of about 2.6-2.8 g/cm3, which is similar to other sedimentary rocks.
5. Texture: Limestone has a granular texture, with individual grains of calcite or other minerals visible to the naked eye. Some types of limestone, such as travertine, have a more crystalline texture.
6. Fossils: Limestone is often rich in fossils, particularly marine fossils such as shells, corals, and brachiopods. These fossils can provide important clues about the geological history of the rock.
7. Durability: Limestone is a relatively durable rock that is resistant to weathering and erosion. However, it can be affected by acid rain and other forms of chemical weathering, which can dissolve the calcite and weaken the rock over time.
8. Overall, limestone is a versatile and widely used rock that has a range of physical properties that make it suitable for a variety of applications, including construction, agriculture, and industry.
9. Limestone hardness is 2.5 and its permeability, porosity is very less hence water is not found in the beds.

Lithology:

- Cream coloured limestone
- Bluff colour limestone Bluish & black
- layers limestone
- Green coloured limestone

3.2.2 Gypsum

Gypsum is a crucial ingredient in cement production. Cement is primarily composed of clinker, which is produced by heating limestone and other materials at high temperatures in a kiln. Gypsum is added to the clinker during the grinding process to control the setting time of the cement and improve its workability.

When cement is mixed with water, it undergoes a chemical reaction called hydration, which results in the formation of a rigid matrix that binds the other ingredients together. However, this reaction can occur too quickly, making the cement difficult to work with and reducing its strength. Gypsum is added to the cement during the grinding process to slow down the reaction and give the workers more time to work with the cement before it sets.

In addition to controlling the setting time of the cement, gypsum also helps to prevent flash setting, a phenomenon where the cement sets too quickly and becomes unusable. This is especially important in hot climates where the cement can set rapidly due to high temperatures. Gypsum is the name given to a mineral categorized as calcium sulphate mineral, and its chemical formula is calcium sulphate dihydrate, $CaSO4 \cdot 2H2O$.

3.2.3 Industrial By-products as Clay Materials

Industrial by-products can also be used as clay materials in the cement manufacturing process. Some of the commonly used industrial by-products as clay materials include Blast Furnace slag, Fly ash, copper slag and Rise Husk ash.

These industrial by-products can be used as a partial replacement for traditional clay materials in the cement manufacturing process. This not only reduces the reliance on natural resources but also reduces the amount of waste that is sent to landfills. The use of industrial by-products in cement production also has the potential to improve the performance of cement and reduce its environmental impact.

3.2.3.1 Fly ash

Fly ash is a fine powder that is a byproduct of burning pulverized coal in electric power generating plants. It is the largest industrial waste by volume generated in the world. Fly ash consists mainly of oxides of silicon (SiO2), aluminum (Al_2O_3), and iron (Fe_2O_3), with small amounts of other elements. It is typically collected from the flue gases by electrostatic precipitators or bag filters before they are released into the atmosphere. Fly ash is generally a fine, light gray powder that is easy to handle and transport. It has a low density and is relatively low in heavy metals and other pollutants. Fly ash has a high silica content, which makes it an excellent pozzolan for use in concrete and other construction materials. Its pozzolanic properties make it a valuable material in the construction industry, where it is often used as a partial replacement for Portland cement.

Physical Properties of Fly Ash:

The physical properties of fly ash can vary depending on the source of coal and the combustion process used. However, some of the common physical properties of fly ash are:

- **Particle size distribution:** Fly ash is composed of fine particles that can range in size from a few microns to several hundred microns. The particle size distribution of fly ash can affect its pozzolanic reactivity and its ability to be used in various applications.
- **Specific gravity:** Fly ash has a low specific gravity, typically ranging from 2.0 to 2.5 g/cm3. This property makes fly ash lightweight and suitable for use in lightweight concrete.
- **Color:** Fly ash is typically a light gray or tan color.
- **Fineness:** Fly ash is a finely divided powder, with a high surface area per unit weight. The fineness of fly ash can affect its reactivity and its ability to be used in various applications.
- **Density:** The density of fly ash can range from 1.0 to 1.4 g/cm3, depending on the specific type of fly ash.
- **Moisture content:** Fly ash can absorb moisture from the environment, which can affect its physical properties and handling characteristics.

Chemical Composition:

The chemical composition of fly ash can vary depending on the source of coal and the combustion process used. However, fly ash is generally composed of oxides of silicon, aluminum, iron, and calcium, as well as other minor elements.

Fly ash is a byproduct of burning coal in power plants and consists primarily of silicon dioxide (SiO_2), aluminum oxide (Al_2O_3), and iron oxide (Fe_2O_3), as well as smaller amounts of calcium oxide (Cao) and other trace elements. The exact composition of fly ash can vary depending on the type of coal burned and the combustion process used, but SiO_2, Al_2O_3, Fe_2O_3, and Cao are commonly found in fly ash samples.

The Chemical composition of Fly Ash depends on the type of Coal used in the burning and dilution at the dump site. The Silica and Cao present in the fly ash at high concentrations that yields the better strength to the concrete when the fly ash utilized in the cement manufacturing. The following data shows the General Composition of Fly Ash on the basis of type of Coal used in the Power Plant this data is given by the authorities of Power plant as shown in the Table 3.1

Table 3.1: General Composition of Fly Ash on the basis of type of Coal used in the Power Plant.

Component	Bituminous	Subbituminous	Lignite
SiO_2 (%)	20–60	40–60	15–45
Al_2O_3 (%)	5–35	20–30	20–25
Fe_2O_3 (%)	10–40	4–10	4–15
Cao (%)	1–12	5–30	15–40
LOI (%)	0–15	0–3	0–5

Various types of coal fly ashes can differ in their chemical composition and physical properties, depending on the type of coal burned, the combustion technology used, and the conditions of combustion. Here are some examples:

Bituminous Coal Fly Ash: Bituminous coal is the most common type of coal used for power generation, and its fly ash typically contains high levels of silica, alumina, iron oxide, and calcium oxide. Bituminous coal fly ash is often used in construction materials, such as concrete, due to its high pozzolanic activity.

Sub-Bituminous Coal Fly Ash: Sub-bituminous coal is a lower quality coal than bituminous coal and is often used for power generation in areas where it is abundant. Sub-bituminous coal fly ash typically contains lower levels of silica and alumina but higher levels of calcium oxide, magnesium oxide, and sulfur trioxide. It is often used in cement production and as a soil amendment. Lignite Coal Fly Ash: Lignite coal is the lowest quality coal and is typically used for power generation in areas where it is abundant. Lignite coal fly ash typically has a higher content of calcium oxide and lower content of silica and alumina. It is often used in cement production, as a soil amendment, and as a filler in plastics and other materials.

Fig 3.2: NTPC Fly Ash Dump Site (Collection Point)

3.2.3.2 Granulated Blast Furnace Slag:

Granulated Blast Furnace Slag (GBFS) is a by-product of the iron-making process and is produced by quenching molten iron slag (a mixture of silica, alumina, calcium oxide, and magnesium oxide) with water. This rapid cooling process results in the formation of a glassy, granular material that is similar in appearance to coarse sand. Blast furnace process is a commonly used method for producing iron from iron ore, iron scrap, limestone, and coke. The coke is burned in the blast furnace to produce carbon monoxide, which reacts with the iron ore to reduce it to molten iron. Along with the molten iron, a non-metallic coproduct called blast furnace slag is also produced in the process.

Blast furnace slag is primarily composed of silicates, aluminosilicates, and calcium-alumina-silicates, and it absorbs much of the sulfur from the charge. The molten slag comprises about 20 percent by mass of iron production. Depending on the method used to cool the molten slag, different forms of slag products are produced, such as air-cooled blast furnace slag (ACBFS), expanded or foamed slag, pelletized slag, and granulated blast furnace slag.

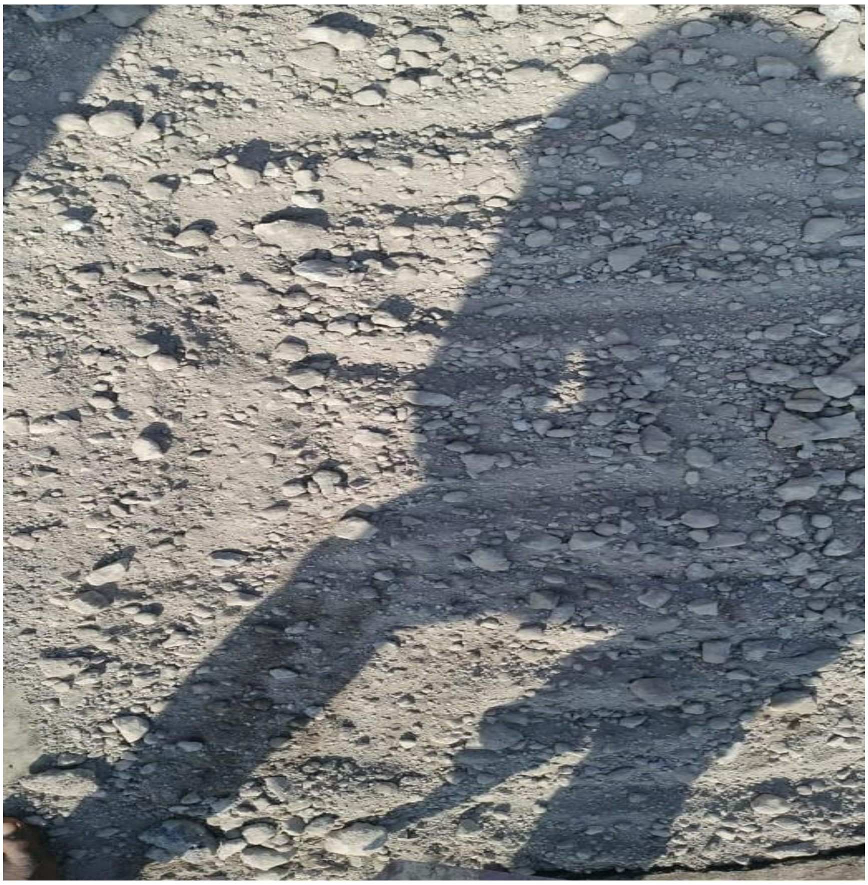


Fig 3.3: Blast Furnace Slag at Collection Point

Physical Properties:

Blast furnace slag is a non-metallic byproduct of iron production that has a range of physical properties.

Here are some of the physical properties of blast furnace slag:

- Color: Blast furnace slag typically has a light color ranging from beige to grey.
- Texture: The texture of blast furnace slag can vary depending on the cooling process used. For example, air-cooled blast furnace slag has a rough, angular texture, while granulated blast furnace slag has a smoother, more glassy texture.
- Density: The density of blast furnace slag can vary depending on the cooling process and the chemical composition. Typically, it has a density of around 2.9-3.4 g/cm^3.
- Hardness: Blast furnace slag is relatively hard and abrasive, with a Mohs hardness of around 6-7.
- Porosity: Blast furnace slag has a low porosity, meaning it is relatively impermeable to water and other liquids.
- Chemical stability: Blast furnace slag is chemically stable and does not react with most materials, making it a suitable material for use in construction applications.
- Durability: Blast furnace slag is highly durable and resistant to weathering, erosion, and chemical attack, making it an ideal material for use in long-lasting infrastructure projects.

Chemical Composition:

Granulated Blast Furnace Slag (GBFS) is a by-product of iron production, which is extensively used as a supplementary cementitious material in the production of cement. The chemical composition of GBFS varies depending on the source and the manufacturing process. GBFS typically contains silica (SiO_2), alumina (Al_2O_3), calcium oxide (CaO), magnesium oxide (MgO), and sulfur trioxide (SO3) as its major chemical components. The chemical composition of Granulated Blast Furnace Slag (GBFS) can vary depending on the specific production process and source of the raw materials. However, in general, GBFS is composed shown in the table 3.2 taken the data from authorities of Vizag Steel Plant

Table 3.2: General Composition Range of Iron Slag Collected from Steel Plant

S.NO	Parameter	Content
1	CaO	33-40%
2	SiO_2	32-38%
3	Al_2O_3	14-20%
4	MgO	7-10%
5	FeO	3.0% Maximum
6	MnO	1.0% Maximum

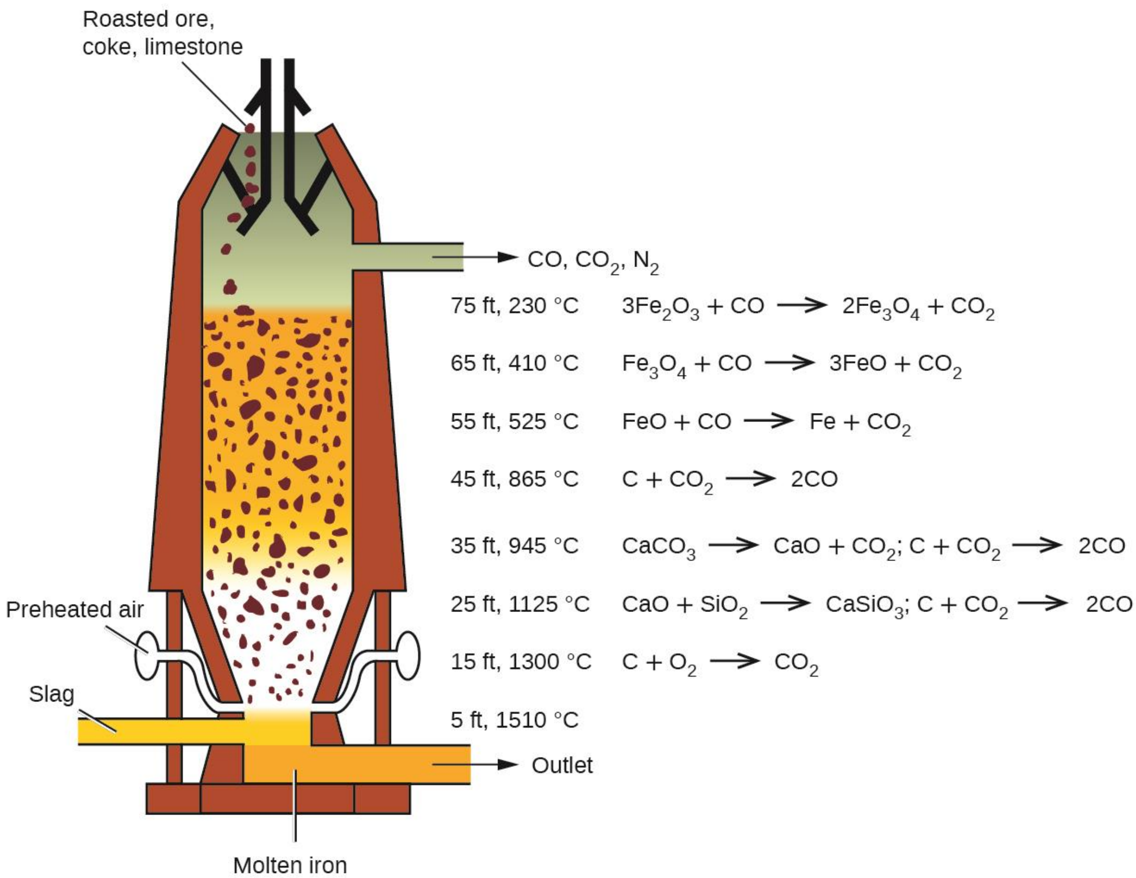


Fig 3.4: Formation of Slag in Blast Furnace in the separation of Iron from Iron ore [19]

<u>3.3 Characterization of Industrial By-Products and clinker</u>

Both fly ash and iron slag contain similar oxides, but their relative concentrations can vary depending on the specific source and production method. Both wastes have the potential to be recycled and used in various applications, such as in construction and infrastructure projects. However, it is important to properly characterize and handle these wastes to ensure their safe and effective use.

Fly ash and Iron slag can be characterized by their respective chemical composition: (The results were determined by using titrimetric methods in Pharmacal Laboratory with reference to Indian standards of Chemical composition tests for cement.)

Titrimetric methods are commonly used in chemical laboratories to determine the chemical composition of the raw materials. The procedure for using titrimetric methods to determine chemical composition typically involves the following steps:

1. Sample preparation: Prepare a representative sample of the material to be analyzed. The sample should be homogeneous and free from any contaminants.
2. Standard solution preparation: Prepare a solution of known concentration of the titrant or reagent. The concentration of the standard solution should be accurately known.
3. Titration: Add the standard solution to the sample until a reaction is complete, as indicated by a change in color or another observable indicator. The volume of the standard solution required to complete the reaction can be used to calculate the concentration of the analyte in the sample.
4. Calculation: Calculate the concentration of the analyte in the sample using the volume of the standard solution and the known concentration of the standard solution.

3.4 Manufacturing Process:

The manufacturing process for making cement involves several steps. First, limestone is heated to a temperature of 1450°C in a kiln, which produces a material called clinker. The clinker is then cooled and ground into a fine powder. This powder is the primary source of lime (Cao) used in the manufacturing process. Next, raw materials are ground to a size of a few microns and then blended with industrial by-products. These industrial by-products can be waste materials from other manufacturing processes, such as slag from steel production or fly ash from power plants. The blending of raw materials and industrial by-products is done to create a mixture with the desired chemical composition.

Finally, a small percentage of gypsum is added to the mixture to regulate the setting time of the final product. The mixture is then heated in a kiln to a temperature of around 1450°C to produce the final product, which can be used in various construction and industrial applications. Overall, the manufacturing process for lime-based products involves a complex series of steps that require careful attention to detail to ensure the final product meets the desired specifications.

3.4.1 Crushing and Grinding the Raw Materials

The production of cement involves various stages, including crushing and grinding, which are fundamental processes in reducing the size of raw materials. Raw materials such as fly ash, iron slag, clinker, and gypsum are used in the production of cement. The first step in the process is primary crushing, where the raw materials are initially reduced in size using a laboratory jaw crusher. This step is essential to prepare the raw materials for further processing.

The laboratory jaw crusher is a powerful machine that can handle even the toughest materials. It is designed to crush materials with a compressive strength of up to 300 MPa, ensuring that even the most robust raw materials can be processed. The raw materials are typically reduced to a size of 10-15 mm in this step. The next step is secondary crushing, where the materials from primary crushing are further reduced in size using a laboratory roll crusher. This step is necessary to reduce the size of the particles to a range of 1-0.5 mm, which is the ideal size for further processing. The laboratory roll crusher is designed to handle the material effectively and efficiently, ensuring that the particle size is reduced to the desired level.

After secondary crushing, the materials are then subjected to grinding using a laboratory pulveriser. The pulveriser is a machine that is used to reduce the size of materials to a range of 50 to 120 microns. This step is critical in the production of high-quality cement, as the finer particles produced by grinding are essential for the final product. To ensure that the final product meets the desired specifications, the material is sieved using a laboratory sieve shaker. In this step, a 0.075 mm IS sieve is used, and the materials that pass through the sieve (-0.075 mm) are stored for further processing, while the retained material (+0.075 mm) is loaded into a ball mill for further size reduction.

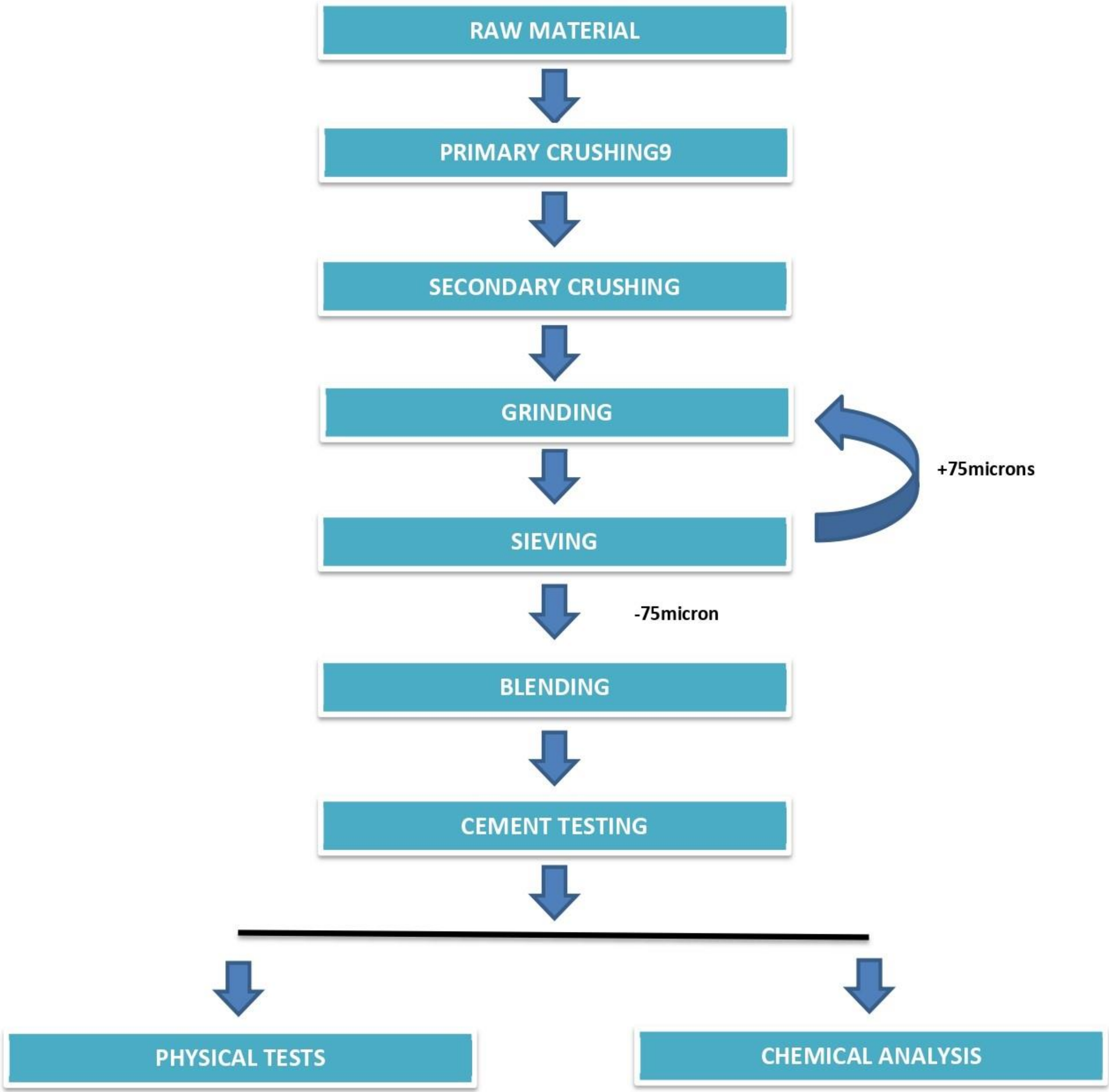


Fig 3.5: Sequence of operations for sampling and testing

JAW CRUSHER:

A primary crusher used in mines and ore processing plants is a jaw crusher. It operates by compressing rock material between two jaw plates: one that is still and the other that is moving. Rocks are compressed and crushed as they go from the moving jaw plate to the stationary jaw plate, and then they are ejected at the machine's base.

In the mining, quarrying, and recycling industries, jaw crushers are frequently employed. They can process materials with compressive strengths of up to 320 MPa. They come in a variety of sizes and capacities and are frequently used in conjunction with other crushing and screening machinery to produce products of the desired size and quality.

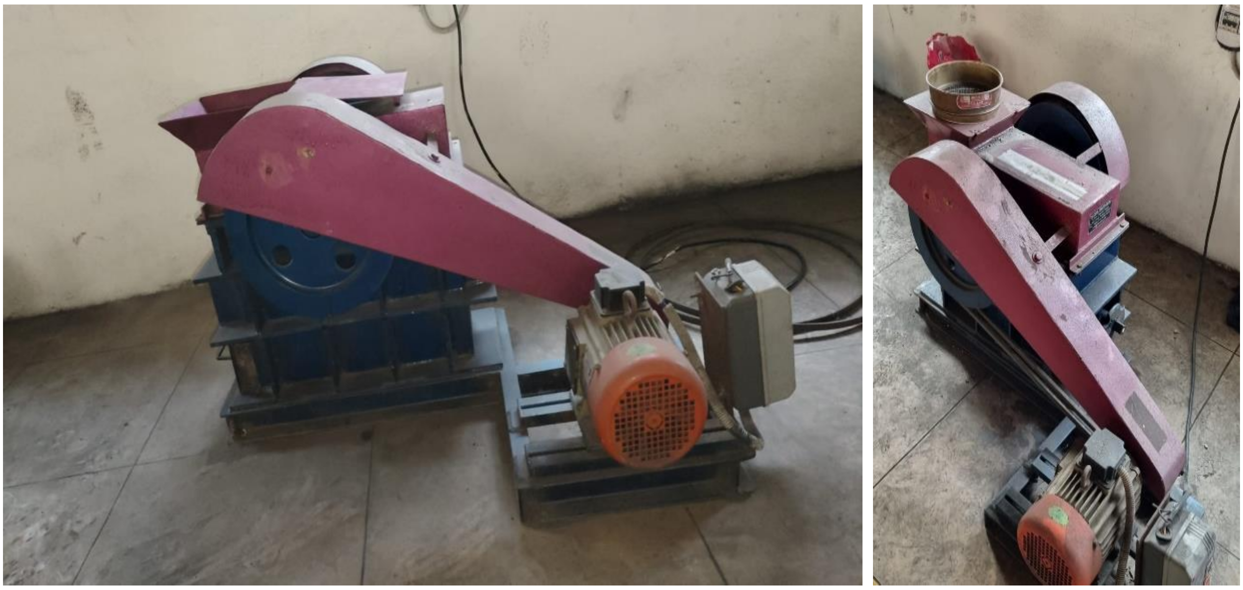


Fig 3.6: Jaw Crusher for primary crushing

ROLL CRUSHER:

A secondary crushing is a post primary crushing operation, in order to achieve further size reduction of raw materials to blend the desired cement which should satisfies the minimum range of particle size. A roll crusher is a type of crushing equipment used to reduce the size of large rocks or other materials into smaller pieces.

It consists of two large metal rolls, called cylinders or drums, that are mounted on horizontal shafts and rotate in opposite directions. The material to be crushed is fed into the top of the rolls and is compressed between the rolls as they rotate, causing it to break apart into smaller pieces.

Roll crushers are commonly used in mining and mineral processing industries, where they are used to crush ore and other materials. They can also be used in other industries, such as construction, for the reduction of materials such as limestone, coal, and cement clinker. Roll crushers are often used in combination with other types of crushers, such as jaw crushers and cone crushers, to achieve a more uniform and finer product size.

Fig 3.7: Roll Crusher for Secondary crushing

PULVERISER:

A mechanical tool called a pulveriser is used to crush, grind, and pulverise materials into tiny particles. It is frequently used to process commodities like minerals, chemicals, plastics, and food items in a variety of industries. The purpose of the pulveriser, which can work in batch or continuous modes, is to reduce the size of materials so that they are simpler to handle or utilise in subsequent procedures.

Fig 3.8: Pulveriser for Grinding

SHIEVE SHAKER:

A sieve shaker is a laboratory instrument used to shake a stack of test sieves to separate particles according to size. The sieves are subjected to mechanical vibration, which causes the particles to shoot up and down through the mesh holes. Particles of various sizes are thus divided into several layers on the sieves. In fields like pharmaceuticals, food processing, and mining, sieve shakers are frequently employed in particle size analysis, quality control, and R&D. They come in diverse shapes and sizes and can fit various test sieve sizes and types, ranging from manually controlled to automatic equipment.

A laboratory sieve shaker has multiple sieves stacked on top of each other, with the smallest mesh size on top and the largest mesh size on the bottom. The number of sieves can vary depending on the specific model and application. The mesh size of the sieve refers to the number of openings per linear inch. The smaller the mesh size, the smaller the particles that can pass through the sieve. Common mesh sizes used in laboratory sieve shakers include 20, 40, 60, 80, 100, and 200. The shaker mechanism can also vary depending on the specific model and manufacturer. Some laboratory sieve shakers use a simple reciprocating motion, while others use a more complex rotary and tapping motion to improve particle separation. The capacity of a laboratory sieve shaker can vary depending on the specific model and application. Some laboratory sieve shakers can accommodate multiple sieves and large sample sizes, while others are designed for smaller samples and fewer sieves.

Fig 3.9: Sieve Shaker

3.4.2 The Process of Clinker Formation

Clinker formation is a critical process in the production of cement. It involves the transformation of raw materials into a substance called clinker, which is the primary ingredient in cement. The raw materials for making clinker are typically limestone, clay, and other minerals, which are quarried and crushed into a fine powder. This powder is then mixed with water to form a slurry, which is heated in a kiln to temperatures of up to 1450°C.

The main stages of clinker formation can be described as follows:

Portland cement is produced through a complex series of chemical reactions in a rotary cement kiln. The raw materials used in cement production are typically limestone ($CaCO_3$), silicon dioxide (SiO_2), aluminium oxide (Al_2O_3), and iron oxide (Fe_2O_3). These raw materials are crushed and then heated in a rotary kiln at temperatures of up to 1,450°C.

The first stage of the process is the preheating of the raw materials. The raw materials are heated to around 800°C, which causes them to release any moisture that they may contain. This is followed by the evaporation of free water, which requires a pressure above atmospheric to vaporize the water from the slurry mixture of raw materials. Water becomes superheated, and the evaporation gradually stops when the temperature rises above 120°C.

The next stage is clay decomposition. “Clay” minerals account for most of the alkalis in the raw materials, the most common of which is kaolinite, $Al_2Si_2O_5(OH)_4$. The detached alkalis react with the acid gases present in the kiln at high temperature. The effective reactions here are:

$$Si_2Al_2O_5(OH)_2 \rightarrow 2\ SiO_2 + Al_2O_3 + 2\ H_2O\ \text{(vapor)}$$

$$KAlSi_3O_8\ \text{(orthoclase)} + 0.5\ SO_2 + 0.25\ O_2 \rightarrow 3\ SiO_2 + 0.5\ Al_2O_3 + 0.5\ K_2SO_4$$

Dolomite decomposition is the next stage. The magnesia in the raw mix exists mainly as dolomite, $CaMg\ (CO_3)_2$, but also as silicate or in carbonate form. Dolomite reacts as follows:

$$CaMg\ (CO_3)_2 \rightarrow CaCO_3 + MgO + CO_2$$

Non-carbonate magnesium compounds (for example, phlogopite) react thus:

$$KMg_3AlSi_3O_{10}(OH)_2 + 0.5\ SO_2 + 0.25\ O_2 \rightarrow 0.5\ K_2SO_4 + 3\ MgO + 0.5\ Al_2O_3 + 3\ SiO_2 + H_2O\ \text{(vapor)}$$

The low-temperature calcite decomposition is the next stage. Calcium carbonate, present in the raw mix as calcite, produces carbon dioxide, the amount of which exceeds half the mass of the finished clinker. This requires a huge heat input. The efficiency of this reaction is one of the factors that determine the output and heat consumption in the kiln. Pure calcite in the kiln decomposes at around 650°C:

$$2\ CaCO_3 + SiO_2 \rightarrow Ca_2SiO_4 + 2\ CO_2$$

Reactive clay decomposition products and small amounts of alkali sulphate/chloride-melt draw the products together by surface tension and act as an ion transfer medium. Here, CO2 is produced, but no free lime (CaO) is formed. In the silicate phases, magnesium reacts with silica to produce forsterite (which goes into solid solution in belite):

$$2\ MgO + SiO_2 \rightarrow Mg_2SiO_4$$

Phosphorus (as apatite in raw mix) reacts with a little free silica and produces whitlockite (which also goes into solid solution in belite):

$$Ca_5(PO_4)_3OH + 0.25\ SiO_2 \rightarrow 1.5\ Ca_3(PO_4)_2 + 0.25\ Ca_2SiO_4 + 0.5\ H_2O\ \text{(vapour)}$$

This stage ends when all the silica in the kiln is used up.

Alumina and iron oxide react with calcium carbonate, and tricalcium aluminate is stable at this stage. Poorly crystallized mayenite ($Ca_{12}Al_{14}O_{33}$)

Portland cement clinker is a "phase assemblage" in which the main mineral crystalline species are:

Alite - roughly Ca_3SiO_5

Belite - roughly Ca_2SiO_4

Tricalcium Aluminate - roughly $Ca_3Al_2O_6$

Tetracalcium aluminoferrite - roughly $Ca_2(Al,Fe)_2O_5$

All these chemical formulae are "rough" because the phases are actually solid solutions, and the atoms of the formula are substituted to a small but significant extent by other elements. For instance, some of the calcium locations in the belite crystal can be occupied by magnesium, and some of the silicon locations can be occupied by aluminium, phosphorus, sulfur, iron etc. Furthermore, the crystals of the phases are usually highly defective, with discontinuities in their structure and missing atoms, this being a desirable characteristic because defects increase the crystal's reactivity.

A typical modern good-quality general purpose grey clinker might contain 72% alite, 9% belite, 7% tricalcium aluminate, 10 % tetra calcium alumina ferrite, 1% salt phases and 1% free calcium oxide, but depending on the properties desired, clinkers with markedly different compositions may be made.

Alite and belite are the "active ingredients" of cement, producing strength when hydrated. They are "primary phases" that remain solid throughout the clinkering process. Tricalcium aluminate and tetra calcium alumina ferrite does not contribute useful properties and can be to some degree deleterious. They are "interstitial phases" that crystallize from the liquid formed by partial melting during clinkering and fill in the spaces between the silicate crystals. They are present purely for the convenience of the manufacturer, because, without the liquid from which they form, the clinker cannot be formed at an economically viable temperature.

3.4.3 Blending the materials with Various Proportions:

Blending of raw materials in cement production is a crucial process that determines the quality of the final product. The traditional approach involves using limestone, clay, and other natural resources in specific proportions to produce cement. However, by using these industrial by-products, we have prepared few cement samples. Such samples contain different concentrations of industrial wastes varies from 0% to 97% for Blast Furnace Slag and 0% to 50% replacement for Fly ash.

Table 3.3: Various proportions of the prepared samples for Fly ash.

S.No.	Sample Name	Fly ash (%)	Clinker (%)	Gypsum (%)
1	A0	0	97	3
2	A10	10	87	3
3	A20	20	77	3
4	A30	30	67	3
5	A40	40	57	3
6	A50	50	47	3

The Raw materials were weighed by a micro weighing machine for an overall weight of each cement sample was 1.4 kgs. The measured raw materials were blended in a ball mill for proper mixing of all raw materials. Finally, the blended cement samples are ready to conduct physical tests and chemical analysis.

Fig 3.10: Raw material weighing and blended samples

Table 3.4: Various proportions of the prepared samples for Blast Furnace Slag.

S.NO	Sample Name	Slag (%)	Clinker (%)	Gypsum (%)
1	S0	0	97	3
2	S10	10	87	3
3	S20	20	77	3
4	S30	30	67	3
5	S40	40	57	3
6	S50	50	47	3
7	S60	60	37	3
8	S70	70	27	3
9	S80	80	17	3
10	S90	90	7	3
11	S100	97	0	3

3.4.4 Physical Tests on Prepared Cement Samples:

Testing is essential for ensuring the quality and performance of cement. Some of the main tests that should be done on cement include fineness, setting time, compressive strength, soundness, consistency, chemical analysis. These tests help to determine the particle size distribution, setting time, load-bearing capacity, stability, workability, chemical composition, and heat production of the cement. By conducting these tests, any issues with the cement can be identified and corrected, ensuring that it meets the required standards for quality and performance.

3.4.4.1 Fineness Test

The fineness of cement significantly impacts the rate of hydration, heat evolution, and strength development, making its evaluation crucial. The rate of hydration and strength development increases with the fineness of cement, and hence, it is essential to ensure that the cement used in construction meets the desired fineness requirements. The sieve test provides accurate measures of cement fineness, and their use can prevent the development of weak concrete structures.

Procedure: The fineness test requires a few key materials, including test sieves, a sieve shaker, a weighing scale, a sample of the material being tested, and a cleaning tool. By performing this test, engineers and construction professionals can better understand the particle size distribution and fineness of the material, which can help to optimize its use in construction applications.

Test sieves: Test sieves are used to separate the material into different particle size fractions. The sieves are made of brass or stainless steel and come in various sizes, such as 45 microns, 90 microns, or 150 microns.

Sieve shaker: A sieve shaker is used to agitate the sieves and ensure that the material is evenly distributed and separated into the appropriate particle size fractions.

Weighing scale: A weighing scale is used to weigh the material before and after the test to determine the percentage of material that passes through each sieve.

Sample of material: A representative sample of the material being tested is required to perform the fineness test. This sample should be collected according to specific procedures to ensure that it is representative of the entire lot of material.

Brush or other cleaning tool: A brush or other cleaning tool is used to remove any particles that may be stuck on the sieves after the test is complete.

Weigh 100 g of cement using a weighing balance and record the weight.

1) Place the 90-micron IS sieve over a tray or container to collect the sieved cement.
2) Break any air-set lumps in the cement sample and place it on the sieve.
3) Sieve the cement continuously in a circular and vertical motion for 15 minutes. Alternatively, a mechanical sieve shaker can also be used.
4) After 15 minutes, remove the sieve and collect the residue left on it.
5) Weigh the residue and record the weight.
6) Calculate the percentage residue as follows:

   % Residue = (Weight of residue / Weight of original sample) x 10

7) Compare the calculated percentage residue with the maximum allowable residue for the type of cement being tested. For ordinary cement, the maximum residue should not exceed 10%.

If the percentage residue is within the allowable limit, the cement is considered to have passed the fineness test. If the percentage residue exceeds the limit, the cement is too coarse and may not perform as desired in concrete structures.

Note: It is important to ensure that the sieving process is done correctly to obtain accurate results. Additionally, it is recommended to perform the test in duplicate or triplicate to minimize errors.

3.4.4.2 Consistency Test

The consistency of cement paste is important because it helps to ensure the quality and performance of concrete structures. If the consistency of the cement paste is not standardized, it can result in variations in the strength and other properties of the concrete, leading to potential durability issues and structural failures. By determining the correct amount of water needed to achieve a standard consistency, engineers and construction professionals can ensure that the concrete mix is optimized for the specific application and can meet the desired performance criteria. Therefore, the consistency test is a crucial step in the quality control process for producing high-quality and durable concrete structures.

Procedure: To perform a consistency test, several materials are required. The main material being tested is the concrete mix itself, which is typically a combination of cement, sand, and water. In addition, water is added to the concrete mix to determine the amount of water required to achieve the desired consistency.

Concrete mix: This is the material being tested for consistency.

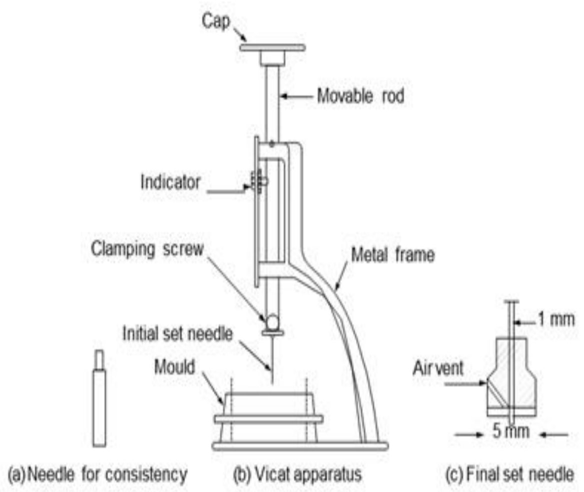


Fig 3.11: Consistency Apparatus [20]

Water: Water is added to the concrete mix to determine the amount of water required to achieve the desired consistency.

Measuring equipment: Measuring cups, beakers, or a scale can be used to measure the amount of water added to the concrete mix.

Mixing equipment: A mixing bowl or a mechanical mixer can be used to mix the concrete and water together.

Trowel or scoop: A trowel or scoop can be used to place the concrete mix into the mould.

Mould: A container or mold of a known size and shape is used to hold the concrete mixture during the test.

Stopwatch: A stopwatch or timer can be used to measure the time taken for the concrete mixture to flow or spread to a certain distance.

Testing apparatus: A variety of testing apparatus, such as a slump cone or a flow table, can be used to perform the consistency test, depending on the specific requirements of the test.

1. Take 400g of cement and mix it with a weighed quantity of water. The ratio of water to cement will depend on the initial consistency of the cement and will be adjusted during the test to achieve the desired consistency.
2. Quickly fill the Vicat mould with the paste made in step 1, making sure there are no voids or air pockets.
3. Smooth off the surface of the paste and insert the plunger gently into the paste so that it contacts the surface.

4. Allow the plunger to penetrate the paste to a depth of 5 to 7 mm, using the Vicat apparatus. This depth should be measured from the bottom of the mould.
5. If the plunger does not penetrate the paste to the required depth, add more water to the cement paste and repeat steps 2-4 until the correct consistency is achieved. The gauging time, which is the time between adding water to the cement and filling the mould, should be between 3 to 5 minutes.
6. Record the amount of water added to the cement paste to achieve the desired consistency. This is the amount of water to be used in all subsequent tests that require a standard consistency.

The consistency test is essential for conducting other tests on cement, including the determination of initial and final setting times, soundness, and compressive strength.

Fig 3.12: Mixing the cement and filling in Mould for consistency testing

Fig 3.13: Penetration of Plunger at exactly 5-7 mm

3.4.4.3 Initial and Final Settling Time

The setting time of cement is a crucial property that determines the time available for mixing, placing, and finishing the cement-based materials. The initial setting time indicates the time available for mixing and placing the cement-based materials, while the final setting time determines the time available for finishing and curing the materials. Hence, it is important to determine the setting time accurately to ensure proper handling and performance of the cement-based materials.

Procedure: Initial settling time refers to the period between the completion of mixing of the concrete and the point at which the concrete starts to stiffen and lose its plasticity. During this time, the concrete is still in a workable state and can be moulded and placed into its final position. The initial settling time is typically measured using the Vicat apparatus, which involves placing a standard cone into the concrete and measuring the time it takes for the cone to penetrate a specific distance into the concrete.

Final settling time, on the other hand, refers to the period between the completion of mixing of the concrete and the point at which the concrete has completely hardened and can no longer be moulded or worked with. The final settling time is also measured using the Vicat apparatus, but this time the penetration depth of the cone is measured at regular intervals until it reaches a specific depth that indicates that the concrete has fully hardened.

Initial setting time test:

1. Take 500g of cement sample and mix it with water content of 0.85P (where P is the percentage of water required as per the consistency test) to form a paste.
2. Fill the paste into the testing mould within 3-5 minutes.
3. Use needle C to penetrate the paste and note the time taken for it to reach a depth of 33-35mm. This time is considered the initial setting time.

Final setting time test:

1. Replace needle C with needle F.
2. Gently lower the attachment to cover the surface of the test block.
3. If the central needle makes an impression and the circular cutting edge fails to do so, the paste has attained hardness.

Record the time at which the centre needle does not penetrate the paste more than 0.5mm. This time is considered the final setting time.

3.4.4.4 Compressive Strength Test

The strength test is a critical test for cement, as it helps in determining the load-bearing capacity of cement-based materials. The compressive strength of cement-mortar at a specific period gives an idea about the durability, stability, and strength of the cement-based materials.

Casting Procedure: To prepare a concrete block using the above recipe, standard sand, cement, and water must be mixed to form a paste. The paste is then compacted into a cube mould using a tamping rod or vibrator, and allowed to cure. After curing, the compressive strength of the block can be measured using a testing machine**.**

Standard sand: Standard sand is a type of sand that has been standardized to ensure consistency in testing. For this particular recipe, 555 grams of standard sand is required.

Cement: Cement is a binding material that is used to hold the sand together and provide strength to the concrete block. For this recipe, 185 grams of cement is required.

Water: Water is needed to mix the cement and sand together to form a paste. The quantity of water required depends on the desired consistency of the paste, which is typically measured using a standard cone test. For this recipe, the water quantity required is P/4 + 3% of the combined weight of the cement and sand mixture.

Cube mould: A cube mould is a container used to hold the concrete block during preparation and curing. For this recipe, a cube mould with a size of 7.06mm and a face area of 50 square centimetres is required.

Fig 3.14: Sieving the Sand for Casting

Tamping rod or vibrator: A tamping rod or vibrator is used to compact the concrete block mixture into the cube mould. This helps to ensure that the mixture is evenly distributed and has no air pockets.

Testing apparatus: Testing apparatus is needed to measure the compressive strength of the concrete block after it has cured. This typically involves using a testing machine that applies a load to the block until it fails.

Fig 3.15: Prepared concrete blocks placing in water

Fig3.16: Image of Flexural Strength Testing Machine and Testing of prepared concrete block

1. Mix the cement, standard sand, and water thoroughly within 3-4 minutes to form a paste.
2. Fill the paste into the cube mould and compact it using a tamping rod or vibrator.
3. Keep the moulds in a controlled environment with a temperature of 27±2°C and at least 90% humidity. If such a facility is not available, keep the moulds under a wet gunny bag to simulate 90% relative humidity.

4. After 24 hours, remove the cubes from the mould and immerse them in clean water till they are tested.
5. Test the compressive strength of three cubes after a specific period (3, 7, or 28 days), reckoned from the completion of vibration.
6. Calculate the average compressive strength of the three cubes to determine the strength of cement at the specific period.

3.4.5 Chemical Composition Tests for Prepared Cement Samples

The chemical composition test of cement is important because it helps in determining the quality and suitability of the cement for various construction applications. The percentage of different oxides present in cement affects its strength, setting time, durability, and other properties. Therefore, it is essential to perform this test to ensure that the cement used in construction meets the required standards and specifications.

The test results are used to verify the quality of the cement and to compare it with the standard values set by the relevant regulatory authorities. If the chemical composition of the cement is not within the required range, it may affect the quality and durability of the concrete made from it. For example, excessive amounts of certain oxides in the cement can cause cracking, shrinkage, or other defects in the concrete.

Sample Solution Preparation: In chemical Analysis for Hydraulic Cement, for every component test (Cao, MgO, SiO_2, Fe_2O_3 etc.,) one common solution to be needed for the Titration process which is known as sample solution.

- Transfer 0.5g of the cement sample to an evaporating dish.
- Moisten the sample with 10ml of distilled water to prevent lumping.
- Add 5 to 10ml of hydrochloric acid to the sample and digest with gentle heat and agitation until the sample is completely dissolved. Dissolution may be aided by light pressure with the flattened end of a glass rod.
- Evaporate the solution to dryness on a steam-bath.
- Treat the residue with 5 to 10ml of hydrochloric acid and then with an equal amount of water, or pour at once upon the residue 10 to 20ml of hydrochloric acid (1:1). Then cover the dish and digest for 10 minutes on the water-bath or hot-plate. Dilute the solution with an equal volume of hot water, immediately filter through an ashless filter

paper (Whatman No. 40 or its equivalent), and wash the separated silica (SiO_2) thoroughly with hot water and reserve the residue.

- Evaporate the filtrate to dryness, baking the residue in an oven for one hour at 105 to 110°C.
- Treat the residue with 10 to 15ml of hydrochloric acid (1:1) and heat the solution on water-bath or hot-plate. Dilute the solution with an equal volume of hot water catch and wash the small amount of silica it contains on another filter paper. Reserve the filtrate and washings for the determination of combined alumina and ferric oxide.

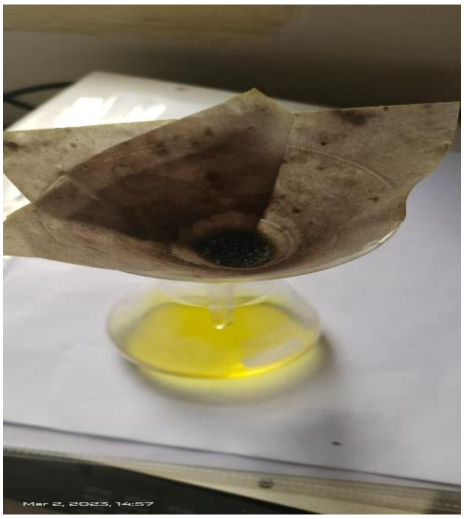


Fig 3.17: Cement sample (0.5g)

Fig 3.18: Filtering the Sample Solution

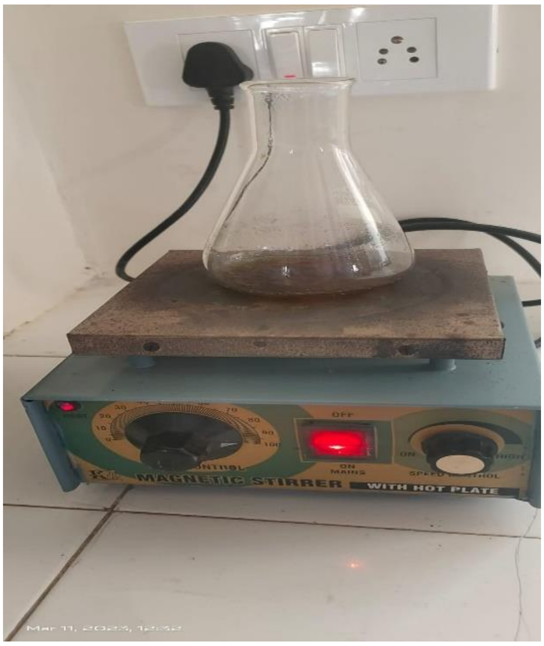


Fig 3.19: Sample Solution preparation on Hot Plate

3.4.5.1 Silica (SiO_2**)**

To start the test, a representative sample of the construction material, weighing 0.5 grams, is transferred to an evaporating dish. It is important to prevent lumping by adding 10 ml of distilled water at room temperature to the sample. Then, 5 to 10 ml of hydrochloric acid is added to the mixture. Hydrochloric acid aids in dissolving the sample. The mixture is then digested with the aid of gentle heat and agitation until the sample is completely dissolved. Light pressure can be applied with the flattened end of a glass rod to aid in the dissolution.

The next step involves evaporating the solution to dryness on a steam-bath. This is done to remove excess water and other volatile components. After evaporation, the residue is treated with 5 to 10 ml of hydrochloric acid and then with an equal amount of water. Alternatively, 10 to 20 ml of hydrochloric acid (1:1) can be poured onto the residue. The dish is then covered, and the mixture is allowed to digest for 10 minutes on a water-bath or hot-plate. Digestion is the process of breaking down the sample into its component parts.

After digestion, the solution is diluted with an equal volume of hot water, and it is immediately filtered through an ashless filter paper, such as Whatman No. 40, or its equivalent. The filtered solution is collected in a separate container, while the separated silica residue is washed thoroughly with hot water. The washed silica residue is then reserved for further analysis.

Fig 3.20: Muffle Furnace for heating the samples

Fig 3.21: Silica dishes with Cement Samples (before heating)

Finally heat the small residue at 1 050 to 1 100°C for a minute or two; cool and weigh. The difference between this weight and the weight of ignited sample represents the amounts of silica.

The silica content of the construction material is calculated using the following formula:

Silica content (%) = [(weight of silica residue) / (weight of sample)] x 100

Fig 3.22: Heated samples in Crucibles

3.4.5.2 Calcium Oxide (CaO):

Take 10 ml of the solution reserved under section 4.5.2.1 in a 250-ml conical flask. Add 5 ml of 1:1 glycerol to the solution with constant stirring. Then, add 5 ml of diethyl amine to the mixture. Next, add 10 ml of 4N sodium hydroxide solution to the mixture and shake it well to adjust the pH to the highly alkaline range of 12 or slightly more. This step is essential to ensure that the reaction proceeds efficiently.

After adjusting the pH, add approximately 50 ml of distilled water and 50 mg of solid Patton-Reeder's indicator to the solution. Titrate the solution against 0.01 M EDTA solution to a sharp change in color from wine red to clear blue. The color change indicates that the titration is complete, and the endpoint has been reached.

To calculate the percentage of CaO in the sample, use the following formula:

$$\text{Calcium Oxide (CaO) percent} = (0.05608 \times 25 \times V) / W$$

Where V is the volume of EDTA solution used in the titration, and W is the weight of the sample in grams.

The factor 0.05608 is derived from the fact that 1 ml of 0.01 M EDTA solution is equivalent to 0.5608 mg of CaO.

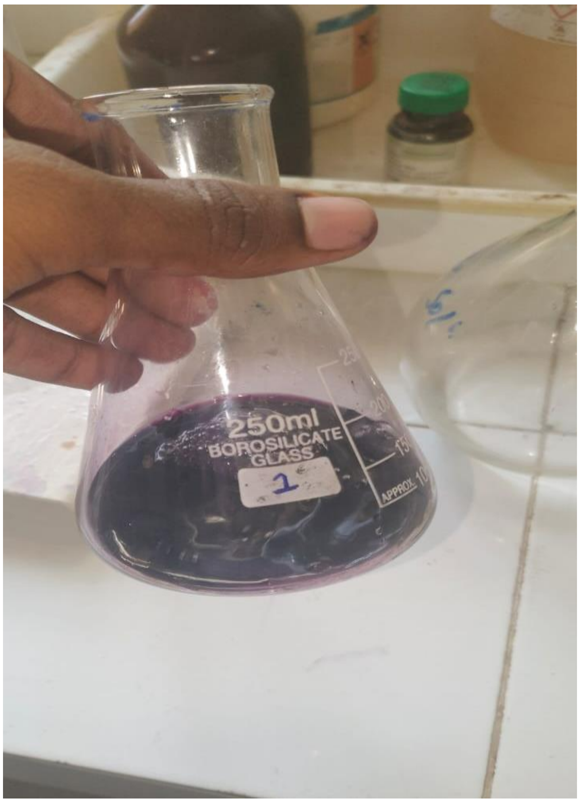


Fig 3.23: End point of the Titration pink colour is obtained by adding P&R reagent

3.4.5.3 Ferric Oxide (Fe_2O_3):

Take 25 ml of the sample solution as mentioned in the silica test and add dilute ammonium hydroxide (1:6) until turbidity appears. Then, clear the turbidity with a minimum amount of dilute hydrochloric acid (1:10) and add a few drops in excess to adjust the pH to approximately 1 to 1.5. Shake the solution well to ensure homogeneity.

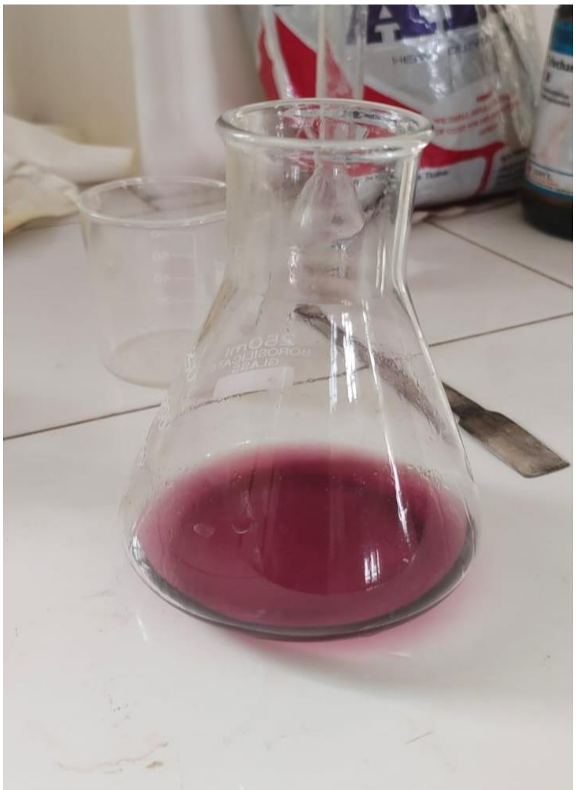
After adjusting the pH, add 100 mg of sulphosalicylic acid to the solution and titrate with 0.01 M EDTA solution carefully to a colorless or pale-yellow solution. The titration is complete when the solution becomes colorless or pale yellow.

To calculate the percentage of Fe_2O_3 in the sample, use the following formula:

Iron Oxide (Fe_2O_3) percent = $(0.7985 \times 25 \times V) / W$

Where V is the volume of EDTA solution used in the titration, and W is the weight of the sample in grams.

The factor 0.7985 is derived from the fact that 1 ml of 0.01 M EDTA solution is equivalent to 0.7985 mg of Fe_2O_3.



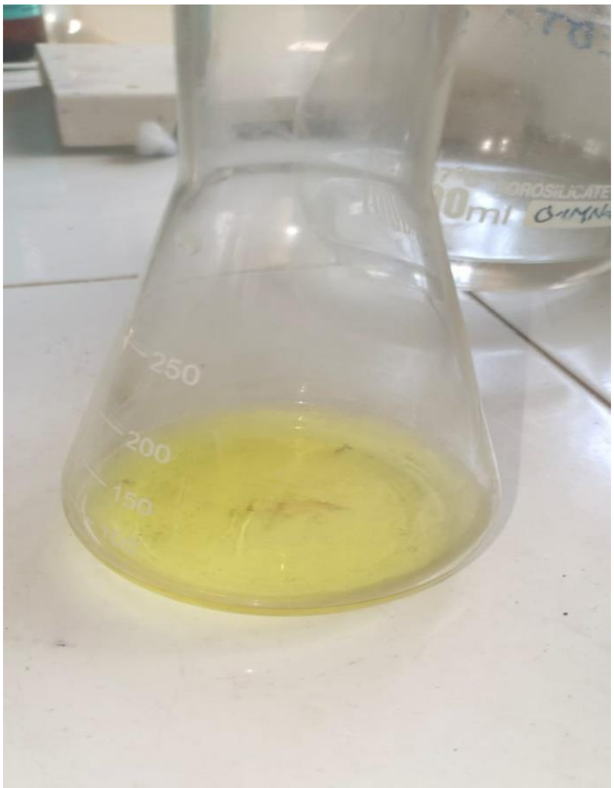


Fig 3.24: Start & End point of the Titration

3.4.5.4 Alumina (Al_2O_3):

The first step is to take 25 ml of the sample solution and titrate the iron content using EDTA and sulphosalicylic acid as the indicator. After adding 15 ml of standard EDTA solution, phosphoric acid, sulphuric acid, and thymol blue, ammonium acetate solution is added until the color changes from red to yellow. Excess ammonium acetate solution is then added to obtain a pH of approximately 6, and the solution is heated to boiling and then cooled. Solid xylenol orange indicator and bismuth nitrate solution are added slowly until the color changes from yellow to red. The solution is then titrated with 0.01 M EDTA solution to a sharp yellow end point red color.

The percentage of Al_2O_3 is calculated using the formula

$$V = V1 - V2 - (V3 \times E)$$

where V is the volume of EDTA solution used, V1 is the volume of the solution titrated, V2 is the volume of blank EDTA solution, V3 is the volume of bismuth nitrate solution, E is the equivalence of bismuth nitrate solution, and W is the weight of the sample in grams. The equivalence of bismuth nitrate solution can be determined by titrating with EDTA solution and calculating the amount required to reach the end point.

Fig 3.25: Sample solutions stored in volumetric Flasks

"E" represents the equivalent value of the bismuth nitrate solution. This value is determined through a titration process using 0.01 M EDTA solution, transfer 100 ml of bismuth nitrate solution to a 500-ml flask and dilute with about 100 ml distilled water. Add a few drops of thymol blue solution and ammonium acetate solution until the colour changes from red to yellow. Add 50 mg of xylenol orange indicator and titrate with 0.01 M EDTA solution until the colour changes from red to yellow.

3.4.5.5 Magnesia (MgO)

10 ml of the sample solution is taken and mixed with 5 ml of 1:1 triethanolamine and 20 ml of buffer solution with a pH of 10. To this solution, 50 mg of solid thymol Eri chrome Black-T indicator is added followed by approximately 50 ml of distilled water. The solution is then titrated against standard 0.01 M EDTA solution until the color changes from pink to Blue. This titration gives the sum of calcium and magnesium oxide present in the solution.

The value of magnesium oxide can be obtained by subtracting the volume of EDTA used in calcium oxide titration from the volume EDTA used in this titration.

1 ml of 0.01 M EDTA 0.4032 mg of MgO

$$\text{Magnesium oxide (MgO) percent} = 0.04032 \times 25 \times (V1 - V) / W$$

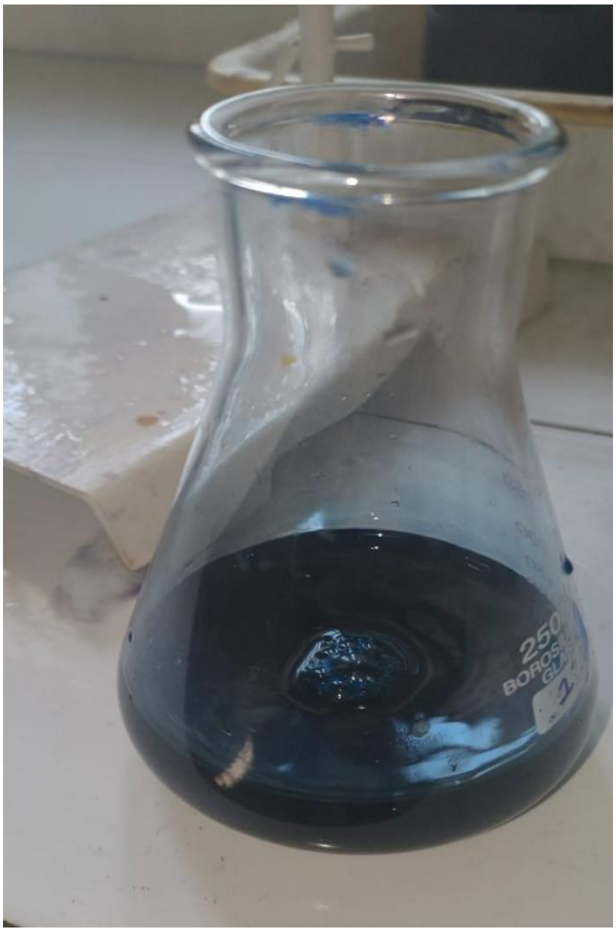


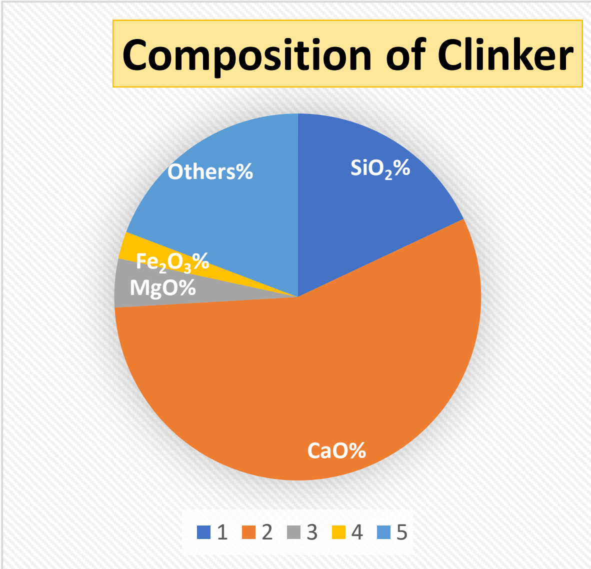
Fig 3.26: End point of the titration

CHAPTER -4

RESULT AND DISCUSSION

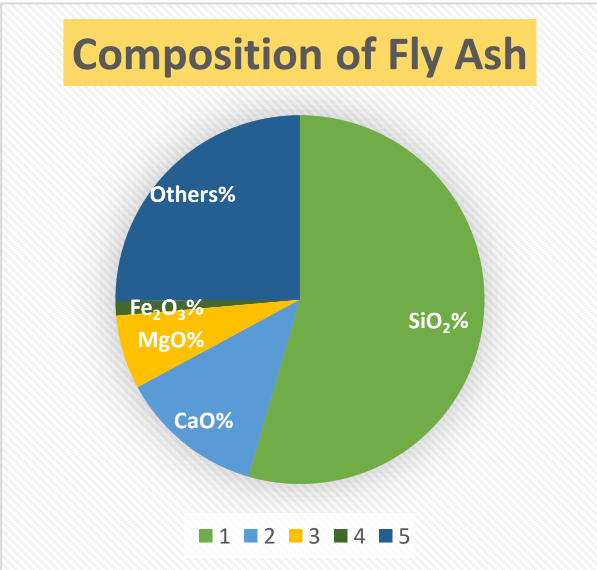
4.1 Characterization Results

4.1.1 Limestone Clinker



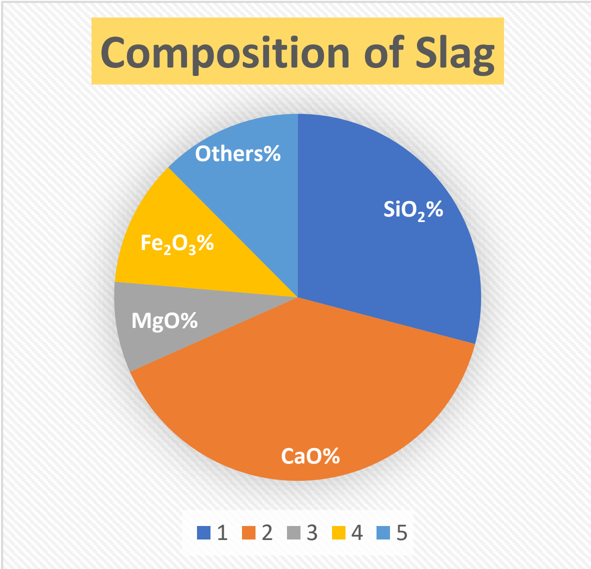
Limestone is a sedimentary rock composed primarily of calcium carbonate (CaCO3) in the form of the mineral calcite. The limestone samples were crushed and ground to a fine powder to facilitate analysis. Particle size distribution of the limestone was determined by the sieve analysis method, which revealed that 25% of the particles were below 5 microns, 50% were below 10 microns, and 90% were below 40 microns. The specific gravity of limestone was found to be 2.71. The calcium carbonate content of limestone is the most important factor determining its suitability for cement production. The high calcium carbonate content of the limestone used in this project makes it a suitable raw material for cement production. The presence of other minerals such as silica, alumina, and iron oxide in the limestone is also important as they contribute to the chemical reactions that take place during cement production.

4.1.2 Fly Ash



Fly ash is a byproduct of coal combustion that is commonly used as a pozzolanic material in cement production. The particle size distribution of the fly ash indicates that 30% of the particles are below 10 microns, 60% are below 20 microns, and 90% are below 40 microns. The specific gravity of the fly ash is 2.3, and its Blaine fineness is 400 m2/kg. These results suggest that the fly ash used in this project has a fine particle size and a high pozzolanic activity, which should make it a suitable replacement for a portion of the cement in the concrete mixtures.

4.1.3 Metal Slag



Metal slag is a byproduct of the steel manufacturing process and contains various chemical components. As for its chemical composition, it contains high amounts of iron oxide (Fe_2O_3), silica (SiO_2), and alumina (Al_2O_3). It also has a significant amount of magnesium oxide (MgO) and calcium oxide (CaO), which makes it a potential alternative to limestone in the cement manufacturing process. In terms of particle size distribution, metal slag has a smaller particle size than fly ash, with 30% of particles below 20 microns, 60% below 30 microns, and 90% below 60 microns. This small particle size distribution indicates that it can contribute to the early strength development of cement. The specific gravity of metal slag is 2.8, which is higher than that of fly ash, indicating that it is denser and can contribute to the weight and density of the final product. The Blaine fineness of metal slag is 380 m2/kg, which is lower than that of fly ash. However, this is still within an acceptable range for cement production.

4.2 Fineness Test Results:

The results of the experiment for fineness testing were conducted in CT&TE lab in MREC and can result in cementitious materials with properties comparable to those of traditional cement. The cementitious materials were produced by blending coal fly ash and metal slag with Portland cement in different proportions (10%, 20%, 30%, 40%, 50%, 60%, 70%, 80%, 90%, and 100%).

The following tables represents the fineness test results on the respective fly ash cement samples. In which, the amount of cement taken for the test, weight of residue retained in the sieve IS 0.090 mm and the percentage of cement retained in the sieve. The Table 4.1 Represents the Fineness Test results for Blast Furnace Slag Cement Samples

Table 4.1: Fineness Test results for Blast Furnace Slag Cement Samples

S.NO	Sample	Weight of cement (gm)	Wt. Retained in 90 mics sieve in gm	% Weight of Residue	Average
1	S0	100	3.38	3.38	5.27
2	S10	100	4.25	4.25	
3	S20	100	7.23	7.23	
4	S30	100	5.65	5.65	
5	S40	100	4.98	4.98	
6	S50	100	6.51	6.51	
7	S60	100	3.27	3.27	
8	S70	100	4.85	4.85	
9	S80	100	6.25	6.25	
10	S90	100	7.32	7.32	
11	S100	100	4.25	4.25	

The following tables represents the fineness test results on the respective slag cement samples. In which, the amount of cement taken for the test, weight of residue retained in the sieve IS 0.090 mm and the percentage of cement retained in the sieve. The Table 4.2 Represents the Fineness Test results for Fly Ash Cement Samples

Table 4.2: Fineness Test results for Fly Ash Cement Samples

S.NO	Sample	Weight of cement (gm)	Wt. Retained in 90 mics sieve in gm	% Weight of Residue	Average
1	A0	100	5	5	5.42
2	A10	100	5.6	5.6	
3	A20	100	7	7	
4	A30	100	4.6	4.6	
5	A40	100	4.3	4.3	
6	A50	100	6	6	

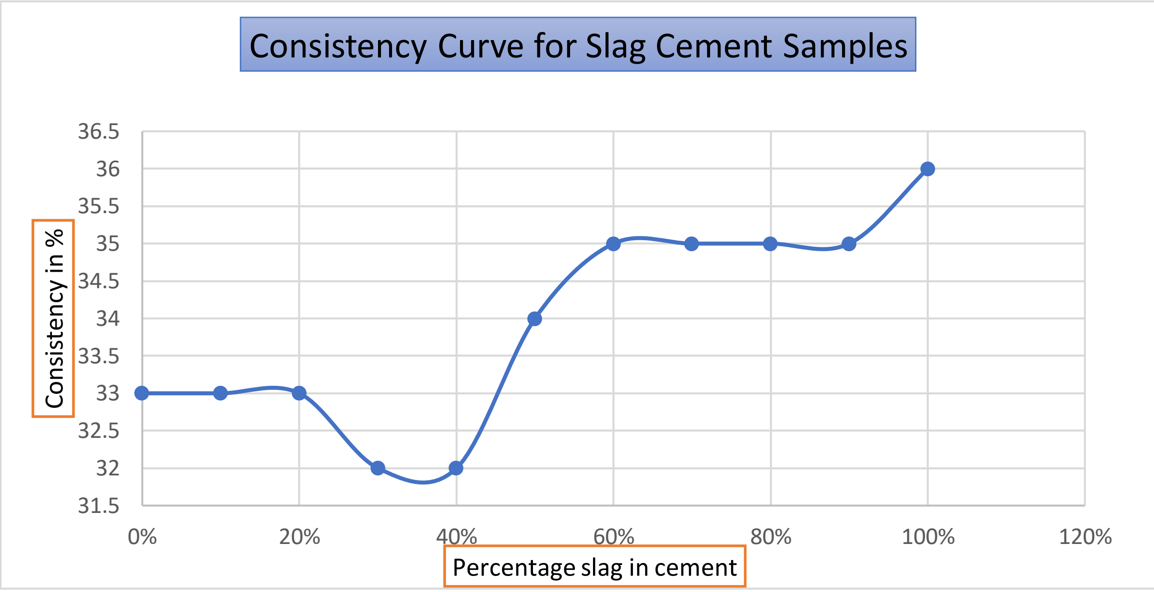
4.3 Consistency Test Results:

Consistency is an important property of cement paste that indicates the flow characteristics of the paste. The consistency of the ash samples was evaluated by measuring the water required to form a paste of standard consistency. This test was conducted in the Civil Engineering CT&TE laboratory in MREC. The results of the test show that as the percentage of ash increases, the water requirement for standard consistency decreases. The consistency test was conducted by adding different percentages of water to the cement paste and measuring the initial and final readings of the penetration of the needle. The percentage of water added to the cement paste at which the penetration of the needle shows 5-7 mm reading is said to be consistency. The results of the consistency test for slag cement samples are presented in Table 4.3. The table shows the amount of water added to the cement paste, the initial reading of the penetration of the needle, the final reading of the penetration of the needle, and the percentage of water added to the cement paste at which the penetration of the needle shows 5-7 mm reading. From the table, it is evident that the consistency of the slag cement samples is within the recommended range for the production of high-quality concrete. As the percentage of water added to the cement paste increases, the consistency of the paste also increases, indicating that the paste becomes more flowable.

Table 4.3: Represents the results of consistency test for slag cement samples

S.NO	Sample	Water Added In ml	Percentage of Water added	Initial Reading	Final Reading
1	0%	132	33	21	7
2	10%	132	33	15	6
3	20%	132	33	13	7
4	30%	128	32	22	7
5	40%	128	32	18	7
6	50%	140	34	16	6
7	60%	140	35	15	7
8	70%	140	35	17	7
9	80%	140	35	16	7
10	90%	140	35	17	7
11	100%	145	36	9	7

Graph 4.1: Characteristic curve for the consistency values of slag cement



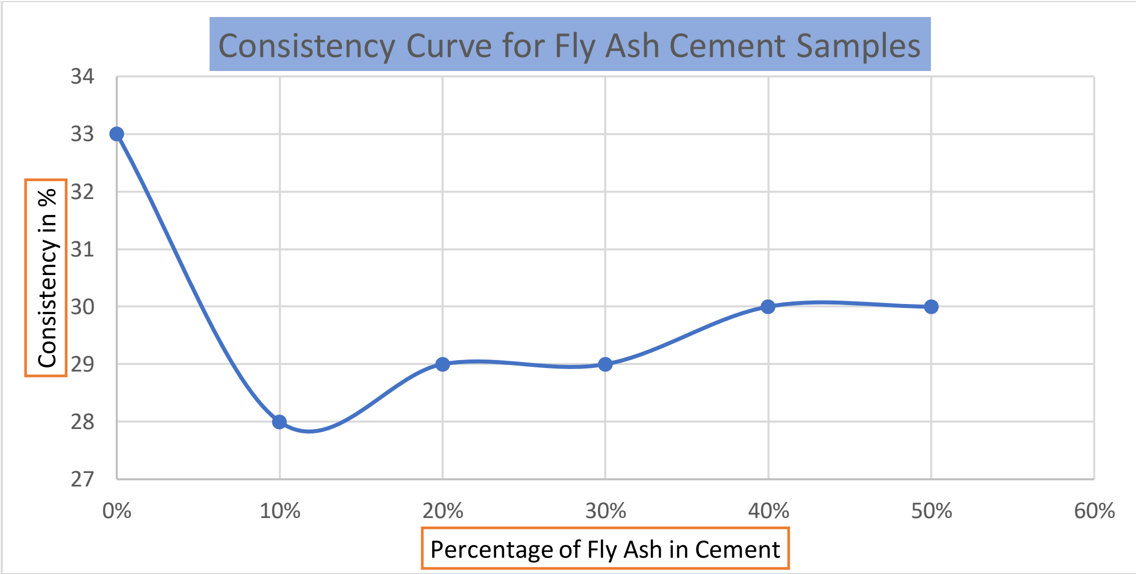
The sample with no slag (0%) required 132 ml of water to achieve standard consistency, while the sample with 100% slag required 145 ml of water. The data also suggests that the addition of slag beyond a certain point does not have a significant effect on the consistency of the paste.

It is interesting to note that the sample with 50% slag required only 140 ml of water to achieve standard consistency, which is higher than the samples with 30% and 40% slag. This could be due to variations in the particle size, chemical composition, or other factors. Overall, the data suggests that the addition of slag can increase the water requirement for achieving standard consistency in cementitious materials. The table 4.4 Consistency test results for fly ash cement samples

Table 4.4: Consistency test results for fly ash cement samples

S.NO	Sample	Water Added in ml	Percentage of Water added	Initial Reading	Final Reading
1	0%	132	33	21	6
2	10%	112	28	16	7
3	20%	116	29	14	5
4	30%	116	29	17	6
5	40%	120	30	17	7
6	50%	120	30	18	6

Graph 4.2: Characteristic curve for the consistency values of Fly Ash cement



The sample with no ash (0%) required 132 ml of water to achieve standard consistency, while the sample with 50% ash required only 120 ml of water. It can be observed that the difference in water requirement between the samples with 30%, 40%, and 50% ash was not significant. This suggests that after a certain point, the addition of ash may not have a significant effect on the consistency of the paste. Overall, the results suggests that the addition of ash can reduce the water requirement for achieving standard consistency in cementitious.

4.4 Initial and Final Settling Time Results

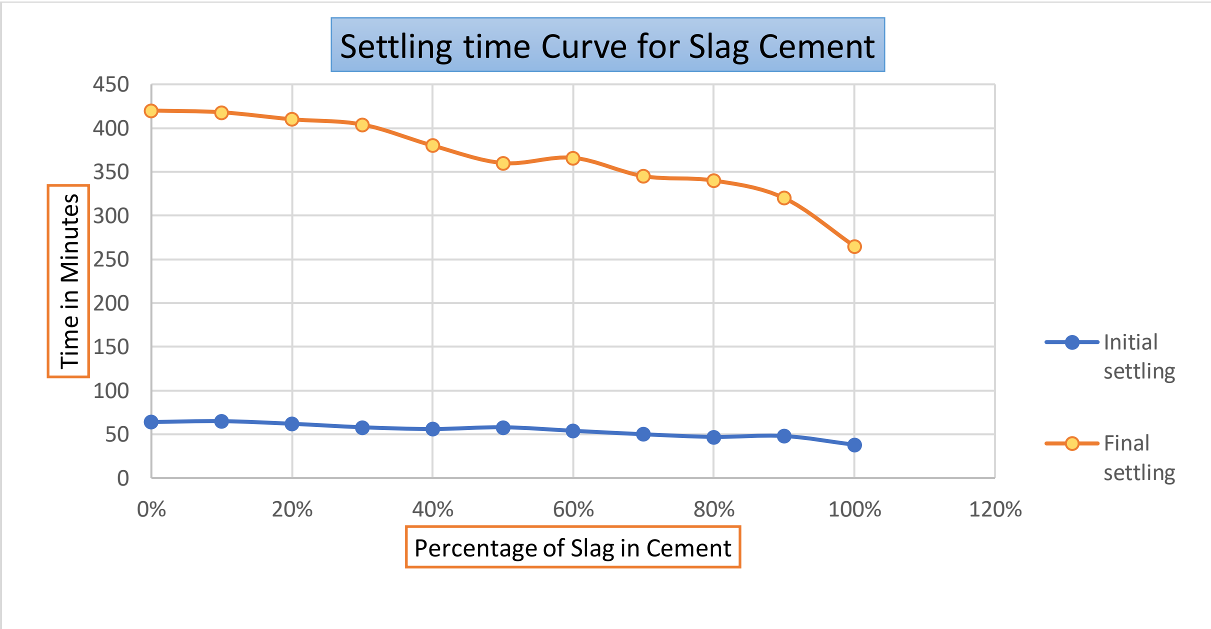
The initial and final settling time of the cement sample were conducted in CT&TE Laboratory in MREC. The results of the experiment indicates that the settling time of the cement clearly depends on the type of raw material used in the cement manufacturing and the amount of slag and fly ash used in the cement.

Table 4.5: Initial and Final Settling Time of the Fly ash and Slag cements

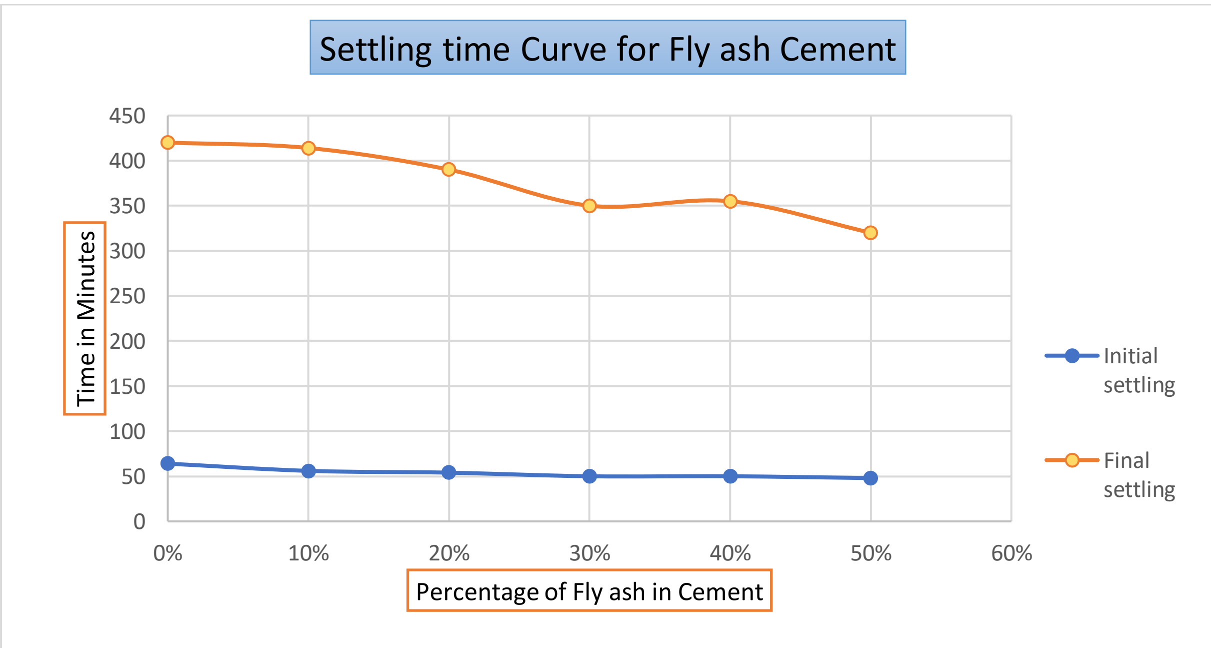
S.NO	Sample	Initial Settling Time in min	Final Settling Time in min
1	A0	64	420
2	A10	56	414
3	A20	54	390
4	A30	50	350
5	A40	50	355
6	A50	48	320
7	S0	64	420
8	S10	65	418
9	S20	62	410
10	S30	58	404
11	S40	56	380
12	S50	58	360
13	S60	54	366
14	S70	50	345
15	S80	47	340
16	S90	48	320
17	S100	38	265

From the above results, settling time of the cement depends on how much industrial by-products added to it. This could be due to variations in the particle size, chemical composition, or other factors. Overall, the data suggests that the addition of slag and fly ash can decrease the initial settling time as well as final settling time of the both cements.

Graph 4.3: characteristic curves for the initial and final settling time of the slag cement samples.



Graph 4.4: Characteristic curves for the initial and final settling time of the slag cement samples.



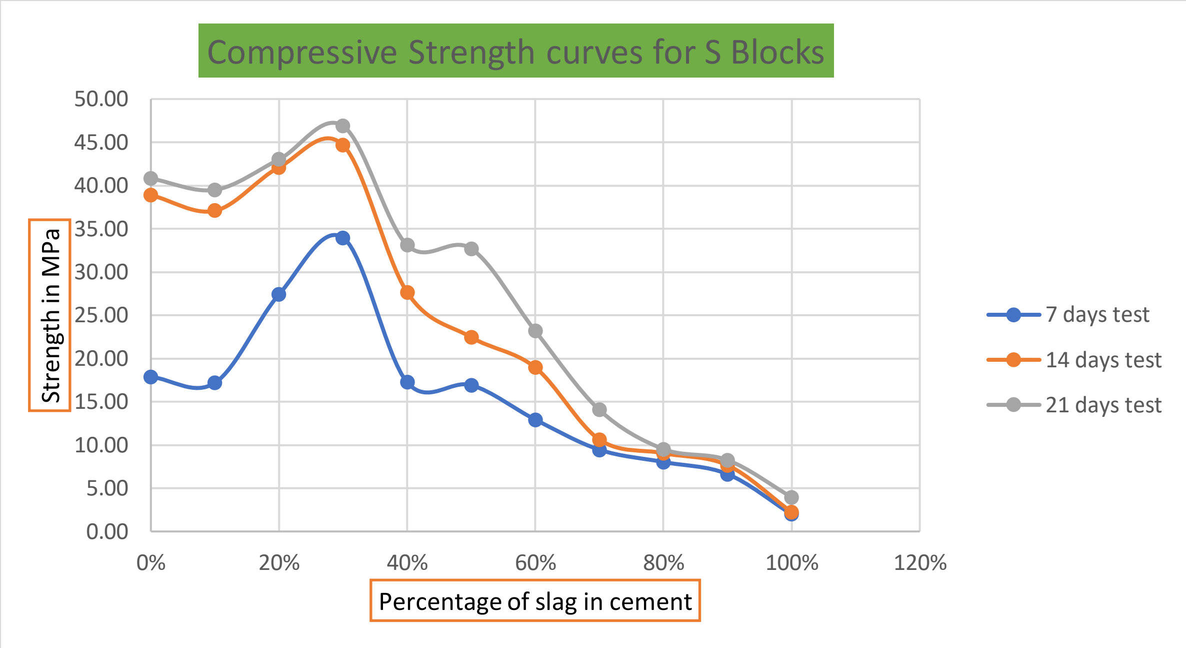
4.5 Compressive Strength Testing Results

The results of the experiment, the utilization of coal fly ash and metal slag as supplementary cementitious materials in cement production for the concrete block strength testing was conducted in CT&TE lab in MREC and can result in cementitious materials with properties comparable to those of traditional cement. The cementitious materials were produced by blending coal fly ash and metal slag with Portland cement in different proportions (10%, 20%, 30%, 40%, 50%, 60%, 70%, 80%, 90%, and 100%).

Table 4.6: Compressive strength testing results for Slag cement mortar cubes.

S.NO	Sample	7 Days		14 Days		21 Days	
		Load KN	Strength MPa	Load KN	Strength MPa	Load KN	Strength MPa
1	S0	89.32	17.87	194.41	38.89	204	40.81
2	S10	85.9	17.19	185.34	37.08	197.45	39.50
3	S20	137.02	27.41	210.48	42.11	215.05	43.02
4	S30	169.74	33.96	223.23	44.66	234.32	46.88
5	S40	86.17	17.24	138.17	27.64	165.45	33.10
6	S50	84.56	16.92	112.23	22.45	163.4	32.69
7	S60	64.62	12.93	94.71	18.95	115.8	23.17
8	S70	47.23	9.45	53.12	10.63	70.23	14.05
9	S80	40.12	8.03	45.23	9.05	47.6	9.52
10	S90	33.15	6.63	38.26	7.65	41.23	8.25
11	S100	9.89	1.98	11.16	2.23	19.8	3.96

Graph 4.5: Characteristic curves for the Strength of slag concrete blocks



The above results show the compressive strength of the slag samples increased with the addition of slag up to 30% and then started to decrease gradually. The highest compressive strength was achieved at 30% slag with a strength of 33.96 MPa after 7 days of curing, which increased to 46.88 MPa after 21 days of curing. It is interesting to note that the compressive strength of the samples with 40% slag was lower than that of the samples with 30% slag. This happens because an excess amount of slag can lead to an increase in porosity and a decrease in density, resulting in lower compressive strength.

The compressive strength is one of the most important parameters to evaluate the quality and performance of cement-based materials. In this project, the compressive strength of the concrete blocks made from fly ash and slag cement was tested. The standard compressive strength of good Portland cement and standard sand mortar cement cube at the end of 3 days should not be less than 11.50 MPa and that at the end of 7 days should not be less than 17.50 MPa.

Furthermore, it can be observed that the compressive strength of the samples with no slag (0%) was the lowest, with a strength of 17.87 KN/m2 after 7 days of curing, which increased to 40.81 KN/m2 after 21 days of curing. This indicates the positive effect of slag on the compressive strength of the samples. which resulted in a 20% increase in compressive strength compared to traditional cement after 21 days of curing.

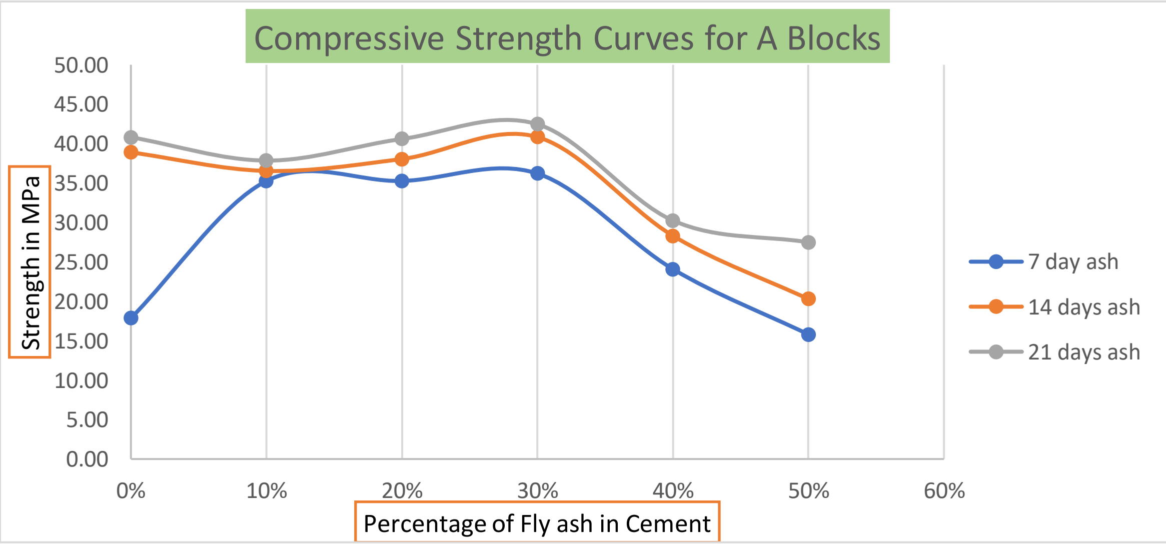
Table 4.7: Compressive strength testing results for fly ash cement mortar cubes.

S.NO	Sample	7 Days		14 Days		21 Days	
		Load KN	Strength MPa	Load KN	Strength MPa	Load KN	Strength MPa
1	A0	89.32	17.87	194.41	38.89	204	40.81
2	A10	176.25	35.26	182.65	36.54	189.2	37.85
3	A20	180.5	35.27	190.19	38.05	202.9	40.59
4	A30	180.99	36.21	204.11	40.83	212.23	42.46
5	A40	120.39	24.09	141.53	28.31	151.19	30.25
6	A50	79	15.08	101.4	20.29	137.4	27.49

The above table shows the compressive strength of the Ash samples increased with the addition of Ash up to 30% and then started to decrease gradually. The highest compressive strength was achieved at 30% Ash with a strength of 36.21 MPa after 7 days of curing, which increased to 42.46 MPa after 21 days of curing. It is interesting to note that the compressive strength of the samples with 40% Ash was lower than that of the samples with 30% Ash. This happens because an excess amount of Ash can lead to an increase in porosity and a decrease in density, resulting in lower compressive strength.

The Standard compressive strength of good Portland cement and standard sand mortar cement cube at the end of 3 days should not be less than 11.50 MPa and that at the end of 7 days should not be less than 17.50 MPa

Graph 4.6: Characteristic curves for the Strength of fly ash concrete blocks



From the above results, it can be observed that the compressive strength of the samples with no Ash (0%) was the lowest, with a strength of 17.87 MPa after 7 days of curing, which increased to 40.81 MPa after 21 days of curing. This indicates the positive effect of Ash on the compressive strength of the samples. which resulted in a 20% increase in compressive strength compared to traditional cement after 21 days of curing.

4.6 Chemical Analysis Results

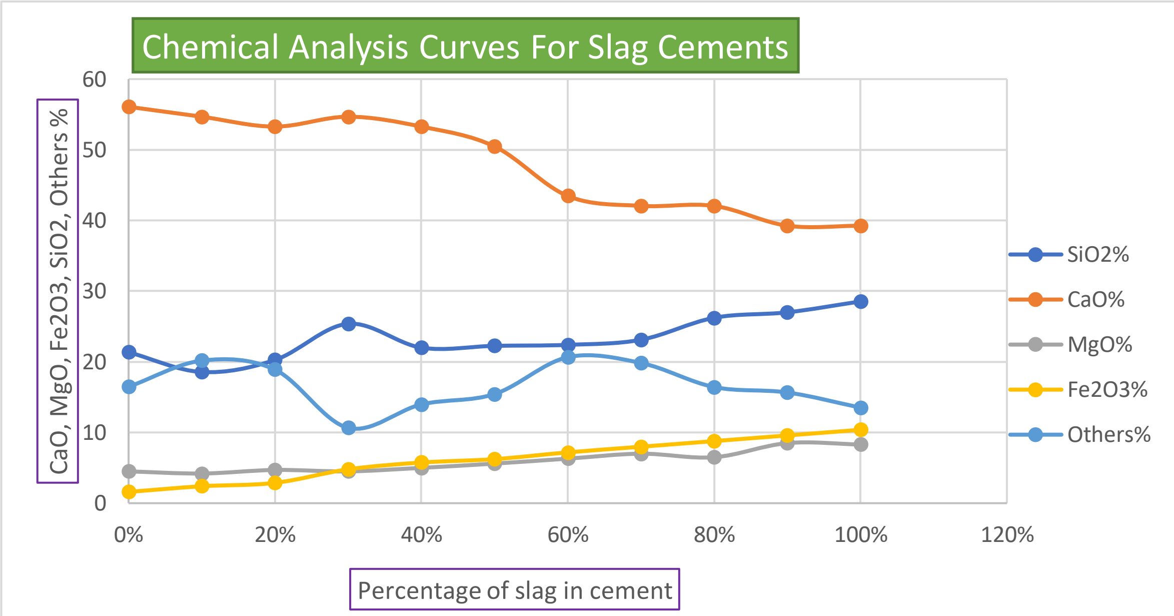
Chemical analysis tests were conducted on fly ash and slag cement samples at the Pharmacy Laboratory of MREC of Pharmacy. The results of these tests can provide valuable insights into the properties and potential uses of these materials. chemical analysis tests on fly ash and slag cement samples, determining the silica, magnesia, calcium oxide, and iron contents. Although the contents of SO_2, Al_2O_3, loss of ignition, etc., could not be estimated, they were categorized as "others" in the graphical representation of the results.

Table 4.8: Chemical Analysis test results for Slag Cement Samples

S.NO	**Sample**	**SiO_2 %**	**Fe_2O_3 %**	**CaO %**	**MgO %**	**Others %**
1	S0	21.36	1.59	56.08	4.51	16.46
2	S10	18.56	2.39	54.67	4.20	20.16
3	S20	20.26	2.87	53.27	4.71	18.88
4	S30	25.38	4.79	54.67	4.53	10.65
5	S40	22	5.74	53.27	5.00	13.97
6	S50	22.26	6.22	50.47	5.61	15.43
7	S60	22.4	7.18	43.46	6.32	20.65
8	S70	23.12	7.98	42.06	7.23	19.83
9	S80	26.23	8.78	42.06	6.51	16.42
10	S90	27	9.58	39.25	8.50	15.66
11	S100	28.54	10.38	39.25	8.30	13.52

Based on the chemical analysis tests conducted on the slag cement samples, the chemical composition of the slag cement samples was found to be satisfactory according to the industrial standards for these materials. slag cement could be used as a replacement for the traditional raw material used in cement manufacturing, limestone clinker. This finding has significant implications for the construction industry, as it could help reduce the environmental impact of cement production while also providing a viable alternative to traditional raw materials.

Graph 4.7: Graphical Representation of Chemical Analysis test results for Slag Cement Samples

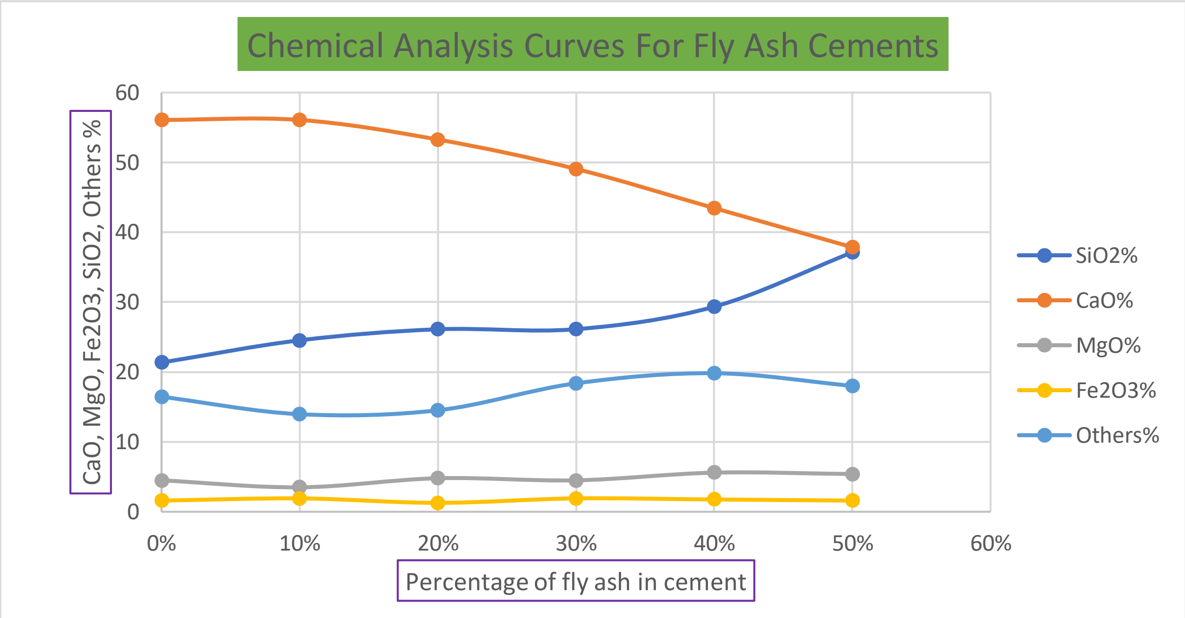


Based on the chemical analysis tests conducted on the Fly Ash cement samples, the chemical composition of these cement samples was found to be satisfactory according to the industrial standards for these materials. slag cement could be used as a replacement for the traditional raw material used in cement manufacturing, limestone clinker. This finding has significant implications for the construction industry, as it could help reduce the environmental impact of cement production while also providing a viable alternative to traditional raw materials.

Table 4.9: Chemical Analysis test results for Fly ash Cement Samples

S.NO	**Sample**	**SiO_2 %**	**Fe_2O_3 %**	**CaO %**	**MgO %**	**Others %**
1	A0	21.36	1.59	56.08	4.51	16.46
2	A10	24.54	1.91	56.08	3.53	13.96
3	A20	26.12	1.27	53.27	4.81	14.52
4	A30	26.13	1.91	49.07	4.5O	18.38
5	A40	29.35	1.75	43.46	5.63	19.83
6	A50	37.13	1.59	37.85	5.41	18.01

Graph 4.8: Graphical Representation of Chemical Analysis test results for Fly ash Cement Samples



CHAPTER - 5

CONCLUSION

The project focused on the utilization of industrial wastes as supplementary cementitious materials in cement production and their feasibility study. The study aimed to explore the potential of using industrial wastes like fly ash and metal slag as alternative sources of cementitious materials to reduce the dependency on traditional cement production methods. The project involved the collection of raw materials from various sources and their characterization to evaluate their suitability for use in cement production. The raw materials used in the study were fly ash, metal slag, and limestone. Fly ash was collected from a local thermal power plant, metal slag was collected from a nearby steel plant, and limestone was sourced from a quarry. The characterization of these materials was done through various tests to assess their chemical composition, particle size distribution, specific gravity, and Blaine fineness. The results of the characterization showed that all three materials were suitable for use in cement production. The next phase of the project involved the production of cement with varying percentages of fly ash and metal slag as supplementary cementitious materials. The cement samples produced were tested for compressive strength, consistency, fineness, and settling time to evaluate their quality and performance.

- The compressive strength tests were done using a compression testing machine and the results showed that the cement samples produced with 20% and 30% fly ash and 20% and 30% metal slag had comparable strengths to that of traditional cement with compressive strengths of 43.02 MPa, 46.88 MPa and 40.59 MPa, 42.46 MPa.
- The consistency tests were done using a flow table and the results showed that the consistency of the cement samples decreased with an increase in the percentage of fly ash and metal slag up to 50% sample for both.
- The fineness tests were done using a Sieve Shaker apparatus and the results showed that the cement samples produced with fly ash and metal slag had comparable fineness to that of traditional cement of an average percentage of 5.35% of residue is remained.
- The settling time tests were done using a Vicat apparatus and the results showed that the cement samples produced with fly ash and metal slag had longer settling times than traditional cement. It can be observed that as the percentage of fly ash and slag increases, the initial and final settling times also increase. This can be attributed to the fact that fly ash and slag have finer particle sizes, which increase the surface area and water demand.

Based on the results of the tests, it can be concluded that the utilization of fly ash and metal slag as supplementary cementitious materials in cement production is feasible and can result in a product with comparable strength and fineness to traditional cement. However, the consistency and settling time of the cement samples produced with fly ash and metal slag were affected, indicating the need for adjustments in the production process to achieve the desired consistency and settling time. In addition to the technical aspects of the study, the project also evaluated the economic feasibility of using industrial wastes as supplementary cementitious materials. The results of the economic analysis showed that the use of industrial wastes can result in cost savings in cement production, making it an attractive option for cement manufacturers. In conclusion, the utilization of industrial wastes like fly ash and metal slag as supplementary cementitious materials in cement production is a feasible and economically viable option. However, further research is needed to optimize the production process to achieve the desired consistency and settling time of the cement product. The findings of this project can serve as a starting point for future research in this area and can help in the development of sustainable and cost-effective cement production methods.

CHAPTER - 6

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