Towards Cement Minimization and Circular Economy

Recycling waste-to-energy bottom ash in construction

Master's thesis in the Master's Programme Innovative and Sustainable Chemical Engineering

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CHALMERS UNIVERSITY OF TECHNOLOGY
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ABSTRACT

The pursuit of carbon neutrality and the reduction of carbon dioxide (CO_2) emissions have become key areas of focus in Sweden, especially within the construction sector. Cement production is recognized as one of the largest industrial contributors to CO_2 emissions globally. To mitigate its environmental impact, ongoing research has focused on the partial replacement of cement with supplementary cementitious materials (SCMs). Among these alternatives, coal fly ash has been extensively studied; however, this project investigates the potential of bottom ash derived from Waste-to-Energy (WtE) which is processed to form Mineral Fraction of Icinerator Bottom Ash (MIBA) as a viable SCM. The MIBA used in this study was initially separated into different particle size fractions, which were subsequently processed to achieve a uniform particle size. Concrete cubes were then produced with a fixed level of SCM substitution. The compressive strength of these cubes was measured and compared to reference samples made with ordinary Portland cement to assess the mechanical performance of the material.

The primary aim of this study is not only to assess the feasibility of substituting cement with bottom ash but also to evaluate the environmental implications of such substitution, particularly the leaching of potentially harmful metals from the resulting concrete. To achieve this, the leaching behavior of heavy metals from concrete cubes containing bottom ash was investigated using a semi-dynamic leaching test (batch leaching method). The extent of metal release was quantified and analyzed using advanced analytical techniques such as Inductively Coupled Plasma Mass Spectrometry (ICP-MS) and Powder X-Ray Diffraction (XRD). The findings contribute to understanding the environmental performance of bottom ash as an SCM and support its potential role in reducing CO_2 emissions in the cement and construction industries.

Key words: Supplementary Cementitious Materials (SCM), Waste-to-Enegry (WtE),Mineral Fraction of Icinerator Bottom Ash (MIBA), Powder X-Ray Diffraction (PXRD), Coupled Plasma Mass Spectrometry (ICP-MS),

Contents

Preface

In this project mechanical and chemical tests have been carried out to investigate if ash from WtE can be used as a cement replacement and/or filler. The project is done in cooperation with Renova AB and Chalmers University of Technology. The project investigates the possibility of using ash to create new more environmentally sustainable building materials.

Mechanical test has been done to investigate the mechanical properties of concrete containing WtE incineration ash. These tests were carried out at the Department of Architecture and Civil Engineering, Division of Building Technology, Chalmers University of Technology, Sweden. Leaching tests and chemical analyses have also been carried out to investigate the leaching of metals from monolithic concrete cubes and Granular Concerte samples containing WtE incineration ash. These tests were carried out at the Department of Architecture and Civil Engineering, Division of Water Energy Technology, Chalmers University of Technology, Sweden. I would like to thank Bruno Oliveira Goncalves and Amir Saeid Mohammadi for their help and supervision in the laboratory work.

Gothenburg, June 2025
Flinto Justin

Notations

Roman upper case letters

SCMs	Supplementary Cementitious Materials
MIBA	Mineral Fraction of Incinerator Bottom Ash
SMFC	Sulphonated Melamine-Formaldehyde Condensate
SNFC	Naphthalene Sulphonate-Formaldehyde Condensates
MLS	Modified lignosulphonates
ASTM	American Society for Testing and Materials
LSP	Liquid Solid Partitioning
ICPMS	Inductively Coupled Plasma Mass Spectrometry
PXRD	Powder X-Ray Diffraction
COPCs	Contaminants of Potential Concerns

1 INTRODUCTION

Sweden is strongly focused on achieving carbon neutrality, with significant efforts directed toward innovative and sustainable carbon capture technologies. By 2030 there should be climate neutral concrete on the market and by 2045 all concrete on the Swedish market should be climate neutral [3]. As part of this initiative, emerging methods are being explored to mitigate CO_2 emissions from major industries. The cement industry, one of the largest contributors to CO_2 emissions, has been a key area of concern. Although carbon capture technologies can be implemented to reduce emissions, integrating new solutions into existing cement production processes is often costly and complex. Additionally, the demand for buildings is rising due to population growth, increasing the need for sustainable construction solutions.This has led to the development of new and environmentally friendly building materials, such as low-carbon binders—alternative cementitious materials made from industrial by-products like fly ash, ground granulated blast furnace slag (GGBS), or municipal incinerator bottom ash (MIBA)—that can reduce dependence on conventional Portland cement and lower the overall carbon footprint of the construction industry [12].

Supplementary cementitious materials (SCMs) are not a new trend in the field of concrete. Materials such as coal fly ash and slags have been extensively studied for their potential use as SCMs. Among these, coal fly ash has been one of the most widely used substitutes for cement. However, with the global shift toward sustainability, the demand for coal is rapidly declining, leading to a reduced availability of coal fly ash. This has driven research toward alternative SCMs such as industrial slags, waste ceramics, bottom ash from incinerators, volcanic ash, rice husk ash, and silica fume[1]. The most important properties to consider when evaluating SCMs are their pozzolanic activity and amorphous structure, as these determine their effectiveness in replacing cement while maintaining strength and durability in construction[12].

one of the waste streams largely available in Sweden is the waste-to-energy ashes. In Sweden approximately 1 million tons of bottom ash is generated from waste incineration annually [36]. These potential source can be processed or activated to gain the potential as a substitution for cement can promote new cement compositions and circular economy . In Sweden, municipal solid waste is incinerated primarily for electricity generation and district heating systems. This process produces large quantities of ash, which has the potential to be utilized as a sustainable alternative to traditional cement in construction. At one waste to energy incineration plant in southwestern Sweden 550 000 metric tonnes of waste are incinerated yearly meaning that there is also a lot of waste ash that could be used [12]. The waste used in the incineration process comes from mixed sources, including households, industries, and imported waste. As a result, the resulting ash can contain a diverse range of materials.Inorganic minerals such as SiO_2, CaO, Al_2O_3 and Fe_2O_3 that are the reason that this material might be a suitable replacement for cement [37]. The use of ash as a cement replacement would be mutually beneficial for both the concrete industry and the ash producers. For the concrete industry it would lead to a decreased need of mining new raw material from nature and a decrease of emission from cement production. The ash producers would also be benefiting since the ash materials could be used in more areas and therefore get new value [12].

There has been little focus on the legal and regulatory hurdles that are crucial for gaining access to the European construction market. Currently, we have specific end-of-waste criteria at the EU level for mineral-based waste-derived SCMs but the frameworks are not harmonized and the defined benchmarks vary between country to country, . As a result, researchers and industry professionals face uncertainty in assessing the hazardous nature of mineral waste and its potential for use as secondary materials in SCMs. In these cases, both the level of potential contamination in the source material (e.g., total heavy metal content) and the threshold at which a potential receptor is considered contaminated should be evaluated.

In this project, we obtained sorted and aged ash from WtE company. We partially substituted cement with this bottom ash (MIBA) and produced monolith samples. The compressive strength of these concrete samples was then analyzed to determine whether they exhibited strength comparable to that of conventional concrete cubes. To analyze the leaching properties of the monolithic samples, leaching tests were conducted on the concrete cubes. Although there is no standardized leaching test specifically for monolithic samples, some existing methods can be applied. However, due to the complexity of these frameworks, this study adapted standardized leaching procedures originally developed for granular municipal and industrial wastes.

1.1 Aim and Objective

1.1.1 Aim

The primary aim of this study is to evaluate the mechanical performance and environmental safety of concrete incorporating 15% WtE bottom ash as a partial replacement for cement.

1.1.2 Objectives

To achieve this aim, the study will pursue the following objectives:

- Investigate the effect of substituting 15% SCM for cement on the compressive strength and long-term durability of concrete compared to conventional concrete.
- Perform standardized leaching tests on SCM-substituted concrete to analyze the release of metals.
- Determine whether the leaching behavior of SCM-substituted concrete meets environmental safety threshold.
- Compare the leaching characteristics and environmental impact of SCM-substituted concrete with that of concrete incorporating coal fly ash as a cement replacement.

1.2 Research Questions

- To investigate the effect of replacing 15% of cement with Municipal Incinerator Bottom Ash (MIBA) on the compressive strength of concrete, and to evaluate how different size fractions of MIBA influence this performance.
- What are the differences in metal leaching behavior between monolithic and gran-

ular forms of bottom ash concrete? How do the two leaching test procedures compare in evaluating environmental impact, and how do these results differ from those of reference samples?

1.3 Limitations

Time constraints were a major limitation during the study. The use of a small ball mill made the grinding process time-consuming, and the extended settling time required for concrete samples further delayed the initiation of the batch leaching tests. Additionally, the final particle size analysis using the sieving method proved challenging due to the agglomeration of finer particles, which affected accurate size measurement. One of the main challenges in the leaching setup was water evaporation. In the initial setup, samples were placed in cotton or cloth filter bags, which absorbed water and led to significant evaporation losses. To address this, plastic holders were later fabricated to support the samples, and the entire setup was enclosed to minimize evaporation. Curing of the concrete cubes also posed difficulties. Initially, common curing method was used in which the cubes were fully submerged in water. However, it was later realized that this method promoted early leaching of the cubes during the curing period. Consequently, adjustments were made to reduce this unintended leaching. Furthermore, Since granular samples cannot be handled as easily as monolithic specimens, they were enclosed in fabric pouches during the leaching tests to contain the material. However, this containment method was found to restrict the free movement of particles and the interaction between the granular material and the leachant. As a result, the fabric acted as a physical barrier, potentially limiting the leaching efficiency.

1.4 Background

1.4.1 Concrete

The concrete that we use in construction are structural material consisting of a hard chemically inert particulate substance, known as aggregate (usually sand and gravel), that is bonded together by binder(Cement) and water[8]. The introduction highlights that the current cement industry is a major contributor to carbon dioxide emissions[12]. In this project, we investigate the feasibility of using SCMs as a replacement for cement. Pozzolanic activity is a key characteristic that supplementary cementitious materials (SCMs) should possess, which determines whether we can substitute cement with SCMs. Pozzolanic activity can be assessed using various chemical methods. Among the chemical methods, one commonly used approach is the lime saturation method. In this test, the supplementary cementitious materials (SCMs) is added to a saturated lime solution. After allowing the pozzolanic reaction to proceed for a specific duration, the remaining amount of lime is measured. The reduction in lime content indicates the extent of the pozzolanic reaction and, consequently, the reactivity of the material[24]. Another method is the modified R3 method which measures the amount of bound water that is in a paste made with the SCMs[12].This method, which measures bound water, is particularly suitable for evaluating waste-to-energy (WtE) ash materials, as these materials typically exhibit relatively low pozzolanic activity. This reduced activity is primarily due to their lower concentrations of SiO_2 and Al_2O_3, which are key components responsible for pozzolanic reactions[23]. If we compare the pozzolanic actvity of different SCMs like Fly ash, industrial slaggs and Bottom ash .Out of the three materials the bottom ash had the most bound water in the modified R3 test followed by fly ash and lastly the slag [12].

1.4.1.1 concrete Ingredients

Concrete is a composite material made by combining several ingredients—primarily cement, aggregates (such as sand and gravel), and water—in specific proportions. These components are thoroughly mixed to form a workable paste that hardens over time. The individual roles of each ingredient in the concrete mix are described in the sections below.

- **Cement**
  Throughout history, different types of binders have been used, with gypsum, clay, and Portland cement being some of the most common and widely utilized examples.Cement is a fine, soft powder that acts as a binder, hardening upon contact with water. Cement is the primary component of concrete and is composed of a blend of various minerals and elements. For example, it contains minerals such as tricalcium silicate (C_3S) – Ca_3SiO_5, which contributes to early strength development, and elements like calcium (Ca), primarily sourced from limestone ($CaCO_3$).[18].

  Portland cement and similar materials are made by heating (about 1350-1400 C) limestone ($CaCO_3$) with clay and then grinding this product (called clinker) with a source of sulfate (most commonly gypsum) [6]. Shale is also added during cement production as a source of silicon and aluminum, along with other raw materials. These elements contribute to the formation of clinker minerals such as tricalcium silicate (C_3S) and tricalcium aluminate (C_3A)We can observe the transform the raw material into various minerals including Alite (Ca_3SiO_5), Belite (Ca_2SiO_4), Aluminate ($M(Al_xO_y)$), Ferrite ($M(Fe_xO_y)$) and notably a large amount of carbon dioxide (CO_2) especially in calcination clinker. Production of the portland cement is summarized in the equation 1.1 below

  $$Calcite + Claymineral + Shale \rightarrow Alite + Belite + Aluminate + Ferrite + CO_2 \tag{1.1}$$

  The reaction between the cement minerals such as Alite and Belite with water will result in the formation of calcium silicate hydrate gel which will work as a glue and hold the concrete matrix together[12]. One of the reaction that can occur where Alite reacts with water to form calcium silicate hydrate gel and slaked lime ($Ca(OH)_2$) is given below in the equation 1.2

  $$Alite + Water \rightarrow CalciumSilicateHydrategel(C - S - H) + SlakedLime \tag{1.2}$$

  The C-S-H phase is the main binding agent in portland cement pastes. The exact structure of C-S-H is not easily determined. Considering the several possibilities by which the atoms and ions are bonded to each other in this phase, a model may be constructed [27].

  As discussed, a significant portion of the carbon footprint comes from cement manufacturing, which also requires high heat energy for production. Therefore, using alternative binders, such as supplementary cementitious materials (SCMs) with similar properties and composition to cement, can be a potential solution to this issue. One of the reasons that WtE ash is seen as a possible replacement or additive in cement is that they contain minerals with elements that are important in the creation of cement. This will be further explained in the section selection of WtE ash materials.

- **Aggregates**
  Aggregates are usually sand which should be free free from soft particles or any unwanted particles, as even small amounts of organic soil compounds can trigger chemical reactions that significantly weaken the concrete[8]. Aggregates are generally designated as either fine (ranging in size from 0.025 to 6.5 mm [0.001 to 0.25 inch]) or coarse (from 6.5 to 38 mm [0.25 to 1.5 inch] or larger) [8].Aggregates typically consist of sand, gravel and/or crushed stone. Properties of the aggregates will also have big impact on the properties of the concrete. Size, shape surface texture and area will impact workability, density, durability and strength among others [14].

- **Superplasticizier**
  Super plasticizers are admixtures used in cement mortars that act as fluidizers, enhancing the workability of concrete while maintaining a fixed water-to-cement ratio.They are variously known as superplasticizers, superfluidizers, superfluidifiers, super water reducers or high range water reducers [40]. They can also increase the strength of concrete without requiring additional water. Because over addition of water can cause the concrete mix to weakens its strength, increases porosity and shrinkage, and reduces overall durability and performance [21].When super plasticizers are added to cement mortar containing very fine particles, they attach to the cement particles, preventing their agglomeration compared to cement suspension without the admixture (containing agglomerates). The suspension with the plasticizer shows a better dispersion and finer particles. This allows the cement particles to disperse evenly and react individually with water, enhancing the hydration process and improving workability. In general Super plasticizers can produce much more workable concrete than plain concrete at the same water/cement ratio and also at the same workability, a considerable decrease in w/c ratio and, therefore, a concrete having higher strength [40].

  Other applications of super plasticizers are, producing flowing concrete, effect on rheology of the mortar, effects on the process of hydration of cement and Setting time. The application of super plasticizers varies based on specific project requirements. The composition of Super plasticizer can be adjusted accordingly to meet different performance needs.Superplasticizers are grouped into types like Sulphonated melamine-formaldehyde condensate (SMFC) and Naphthalene sulphonate-formaldehyde condensates (SNFC), which are older formulations made from formaldehyde condensates. Modified lignosulphonates (MLS) types come from modified natural lignin, while newer versions—like polycarboxylates and polycyclic sulphonates—provide better performance and help concrete stay workable for longer [40].

1.4.2 Curing Methods and Strength test of Concrete

- **Cement Curing**
  Curing is the process or operation which controls the loss of moisture from concrete after it has been placed in position, or in the manufacture of concrete products, thereby providing time for the hydration of the cement to occur [9]. Hydration of cement is a time-dependent process that can take hours, days, or even

weeks to complete. To ensure proper hydration, it is essential to maintain constant moisture around the concrete sample, ideally in an environment with 100% relative humidity. The most commonly used method to achieve this is water curing, where the concrete samples are fully submerged in a suitable curing liquid bath to provide continuous moisture and support optimal hydration.Curing may also encompass the control of temperature since this affects the rate at which cement hydrates [9].Water curing is one of the most commonly used methods for maintaining moisture during the hydration process. It can be carried out in various ways, such as continuous sprinkling, immersing the samples in water ponds or tanks, or covering them with wet cloths or burlap to retain moisture on the surface. The period and try of curing methods will depend on the properties required of the concrete, the purpose for which it is to be used, and the ambient conditions, that is the temperature and relative humidity of the surrounding atmosphere [9].

- **Strength Test**
  The compressive strength test determines the load-carrying capacity of concrete under compression. It is a fundamental quality control measure used to assess the performance and durability of concrete mixtures. The procedure aligns with SS EN 196 [32] (a European standard for testing cement), ensuring consistent and reliable results. This test can assess the durability and service Life of concrete structure: For instance, highways and bridges require high-strength concrete to withstand heavy traffic and harsh weather [25]. When new materials or industrial by-products (e.g., fly ash, slag, or MIBA) are introduced as partial cement replacements, compressive strength tests evaluate their impact on concrete performance.

1.4.3 Waste to Energy Process

Waste-to-energy is a sustainable solution for recycling and maximizing the utilization of waste. The process involves incinerating waste to generate steam, which is then used for electricity production and district heating systems. Various household wastes, including plastics, paper, and other raw materials, are burned in the incinerator to produce energy. However, since the waste being incinerated is heterogeneous, its composition varies. In addition to steam, the process also generates fly ash, which contains harmful materials, and bottom ash, which consists of unburned residues and incineration byproducts. Proper treatment of both fly ash and bottom ash is necessary to ensure environmental safety.

The waste collected from various areas is stored in a bunker before being fed into the incinerator reactors using a collector. The incineration process can take place in rotary kilns, fluidized beds, or grate furnaces. Grate furnaces are commonly used for waste incineration, as they operate at temperatures below 1000°C. Incineration heat turns water into steam, which powers turbines to produce electricity and heat buildings. An overview of the WtE incineration plant is given in Figure 1.1 [28].

After the incineration process, the Bottom ash/ slag is cooled with water and collected. The slag is then sorted to remove metals and other unburned or unwanted materials before being stored in landfills for approximately 6 months. Fly ash generally contains larger amounts of metals and chlorides compared to bottom ash and is therefore frequently regarded as hazardous waste, which limits its utilization. In contrast, the bottom ash can after treatment be used for different construction purposes. The treatment procedure involves sorting out metal pieces followed by natural weathering ("aging") to make the bottom ash more stable to metal leaching by means of e.g. carbonation [36]. The metal content remains unchanged during this process, but as the pH drops to a slightly alkaline level, metals become less soluble. In this condition, the bottom ash is referred to as Mineral Fraction of Incinerator Bottom Ash (MIBA)(MIBA)[36].

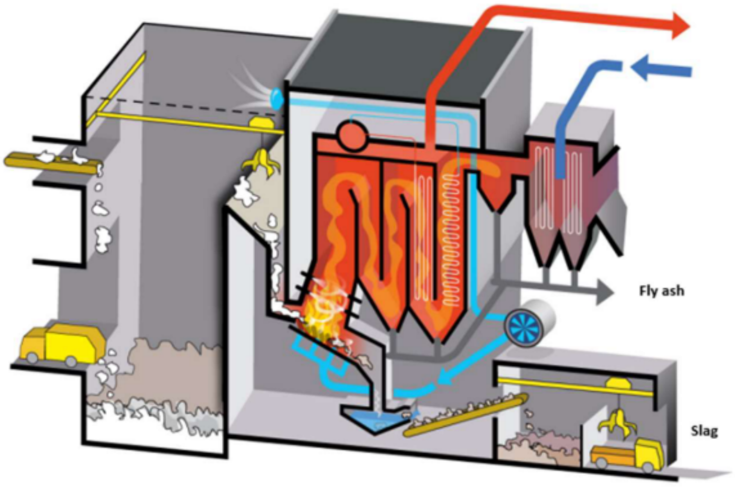


Figure 1.1: Overview of the WtE incineration process [28]

1.4.4 Leaching Test

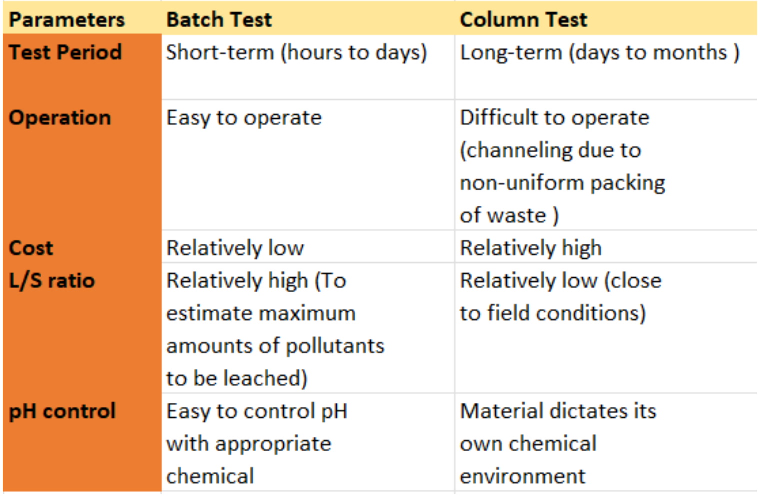
Leaching is the process of extracting or separating soluble materials from an insoluble solid using a suitable solvent. The solvent used in this process is called the leachant, while the solution obtained after leaching is known as leachate.The release of constituents is governed by a combination of chemical processes and mass transfer mechanisms based on the chemical composition and physical properties of the solid material along with the pH, redox and composition (i.e., dissolved constituents) of the contacting liquid [35].

Leaching of metals from concrete samples is a major environmental concern, especially when Supplementary Cementitious Materials (SCMs) are used as a partial replacement for cement. SCMs often contain trace amounts of metals, which may leach out over time and pose risks to the environment. Therefore, it is essential that the leaching behavior of such materials complies with environmental regulations. However, one of the key challenges in this area is the lack of a standardized leaching test specifically designed for concrete, particularly for monolithic forms. This makes it difficult to accurately assess the environmental safety of concrete incorporating SCMs, highlighting the need for adapted or modified testing procedures. But leaching test methods developed for solid wastes can also be adapted for testing concrete samples. These tests follow specific standards and are essential for assessing every waste material to identify environmentally hazardous substances that should not be released into the environment[35].Many batch leaching test protocols have been developed to simulate the leaching processes of waste materials in landfill or other disposal scenarios to evaluate potential risks to human and/or groundwater [38]. The primary focus of leaching tests is on metals and ions such as Chloride ions (Cl^-), that may leach from the system under consideration.

1.4.4.1 Types of Leaching Tests

In general there is two types of standarisied leaching test that are batch and column test which is are illustrated below. It is important to note that most of these tests are designed for granular samples rather than solid materials[1].

The main differences among these tests are leaching solution, liquid to solid (L/S) ratio, and number and duration of extraction[38].The comparison of batch test with column test is given in the figure 1.2 below



Parameters	Batch Test	Column Test
Test Period	Short-term (hours to days)	Long-term (days to months)
Operation	Easy to operate	Difficult to operate (channeling due to non-uniform packing of waste)
Cost	Relatively low	Relatively high
L/S ratio	Relatively high (To estimate maximum amounts of pollutants to be leached)	Relatively low (close to field conditions)
pH control	Easy to control pH with appropriate chemical	Material dictates its own chemical environment

Figure 1.2: Comparison of Batch test with Column test

- **Batch Leaching Test**
  The batch tests typically involve mixing size-reduced waste with extraction solution and then agitating the mixture. These tests generally are performed for a short period of time (typically for hours or days) and therefore are often called short -term tests. Some of the examples of batch tests are Extraction Procedure Toxicity (EP-Tox; US EPA Method 1310, 2001) and Toxicity Characteristic Leaching Procedure (TCLP; US EPA Method 1311, 2001)[38] .
- **Column Leaching Test**
  The column test is a type of leaching test in which the solid or waste material is packed into a column, and the leachant is continuously passed through it. This setup allows for extended contact time between the leachant and the solid matrix, enhancing the leaching process. The continuous flow of the leachant improves the leaching rate, providing a more accurate assessment of the material's leaching behavior over time. Unlike the batch leaching tests, the leaching solution is under continuous flux. Therefore, this test is often called a dynamic test and may be more representative of field conditions [38].

1.4.4.2 Factors affecting leaching test

The leaching process is influenced by a range of internal and external factors that together determine the rate and extent of material release from solid matrices. External factors include environmental exposures such as high concentrations of hydrogen ions (H^+), carbon dioxide (CO_2), and chloride ions (Cl^-), which can aggressively interact with the material and accelerate leaching[35]. Additionally, the transport of water into and out of the solid surface significantly affects the leaching process, especially under fluctuating environmental conditions. Physical properties like particle size, porosity, and permeability are also crucial, particularly when dealing with monolithic samples, as they govern the ease with which leachants penetrate the material [35].

On the other hand, internal factors such as mass transfer characteristics play a vital role. These include the diffusivity of substances within the solid matrix, the degree of local equilibrium achieved during the leaching process, and the available surface area for interactions. Another critical aspect is the composition of the leachant itself. The type of leachant whether it is neutral water or an acidic solution can greatly influence the chemical reactions occurring at the solid-liquid interface and, consequently, the extent of metal release [35]. Together, these internal and external factors must be carefully considered to understand and predict the leaching behavior of materials like concrete incorporating supplementary cementitious materials.

At the material interface, interaction of the solid materials with the surroundings can lead to alteration of the material release behavior. In some cases, reactions with components of the groundwater or subsurface atmosphere may increase release [35].Although these factors represents a monolith or solid cube, these factors also apply to the waste or system under consideration, which consists of fine granules. The same fundamental principles govern finer-grained materials, where leachant interaction and leaching follow a percolation model which means fluid (such as lechant) moves through the granular materials.

Leaching test methods may be categorized by whether the intent of the method is to establish equilibrium between the material and the leachant (e.g., “equilibrium-based test”) [35]. Another is "Dynamic leaching method" [35] in which the leachant liquid is changed in regular interval of time which fresh leachants.A key feature of this method is that it focuses on mass transport rather than equilibrium, with leaching evaluated as a function of time.In order to determine the chemical stability of materials, equilibrium leaching is commonly evaluated using the liquid-to-solid (L/S) ratio and pH. Dynamic tests, on the other hand, are particularly well-suited to investigate the rate or amount of leaching over time.

- **Influence of PH on equilibrium**
  pH is considered a principal variable controlling the equilibrium between the liquid and solid phases for many inorganic contaminants.The pH of a solid is largely influenced by the presence of inorganic materials. These inorganics can exist in various forms, including being incorporated within minerals (e.g., strontium or barium substituting for calcium), adsorbed onto mineral surfaces such as iron oxy(hydr)oxides or organic matter[35], precipitated as low-solubility solids within pore spaces, or dissolved in the liquid phase retained within the pore structure.

- **Influence of Liquid-to-solid on equilibrium**
  The water-to-solid ratio is a fundamental consideration in leaching tests. Sufficient water should be used to ensure complete contact with all particle surfaces and allow percolation through the porous structure of the solid.Changes in pore water chemistry as more soluble components are released can alter the dissolution of the more stable mineral phases and subsequent pore solutions and leaching of Contaminants of Potential Concerns (COPCs), as infiltrating water continues to

percolate [35]. The column test and batch test provide valuable insights into the influence of the liquid-to-solid (L/S) ratio on leaching behavior. Column tests allow precise control over water contact and enable the analysis of aqueous equilibrium as a function of the fluid volume passing through the solid mass, normalized as the L/S ratio. At lowL/S, concentrations provide insight into the composition of the initial pore solution; while concentrations at an L/S greater than or equal to 5 mL/g-dry indicate the effects of long-term exposure on Liquid solid partitioning (LSP) [35].

1.4.4.3 Leaching Test for Monolith Concerte samples

Since the existing leaching tests are primarily designed for granular waste materials (EN 12457, CEN/TS 16637-2) several studies have been conducted to develop standardized methods specifically for monolithic samples. However, in these researches they adopted key aspects of established tests methods and develop a modified testing method suited to monolith samples. Two of the most important test methods for monolith samples, as per European regulations, are detailed in the section below.

- **Semi Dynamic Tank Test**

  This method is designed to provide the mass transfer rates (release rates) of inorganic analytes contained in a monolithic or compacted granular material, under diffusion- controlled release conditions, as a function of leaching time [35].The samples used are monolithic in nature, and the eluate is replaced at regular intervals. In this method, the monolith samples must be fully immersed in the eluant to ensure optimal solid-to-liquid contact time. The leaching process occurs under mass transfer-controlled conditions, allowing for accurate assessment of leaching.

  The geometry of the samples should ideally be in the form of cubes, wafers, or cylinders. Using such shapes allows for better representation of 3D mass transfer conditions, which improves the accuracy and reliability of the leaching assessment.. In all cases, a minimum sample size of 5 cm in the direction of mass transfer must be employed and the liquid-surface-area (LSa) ratio must be maintained at 9 ± 1 mL/cm^2 [35] . The sample should be immersed 98 percentage into the eluant. Samples (eluates) are collected at nine-time intervals: 2 hours, 24 hours, 48 hours (2 days), 5 days, 7 days, 14 days, 21 days (7 days after previous sample),28 days (14 days after previous sample) and 42 days (14 days after previous sample). Each eluate samples (eluates) are collected at nine-time intervals: 2 hours,24 hours,48 hours (2 days),5 days,7 days ,14 days,21 days (7 days after previous sample),28 days (14 days after previous sample) and 42 days (14 days after previous sample) Each eluate is analysed using ICP-MS (Inductively Coupled Plasma Mass Spectrometry) to determine heavy metal concentrations. Additionally, the pH of the collected eluates is measured at each interval[35]. The concentration of leached heavy metals at each time interval is recorded. A cumulative leaching curve is plotted, with: Cumulative time (days) on the x-axis and Heavy metal concentration (mg/L or μg/L) on the y-axis. This plot helps visualize the leaching behaviour over time, showing whether heavy metal release stabilizes or continues increasing. In our project, we modified this leaching test method and adapted it for both monolithic and granular samples; further details will be provided in later chapters.

- **Up Flow Precolation Column Test**

This method is suitable to a wide range of granular solid materials. Example materials include industrial wastes, soils, sludges, combustion residues, sediments, construction materials, and mining wastes [35]. In this process Eluant is introduced into a column of moderately-packed granular material in an up-flow pumping mode, with eluate collection performed as a function of the cumulative LS ratio. Up-flow pumping is used to minimize air entrainment and flow channeling.

A column leaching test is conducted to evaluate the release of heavy metals from granulated concrete samples under controlled flow conditions. A cylindrical column with a length of 30 cm and a diameter of 5 cm is used, resulting in a total volume of 588.75 cm³.The mass of granular particles must be greater than 1/120 of the column diameter to ensure uniform packing.The column is packed in layers as follows [35]:

o Bottom layer: 1 cm of silica sand.

o Middle layer: 300 g of test sample (granulated concrete).

o Top layer: 1 cm of silica sand.

The leaching solution is pumped from the bottom of the column upward, ensuring consistent percolation through the test sample. The flow rate of the pump is controlled to achieve an eluate production rate of 0.75 ± 0.25 L/S per day [35]. Nine eluate fractions are collected up to L/S ratio of 10 ± 0.2 mL/g-dry. The cumulative liquid-to-solid ($\sum$ L/S) ratios using for the test are 0.2, 0.5, 1, 1.5, 2.0, 4.5, 5, 9.5, 10 respectively. The corresponding flow rate is calculated for each L/S ratio, determining the volume of eluate to be collected. The time required to collect the eluate is determined using the following equation 1.3:

$$T = (M_{dry} - (\sum L/S_i))/R_i \tag{1.3}$$

M_{dry} is the mass of dry material, R_i is the pump rate at specific interval i and target cumulative $\sum$ L/Si is the target cumulative ratio for interval i. The collected eluate is analysed using ICP-MS (Inductively Coupled Plasma Mass Spectrometry) to determine heavy metal concentrations. Additionally, the pH of the collected eluates is measured at each interval [35]. The concentration of leached heavy metals at each time interval is recorded. A cumulative leaching curve is plotted, with: Cumulative time (days) on the x-axis and Heavy metal concentration (mg/L or μg/L) on the y-axis. This plot helps visualize the leaching behaviour over time, showing whether heavy metal release stabilizes or continues increasing.

2 RAW MATERIALS AND METHODOLOGY

2.1 Raw materials

The raw materials used to produce the concrete cubes include pretreated MIBA samples, base cement, sand as fine aggregate (with particle sizes less than 2 mm), a superplasticizer, and water. The MIBA was collected from a Waste-to-Energy (WtE) plant, and particles larger than 2 mm were excluded, as they could not be milled using the rotating ball mill available in our laboratory. Basic information about each of these raw materials is provided in the background section above.

2.2 Methodology

2.2.1 Pre treatment of MIBA

2.2.1.1 Drying

The sample obtained was initially wet, and the primary objective was to determine the optimum drying time required to completely dry it. For this purpose, 300g of the sample was placed in a beaker and dried for 24 hours at 105 °c. The combined weight of the sample and the container was recorded both before and after the drying process. The sample was then subjected to an additional 24 hours of drying, making a total of 48 hours, and the weight was measured again. If the weight after 48 hours remained the same as that after the first 24 hours, it was concluded that the sample had reached a constant weight, indicating that the optimum drying time had been achieved.For detailed information on the calculations, refer to the Appendix 8.1.

2.2.1.2 Separation and Size Reduction

After the drying process, The dried MIBA sample was sieved for 4 minutes using sieves of sizes 2 mm, 1 mm, and 0.5 mm, respectively. The sieving process was conducted in six batches with different sample weights. More detailed calculations and observations about separation, Size distribution are mentioned in Appendix A 8.2.1. Particles larger than 2mm were discarded as they could not be milled using the rotating ball mill. While the remaining fractions were separated based on size and collected in different containers. Each size fraction was then weighed, and the data was used to plot a particle size distribution curve, providing insight into the distribution of particle sizes within the sample.

The three size fractions obtained (particles >1mm, particles between 0.5mm and 1mm, and particles smaller than 05mm)were each milled separately into fine powder (approx 75 μm) as shown in figure 2.1 to achieve a decent comparison to cement. Different milling methods were employed for each size fraction to ensure efficient and consistent size reduction across all samples.

Figure 2.1: Ball milled MIBA samples from left >1mm , > 0.5mm and < 0.5mm

- **Sample with particles greater than 0.5 mm**
  For particles larger than 0.5mm, ball milling was performed to reduce the particle size comparable to cement. About 20g of MIBA sample were put in a stainless-steel container along with 31 stainless-steel balls and was tightly secured into the milling machine[12]. The counterweight was set to match the total weight of the sample holder and the contents inside. After this the milling machine was run for 20 minutes at 500 rpm and switching rotating direction every 5 minutes[12].After milling, the particle size was assessed using a 75μm sieve, ensuring that at least 95% of the material passed through. For detailed information on the particle size analysis of the milled samples, refer to the Appendix A 8.2.2. We should note that this size analyses method cannot completely say that the size of the particle is within range of 75 micrometres because the powdered sample can get agglomerate together which will affect the size analysis method.
- **Sample with particles less than 0.5mm**
  For particles smaller than 0.5mm, a trial-and-error approach was adopted to determine the optimal milling parameters. Detailed information on the trial-and-error method can be found in the Appendix A 8.2.3. Based on the results, the optimal conditions were found to be ball milling at 250 rpm using 20g of the sample, 31 balls, and a milling time of 10 minutes.
- **Sample with particles greater than 1 mm**
  Since the Retsch PM 100 milling machine could not handle particle sizes larger than 1mm, particles exceeding this size were first processed using a jaw crusher. The goal was to reduce their size to below 1mm so that all material could pass through a 1mm sieve. As a result, approximately 95% of the sample successfully passed through the sieve. After jaw crushing, the material was separated into two fractions: particles larger than 0.5mm and particles smaller than 0.5mm. The fraction greater than 0.5mm was then subjected to the same ball milling procedure (500 rpm with 31 balls for 20 minutes). The remaining sample with size less than 0.5mm is treated with the new recipe.

2.2.2 Production of Concrete Test Cubes

To conduct strength tests, concrete cubes incorporating MIBA as a substitute were cast in accordance with the SS-EN 196 standard [7].The aim was to evaluate compressive strength at curing intervals of 7, 28, and 56 days and leaching tests. The Recipe in the table 2.2 below is according to EN 196 standard for 6 cubes of size 4mm × 4mm × 4mm. The mix design included sand with a particle size of less than 2 mm (sieved), deionized water, base cement, and MIBA . However, due to the small quantity of material prepared and the laboratory-scale limitations associated with mechanochemical grinding, the standard procedure was slightly modified. A total of 60 concrete cubes were prepared, with 15% of the cement replaced by MIBA and . Among these, 6 cubes were made using 100% base cement, serving as reference samples for comparison in strength testing and also 6 cubes with 15 % substitution of cement with Coal Fly Ash for comparison in Leaching of metals from the samples . Base cement was selected due to its widespread use and reliability in construction applications.

Table 2.1: Recipes used when casting cubes with 15 % MIBA substitution

Ingredients	**15% MIBA**
MIBA	33.75g
Bascement	191.25g
Sand	67.5g
Water	112.5g

After the production curing of concerte samples should be done since the samples are made using waste materials, the risk of leaching is higher during the curing period. In such cases, if the leachate is to be measured, traditional water curing may not be suitable, as it can interfere with accurate leaching analysis. Therefore, an alternative curing method is often used—maintaining the samples in an environment with 100% relative humidity without fully submerging them in water as shown in the figure 2.2 . This approach helps preserve the moisture necessary for hydration while preventing dilution or loss of leachate, allowing for more accurate monitoring of leaching behavior.

Figure 2.2: Curing of cubes without Submerging them in water

2.2.2.1 Strength Test of Concrete Cubes

The compressive strength of concrete is measured by testing cubes or cylinders in a laboratory.the strength of the concrete cubes was tested after 7, 28 and 56 days of curing time according to SS-EN 196 standards [32].In each specific intervals were picked out from the water storage container. Using paper towels the cubes where dried before continuing with the test. The cubes were put into a mount and the area that was not covered by the walls of the mold was avoided to be put against the metal plates in the mount. The sample was compressed in a Matest compression machine until mechanical failure [12]. At this point the maximum load and maximum strength was noted down.

2.2.3 Leaching Test

Since our study also considers the end-of-life disposal of concrete, it is important to evaluate the material not only in its monolithic form but also as granular fragments.

As outlined in the background's leaching section, a semi-dynamic (batch leaching) test is suitable for monolithic samples. Although a percolation column test is typically recommended for granular materials, its standardized procedure is time-consuming and complex. Therefore, we adopted and modified the semi-dynamic test based on the EPA/600/R-10/170 protocol [35], applying it to both monolithic and granular samples in this study.

For the leaching tests, both monolithic and granular samples were fully immersed in beakers, with approximately 98% of each sample submerged in water, maintaining a liquid-to-surface area ratio of 10 (ml/cm^2). Granular samples are made by manually crushing the monolith sample using a hammer until 98% of the material passed through a 2 mm sieve. We prepared four types of samples for both monolith and granular samples with varying particle sizes including samples greater than 1 mm, greater than 0.5 mm, less than 0.5 mm, and a mix incorporating coal fly ash. In total, eight beakers each containing 960 mL of leachant which is water. The parameters for this leaching test are summarized in the table 2.3below.

Table 2.2: key parameters used in your leaching tests for both monolithic and granular concrete samples

Parameter	Description
Sample Types	Monolithic and Granular
Liquid-to-Surface Area Ratio	10 mL/cm^2
Particle Size Fractions	>1 mm, >0.5 mm, <0.5 mm, Mix with Coal Fly Ash
Number of Sample Types	4 for monolith, 4 for granular
Volume of leachant per Beaker	960 ml

To ensure uniform exposure, the monolithic samples were suspended in the beakers using a custom-made plastic holder, allowing all sides of the sample to be in contact with the water. The granular samples were placed in a permeable cloth bag, which allowed water to flow freely in and out, ensuring interaction between the water and the sample material while preventing particle dispersion in the medium. The beakers should be closed properly so that water does not evaporate from the system. The eluate samples were collected from each beaker at regular time intervals, specifically at 2 hours, 1 day, 2 days, 5 days, 7 days, 21 days, 28 days and 46 days. At each interval, pH and electrical conductivity of the eluate were also measured to monitor changes in the chemical environment during the leaching process. The collected eluates were analyzed for metal content using the inductively coupled plasma mass spectrometry (ICP-MS) method. Furthermore, cumulative metal release was plotted against cumulative leaching time to assess leaching behavior during the test period. After each sampling, the water in the beakers was replaced with fresh deionized water to maintain consistent test conditions and accelerate the leaching process.

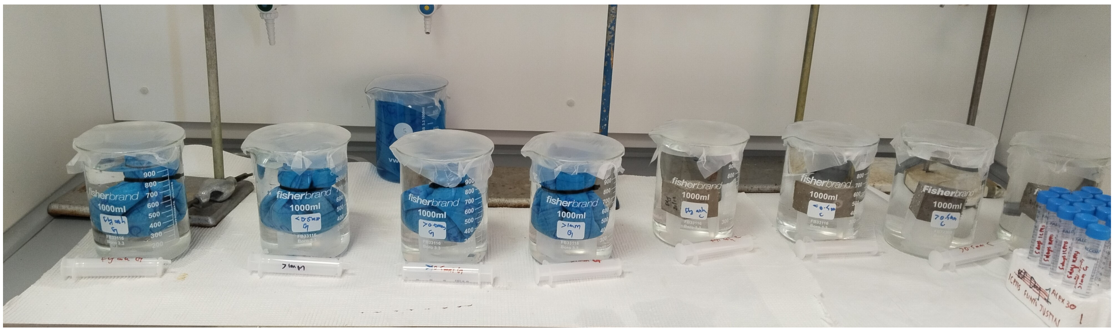


Figure 2.3: Experimental setup for Batch leaching Test

3 ANALYTICAL METHODS

Following analytical methods are used for the chemical analysis of samples that we have obtained.

3.1 Powder X-ray diffraction

Powder X-ray diffraction (PXRD) is a technique that involves directing X-rays at a sample at its crystalline phases. The resulting data is displayed as a diffractogram, which plots intensity against the angle theta[16]. By comparing the positions and intensities of the peaks with those in a reference database, the various crystalline phases present in the sample can be identified.The acquire diffractograms were later analyzed using the program DIFFRAC.EVA where the search by name function was used to search for phases that was expected to be found in in each sample [4]

In this project, Powder X-ray Diffraction (PXRD) analysis was conducted to examine the mineralogical composition and phase structure of the three different MIBA fractions. This characterization was essential to help explain the observed differences in compressive strength when these fractions were used as partial cement replacements in concrete cubes. MIBA powder of different fractions is placed into a circular mold and evenly distributed to ensure uniform packing. The mold is then placed into the sample holder and inserted into the XRD machine for analysis. Since we did not have access to DIFFRAC.EVA software, the phase identification and peak analysis of our samples were carried out by comparing the XRD patterns with those reported in recent research studies. This approach allowed us to match the observed peaks with known mineral phases documented in the literature[16].

3.2 ICP-MS

ICP-MS is an extremely sensitive and accurate analytical method for finding metals and a number of non-metals in liquid samples at trace and ultra-trace levels. It works by ionising the sample with an inductively coupled plasma, and then using a mass spectrometer to analyse the ions according to their mass-to-charge ratio[10]. ICP-MS is widely used in geochemistry, leaching investigations, and environmental monitoring to evaluate the release of elements over time because of its strong detection limits, broad dynamic range, and multi-element capabilities.

A total of 64 samples were collected from the tank leaching test, covering 4 sets of monoliths and 4 sets of granules for each cube type. Sampling was carried out at the following intervals: 2 hours, 1 day, 2 days, 5 days, 7 days, 14 days, 21 days, and 28 days. Each sample was diluted 45-fold with Milli-Q water and acidified with nitric acid. Once diluted, the samples were placed in trays and loaded into the ICP-MS system for analysis.

3.3 pH Measurement

pH measures the concentration of hydrogen ions in a solution, indicating whether the solution is acidic or alkaline. The pH scale ranges from 0 to 14, with values below 7 indicating acidity and values above 7 indicating alkalinity. Several factors can affect pH measurement, including ionic strength, temperature, and the condition of the electrode. Therefore, proper storage and maintenance of electrodes are essential to ensure consistent and accurate results[5].

pH is typically measured using a pH meter, which includes a probe—usually a combination electrode—that detects the voltage difference between a reference and a sensing electrode. This voltage is then converted into a pH value. Regular calibration of the pH meter with standard buffer solutions is necessary to maintain measurement accuracy and reliability.In this project, the pH of eight samples was measured at different time intervals using a pH meter to study the leaching behavior of metal ions from solid materials into Milli-Q water.

3.4 Conductivity Measurement

Conductivity measures a solution's ability to conduct electric current, which is directly related to the concentration of dissolved ions such as chloride (Cl^-), acids, or bases. The unit of conductivity is typically expressed in microsiemens per centimeter (μS/cm) or millisiemens per centimeter (mS/cm), depending on the ionic strength of the solution [15] . Conductivity is an important parameter for assessing water quality and studying chemical leaching processes.

Generally conductivity is measured using conductivity meter which applies a voltage across two electrodes submerged in the solution. The resulting current is proportional to the ionic concentration. In our project we measured the conductivity of our samples to provides insight into the total ion release from solid materials into the aqueous phase, which can be linked to the dissolution of metals or salts over time[35].

4 RESULTS AND DISCUSSIONS

4.1 Characterization of MIBA

4.1.1 Size Distribution

After sieving, the cumulative weight percentage was calculated and plotted against sieve size, as shown in the figure 3.1. Batches 1,2,3,5 and 6 display a similar trend; however, Batch 4 shows a slightly different pattern due to its smaller sample quantity. Despite this variation, the cumulative weight percentages for Batch 4 follow a similar ratio to the other batches. From the graph, we observe that approximately 55–60% of the sample passes through the 2 mm sieve, around 35–38% passes through the 1 mm sieve, and 20–21% passes through the 0.5 mm sieve. The remaining material is retained in the tray. For the experiment, particles larger than 2 mm were discarded as they could not be milled using the rotating ball mill. Although particles larger than 1 mm are also unsuitable for direct ball milling, we used a jaw crusher to reduce their size to below 1 mm, allowing further milling in the ball mill. As a result, only about 50% of MIBA sample was usable, since the remaining 50% consisted of particles larger than 2 mm. But it would be particularly interesting to explore the use of advanced milling equipment for processing the larger than 2 mm fraction, as this size range exhibits a similar composition and mineralogy to the finer fractions, as discussed in Section 3.1.2 below. With appropriate milling, these coarser particles could be effectively utilized, expanding the potential application of the entire MIBA sample.

Additionally, after sieving and milling to the required size, we observed noticeable color differences among the size fractions. These variations in color are explained in detail through mineralogical by PXRD (Powder X-Ray Diffraction) analyses in the following sections 3.1.2.1.

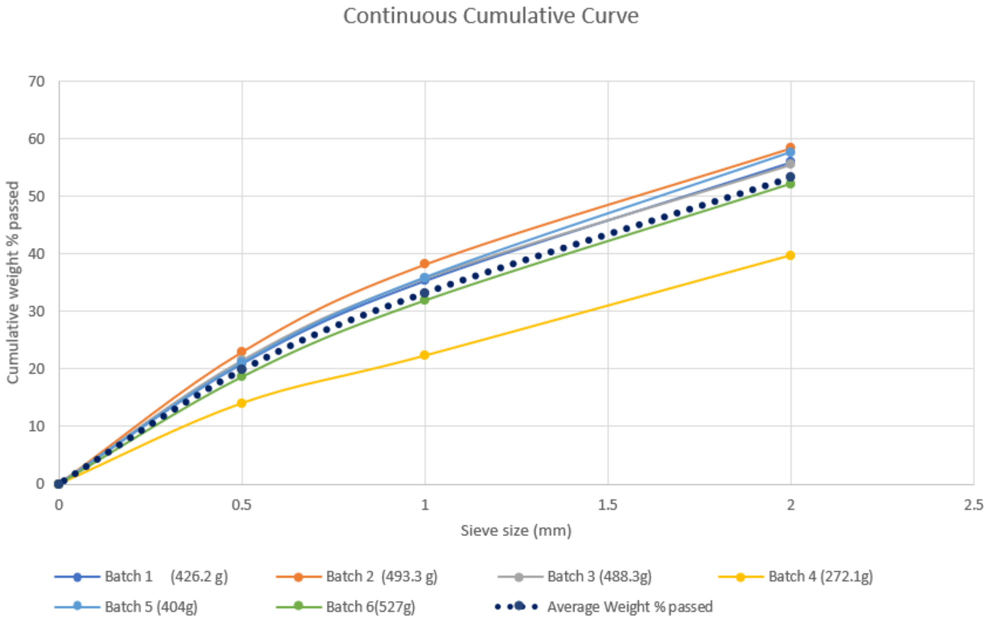


Figure 4.1: Cumulative Weight Percentage obtained from 6 batches

4.1.2 Mineralogy

The elements that is present in different size fraction after milling is shown in the table 4.1 below.

Table 4.1: Total elemental content of the MIBA in different fractions mg/Kg Ds [37]

Element	< 0.5mm MIBA	> 0.5mm MIBA	> 1mm MIBA	>2mm MIBA
Aluminum (Al)	65600	64500	65100	58200
Arsenic (As)	100	50	50.5	40
Barium (Ba)	2000	2200	2300	3100
Beryllium (Be)	1	2	1.3	1.05
Calcium (Ca)	139400	120800	121500	123600
Cadmium (Cd)	11	5	21.1	2
Cobalt (Co)	240	210	160	55
Chromium (Cr)	1300	1500	1200	800
Copper (Cu)	10200	5900	8500	3900
Iron (Fe)	69900	102800	77600	100700
Mercury (Hg)	0.05	0.04	0.04	0.01
Potassium (K)	8900	10000	10800	8700
Magnesium (Mg)	11600	12700	12400	13300
Manganese (Mn)	1400	1700	1400	2400
Molybdenum (Mo)	82.7	87.3	77.9	49
Sodium (Na)	15300	18700	1400	26300
Nickel (Ni)	800	700	600	400
Phosphorous (P)	5200	5000	5500	4800
Lead (Pb)	1100	1000	1100	500
Sulfur (S)	11700	5600	6000	4200
Silicon (Si)	148600	193100	180500	188800
Zinc (Zn)	8700	6600	6400	5100
Titanium (Ti)	9500	10100	9800	9820

When observing the table we can see that Silicon (Si) and Calcium (Ca) are the dominant elements across all MIBA particle size fractions. Iron (Fe) and Aluminum (Al) are also present in high concentrations, indicating the significant presence of metal-rich waste materials. When we analyses the chemical composition of municipal incineration wastes we can see that the predominant oxides present are SiO_2, CaO, Al_2O_3, and Fe_2O_3 which validate our conclusions [34] . Although elements like Lead (Pb), Cadmium (Cd), Mercury (Hg), and Arsenic (As) occur in much lower concentrations, their environmental toxicity is significant. Notably, their concentrations tend to decrease as particle size increases, suggesting a higher affinity for finer particles. Studies shows that metals like As, Cd, Cr, and Pb were enriched in the finest particle fractions of bottom ash. This enrichment is likely due to vaporization and subsequent condensation of these metals onto smaller particles during the incineration process [26]. Sodium (Na) and Potassium (K) show an opposite trend, with their concentrations increasing with particle size. Sodium, in particular, is most abundant in particles larger than 2 mm. Research has demonstrated that sodium (Na) and potassium (K) concentrations tend to increase with particle size in bottom ash samples.[17].

Overall, finer particles (<0.5 mm) contain higher levels of toxic and volatile elements. Approximately 43% of the total elemental content is concentrated in these finer fractions. This distribution provides valuable insight for determining the reuse potential of MIBA in applications such as concrete production, where different size fractions may be selected based on their elemental composition and associated environmental risks.

4.1.2.1 PXRD

Powdered-XRD analysis was conducted to better understand the crystalline and amorphous structures present in the different size fractions of the samples. This analysis also helps establish a relationship between the mineralogical composition and the reactivity of the materials, which is essential for evaluating their suitability as supplementary cementitious materials. The X-ray Diffraction (XRD) patterns presented below 4.2 show the mineralogical composition of MIBA powders with three different particle size fractions are given below . note that all the size fractions are milled into same size (70μm).

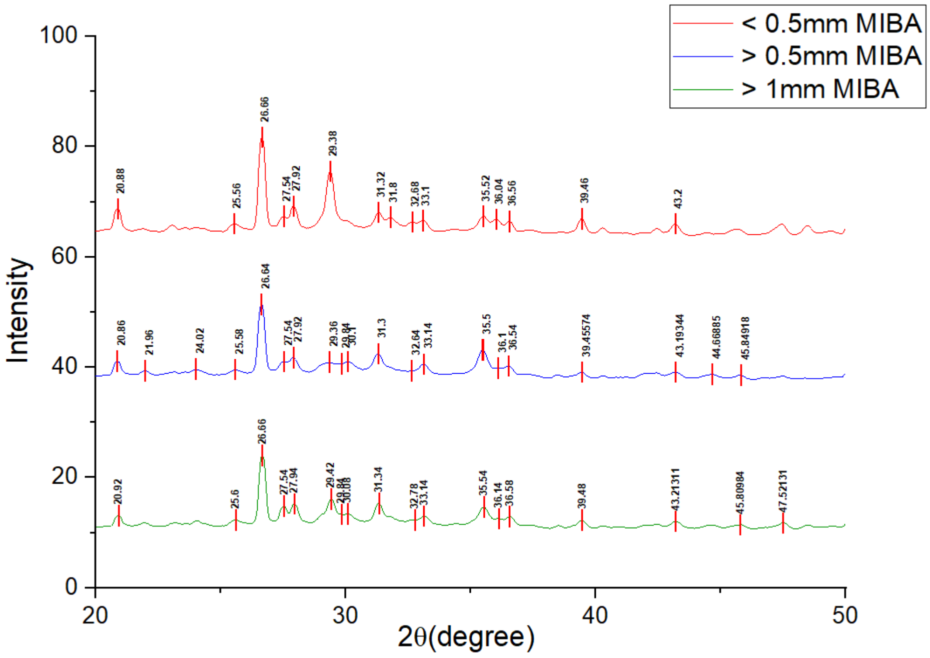


Figure 4.2: XRD Diffractogram of different size fractions of MIBA

The major diffraction peaks observed across all size fractions correspond to quartz (SiO_2), with dominant reflections at 2θ values of approximately 26.6°, 20.8°, and 36.5° which is a major crystalline phase commonly found in incinerator bottom ashes [41].The presence of quartz in MIBA does not contribute directly to cement hydration but can act as an inert filler and potentially influence the microstructure[14]. Additionally, the presence of calcite ($CaCO_3$) was identified by a peak at around 29.4°. Minor peaks observed between 27° to 33° suggest the possible presence of feldspars, aluminosilicates (e.g mullite-$3Al_2O_3{\cdot}2SiO_2$), and metal oxides such as Fe_2O_3 or Al_2O_3 spinel-type phases[20].Although these are not typically found in ordinary Portland cement, these phases may contribute marginally to long-term strength if properly activated. They can also influence the chemical durability and mechanical properties of the concrete composite[27].The PXRD patterns for the >0.5 mm and >1 mm MIBA fractions display sharp and well-defined peaks, indicating a higher crystalline content. This high crystallinity is associated with reduced pozzolanic reactivity, as reported by Eriksson [12]. In contrast, the <0.5 mm fraction shows lower peak intensities and increased background noise in the diffractogram, suggesting a higher proportion of amorphous material. These observations are consistent with existing research, which highlights that finer MIBA particles tend to have improved pozzolanic activity due to their greater surface area and the enhanced presence of reactive aluminosilicate phases [30].

4.2 Characterization of Concerte Cubes

4.2.1 Compressive Strength Test

The compressive strength data, measured in MPa, is presented in the figure 4.3 below corresponding to three curing periods: 7, 28, and 56 days. For detailed table refer Appendix 8.3.

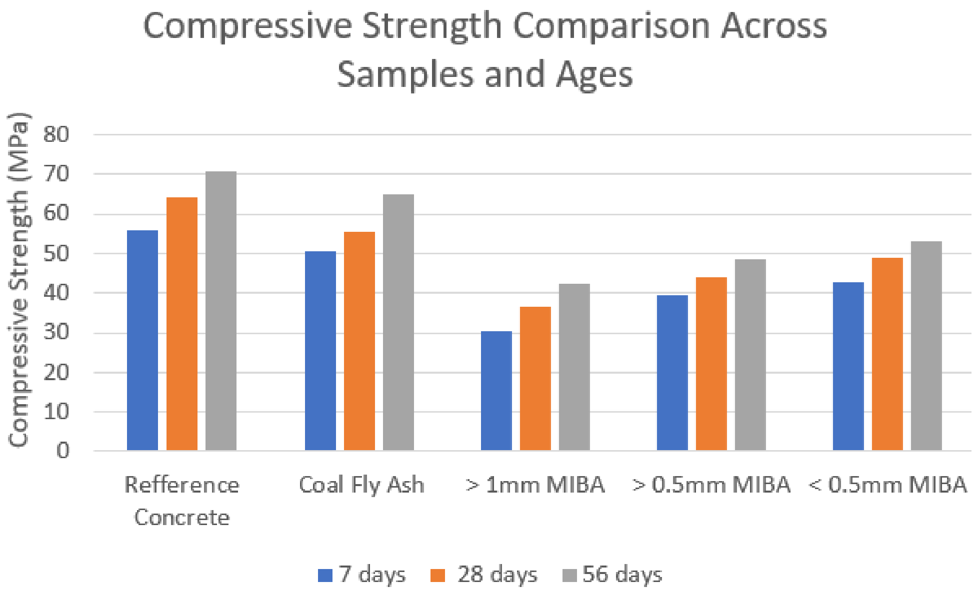


Figure 4.3: Compressive Strength Comparison Across samples and ages

From the graph, it is evident that none of the alternative samples reach the compressive strength of the reference concrete. There is a significant variation in strength among the different MIBA size fractions. In all samples, compressive strength increases over time, which reflects continued hydration and strength development with age. Interestingly, this increase appears to follow a steady, consistent trend for most samples. However, at 56 days, the compressive strength of the coal fly ash mix rises sharply, reaching approximately 91% of the reference concrete's strength, indicating strong long-term pozzolanic activity.Finer MIBA particles show notably better performance compared to coarser ones.

The previous research[12] further supports the importance of particle size. In his study, using 15% unprocessed MIBA (without size separation) as a cement substitute led to reduced performance, with compressive strength reaching only 66% of that of the reference mix after 56 days. This contrast emphasizes the advantage of using finely ground MIBA to enhance the mechanical properties of concrete.

These findings support that potential use of coal fly ash and finely ground MIBA as alternatives in concrete production[30]. While coarse MIBA showed limitations, finer fractions performed considerably better and may be considered for structural applications where strength requirements permit.

4.3 Leaching Test

After the leaching test, pH and conductivity of eluate were measured at consecutive intervals and plotted for different samples. To determine which metals had leached out, an ICP-MS (Inductively Coupled Plasma Mass Spectrometry) analysis was also conducted.

4.3.1 pH Measurement

pH measurements are included in standardized testing methods because they help determine criteria for the safe disposal of materials[11]. The release of metals into the leachant is highly influenced by pH levels. At lower pH values, metals such as cadmium, mercury, and copper become more soluble and are more likely to leach out. Conversely, at higher pH, these metals tend to precipitate, reducing their mobility. However, certain metals like lead may become more soluble under highly alkaline conditions, leading to increased leaching in some cases[22]. So for our analysis we plotted Separate graphs to illustrate the pH variation of granular and monolith samples over the defined sampling intervals. For detailed observation table see Appendix B 8.4.

4.3.1.1 pH Trends in Monolith Samples

The graph of pH of the eluate from the monoilth samples at a regular interval of time is represented in Figure 4.4 below. From the graph we can see that pH of eluate from monolith samples staying within the range of approximately 9.5–11.5. This pH range is supported by van der Sloot et al [39] , who state that leaching behavior is particularly significant within this interval. The study also highlights that elevated pH levels can lead to the dissolution of certain metals from the cement matrix, influencing their mobility and release.

All samples display more fluctuation in pH over time.The >1 mm MIBA concrete monolith show the lowest dip around sampling interval of 7 days, indicating some potential buffering or interaction that momentarily reduces alkalinity. The coal fly ash concrete cube sample again shows consistently higher pH, indicating a more alkaline metals or compounds that is leaching out to the environment. At the end of the sampling period ie day 28, the pH of all samples had decreased.

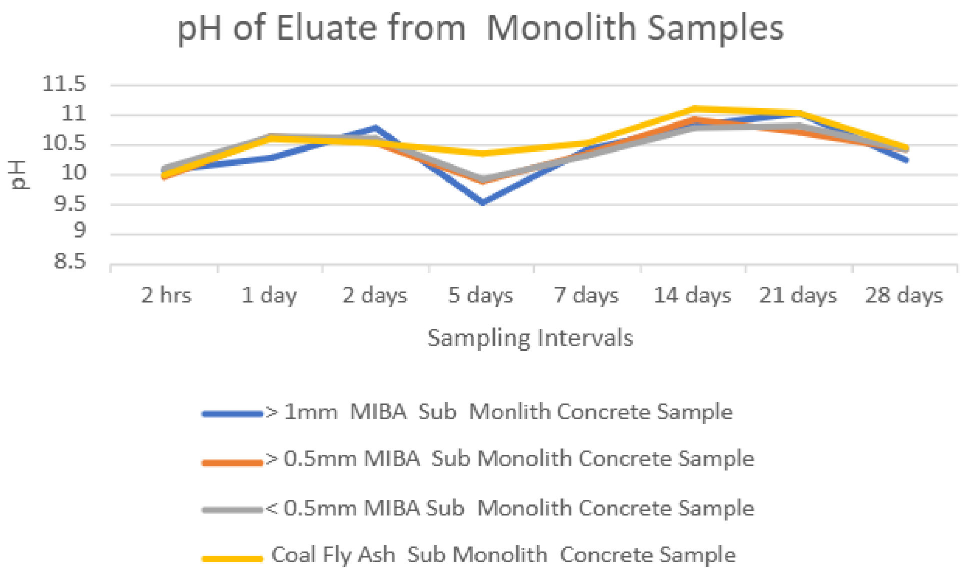


Figure 4.4: pH of eluate from cube sample at regular sampling Interval

4.3.1.2 pH Trends in Granular Samples

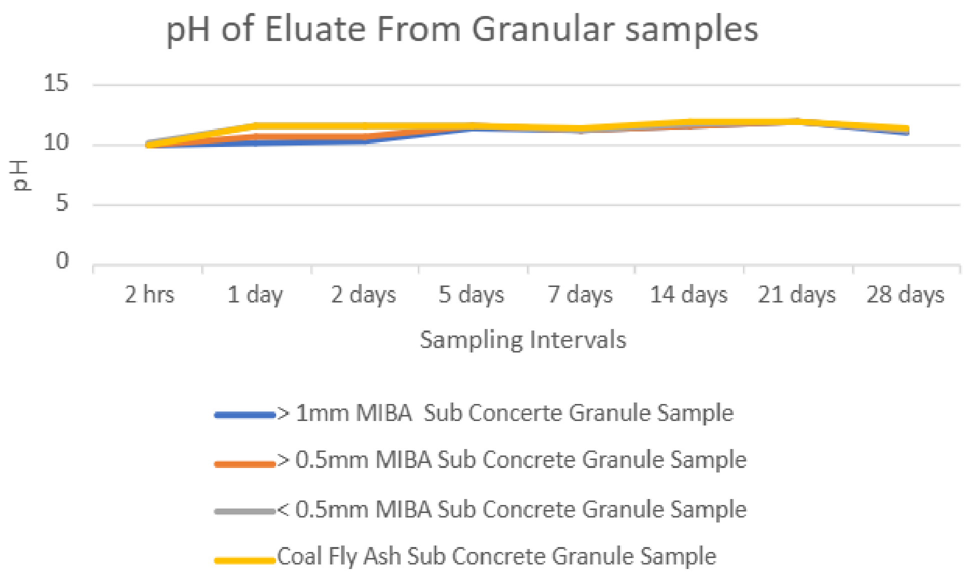


Figure 4.5: pH of eluate from granular sample at regular sampling Interval

The graph indicating the pH of the granular sample eluate at a regular interval of time is represented in Figure 4.5 above. Throughout the sampling intervals, all granule samples show high alkaline pH values (10-13), regardless of the type of ash or particle size. The highest and most consistent pH (12–13) is maintained by the coal fly ash concrete particles. Continuous leaching of alkaline compounds is suggested by the small increase in pH of MIBA-based granules over time, particularly those that are >1 mm and <0.5 mm. Although the initial pH of larger particles (>1 mm) is slightly lower than that of tiny particles, the differences in particle sizes are rather small. As similar in case of Monolith sample at last sampling interval we can observe a slit decrease in the pH

So in general we can see that Granule samples leach more alkaline or metal content initially and maintain higher pH than monolith, likely due to higher surface area exposure to leaching fluid. Coal fly ash-based samples consistently show the highest and most stable pH, indicating more leaching potential than MIBA substituted samples. MIBA-based samples, particularly the coarser fraction, show more variability in pH, which may suggest heterogeneous composition or slower dissolution kinetics. These pH profiles are important as they indicate the long-term leaching behavior and chemical durability of the concrete materials. High and stable pH may help immobilize certain heavy metals but could also affect surrounding environments if leachate escapes[39]. In studies examining the leaching behavior of waste-derived aggregates, it has been highlighted that pH tends to decrease over time, indicating a reduction in the leaching rate[29]. Similarly, in our testing, we observed a decline in pH after 28 days compared to earlier sampling intervals for both monolith and granular samples. This trend suggests that the leaching rate decreases as the curing or exposure time increases.

4.3.2 Conductivity Measurement

Conductivity reflects the total concentration of dissolved ions (e.g., Ca^{2+}, Na^{+}, Cl^{-}), providing insight into the rate and extent of leaching. It is often evaluated alongside pH and metal concentrations to better understand the underlying chemical processes, such as dissolution and precipitation, that govern leaching behavior. Separate graphs were plotted to illustrate the variation of conductivity of eluate collected from granular and monolith samples over the defined sampling intervals. For detailed observation table see Appendix B8.4.2.

4.3.2.1 Conductivity Trends in monolith Samples

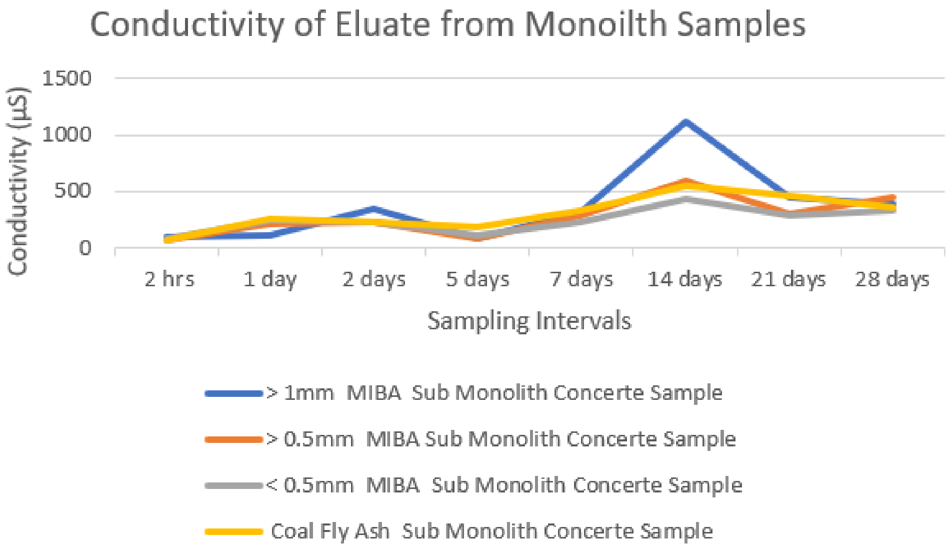


Figure 4.6: Conductivity of eluate from Cube samples at regular sampling Interval

The graph indicating conductivity of the eluate from the monolith samples at a regular interval of time is represented in Figure 4.6 above. The monolith samples eluate exhibit lower conductivity compared to granular samples, with higher value reaching up to 1200 μS. Among them, the >1 mm MIBA concrete monoliths eluate show the highest conductivity, peaking at around 1000 μS. While other momolith samples eluate display a general increasing trend, the coal fly ash and <0.5 mm MIBA concrete eluates show fluctuations in conductivity over time.

4.3.2.2 Conductivity Trends in Granular Samples

The graph indicating the conductivity of granular sample eluate at a regular interval of time is represented in Figure 4.7 below. The conductivity of granular samples is comparatively higher than that of the cube samples, indicating greater ionic leaching.

Coal fly ash granules exhibit the highest conductivity, reaching up to 4500 μS at the 21-day sampling interval, which suggests a higher concentration of soluble ions. Crushing concrete into granular form increases its surface area, which in turn promotes the release of soluble ions into the surrounding liquid, resulting in elevated conductivity levels. This effect has been observed in several studies. For instance, Kosson et al [33] found that incorporating fly ash in cement-based materials influences leaching behavior, particularly by enhancing the mobility of ions and increasing the conductivity of the leachate. Likewise, Van der Sloot et al [39] noted that concrete granules containing fly ash tend to show higher conductivity, likely due to the exposure of mineral phases that are either unreacted or only weakly bound within the matrix. These observations support the findings in the present study, where high conductivity in fly ash-containing concrete granules aligns with expectations based on established research.

MIBA-based granules, regardless of particle size, show a general increasing trend in conductivity. However, finer particles (< 0.5mm MIBA) display more fluctuations. A sharp rise in conductivity is observed between sampling intervals ie after 5 days and after 14 days for most MIBA granule types, followed by either stabilization or a continued increase.

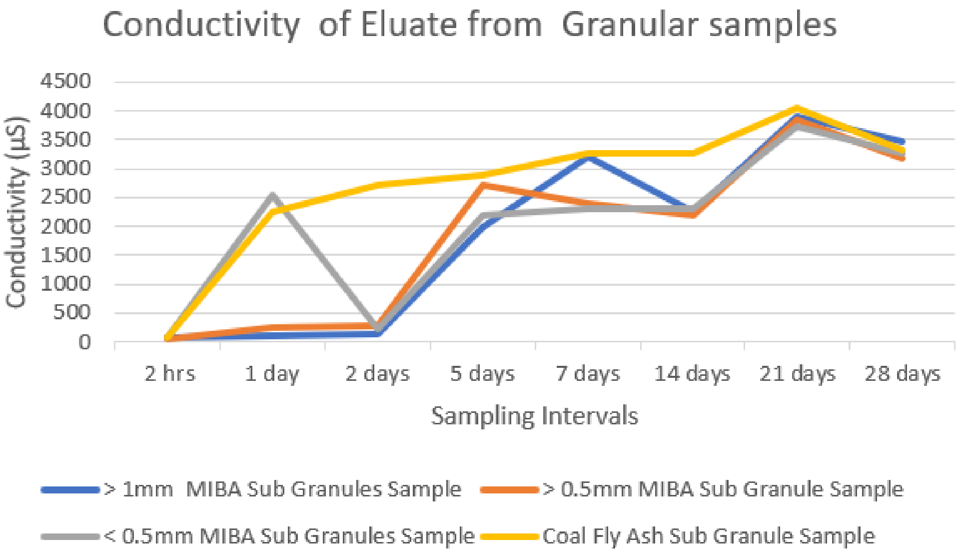


Figure 4.7: Conductivity of eluate from Granular samples at regular sampling Interval

In general, when we compare granular and monolith leaching behaviour we can say that granules leach more ions than monolith samples, confirmed by consistently higher conductivity values across all ash types and sizes. Coal fly ash consistently shows higher conductivity than MIBA, indicating higher leaching behaviour than any other samples. And as in case of pH conductivity of both the samples reduced at last sampling interval.

4.3.3 Analysis of Eluate from Monolith and Granular Samples

The eluates collected at each sampling interval were analyzed using Inductively Coupled Plasma Mass Spectrometry (ICP-MS) to determine the concentrations of metals leached from both monolithic and granular samples.

There are standardized threshold limits for metal leaching in Sweden, particularly relevant to the concrete and construction sectors[2]. However, these regulatory limits are often set at very low levels, making direct comparison with the leaching results obtained in this study difficult. Furthermore, certain metals that leached at relatively higher levels in our samples are specifically included in these standards, which complicates the interpretation. To provide a more comprehensive evaluation and to contextualize our findings within a broader framework, we incorporated additional threshold values reported in several other research studies.

we referred comparative tables provided by Snelling's et al[31]. that summarize regulatory values from various countries. Although the original report does not specify the testing method used to derive these values, this comparison offers a practical approach for assessing the potential environmental risk associated with the monoliths as well as granular samples in case of lack of a clear and consistent framework for acceptable leaching levels for such matrices.

For granular samples, the increased exposed surface area typically leads to higher metal leaching compared to monolithic samples[35]. As a result, applying higher threshold values may be more appropriate for evaluating granular materials. Although the thresholds proposed in the Lamber's report[13], were originally established for incineration waste intended for landfilling, we have used them as a reference point to compare with the leaching behavior observed in concrete applications. This approach provides a broader perspective for assessing the environmental acceptability of MIBA when used as a supplementary cementitious material. This classification system groups granular waste into two categories according to the concentration of leached metals: Category 1, which allows unrestricted use in landfills, and Category 2, which permits only restricted application due to higher environmental risk. These thresholds serve as benchmarks for assessing the environmental acceptability of granular materials derived from waste.

The granular leaching tests conducted in this study were designed to simulate a worst-case scenario for the potential release of contaminants, assuming the concrete is crushed or fragmented—for example, during disposal or accidental damage. While these granular samples do not represent the actual end-of-life state of concrete, they provide a conservative estimate of leaching behavior under extreme conditions. Comparing the leaching concentrations to established threshold values offers valuable insights into the long-term environmental impact and regulatory compliance of these materials. This classification approach aligns with our research objective of evaluating the environmental performance of waste-derived construction materials under disposal-relevant conditions.

We also compared the leaching behavior between the monolithic and granular forms of each sample and included coal fly ash-substituted samples as a reference. Furthermore, since some metal release was observed during the curing phase, we measured the metal content in the curing liquid over a 7-day period. Although not part of the standardized leaching protocol, these results provided additional context for understanding early-stage leaching phenomena and benchmarking them against controlled leaching tests.

In this section, we focus on the metals that only exhibited significant variations.These include barium (Ba), molybdenum (Mo), cadmium (Cd), and copper (Cu) from our observations. Other metals and its trends are explained in the Appendix C 8.5. The selection of these specific metals was based on several criteria:

- Their cumulative leaching concentrations exceeded established threshold values, indicating potential environmental risks.
- They exhibited significant fluctuations across sampling intervals, suggesting inconsistent leaching behavior.
- Some of these metals are not included in the Swedish standard threshold lists, necessitating further evaluation through alternative references.

4.3.3.1 Leaching of Barium

The cumulative leaching of barium (Ba) (mg/Kg of sample) from monolith and granular samples is plotted against the sampling interval is shown in te figure 4.8 below. The Swedish standards, barium is not included among the listed threshold values. This omission prompted us to consider additional reference thresholds from other sources to adequately assess its environmental impact.

Figure 4.8: Cumulative leaching Curves of Barium from Monolith (left) and Granular (right) Samples

- **Monolith sample**
  According to Snelling's report, the threshold limit for Ba leaching from concrete is 25 mg/kg. By observing the graph we can see that all monolithic as MIBA-substituted samples exhibitS Ba leaching values well below this threshold, indicating a low environmental risk during the material's service life in concrete applications.

  Among the monolithic samples, the >1 mm MIBA-substituted concrete exhibited the highest Ba leaching, followed by the > 0.5 mm MIBA sample. In contrast, the Coal Fly Ash monolith, used as a reference, demonstrated lower leaching levels than both MIBA-based samples.However, it was observed that the <0.5 mm MIBA sample leached less barium than the coal fly ash-substituted samples. This suggests that coal fly ash exhibits a leaching behavior comparable to that of MIBA-substituted samples, at least with respect to barium release. The relatively low leaching values for all monoliths are attributed to dense physical packing and limited pore connectivity, which effectively restrict the migration of Ba ions into the leachate[39].

- **Granular sample**
  when considering the granular samples, the leaching behavior changes significantly. When we consider the Snellings report the leaching rate of barium is below the threshold. But based on the thresholds reported by Lamers, where Category 1 has a limit of 3.4 mg/kg and Category 2 has a limit of 55 mg/kg, all our granular MIBA-substituted samples fall within Category 2. This classification suggests restricted applications due to the elevated environmental risk posed by the higher Barium leaching. When compared to the Coal Fly Ash-substituted granules (30.36 mg/Kg of granules), the > 1mm MIBA-substituted granules exhibit a higher rate of Barium leaching. This indicates that Coal Fly Ash releases similar amount of Ba when compared to MIBA in granular form.

In general we can say that monolithic MIBA-substituted concrete poses minimal leaching risk and complies with standard environmental thresholds. granular MIBA samples leach substantially more Ba, especially in finer fractions, surpassing the limits set for unrestricted use. Coal Fly Ash and MIBA samples exhibits similar Ba mobility , in both monolithic and granular forms. These findings underscore the importance of considering the full lifecycle of concrete materials. While MIBA-substituted concrete may be environmentally safe during its use phase, post-use handling (e.g., demolition and disposal) can significantly increase Ba leaching, due to structural breakdown and increased surface exposure. Therefore, end-of-life strategies must be addressed in the sustainable management of MIBA-containing materials.

4.3.3.2 Leaching of Molybdenum

The cumulative leaching of Molybdenum (Mo) (mg/Kg of sample) from monolith and granular samples is plotted against the sampling interval is shown in te figure 4.9 below. The Swedish standards, barium is not included among the listed threshold values. This omission prompted us to consider additional reference thresholds from other sources to adequately assess its environmental impact.

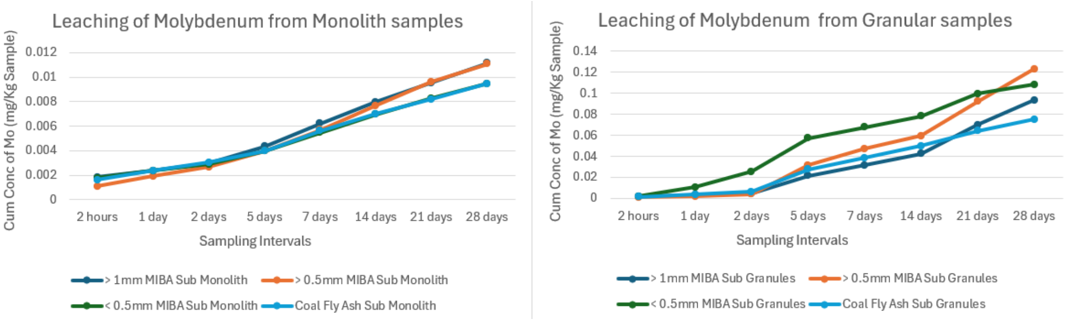


Figure 4.9: Cumulative leaching Curves of Molybdenum from Monolith (left) and Granular (right) Samples

- **Monolith samples**
  According to Snelling's report, the threshold limit for Molybdenum (Mo) leaching from concrete is 0.6 mg/kg. Based on the graph, all monolithic samples—including

  those substituted with MIBA—show Mo leaching well below this limit even after 28 days, indicating a low environmental risk.

  The MIBA-substituted monoliths exhibited similarly low leaching values, with minimal variation among different particle size fractions. The Coal Fly Ash monolith, used as the reference material, demonstrated slightly lower Mo leaching compared to the MIBA samples, particularly during the early sampling intervals. However, the overall differences remained minor throughout the leaching period, suggesting comparable environmental performance among all monolithic samples.

- **Granular Samples**
  When comparing with Snelling's threshold the rate of leaching is less in case of all granular samples. According to Lamers, the threshold limits for Mo leaching in granular materials are 0.21 mg/kg for Category 1 and 0.84 mg/kg for Category 2. From the graph, the >0.5 mm and <0.5 mm MIBA granules leach 0.12 mg/kg and 0.1082 mg/kg of Mo, respectively. These values are approaching the Category 1 threshold, indicating that Mo is a critical element in these samples. With extended leaching periods or under more aggressive environmental conditions, these values could potentially exceed the threshold and fall into Category 2, resulting in restricted applications due to higher environmental risk[13].

  For the granular samples, Coal Fly Ash exhibits the lowest Molybdenum (Mo) leaching, indicating better environmental stability compared to MIBA-substituted granules. Among the MIBA variants, the >0.5 mm MIBA granules show the highest Mo leaching at 28 days. This elevated leaching rate can be attributed to the mineralogical composition, as previously discussed, where the >0.5 mm fraction contains the highest Mo concentration (87.3 mg/kg dry solids) among all MIBA size fractions. Additionally, the greater surface area exposure in granular form compared to monolithic form facilitates increased leachability.

In genral we can say that all monolithic samples, including those with MIBA substitution, showed Molybdenum (Mo) leaching well below the 0.6 mg/kg threshold set by Snellings, indicating low environmental risk. While Coal Fly Ash monoliths leached slightly less Mo than MIBA samples, especially early on, the differences were minimal, suggesting similar environmental performance across all monolithic materials. When we consider Granular samples, although MIBA-substituted granular samples currently fall within acceptable Mo leaching limits, the observed trends—particularly in finer fractions—raise concerns about their long-term environmental impact. This is especially relevant when considering the scenarios like demolish and cracks, where prolonged exposure could lead to increased Mo release. These findings highlight the need for thorough assessment of long-term leaching behavior before promoting the widespread use of MIBA in construction applications.

4.3.3.3 Leaching of Cadmium

The cumulative leaching of Cadmium (Cd) (mg/Kg of sample) from monolith and granular samples is plotted against the sampling interval is shown in te figure 4.10 below.

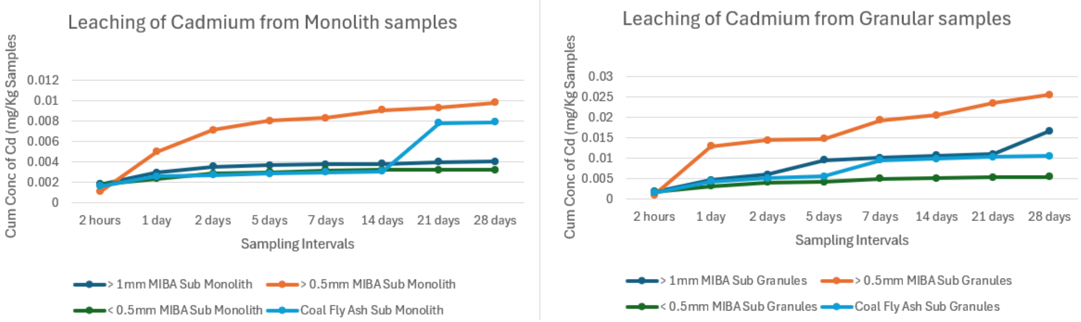


Figure 4.10: Cumulative leaching Curves of Cadmium from Monolith (left) and Granular (right) Samples

- **Monolith samples**
  Upon analyzing the graph, it is evident that all monolith samples—including those substituted with Coal Fly Ash—exhibit Cadmium (Cd) leaching levels well below the threshold of 0.05 mg/kg as proposed by Snelling's and also according to Swedish standards that is 0.2 mg/kg.

  Among the monolithic samples, the >0.5 mm MIBA-substituted concrete shows the highest Cd leaching after 28 days.As shown in the elemental data in Table 4.1, the cadmium (Cd) content is lower(5mg/Kg Ds) in the >0.5 mm MIBA fraction. However, this fraction exhibits a higher leaching rate, which can be explained by the findings of Kosson et al[22]. They noted that concrete incorporating such fractions may have larger voids or reduced packing density, leading to increased water ingress and enhanced diffusion pathways for leachates. This structural porosity can significantly contribute to the elevated Cd leaching, despite the lower total Cd content in the material.

  When compared with the Coal Fly Ash-substituted monolith, MIBA samples demonstrate comparable leaching behavior in the >0.5 mm and <0.5 mm size fractions. However, a notable spike in Cd leaching is observed in the Coal Fly Ash monolith at 21 days, suggesting a delayed release pattern of Cd from the reference material. Cadmium (Cd) may initially be retained through adsorption onto particle surfaces or encapsulated within secondary mineral phases such as ettringite and calcite. However, over time, as these minerals undergo transformation or dissolution during extended leaching periods, the previously immobilized Cd is gradually released into the leachate [19].

- **Granular samples**
  As in case of monolith samples the leaching of cd is less than the threshold according to snelling's as well as Swedish standards. According to Lamber's the Category 1 threshold for cadmium (Cd) leaching is 0.026 mg/kg, while the Category 2 threshold is 0.061 mg/kg[13]. Among the granular samples, the >0.5 mm MIBA-substituted fraction exhibits the highest Cd leaching at 28 days, with a value of 0.0255 mg/kg. This places it just within the limits of Category 1 (Critical element). All other granular samples, including those containing Coal Fly Ash, also fall within Category 1, with lower leaching values.

  This indicates that while the > 0.5 mm MIBA fraction is still within the acceptable range, it is approaching the upper limit of Category 1, suggesting potential restrictions in certain applications. Importantly, although this fraction showed low leaching in monolithic form, its transformation into a granular state—such as during demolition or disposal—could increase its environmental risk.

The > 0.5 mm MIBA granular samples exhibit the highest rate of cadmium leaching compared to all other samples. In contrast, the Coal Fly Ash-substituted granular samples show a moderate leaching rate, which is slightly higher than that of the < 0.5 mm MIBA granular samples. This suggests that the Coal Fly Ash granules contain relatively less mobile cadmium, indicating better immobilization of Cd in granular form compared to MIBA-substituted materials.

Overall, the results underscore that while MIBA-substituted concrete is environmentally safe in monolithic form especially < 0.5mm MIBA samples, granular samples may increase leaching risks, particularly for coarser MIBA fractions.

4.3.3.4 Leaching of Copper

The cumulative leaching of Copper (Cu) (mg/Kg of sample) from monolith and granular samples is plotted against the sampling interval is shown in te figure 4.11 below.

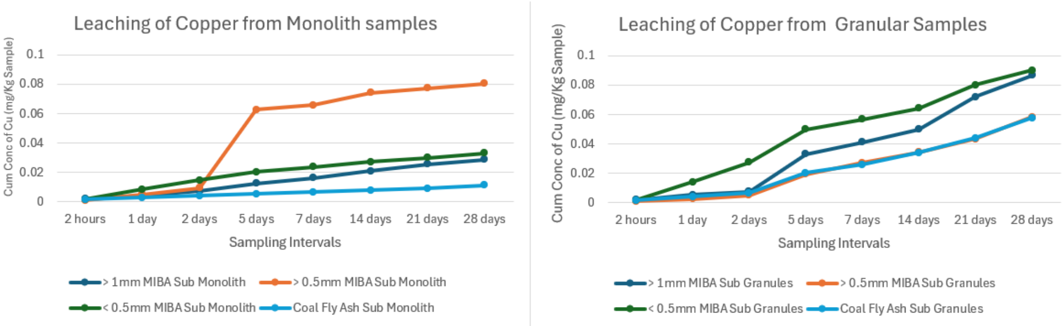


Figure 4.11: Cumulative leaching Curves of Copper from Monolith (left) and Granular (right) Samples

- **Monolith samples**
  When considering the Swedish standards, all samples fall below the Copper threshold of 40 mg/kg. Additionally, all monolithic samples in the graph show cumulative copper leaching well below the 3 mg/kg threshold established by Snelling. This indicates a minimal environmental risk associated with their use, supporting their safe application in construction and related fields.

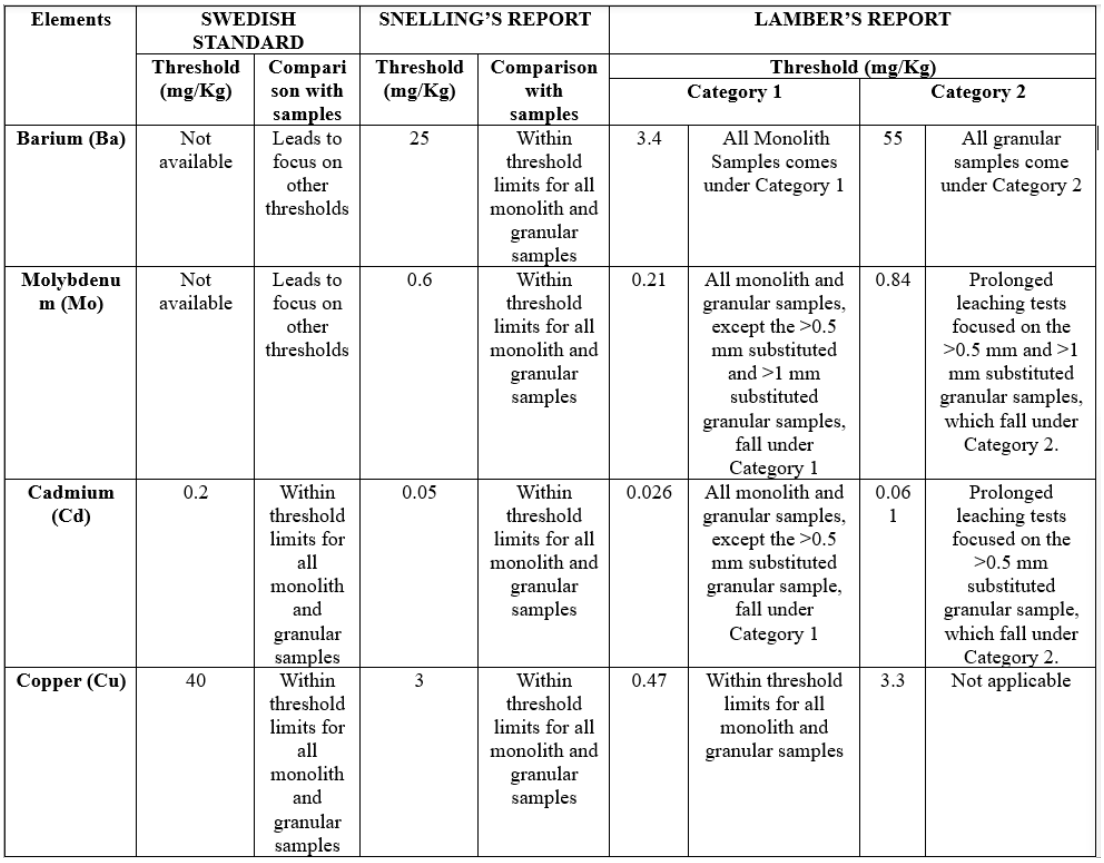
  From the above graph, it is evident that the > 0.5 mm MIBA-substituted monolith exhibits the highest copper (Cu) leaching over time, with a sharp increase observed between day 2 and day 5 . This sudden increase is likely due to the destabilization of mineral phases,such as hydrotalcite or amorphous aluminosilicates-where Cu may initially be immobilized. As leaching progresses, changes in pH and ionic strength within the pore solution can lead to the degradation of these stabilizing phases, resulting in a rapid release of Cu[39]. Moreover, pH measurements during each interval indicate that the environment remains within an alkaline range, a condition known to favor the mobilization of Cu, further contributing to the increased leaching during this period[39]. In contrast, the <0.5 mm MIBA and >1 mm MIBA monoliths show a more gradual and steady leaching trend, suggesting a more controlled release of copper throughout the testing period.

The Coal Fly Ash-substituted monolith exhibits the lowest copper (Cu) leaching among all samples, indicating more effective immobilization of Cu in the reference material compared to the MIBA-substituted samples.

- **Granular Samples**
  According to Swedish standards as well as Snelling's threshold the leaching rate is lower for Cu in granular form. When Comparing with Lamers, the threshold for Category 1 copper (Cu) leaching rate is 0.47 mg/kg, and 3.3 mg/kg for Category 2. All granular samples exhibit Cu leaching rates below the Category 1 threshold, indicating that their use poses no environmental restrictions. This suggests that, even in granular form, the material is likely to pose minimal environmental risk.

  The <0.5 mm MIBA granular sample exhibits the highest copper (Cu) leaching among all granular samples. The >0.5 mm MIBA and Coal Fly Ash granular samples show moderate leaching levels. When comparing all samples to the Coal Fly Ash reference, it consistently demonstrates a lower Cu leaching rate, indicating better Cu immobilization in the fly ash-based material.

Overall, copper (Cu) leaching is notably lower in monolith samples compared to granular forms. Nevertheless, all samples—regardless of whether they are monolithic or granular—exhibit Cu leaching levels that remain within the acceptable threshold limits. This suggests minimal environmental risk both during the service life and after disposal of the materials.



Elements	SWEDISH STANDARD		SNELLING'S REPORT		LAMBER'S REPORT			
	Threshold (mg/Kg)	Comparison with samples	Threshold (mg/Kg)	Comparison with samples	Threshold (mg/Kg)			
					Category 1		Category 2	
Barium (Ba)	Not available	Leads to focus on other thresholds	25	Within threshold limits for all monolith and granular samples	3.4	All Monolith Samples comes under Category 1	55	All granular samples come under Category 2
Molybdenum (Mo)	Not available	Leads to focus on other thresholds	0.6	Within threshold limits for all monolith and granular samples	0.21	All monolith and granular samples, except the >0.5 mm substituted and >1 mm substituted granular samples, fall under Category 1	0.84	Prolonged leaching tests focused on the >0.5 mm and >1 mm substituted granular samples, which fall under Category 2.
Cadmium (Cd)	0.2	Within threshold limits for all monolith and granular samples	0.05	Within threshold limits for all monolith and granular samples	0.026	All monolith and granular samples, except the >0.5 mm substituted granular sample, fall under Category 1	0.061	Prolonged leaching tests focused on the >0.5 mm substituted granular sample, which fall under Category 2.
Copper (Cu)	40	Within threshold limits for all monolith and granular samples	3	Within threshold limits for all monolith and granular samples	0.47	Within threshold limits for all monolith and granular samples	3.3	Not applicable

Figure 4.12: Summarized table comparing leaching rates of different samples against various threshold values

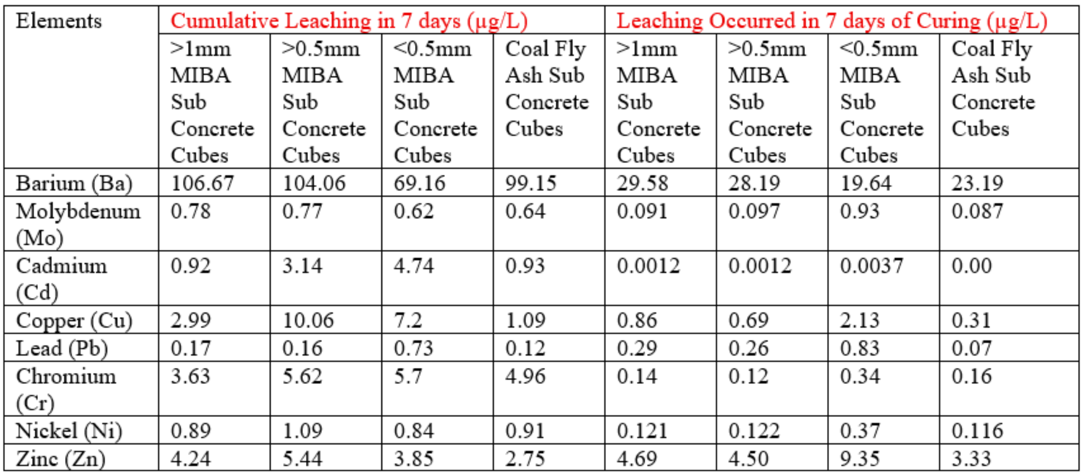
To provide a clearer understanding of the metal leaching behavior in relation to different threshold values, a summarized table is presented in the figure 4.12 above.

4.3.4 Comparison of Leaching Rates During Curing and Leaching Experiments

Initially, the samples were fully immersed in a water bath for 7 days, as this is a commonly adopted curing method known to provide optimal hydration conditions. Each set of four samples was placed in a separate container, resulting in a total of eight cubes per container. It was acknowledged that leaching can occur during this type of curing process. To quantify and assess this early-stage leaching, the curing water was collected and analyzed. These results were then compared with the cumulative metal leaching data obtained from the 7-day leaching experiment.

Table 4.12 presents a comparative table of leaching rates for selected elements after 7 days. The leaching data during the curing phase was obtained by analyzing the curing liquid (Milli-Q water) using ICP-MS. It is important to note that this data reflects the total leaching from eight concrete cubes placed in a single container. Since the exact volume of curing water was not measured, and to facilitate comparison, the total leaching values were divided by eight to estimate the leaching rate per individual cube.

In contrast, the cumulative leaching values from the dedicated leaching experiment represent the release from a single concrete cube into a controlled and consistent volume of water. Consequently, the values observed during the curing phase may appear higher—not necessarily due to greater leachability, but because they initially represent combined leaching from multiple samples. Therefore, any direct numerical comparison between the two datasets must consider the differences in sample quantity and water volume, which naturally influence the observed leaching values.



Elements	Cumulative Leaching in 7 days (μg/L)				Leaching Occurred in 7 days of Curing (μg/L)			
	>1mm MIBA Sub Concrete Cubes	>0.5mm MIBA Sub Concrete Cubes	<0.5mm MIBA Sub Concrete Cubes	Coal Fly Ash Sub Concrete Cubes	>1mm MIBA Sub Concrete Cubes	>0.5mm MIBA Sub Concrete Cubes	<0.5mm MIBA Sub Concrete Cubes	Coal Fly Ash Sub Concrete Cubes
Barium (Ba)	106.67	104.06	69.16	99.15	29.58	28.19	19.64	23.19
Molybdenum (Mo)	0.78	0.77	0.62	0.64	0.091	0.097	0.93	0.087
Cadmium (Cd)	0.92	3.14	4.74	0.93	0.0012	0.0012	0.0037	0.00
Copper (Cu)	2.99	10.06	7.2	1.09	0.86	0.69	2.13	0.31
Lead (Pb)	0.17	0.16	0.73	0.12	0.29	0.26	0.83	0.07
Chromium (Cr)	3.63	5.62	5.7	4.96	0.14	0.12	0.34	0.16
Nickel (Ni)	0.89	1.09	0.84	0.91	0.121	0.122	0.37	0.116
Zinc (Zn)	4.24	5.44	3.85	2.75	4.69	4.50	9.35	3.33

Figure 4.13: Cumulative leaching of Metals during curing and leaching experiment

Based on the table, we can observe that metals such as Barium (Ba), Copper (Cu), and Zinc (Zn) exhibit significantly higher leaching during the curing period, indicating a rapid initial release. In contrast, Cadmium (Cd) and Chromium (Cr) display higher concentrations in the leaching experiment, suggesting a delayed leaching pattern. Molybdenum (Mo) and Nickel (Ni) show relatively consistent leaching behavior across both periods.

Although the curing leachate concentrations are naturally higher due to the presence of eight cubes per container (as opposed to a single cube in the leaching test), the difference in magnitude between the two datasets provides valuable insight. It allows us to assess whether metals are predominantly released early during curing or leach out more gradually over time, offering a deeper understanding of the leaching dynamics—namely, immediate versus delayed release.

To mitigate the initial burst of leaching during the early curing phase, alternative curing methods could be considered. For instance, using sealed or dry curing (e.g., wrapping the specimens in plastic or curing them in a controlled humidity chamber) instead of immersion in water may reduce the early dissolution of mobile metal species[9]. This approach could help retain more of the metals within the concrete matrix during the critical hydration and setting stages.

Extended curing periods under such controlled conditions may also enhance the incorporation of metals into more stable hydration products, such as calcium silicate hydrates (C-S-H) or ettringite phases[31]. This would likely reduce their mobility and, therefore, their subsequent leaching potential. In other words, longer and more controlled curing could promote the stabilization of metals, resulting in decreased leaching from the cured concrete[38].

While some metals may still eventually leach out under certain environmental conditions, improving early curing practices could significantly reduce the extent of leaching, especially for those elements that are more mobile during the initial stages of hydration.

5 Conclusion

The MIBA-substituted samples demonstrated a notable improvement in compressive strength when separated into different size fractions. Specifically, the concrete substituted with the <0.5 mm MIBA fraction and 15% cement replacement achieved approximately 75% of the strength of the reference concrete which is not enough to consider it as a SCMs. The differences in strength among the samples can be explained by the elemental analysis and mineralogical PXRD analyses. Also these analysis gives the possibility of using MIBA fractions as fillers instead of SCM, which highlighted distinct characteristics across the three MIBA fractions.

To assess environmental performance, leaching tests were conducted on both monolithic and granular samples, representing two scenarios: current use and end-of-life conditions, respectively. These batch leaching tests confirmed that metals leach from both monolithic and granular forms, with significantly higher leaching observed in the granular samples. This is attributed to increased surface area and greater exposure to the leaching environment.

In monolithic samples, leaching levels for all measured metals remained within acceptable thresholds, indicating low environmental risk during use. However, in granular samples, Barium (Ba) leaching in particular exceeded the Category 1 threshold, placing the material in Category 2, which implies restricted handling due to higher environmental risk. Additionally, Molybdenum (Mo) leaching in both >0.5 mm and <0.5 mm MIBA-substituted granular samples suggests that, with extended leaching periods, these may also surpass Category 1 limits. Cadmium (Cd) leaching from the >0.5 mm fraction followed a similar pattern. These findings indicate that prolonged exposure could make Mo and Cd, in addition to Ba, critical elements influencing the safe use of MIBA in concrete at end-of-life stages. When considering the overall MIBA samples < 0.5mm can be considered to be better choice in concrete production.

When comparing leaching behavior during the initial 7-day curing period and the 28-day leaching experiment, elements like Barium (Ba), Zinc (Zn), and Copper (Cu) showed higher leaching during the curing phase. This suggests an early release of certain metals, offering insight into the immediate vs. delayed leaching dynamics. It is also important to note that the water used in the curing process contained leachate from eight cubes per container, whereas the leaching test was performed on individual monoliths—this difference influences the concentration values and must be considered when comparing data. The influence of pH and ionic strength during the tests also played a critical role in metal mobility and solubility, contributing to the observed variations.

Importantly, while MIBA-substituted samples exhibited slightly higher metal leaching compared to those containing Coal Fly Ash, the measured values generally remained below regulatory threshold limits and were comparable to those of Coal Fly Ash. This suggests that, despite having higher total elemental contents, MIBA can still serve as a viable alternative with acceptable environmental performance under both conditions as monolith and granular

6 Future Works

Further investigation into the use of MIBA as a filler in concrete is recommended. In particular, the effects of combining different particle size fractions—such as <0.5 mm and >1 mm—should be studied to evaluate their influence on concrete performance. Additionally, the leaching behavior of heavy metals from various substituted fractions warrants comprehensive examination. Given that pH is a key factor influencing metal leaching, it would be valuable to explore the relationship between pH variations and leaching rates. Future studies should include systematic pH measurements at different time intervals during the sampling process, coupled with concurrent analysis of metal release. This combined approach would provide a deeper understanding of the environmental impact and long-term behavior of MIBA-incorporated concrete.

7 Acknowledgement

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Appendix A

8.1 Drying

The initial weight of the wet sample along with the beaker was 1017g. After drying the sample for 24 hours at 105 °C, the weight was reduced to 966g. The percentage weight loss after 24 hours of drying was calculated as:

Weight of sample before drying = 300g

Weight of beaker = 717g

Weight of sample after 24 hours of drying = 966-717 = 249g

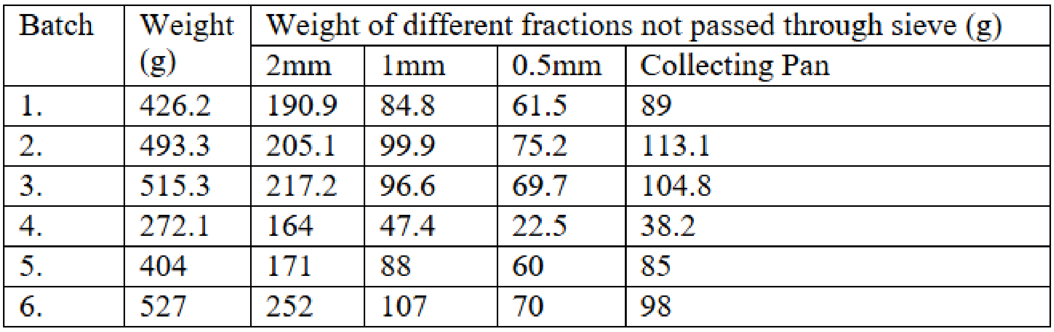
$$\text{Percentage weight loss} = \frac{300 - 249}{300} \times 100 = 17\% \tag{8.1}$$

The sample was then dried for an additional 24 hours (totaling 48 hours), but the weight remained unchanged at 966g. Since there was no further reduction in weight, it was concluded that 24 hours of drying at 105 °C is sufficient for complete drying of the sample.

8.2 Separation and Size Reduction

8.2.1 Size distribution

For size distrubution we have divided the whole sample into six batches of different weights. And sieved them with sievers of size 2mm, 1mm , 0.5mm and collecting pan. The size fractions obtained from the different batches are shown in the figure 6.1 below.



Batch	Weight (g)	Weight of different fractions not passed through sieve (g)			
		2mm	1mm	0.5mm	Collecting Pan
1.	426.2	190.9	84.8	61.5	89
2.	493.3	205.1	99.9	75.2	113.1
3.	515.3	217.2	96.6	69.7	104.8
4.	272.1	164	47.4	22.5	38.2
5.	404	171	88	60	85
6.	527	252	107	70	98

Figure 8.1: Size fractions in (grams) obtained from 6 batches

Batch 1

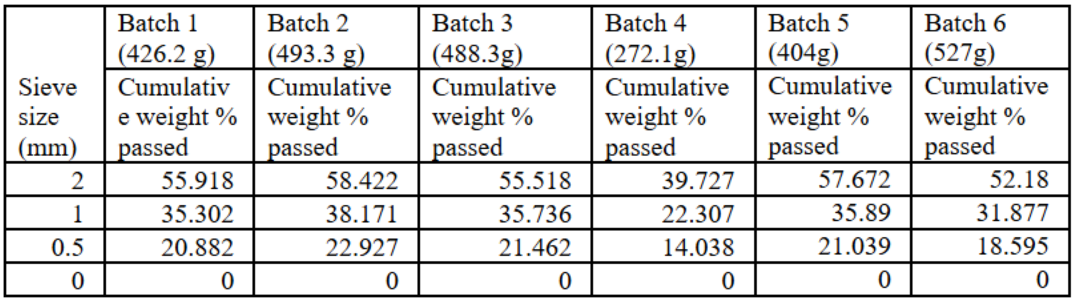
Weight of the batch = 426.2 g

Weight of sample retained in the sieve = 190.9g

$$\text{Weight Percentage of sample retained in 2mm sieve } = (9426.2 - 190.9)\times 100 = 44.791\% \tag{8.2}$$

$$\text{Weight Percentage of sample passed through the sieve of 2mm } = (100 - 44.791) = 55.918\% \tag{8.3}$$

Similarly, determine the weight percentage of each size fraction that passes through the sieve and calculate the cumulative weight percentage passed for each batch. The cumulative weight percentage passed for the different batches are presented in the figure 6.2 below.



Sieve size (mm)	Batch 1 (426.2 g)	Batch 2 (493.3 g)	Batch 3 (488.3g)	Batch 4 (272.1g)	Batch 5 (404g)	Batch 6 (527g)
	Cumulative weight % passed	Cumulative weight % passed	Cumulative weight % passed	Cumulative weight % passed	Cumulative weight % passed	Cumulative weight % passed
2	55.918	58.422	55.518	39.727	57.672	52.18
1	35.302	38.171	35.736	22.307	35.89	31.877
0.5	20.882	22.927	21.462	14.038	21.039	18.595
0	0	0	0	0	0	0

Figure 8.2: Cumulative Weight Percentage obtained from 6 batches

8.2.2 Sample with particles greater than 0.5 mm

Using a trial-and-error method, the milled particles were tested with sieves of various mesh sizes-20μm, 45μm, 75μm, and 125μm. It was observed that the particles only successfully passed through the 75μm mesh. However, agglomeration of the fine particles was noted during the sieving process, which led to partial blockage of the mesh and hindered the smooth passage of particles.

Weight of milled sample = 10g

Weight of sample passed through the sieve = 4.3g

$$\text{Percentage weight of particles passed} = \frac{10-5.7}{10} \times 100 = 43\% \tag{8.4}$$

8.2.3 Sample with particles less than 0.5 mm

8.2.3.1 Trial 1

20g 31 balls for 10 minutes and 500 rpm

The milled sample is passed through the 75μm siever

Total amount of sample = 18g

particles passed through the siever = 11g

particles remained the siever = 7g

$$\text{Percentage weight of particles passed} = \frac{18-7}{18} \times 100 = 61.12\% \tag{8.5}$$

8.2.3.2 Trial 2

20g 46 balls for 10 minutes and 500 rpm

The milled sample is passed through the 75μm siever

Total amount of sample = 18g

particles passed through the siever = 12g

particles remained the siever = 6g

$$\text{Percentage weight of particles passed } = \frac{18-6}{18} \times 100 = 66\% \tag{8.6}$$

8.2.3.3 Trial 3

20g 31 balls for 10 minutes and 250 rpm

The milled sample is passed through the 75μm siever

Total amount of sample = 20g

particles passed through the siever = 19g

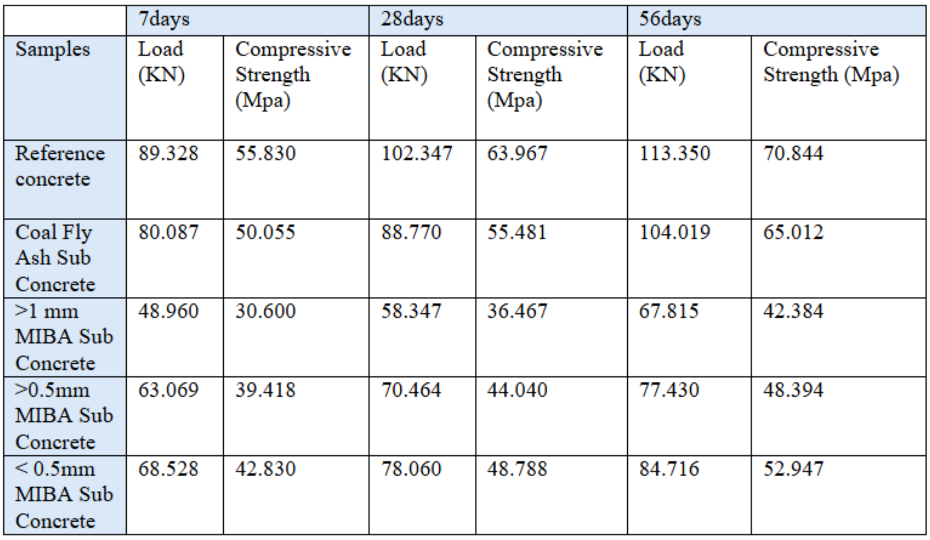
particles remained the siever = 1g

$$\text{Percentage weight of particles passed } = \frac{20-1}{20} \times 100 = 95\% \tag{8.7}$$

Appendix B

8.3 Compressive Strength Test

The compressive strength and corresponding loads on each testing days are mentioned in the figure 5.3 below



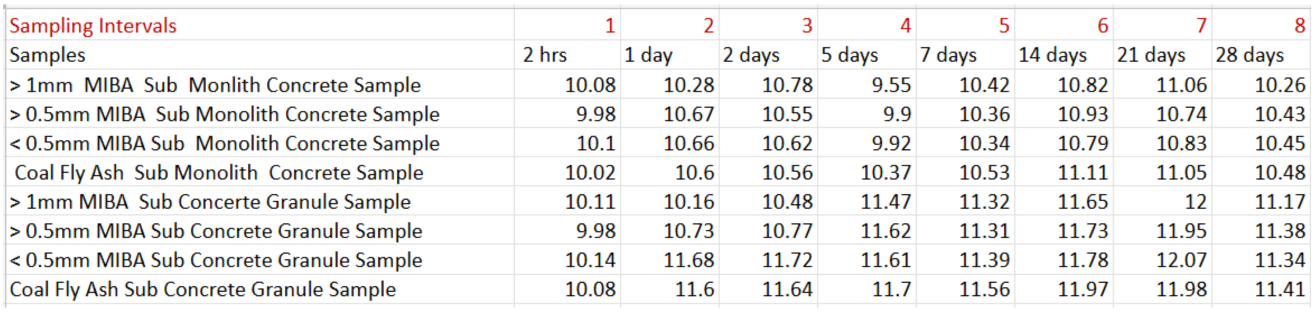
	7days		28days		56days	
Samples	Load (KN)	Compressive Strength (Mpa)	Load (KN)	Compressive Strength (Mpa)	Load (KN)	Compressive Strength (Mpa)
Reference concrete	89.328	55.830	102.347	63.967	113.350	70.844
Coal Fly Ash Sub Concrete	80.087	50.055	88.770	55.481	104.019	65.012
>1 mm MIBA Sub Concrete	48.960	30.600	58.347	36.467	67.815	42.384
>0.5mm MIBA Sub Concrete	63.069	39.418	70.464	44.040	77.430	48.394
< 0.5mm MIBA Sub Concrete	68.528	42.830	78.060	48.788	84.716	52.947

Figure 8.3: Compressive Strength and Load of samples

8.4 Leaching Test

8.4.1 pH of eluate from monolith and granular samples

The obsevation table for the pH measured at a specific interval of sampling time is given in Figure 5.4 below.

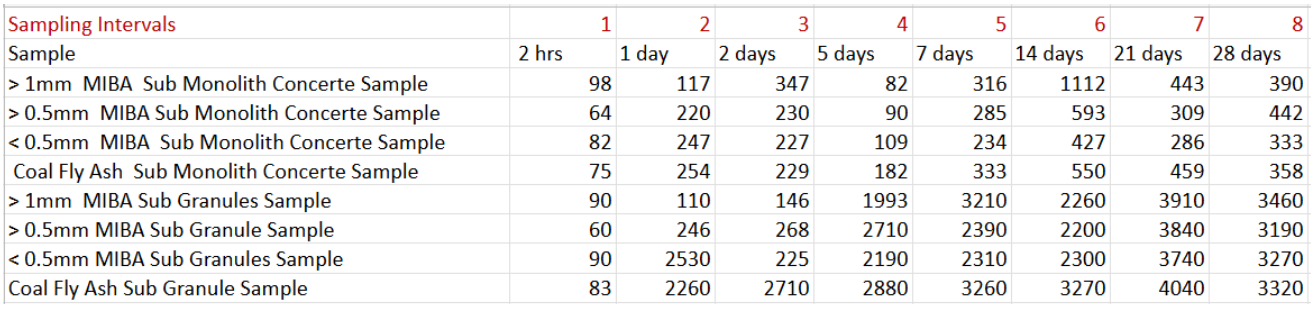


Sampling Intervals	1	2	3	4	5	6	7	8
Samples	2 hrs	1 day	2 days	5 days	7 days	14 days	21 days	28 days
> 1mm MIBA Sub Monlith Concrete Sample	10.08	10.28	10.78	9.55	10.42	10.82	11.06	10.26
> 0.5mm MIBA Sub Monolith Concrete Sample	9.98	10.67	10.55	9.9	10.36	10.93	10.74	10.43
< 0.5mm MIBA Sub Monolith Concrete Sample	10.1	10.66	10.62	9.92	10.34	10.79	10.83	10.45
Coal Fly Ash Sub Monolith Concrete Sample	10.02	10.6	10.56	10.37	10.53	11.11	11.05	10.48
> 1mm MIBA Sub Concerte Granule Sample	10.11	10.16	10.48	11.47	11.32	11.65	12	11.17
> 0.5mm MIBA Sub Concrete Granule Sample	9.98	10.73	10.77	11.62	11.31	11.73	11.95	11.38
< 0.5mm MIBA Sub Concrete Granule Sample	10.14	11.68	11.72	11.61	11.39	11.78	12.07	11.34
Coal Fly Ash Sub Concrete Granule Sample	10.08	11.6	11.64	11.7	11.56	11.97	11.98	11.41

Figure 8.4: pH of the samples at regular sampling time

8.4.2 Conductivity of eluate from monolith and granular samples

The obsevation table for the Conductivity measured at a specific interval of sampling time is given below in Fig. 5.4 below.



Sampling Intervals	1	2	3	4	5	6	7	8
Sample	2 hrs	1 day	2 days	5 days	7 days	14 days	21 days	28 days
> 1mm MIBA Sub Monolith Concerte Sample	98	117	347	82	316	1112	443	390
> 0.5mm MIBA Sub Monolith Concerte Sample	64	220	230	90	285	593	309	442
< 0.5mm MIBA Sub Monolith Concerte Sample	82	247	227	109	234	427	286	333
Coal Fly Ash Sub Monolith Concerte Sample	75	254	229	182	333	550	459	358
> 1mm MIBA Sub Granules Sample	90	110	146	1993	3210	2260	3910	3460
> 0.5mm MIBA Sub Granule Sample	60	246	268	2710	2390	2200	3840	3190
< 0.5mm MIBA Sub Granules Sample	90	2530	225	2190	2310	2300	3740	3270
Coal Fly Ash Sub Granule Sample	83	2260	2710	2880	3260	3270	4040	3320

Figure 8.5: Conductivity (in μS)of the samples at regular sampling time

Appendix C

8.5 Leaching Analysis

The following metals are with the threshold of corresponding threshold by Snellings for monolith samples and comes under Category 1 for granular samples according to lambert Which means these metasl will not cause any envirinmental risk will using it as cubes as well ass end life scenario as Granuless.When compared with Coal Fly Ash substituted samples(Both monlith and Granules) the leaching of these metals are compartively lower than that of MIBA substituted Samples.

8.5.1 Leaching of Zinc

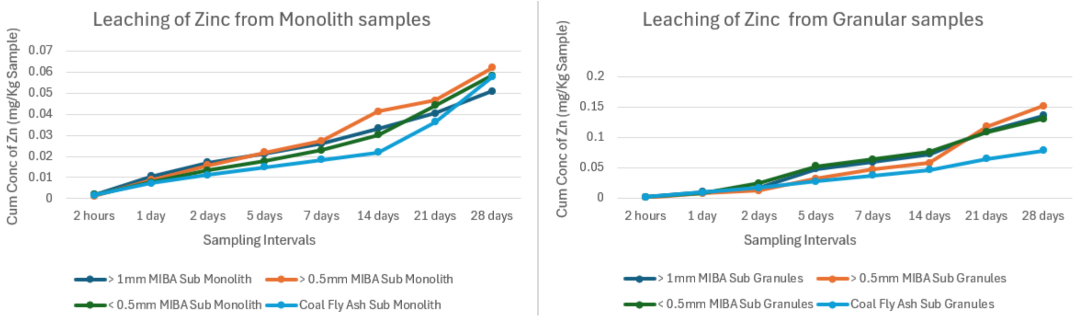


Figure 8.6: Cumulative leaching Curves Of Zinc from Monolith (left) and Granular (right) Samples

From the cumulative leaching graph 6.6 given above, for zinc both monolith and granular samples over a 28-day period indicates a gradual increase in cumulative concentration across all sample types. In the monolith samples, zinc leaching is relatively low, with the > 0.5 mm MIBA sub-monolith showing the highest leaching. In contrast, the granular samples show higher zinc leaching overall, particularly for the > 0.5 mm MIBA sub-granules by day 28.

8.5.2 Leaching of Chromium

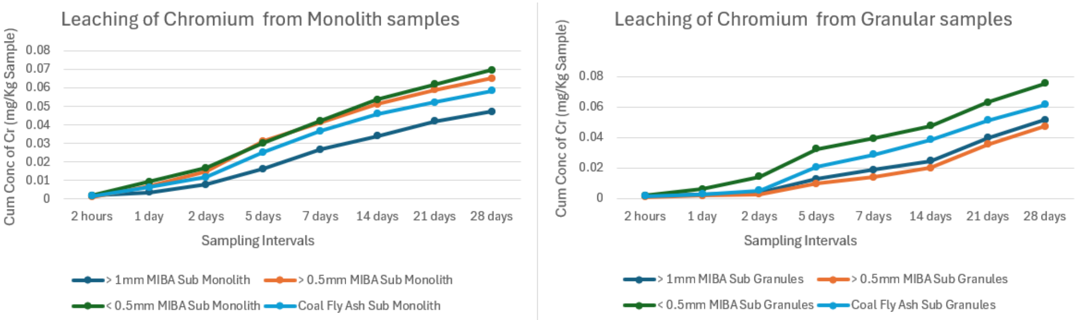


Figure 8.7: Cumulative leaching Curves Of Chromium from Monolith (left) and Granular (right) Samples

The chromium leaching trends from both monolith and granular samples show a steady increase over time, with granular samples generally exhibiting higher leaching levels than their monolithic counterparts. In monlith samples < 0.5 mm MIBA sub-monolith showing the highest release. In granular form, the <0.5 mm MIBA sub-granules display the highest chromium leaching, by day 28.

8.5.3 Leaching of Lead

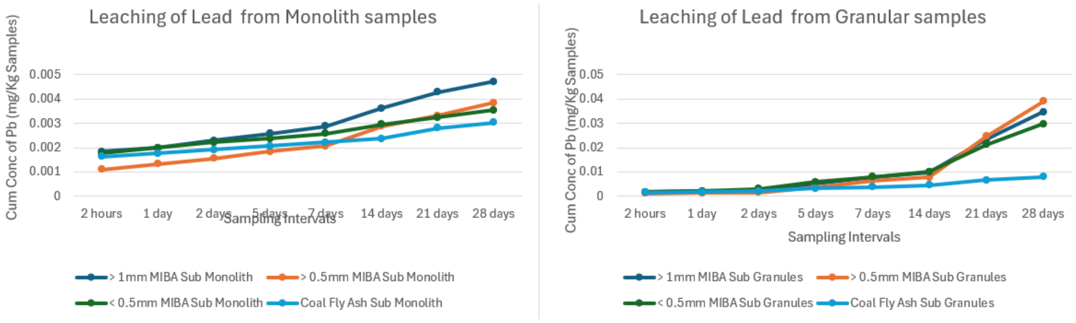


Figure 8.8: Cumulative leaching Curves Of Lead from Monolith (left) and Granular (right) Samples

Lead (Pb) leaching from both monolith and granular samples remains low throughout the testing period. In monolith samples with > 1 mm MIBA sub-monolith showing the highest levels. Granular samples, on the other hand, display a more noticeable rise after 14 days, especially the > 0.5 mm MIBA sub-granules after 28 days.

8.5.4 Leaching of Nickel

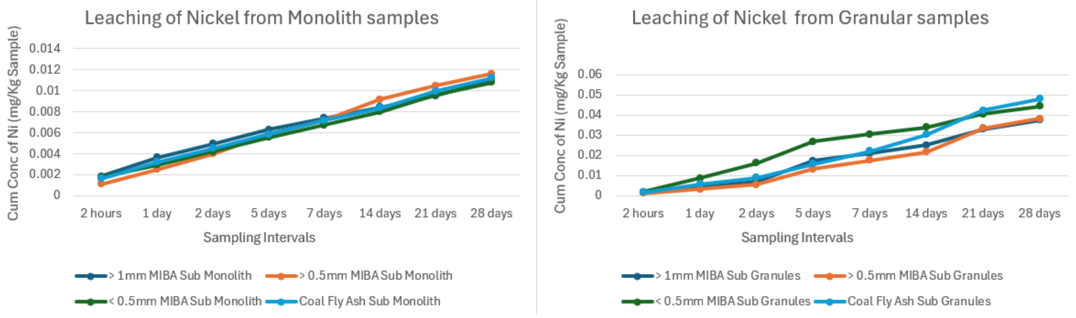


Figure 8.9: Cumulative leaching Curves Of Nickel from Monolith (left) and Granular (right) Samples

From the above graph 6.9 it is evident that for lead (Pb)- leaching from both monolith and granular samples remains low throughout the testing period. In monolith samples, concentrations stay under 0.005 mg/kg, with the >1 mm MIBA sub-monolith showing the highest levels. Granular samples, on the other hand, display a more noticeable rise after 14 days, especially the >0.5 mm MIBA sub-granules, which approach 0.04 mg/kg by day 28.

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