

**INFLUENCE OF TREATMENT METHODS ON THE
PERFORMANCE OF EXCAVATION SOIL AS FINE
AGGREGATE IN MORTAR**

A THESIS

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P. PRIYADHARSHINI

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INDIAN INSTITUTE OF TECHNOLOGY MADRAS

CHENNAI 600 036

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THESIS CERTIFICATE

This is to certify that the thesis entitled “**INFLUENCE OF TREATMENT METHODS ON THE PERFORMANCE OF EXCAVATION SOIL AS FINE AGGREGATE IN MORTAR**”, submitted by **P. Priyadharshini** to the Indian Institute of Technology Madras, for the award of the degree of **Doctor of Philosophy** is a bonafide record of research work carried out by her under our supervision. The contents of this thesis, in full or in parts, have not been submitted to any other Institute or University for the award of any degree or diploma.

Chennai 600 036

1. **Prof. K. Ramamurthy**

Date:

2. **Prof. R. G. Robinson**

(Research Guides)

Department of Civil Engineering

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ABSTRACT

KEYWORDS: *Excavation soil, fine aggregate, clay, montmorillonite, geopolymer, stabilization, lime, GGBS, cement mortar, wet sieving, residual clay, wash water, heat treatment, dry density, compressive strength, water absorption, drying shrinkage.*

Based on origin of the material, alternative fine aggregates can be classified into three broad categories viz., natural origin materials, recycled materials and industrial by-products. Crushed stone is the widely accepted alternative that lies in the category of naturally originated material. The availability of other sources of natural aggregates such as dune sand, offshore soil, marine sediments are location specific. In this category of materials, excavation soil which is composed of rock, sand and clay from earth works like underground constructions, tunnelling, metro developments and mine spoils is available all over the world in huge volume (Magnusson et al. 2015). Considering the alternative materials from recycling of concrete/ brick or the by-products of industries such as foundry sand/ bottom ash, it could be noted that each of these materials have their own disadvantage such as the presence of deleterious materials like chlorides, clay, carbon, fines, etc., (Dias et al. 2008; Monosi et al. 2013). The particle size distribution of the material could also affect the properties of mortar and concrete made of these alternatives (Katz et al. 2006). These result in need for treatment or processing of most of the available alternatives before it can be confidently used as fine aggregate in concrete.

Excavated soils are globally available material which has not been well explored for its use as fine aggregate in concrete. Raw earth or soil is widely used in various applications based on the quality of the material. It is mainly applied in backfilling, if the soil does not contain any expansive clay. Rammed earth, stabilized blocks and adobe are made of raw earth; however, poor resistant to moisture restricts their usage (Pacheco-Torgal and Jalali 2012). Calcined soil with high clay content are used in alkali activated materials which uses Al-Si as geopolymeric precursors (Li et al. 2016). However, presence of fines and clay is an important draw back in considering materials like excavation soil as a fine aggregate in cement mortar applications. Hence, a systematic study of different treatment methods that can

be employed on excavation soil is essential. Such an effort can help in providing value addition to excavation soils, and thereby serve as a potential alternative to river sand.

Excavation soils cannot be directly used in cement mortar which causes excessive shrinkage and poor strength due to the presence of excessive fines (with clay). However, the unprocessed excavation soils can be used as fine aggregates in geopolymer mortar, as alkali activation of reactive Si-Al in the clay particles helps in the formation of alumina-silicate inorganic polymers. Hence, as an initial step the excavation soils without any treatment were used as fine aggregate in fly ash based geopolymer mortar. Three types of plastic soils with varying plasticity and mineralogy have been chosen. The behaviour in geopolymer mortar has been studied for the effects of type of soil, fly ash to fine aggregate (F/A) ratio, curing temperature and molarity of NaOH. The interaction effect of these parameters with four different fine aggregates (river sand, low, medium and high plastic soils) were identified and discussed. Their fresh and hardened properties have been compared with conventional geopolymer mortar made with river sand as filler. Mortar with clayey fine aggregates helps to achieve better properties at a lower dry density range. It was observed that all the three types of soil could be used as a fine aggregate material in the geopolymer mortar.

Using excavated soil in geopolymer mortar as fine aggregate may be the simplest application for the direct use of untreated excavated soil. However, geopolymer is not widely used and hence, as a next step the performance of treated excavation soil in cement mortar was evaluated. Initially, dry sieving has been done using 600 μm size sieve to remove clay and fines. Even after dry sieving, particles $< 75 \mu\text{m}$ (silt + clay) were still present in the soil. Hence, stabilization which is a common treatment method used for improving the poor quality soil in geotechnical engineering applications, was employed. Stabilization using lime (2 – 10%) and GGBS (5 – 20%) were tried as treatment methods on unprocessed and sieved soil samples, for observing its effectiveness in reducing the plastic characteristics of soil. The treated soils were then used as fine aggregate in cement mortar. The mortar properties such as dry density, compressive strength, water absorption, and shrinkage strain were compared with control mortar made of river sand. Though there was improvement in mortar properties, drying shrinkage could not reach the value of river sand mortar owing to the presence of superfluous fines content and reaction products in mortar with dry sieved and stabilized high plastic soil.

To further improve the properties of cement mortar, clay and fines were completely eliminated from excavation soil using wet sieving method. Wet sieving of excavation soil resulted in three products namely, sand ($>75\text{ }\mu\text{m}$), wash water and fines (silt and clay sized particles) with particle size less than $75\text{ }\mu\text{m}$. Low (LP) and medium plastic (MP) soil were used in the wet sieving process. High plastic soil with more than 50% of particles finer than $75\text{ }\mu\text{m}$ including 41% of clay size (mostly swelling type which clogs the sieve), made it difficult to treat in wet sieving method. The wet sieved sand ($>75\text{ }\mu\text{m}$) retained in $75\text{ }\mu\text{m}$ sieve was used as fine aggregate in mortar preparation. With wet sieving process, properties of mortar with excavation soil could reach a value close to that of mortar with river sand. Properties of mortar with wet sieved sand were highly influenced by the particle size distribution of the excavation soil and vary with the source. This could be adjusted by simple granulometric corrections to the wet sieved sand. The residual slurry (referred as residual clay) with fines and clay was studied for its applicability as pozzolanic material in cement mortar. Wash water was analysed for the presence of ions and used as mix water in mortar production.

Though wet sieving method was effective in LP and MP soils, it was not suitable for soil with high clay content and results in removal of most of the material as residual clay. Hence, thermal treatment was adopted to study its effectiveness on medium and high plastic soil. The influence of temperature (200 to 1000°C) and duration (30 mins to 180 mins) of thermal treatment of soil on cement mortar properties were studied. Treatment temperature was identified as the major influential parameter affecting the structural modification of clay sized particles in soil. The removal of adsorbed water around the clay particles leads to particle growth (Yilmaz, 2003) which resulted in reduction of clay content. Sintering of clay particles and growth in particle size helps in shrinkage reduction of mortars with expansive clay. Plastic shrinkage and durability studies are required for fine-tuning the treatment method that is most appropriate for a given soil.

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ABBREVIATIONS

LP	Low plastic
MP	Medium plastic
HP	High plastic
F/A	Fly ash to fine aggregate ratio
A/C	Fine aggregate to cement ratio
SAS	Statistical Analysis Software
ANOVA	Analysis of Variance
XRD	X-ray diffraction
SEM	Scanning Electron Microscope
MIP	Mercury Intrusion Porosimetry

NOTATIONS

NaOH	sodium hydroxide
Na ₂ SiO ₃	sodium silicate
Ca (OH) ₂	calciumhydroxide
γ	surface tension of mercury
θ	cosine angle
P	pressure
l_1	first reading of length
l_2	final reading of length at different time interval
L _d	actual initial length of the specimen

CHAPTER 1

INTRODUCTION

1.1 GENERAL

Concrete is the second most used material by mankind, just next to water. As aggregates occupy a major portion of it, depletion of river sand all over the world has made a great impact on the construction industry. Aggregate is said to be the largest volume of solid material extracted globally (UNEP, 2014). Depletion of natural resources caused the extinction of good quality raw materials, posing problems to the upcoming infrastructural developments (Kou and Poon, 2009; He et al. 2012; Suresh et al. 2011). Indirect estimation of aggregate consumption can be made through cement production which is 4.2 billion tonnes in 2016 (USGS, 2017). It means that almost 25.2 to 29.4 billion tonnes could be the aggregate consumption in 2016. In addition to this, aggregates are also used in applications like road embankments, asphalt pavements and other industries. All together this estimate could exceed 40 billion tonnes per year which is more than twice the sediments carried by world rivers every year (UNEP, 2014; Milliman and Syvitski, 1992). Due to the increase in cost of raw materials and the continuous depletion of natural resources, use of waste materials became a potential alternative. In 2016, Ministry of Environment, Forest and Climate change of India released the ‘Sustainable Sand Mining Management Guidelines’. This guideline stresses on controlled mining of river sand and use of alternatives such as fly ash, crushed stone, slag and so on. Added to the problem of river sand depletion, the transportation of aggregate from source to the site location increases the cost of the projects. These highlight the need for identification of alternative materials that can be a potential replacement for aggregates in construction.

1.2 ALTERNATIVES TO RIVER SAND

Based on the origin, the materials which have been tried as alternative fine aggregate in mortar and concrete can be classified into three broad categories as illustrated in Figure 1.1. It can be a material of natural origin or recycled materials that are otherwise dumped as waste or an industrial by-product. Natural material that is formed naturally without manual processing is available all over the world. While finding alternative source of construction materials is an immense problem in current scenario, other being difficulty in locating places for disposing of waste/demolished materials. Accumulation of waste has become a prime

problem and some of such wastes have been already studied for its applicability as fine aggregate in concrete. Due to industrialisation, natural resources are getting depleted and industrial by-products are getting accumulated as waste. Use of industrial by-products in building materials could be a sustainable solution for waste disposal and to conserve our environment. A detailed review of each of these materials is presented in Chapter 2.

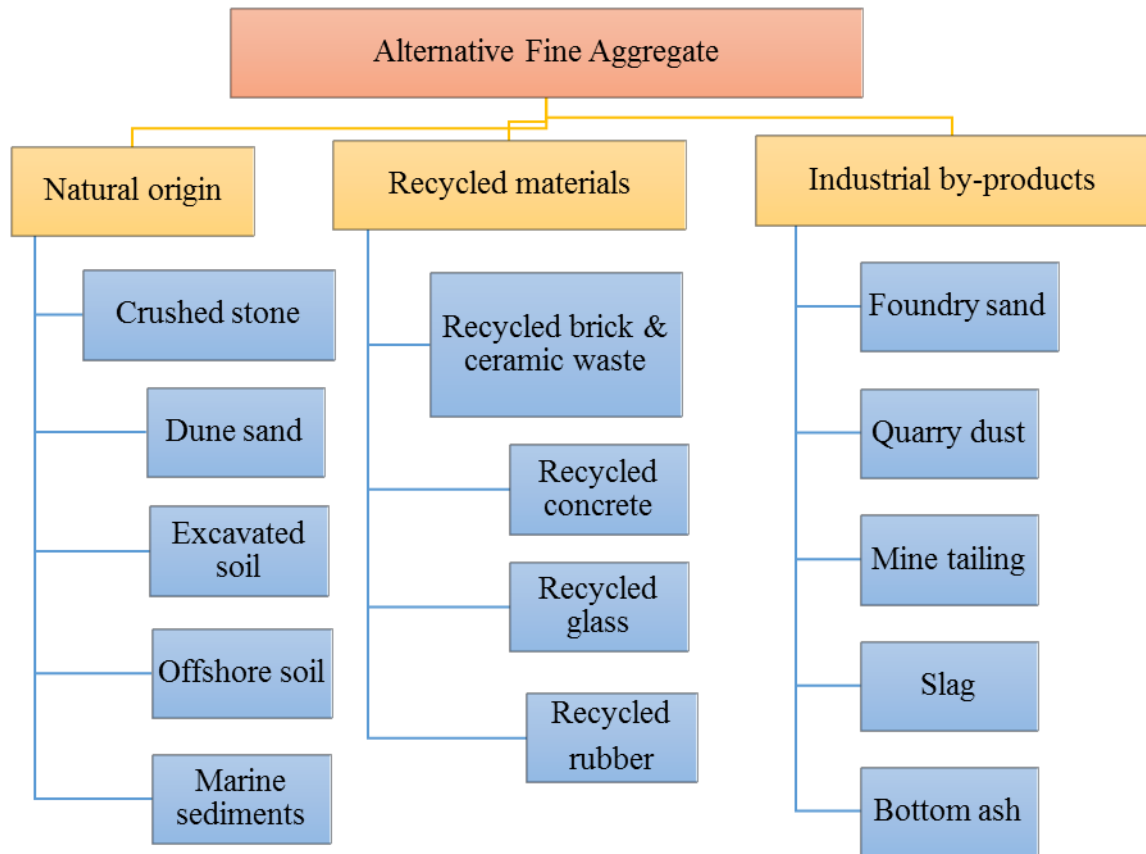


Figure 1.1 Classification of alternative fine aggregates based on their origin

Main disadvantage of most of the alternative fine aggregate materials is the presence of excessive fines or deleterious materials. Though concrete produced with alternative fine aggregates could match with the properties of concrete with river sand, it is done mostly in partial replacement. However, the main aim of this study is to utilize maximum of the other natural resources, recycled materials and by-products to reduce the river sand depletion. Hence, there should be treatment methods to enhance the properties of these alternatives to increase their replacement level in concrete.

1.3 EXCAVATION SOIL: A POTENTIAL FINE AGGREGATE ALTERNATIVE

Excavated soils are local materials which have not been well explored for its use as fine aggregate alternative. Soils from earth works like underground constructions, tunnelling,

metro developments and mine spoils fall under this category. Excavation soil is composed mostly of rock, sand, and clay, and are generally dumped in vacant land or used for refilling in construction sites, based on its quality. When dumped, they are not usually compacted, thereby, posing problems of sliding over the nearby areas. Proper data are not available on excavated soil generation and disposal. Transportation and disposal tax of such soil also adds to the cost of the project. Many developed countries impose tax for the disposal of soil wastes generated from excavation works (Magnusson et al. 2015). Raw earth or soil is widely used in various applications based on the quality of the material. It is mainly used in backfilling, if the soil does not contain any expansive clay. Rammed earth, stabilized blocks and adobe are made of raw earth; however, poor resistant to moisture restricts their usage (Pacheco-Torgal and Jalali 2012). Plastering with soil as fine aggregate is possible by maintaining the proportion of soil to lime as minimum as possible and the application is restricted to interior walls and needs proper maintenance plan (Stazi et al. 2016). Calcined soil with high clay content is used in alkali activated materials which uses Al-Si as geopolymeric precursors (Li et al. 2016). However, the presence of fines and clay is an important draw back in considering materials like excavation soil as a fine aggregate in mortar/ concrete. Hence, a systematic study is required on treatment methods of excavation soil which helps in transforming the soil in to a potential alternative to river sand.

1.3.1 Environmental Problem

More than economical issue, dumping of excavation soil should also be looked-up as a matter of environmental concern. Mine soil or overburden is considered to be the major source of excavation soil which is the material that lies above the area of economic or scientific interest of mining. TERI (2001) indicates that, huge volume of overburden is produced as most of the mineral are extracted from open cast mines (TERI, 2001). In India, every one million ton of coal extracted by surface mining method damages a surface area of 4 hectares (Ghosh, 1996), which results in huge volume of mine spoil being dumped near the mining area. This gives a clear picture of the intensity of the problem in disposing excavation soils from different sources. Some of the negative effects of mines overburden include environmental pollution, loss of biodiversity, and problems to the nearby occupants. Similar effects can be expected in various sources of excavation soil that affect the environmental equilibrium.

1.4 BARRIERS IN USING EXCAVATION SOIL

Presence of impurities in the form of fines and clay in excavated soils is the major problem. It causes deleterious effects when used as fine aggregate in concrete. There is also possibility for the presence of other impurities like chlorides and sulphates based on the source of excavation. Initial investment cost to setup treatment plants for this soil and additional maintenance cost would also be a barrier for the establishment. The undesirable components that are separated from the excavation soil require disposal or alternative use.

1.4.1 Fines and Clay in Soil

Particles which are less than 75 μ m come under the category of fines including clay. Fines are relatively non-reactive and affect the particle packing when the optimum level for a particular concrete mix is exceeded (Katz and Baum 2006). Clay is a very small (<2 μ m) layered crystalline colloidal particle formed by weathering of certain rocks, mainly formed of two fundamental layers of silica (tetrahedral) and alumina (octahedral). The behavior of the major clay minerals such as kaolinite, illite and montmorillonite vary based on the arrangement of the silica and alumina layer, bonding and the free cations in their crystal lattice. Clay surface usually has negative charge. When placed in water, the dipolar water molecules get attracted to the charged clay surfaces, forming diffuse double layer (Mitchell and Kenichi, 2005). Free cations and interlayer space decide the swelling behavior of clay minerals. Free cations attract the dipolar water molecules to balance the charge. However, with reduced interlayer space, the attraction between clay layers will be powerful, not allowing the entry of water molecules. Swelling does not occur in this case. For instance, Kaolinite shows lower ion exchange capacity due to its charge balanced structure and hydrogen bonding, whereas, montmorillonite layers joined by Van der Waals force with charge imbalance impart high cation exchange capacity.

1.4.2 Effect of Fines and Clay on Concrete Properties

Presence of clay can have notable effect on the fresh and hardened properties of concrete. The magnitude of problem depends on the type of minerology (Li et al. 2009; Norvell et al. 2007). Parsons (1933) observed that 10% of clay replaced for cement caused 10% reduction in compressive strength of concrete, whereas, 7.5% of fine aggregate replaced with clay resulted in 37% increase in strength. There was no appreciable effect on water absorption or permeability of concrete with clay. Similar results were observed by Lyse (1934) and

demands on the relaxation of limits of clay and fines content in fine aggregates used for concrete production were made. In contradiction, (Li et al. 2009) found that clay in manufactured sand results in reduced workability, increased drying shrinkage and increased freeze-thaw damages. Further, in a detailed study by Norvell et al. 2007, it was concluded that not all clay sized particles affect the concrete properties. It is only the swelling type smectite clay mineral that causes negative effects. With non-reactive fines, there is an improvement in workability and dimensional stability when water/cement ratio is maintained by using super plasticizers (Chan and Wu, 2000; Seleem and El-Hefnawy, 2003). It is also argued that the presence of clay has no major influence other than increment in water demand to maintain workability (Courard et al. 2011) and the water demand is directly proportional to the amount of reactive clay (swelling type) content. Fernandes et al. (2007) studied the effect of different clay minerals like kaolinite, montmorillonite (20%) on concrete properties. The results showed that the strength reduction with kaolinite can be related to the increase in water demand or compaction difficulty and there are no neo-formed products of clay. Whereas, montmorillonite mineral does not follow this relation suggesting that this type of clay is deleterious beyond just increment in water content. Super plasticizer also does not help in reducing water demand, as clay with its ability for cation exchange can make use of the organic materials in super plasticizers, increasing the dosage drastically. Other than cost implications, this result in excessive delay in setting time and poor strength (Norvell et al. (2007) and Olanitori (2006) suggested to either wash the sand or to use higher cement dosage, whichever is cost effective, to compensate for the strength loss due to clay bearing aggregates.

1.5 MOTIVATION FOR THE PRESENT STUDY

Excavation soil generated from earth works of mining industries, tunneling and large volume excavation for construction which contains basically sand particles with clay and fines. Though these are sand equivalent materials, presence of fines and clay makes them deleterious to mortar production. The problem of excess fines and clay in excavation soil can be avoided to some extent if replaced partially for river sand. However, the aim is to conserve depleting river sand and use the available alternatives in larger volume. Hence, treatment methods to enhance the properties of alternative fine aggregates will be the proper solution, to use them as complete replacement for river sand in cementitious composites. Though there exist few studies addressing such issues of fines on fine aggregate alternatives, there is a need

for a systematic study on different treatment methods and their relative effectiveness on different soil types. There is also gap in understanding the effect of processed material on properties of mortar. Hence, an attempt is made to study the influence of unprocessed and processed excavation soil as a complete replacement for fine aggregate in mortar. This also helps in identifying the adoptability of different treatment methods based on the property of excavation soil.

1.6 ORGANISATION OF THE THESIS

The thesis consists of 8 chapters.

Chapter 1 is the introductory chapter which provides the motivation for undertaking the present study. The organisation of thesis is also elaborated here.

Chapter 2 reviews past related research findings on the use of various materials of natural origin, recycled wastes and industrial by-products as fine aggregate alternatives. This also includes treatment methods adopted to improve the material property and the influence of treated and untreated material on fresh, hardened and durability properties of concrete.

Chapter 3 defines the objectives and scope of the present study and provides the methodology employed to achieve the objectives.

In **Chapter 4**, experimental investigation on use of three different unprocessed excavation soils of various plasticity, as fine aggregate in fly ash based geopolymer mortar are discussed. Properties of soil based geopolymer mortar, such as, flow, dry density, compressive strength, water absorption and drying shrinkage are compared with that of geopolymer mortar with river sand.

Chapter 5 discusses the use of “stabilization” as a treatment method for excavation soil prior to its use as fine aggregate in cement mortar. Lime and slag at various dosages are tried as stabilizers. The stabilized soils are used as fine aggregate in cement mortar. To evaluate their performance, the mortar properties are compared with that of control mortar with river sand.

Chapter 6 explains the applicability and effectiveness of washing and sieving of excavation soil. The wet sieved soils, wash water and residual clay are studied for their effectiveness as fine aggregate, mix water and pozzolanic material respectively.

Chapter 7 deals with thermally treating the soils with high plasticity to suppress the clay reactivity and using them as fine aggregate in cement mortar. The soils are treated at temperature range of 200 to 1000°C for duration of 30 to 180 minutes. The transition of clay-silt size particles is studied using hydrometer analysis. The treated soils are used in cement mortar and the properties of mortar with thermally treated soil are compared with control mortar.

Chapter 8 suggests the concluding remarks and scope for further studies.

CHAPTER 2

REVIEW OF LITERATURE

2.1 GENERAL

Current state-of-the-art on materials that are a potential replacement for fine aggregates is presented, with emphasis on its processing techniques, characterisation and material properties. Initially, the materials are classified based on their origin as materials of natural origin, recycled wastes and industrial by-products. Later the review is organised as per the classification (Figure 1.1) with detailed discussions on the material source, treatment methods, physical properties and concrete performance. Also, effort is made to bring out the common issues of using untreated materials and practical solutions to fix the drawbacks of using these materials in cementitious systems. The need for the research on excavation soil to address the problem due to fines is finally highlighted.

2.2 SOURCES OF ALTERNATIVE FINE AGGREGATES

2.2.1 Natural Origin

Natural alternative materials are those formed without manual processing and are available in huge volume. However, not all the materials would be economically viable due to the location specific availability and hence, high transportation cost. Transportation or importing from other countries will increase the cost of freshwater sand by 10% in coastal areas like China, Sri Lanka (Table 2.1). Crushed stone remains the dominant choice for use as aggregate in construction. Recycled concretes are being increasingly substituted for virgin aggregates. However, the percentage of total aggregate supplied by recycled materials, still remained very small in 2016 (USGS 2017). An increase of 11% in the usage of crushed stone is observed in the year 2017.

Dune sand is available in 11 different countries such as Arab, Republic of Botswana, India, China and Australia. Khan (1982) studied the use of desert sand for highway construction in the arid zones of Sahara region. Offshore soil from 15 m below sea level will not affect the environment and ecology, unlike extraction of beach sand (Garel et al. 2010). This could be a potential material in seashore construction activities. This is the case with marine sediments also. However, the marine sediments has excess clay content that demands

proper treatment before use. The accumulation of sediments naturally in ports makes the dredging a frequent process which results in huge volume of these materials.

Table 2.1 Sources and availability of materials of natural origin

Author, Year	Material	Source	Need and availability
USGS, (2017)	Crushed stone	Crushing rocks from quarries. Parent rock may be limestone, quartz, granite or basalt.	Largely available all over the world. However, use of limestone and dolomite is limited. In 2016, 1.48 billion tons of crushed stones was produced in US alone.
Khan, (1982); Padmakumar et al. (2012)	Dune sand	Formed as sediments from sandy parent material through erosion, transportation and deposition by weathering action of wind.	Arid and semi-arid region like Arab, Republic of Botswana, India, China and Australia. This occupies quarter of total land area of the world.
Garel et al. (2010); Yin et al. (2011); Ratnayake et al. (2014);	Offshore soil	Similar to quarried aggregates as they are formed by deposition of particles carried by any river that ends up in the ocean. In addition, they also have shelly sediments and chlorides that are objectionable to concrete.	Places like coastal China, Srilanka which is in immediate need for alternative aggregates for the growing infrastructure needs.
Magnusson et al. (2015); Osunade, (2002)	Excavated soil	From any excavation work of earth crust, mainly tunneling, major infrastructure and metro projects.	Laterite soil is a type of excavated soil (mostly low to medium plastic) from road cut works and construction sites and available abundantly in continents of Asia, America and Africa. Similarly, different types of excavation soil are available all over the world.
Aoual-Benslafa et al. (2014); Limeir et al. (2012)	Marine sediments	Dredging of marine sediments is a common practice in the Harbour to keep it operational.	The dredged sediments (DS) is normally disposed somewhere near the Port to reduce the transportation cost.

Excavated soil varies widely based on the place of excavation and mostly comprises of rock, stones, gravel, sand and clay. The quality of excavated soil varies from low plastic to high plastic based on the mineralogical composition of clay, organic matter and fines content. Life cycle assessment (LCA) and cleaner production (CP) of excavated soil from earth works were studied by Cabello Eras et al. (2013). It was emphasised that implementation of such strategies reduces the overall construction cost of the project. CP mainly focuses on recycling and reusing of these wastes by pre-treating them. For example, soil reuse program (SRP) to use the soil from construction site was implemented successfully in a rail transit system construction in New Jersey (USA) (Lafebre et al. 1998). Magnusson et al. (2015) has given a conceptual model for construction materials flow which emphasises on reuse of excavated soil in construction projects. They concluded that scientific community does not see the excavated soil in source perspective rather as a waste material to be disposed of. Most of these materials are stabilized with calcium containing materials and used as sub-base materials for roads (Bell, 1996).

2.2.2 Recycled Materials

While finding alternative source of construction materials is an immense problem in the current scenario, another one is the difficulty in locating places for disposing of demolished materials. Accumulation of waste has become a prime problem of the society and some of these wastes are listed in Table 2.2.

Recycled materials have been studied extensively in the laboratory. However, lack of confidence on these materials makes it difficult to implement them practically. Silva et al. (2017) discussed about the barriers in using recycled aggregates (RA) in concrete production. They include limited standards and specifications, low quality, client perception, and, poor market supply and demand. Construction and demolition (C&D) waste constitutes 70% of the total solid wastes in developed countries (EPA, 2014). As a part of construction and demolition waste, masonry demolition results in huge volume of brick wastes that are used in landfills. 7% of total world's waste generation is glass, which is growing due to the increased use of glass panels in construction industries. Though waste glass can be recycled in glass industries, there are restrictions due to the presence of impurities, cost and mixed colour. On the other side, disposing wastes like worn out tires has become a serious issue due to infections caused by sanitary problems and dwelling of insects in such places (Thomas et

al. 2015). In 1990's, waste rubber had been tried to be utilized in sub-bases of highways, sound barriers and other transportation structures. Raghavan et al. (1998) considered using shredded rubber in cementitious system which could help in reducing brittle failure and plastic shrinkage cracking.

Table 2.2 Sources and availability of recycled materials

Author, Year	Material	Source	Availability
Bektas et al. (2009); Gonzalez-Corominas and Etxeberria, (2014)	Brick and ceramics	This is a part of construction and demolition (C&D) waste, mainly from masonry demolition. Brick and tile manufacturing industries produces huge volume of rejects.	C&D waste counts 180 million tons per year in first 15 countries of European Union, with significant portion of masonry rubble. Similar case could be expected in most part of the world as brick was used in most of the old structures which are in the condition of demolition.
EPA, (2014); Eurostat, (2015); Silva et al. (2017); Zheng et al. (2017)	Concrete	Demolition of structures which are standing beyond their lifespan and poses risk of damage. Renovation of older model structures for increasing capacity and, improving architecture.	Though data is not available all over the world, C&D waste is a concern globally. In EU, 30% of total waste produced is concrete waste. US produce 373 MT of demolished concrete and China accounts for 2.36 billion tons.
EPA, (2014); Rashad, (2014)	Glass	Disposal of packing items, filament glass, lighting elements, residential/ commercial building glass panels.	Of the total solid waste produced in the world, 7% is made of glass. In the total glass waste produced, 3.3% was recycled to make new containers or secondary applications like aggregates and insulations. Remaining 96.7% is dumped directly in landfills.
Siddique and Naik, (2004); Thomas and Gupta, (2016)	Rubber	Produced mainly from scrap tires.	1000 million tires end their life time every year all over the world and only 50% of them were recycled. By 2030, this is estimated to be 5000 million tires discarded every year.

2.2.3 Industrial By-products

In India, 960 MT of solid waste is generated every year from different industries of which 290 MT of waste is from mining industries alone (Pappu et al.2007). Use of industrial by-products in building materials could be a sustainable solution for waste disposal. Different industrial by-products, which could be used as a replacement for fine aggregates in cementitious system are given in Figure 1.1. Table 2.3 gives the available data on sources and availability of these materials in the world.

The high-quality silica sand from foundry with more than 80% silica content are bonded with clay/chemicals. After certain number of recycling, they cannot be used for further moulding and casting process. In US alone, 100 MT of sand are used in foundry industries that will be discarded as foundry sand. Properties of foundry soil depends on the metal used in the casting, type of casting and furnace used (Ganesh Prabhu et al. 2014; Siddique et al. 2015). Leaching of heavy metals from foundry sand has been extensively studied by Siddique et al. (2010) and use of such materials in concrete has been suggested for its safe disposal without affecting the environment.

Mine tailing has mineral-ore, rock, soil and clay based on the source. In recent years, the problem of mine tailing persists in almost all countries that have mines, due to environmental hazardous associated (Kossoff et al. 2014). Mine waste finds its applications in land reclamation and backfilling of opencast quarries (Skarzynska, 1995).

Quarry dust is often disposed in landfills around the quarry region as a slurry of dust and water (Naganathan et al. 2012). Proper data on the quantity of quarry dust produced is not available (Table 2.3). However, Gameiro et al. (2014) emphasize that 80% of the rock extracted is treated as waste. This gives an idea on the huge amount of waste from these industries. Positively in geotechnical applications, quarry dust helps in stabilizing and altering the grading of soil by which improvement in the strength and reduction in plasticity is achieved (Amadi, 2014).

Industrial slags are formed by quenching of molten metals from furnace in water to form granular products. Leaching of heavy metals from such by-products filled in lands would cause problems to ground water. Using them in building materials would bind them and the leaching of elements from formed products are within same limits (Tripathi et al. 2013). The quenched, cooled and dried slag is broken into sand sized particles to be used as

fine aggregate replacement material (Saikia et al. 2008; Aboubakar et al. 2017). This can also be ground to powder, pelletized with binders like fly ash, and the formed solids are then crushed to sand sized particles to replace river sand in concrete (Pang et al. 2015).

Table 2.3 Sources and availability of industrial by-products

Author, Year	Material	Source	Availability
Bhardwaj and Kumar, (2017); Ganesh Prabhu et al. (2014)	Foundry sand	By-product of metal alloy casting industries	US utilize 100 MT of foundry sand and discard 10 MT every year. India is the third largest producer of foundry sand waste next to China. However, there is no proper monitoring system to report the total generation.
Gameiro et al. (2014); Naganathan et al. (2012)	Quarry dust	Crushing of stones in quarries produces quarry dust as a secondary product.	Though known to be available all over the world wherever quarrying exists, there is no proper data available on quarry dust.
Kossoff et al. (2014); Wang et al. (2014)	Mine tailing	Mine tailing is the solid material generated in surface mines when overlies were removed to access the ore. The quality highly depends on the type of mineral ore extracted.	Billion tons already stored all over the world and million tons being still produced every year.
Dash et al. (2016); MoEF, (2010)	Slag	Slag is an industrial by-product from the process of manufacturing different metals like lead (Pb), copper (Cu), zinc (Zn), steel and iron. The toxic metals present in such slags are declared hazardous to human health and environment and hence has to be handled carefully.	Including the major slag producers of world, 33 MT of copper slag is generated yearly. Iron and steel industries alone produces 250 MT of slag every year. China stands first in the production of slag from steel manufacturing, followed by Japan, India, USA and Russia.
IEA, (2015); WEC, (2016)	Bottom ash	Collected from settling pond of coal thermal power stations.	15-20% of total coal ash (777 MT) is bottom ash which is just landfilled.

World depends on coal resources for 40% of its total energy needs. China stands first in the production of coal-based energy followed by US, European Union, India and Russia. Of these, Indian coals possess high ash content, resulting in huge volume of coal ashes

including fly ash and bottom ash (IEA, 2015). Though fly ash has been widely accepted as construction material, bottom ash needs proven data for practical utilization (Singh and Siddique, 2013). Coal combustion bottom ash is obtained in the size range of 0 – 5 mm and collected from the bottom of the furnace. This can be used as fine aggregate. At present, unutilized bottom ash is land filled, thereby occupying large area for disposal and causing environmental hazards.

2.3 TREATMENT METHODS FOR ALTERNATIVE AGGREGATES

Without treatment of alternative fine aggregates, only partial replacement studies have been attempted to produce concrete of equivalent properties as that of concrete made of river sand. There are studies on different treatment methods to enhance the properties of these alternatives to increase their replacement level in concrete. The treatment method varies for different materials based on their material properties and requirement. It can be physical methods like sieving, washing, or chemical treatments, which are explained in this Section.

2.3.1 Natural Origin

There are different methods used by researchers to address the problems of these natural materials due to the presence of excessive fines or deleterious materials as given in Table 2.4.

2.3.1.1 Washing and sieving

Cepuritis and Mørtzell, (2016) washed the crushed stone aggregate to remove the fines. This is a common practice followed in most quarries, resulting in quarry dust as waste. Other than desalination of offshore sand, washing is also adopted to remove chlorides and sediments (Sun et al. 2011; Dias et al. 2008; Ratnayake et al. 2014). Washed marine sediments performed better due to the removal of fines, clay and organic matters (Olin-Estes and Palermo, 2001). Ozer-Erdogan et al. (2016) suggest that each harbour with intensive dredging operations should be equipped with a washing plant to utilize the generated waste in a better way.

Excavated soil invariably contains fines and clay in the system. Clay is ambiguous to understand and its behaviour in concrete is not much explored. Clay means a cluster of different minerals with diversity in properties and cannot be acknowledged with one particular performance. Without any treatment, clay increases the water demand and reduces the strength and durability by influencing the interfacial transition zone (Nehdi, 2014).

Washing is a simple and viable treatment method to remove undesirable elements like clay from the feasible fine aggregate materials, provided the disposal of secondary waste sludge is taken care of without affecting the environment.

Table 2.4 Treatment methods adopted in natural fine aggregate alternatives

Author, Year	Material	Treatment methods				Granulometry
		Washing + Sieving	Decantation	Chemical treatment	Thermal treatment	
Khouadjia et al. (2015)	Crushed stone					✓
Cepuritis et al. (2015)						✓
Cepuritis and Mørtzell, (2016)		✓				
Khan, (1982)	Dune sand			✓		
Belferrag et al. (2016)						✓
Bédérina et al. (2005)						✓
Ratnayake et al. (2014)	Offshore sand	✓				
Dias et al. (2008)		✓				
Katano et al. (2012)				✓		
Balogun and Adepegba, (1982)	Excavated soil					✓
Osunade, (2002)						✓
Kanema et al. (2016)				✓		
Dubois et al. (2009)	Marine sediments		✓			✓
Hamer and Karius, (2002)					✓	
Aoual-Benslafa et al. (2014)			✓			
Olin-Estes and Palermo, (2001)		✓	✓	✓		
Ozer-Erdogan et al. (2016)		✓	✓			
Sannier et al. (2009)			✓	✓	✓	✓
Tribout et al. (2011)				✓	✓	

2.3.1.2 Decantation

Decantation is the method adopted to remove the water and reduce the volume of marine sediments. Natural decantation of sediments helps in removing the excess water from sediments (Sannier et al. 2009). This involves placing the sediment in a drain and allowing dewatering to happen by the self-weight of the sediments. The water is then left to evaporate naturally. Decantation not only helps in removing water from sediments, it also removes dissolved solids by evacuation. Marine sediments are thermally treated at 450 °C to remove the organic contaminants (Sannier et al. 2009) and at 700 °C to reduce the volume by stabilizing them (Tribout et al. 2011). Clay (smectite, illite, chlorite and kaolinite) and other minerals present in marine sediments can be thermally stabilized and dimensional changes of blocks could be arrested (Hamer and Karius, 2002).

2.3.1.3 Chemical and thermal treatment

Chemical stabilization method is used mainly on aggregates applied for geotechnical applications. Earlier in 1982, Khan stabilized dune sand with cement to construct roads in desert regions and found it to be successful. Sannier et al. (2009) treated marine sediments with hydraulic binders to stabilize and solidify them for use in roadworks/ embankments. A combined chemical (phosphate) and thermal (at 700°C) stabilization are carried out by Tribout et al. (2011) on marine sediments to treat heavy metals and organic matters respectively. However, phosphatizing and/or calcination make the treatment expensive and results in a need to go for alternative methods (Sannier et al. 2009).

2.3.1.4 Granulometry

Granulometric adjustment is done to redefine the particle size distribution by adding different sized particles or by partial replacement. For example, river sand was mixed with crushed stone aggregate to compensate for the presence of higher fines content (Khouadjia et al. 2015). Cepuritis et al. (2015) adopted air classification method to avoid the problems in washing, which involved acceleration of air inside a chamber and separating different size fractions with gravity and centrifugal force. Together with vertical shaft impact (VSI) crusher and micro-proportioning using air classifier, this technique works well for all types of rocks in crushed stone aggregate. Granulometric adjustment is feasible with any alternative material in the category of natural origin. However, the dilution of material results in reduced volume usage and stresses the use of other materials that may have to be transported at a high cost.

2.3.2 Recycled Materials

2.3.2.1 Sieving

Based on source, fine recycled concrete aggregate (FRA) may have various contaminants like Pb, Zn, Ni and Co. Hence, it becomes mandatory to screen materials based on size and use them in specific applications (Bianchini et al. 2005). Presence of old mortar coating on the recycled concrete aggregate is a main point of concern in fine recycled concrete aggregate (FRA). Mechanical grinding or scrubbing and sieving can be applied to remove up to 70% of such mortar (Hansen, 1990; Purushothaman et al. 2015). Washing is adopted for recycled materials like rubber and glass to remove the contaminants (Eldin and Senouci, 1993). Rostami et al. (1993) improved the washing process by mixing latex cleaner with the water for cleaning recycled rubber particles (Table 2.5).

2.3.2.2 Chemical and thermal treatment

Pre-soaking the FRA in an acidic environment with three different acids, hydrochloric acid (HCl), sulfuric acid (H₂SO₄) and phosphoric acid (H₃PO₄) has been employed by researchers. Later, the treated aggregates were soaked in water for 24 hours and washed to remove the old mortar with acidic concentration. Though this method imparts some chlorides and sulphates to the system, it is said to be well within the limit (Tam et al. 2007; Juan and Gutiérrez, 2009; Purushothaman et al. 2015). With acid treatment, mortar content could be brought down from 47 to 13% (Akbarnezhad et al. 2011). Modification of surface of FRA increases the bonding between the aggregate and cement paste.

Ling and Poon, (2014) used acid washing to remove lead (Pb) from the crushed cathode ray tube (CRT) glasses and brought it under acceptable limit as per toxicity characteristic leaching procedure (TCLP). Such treatments in alkaline medium (NaOH) also helped in improving the surface texture of rubber aggregates. Since, rubber particles have smooth surface, they need treatment for surface improvement to enhance the bonding between the paste and aggregate (Naik and Singh, 1991).

Table 2.5 Treatment methods adopted in recycled materials

Author, Year	Material	Treatment methods				Coating
		Sieving		Chemical treatment	Thermal treatment	
		Dry	Wet (Washing)			
Hansen, (1990)	Concrete	✓				
Bianchini et al. (2005)		✓				
Tam et al. (2007)				✓		
Juan and Gutiérrez, (2009)				✓		
Akbarnezhad et al. (2011)					✓	
Purushothaman et al. (2000)				✓	✓	
Spaeth and Djerbi Tegger, (2013)						✓
Santos et al. (2017)						✓
Ling and Poon, (2012)	Glass			✓		
Naik and Singh, (1991)	Rubber			✓		
Rostami et al. (1993)			✓	✓		
Eldin and Senouci,(1993)			✓			
Segre and Joeke, (2000)				✓		

Treating FRA thermally to 300°C makes the older mortar brittle, followed by mechanical scrubbing improved the material properties (Table 2.5). Microwave was used, exploiting the electromagnetic properties of old mortar and the aggregate particles. Microwave energy absorption rate of mortar is higher compared to aggregate. Hence, mortar gets heated up faster, causing differential stresses. This results in delamination of older mortar and separation of aggregate particles (Akbarnezhad et al. 2011).

2.3.2.3 Coating

Though removal of older mortar from FRA gives better quality aggregates, it results in generation of secondary waste. Coating can be done to avoid this, while enhancing the aggregate property without separating the older mortar. This was tried with polymer products which are water repellent to bring down the water absorption of FRA (Spaeth and Djerbi Tegger 2013; Santos et al. 2017). Polydiorganosiloxanes and alkylalkoxysilanes were two important polymers used for impregnation. Polymer coated aggregates were then dried for 24

hours, followed by oven drying at 50°C for 24 hours (Spaeth and Djerbi Tegguer 2013). Molten paraffin wax which does not need long drying period was also tried (Santos et al. 2017). Coating improved the interfacial transition zone between the fine recycled aggregate and the cement paste.

2.3.3 Industrial By-products

Alternative fine aggregate that are industrial by-products, have few research works witnessing property enhancement by pre-treatment. Sieving/washing and combination of both, are the available treatments used on these materials. Slag and bottom ash have been dry sieved to adjust the particle size distribution. The excess fines in these materials affected the mortar/ concrete properties. Hence, particular size particles (retained 0.15 mm or between 0.25 to 0.1 mm) have been dry sieved and used as fine aggregate material (Qasrawi et al. 2009; Modolo et al. 2013). Washing treatment on bottom ash has been used to remove soluble salts in a continuous shower with a running capacity of 10 tonne/hour. Monosi et al. 2013 used combination of washing and sieving (wet sieving) to remove the fines, contaminant and clay particles in foundry sand that are less than 75 µm in size.

2.4 MATERIAL PROPERTIES

2.4.1 Natural Origin

Table 2.6 shows the physical properties of alternative fine aggregates of natural origin. The properties of crushed stone vary with characteristics of the parent rock, even within same mineralogy. For example, granite-based crushed stone shows huge variation in density from 1890 to 2610 kg/m³ (Kou and Poon, 2009; Li et al. 2009). The percentage of silica, which is a major component of river sand varies from 0.34% (limestone) to 99% (quartzite). Similar differences are observed in other materials too, which makes these materials entirely different from river sand. Materials of natural origin are rounded in shape, except crushed stone which is mostly angular due to crushing process. However, by using impact crushing, it is possible to produce cubical particles (Alexander and Mindess, 2005). Shen et al. (2016) clarified that though crushed stone looks angular in macro-scale; it shows higher roundness in micro-level.

Table 2.6 Physical properties of naturally occurring fine aggregate alternatives

Reference	Material	Silica content (%)	Particle size		Specific gravity	Bulk density (kg/m ³)	Water absorption (%)
			FM*	Mean size (mm)			
Li et al. (2011)	Crushed stone (Lime stone Quartzite Granite Basalt)	4.98-5.10 99.22 54.23 50.53	3.01-3.43 3 2.75 3.54		-	1842 1876 1893 1987	-
Kou and Poon (2009)	Crushed stone (Granite)	-	3.56		-	2610	0.89
Benabed et al. (2012)	Crushed stone (Limestone)	1	2.21		2.68	1541	-
Li et al. (2009)		5.05			2.665	-	-
Khouadjia et al. (2015)		0.34 – 1.37	2.6 – 3		2.45 – 2.56	1489 - 1618	-
Ding et al. (2016)		2.77 – 3.34	-		-	1720 - 1820	0.7 – 1
Celik and Marar (1996)		-	3.39		2.65	-	-
Al-Ansary et al. (2012)	Dune sand	18.41-70.08		0.25	2.63	1687	0.5
Al-Harthy et al. (2007)		-		<0.6	2.56 – 2.66	-	0.4 – 2.04
Benabed et al. (2012)		-	0.78		2.65	1520	-
Belferrag et al. (2016)		21.09	1.33/1		2.5/2.6	1487	1.02
Bederina et al. (2005)		21.47	1.18		-	1428	-
Luo et al. (2013)		94.8	1		2.72	-	3.92
Khouadjia et al. (2015)		86.45	0.5		-	1554	-
Padmakumar et al. (2012)		80.37-93.84		0.05-0.25	-	-	-
Zhang et al. (2006)		86.92		< 0.3	-	1470-1520	-
Dias et al. (2008)	Offshore sand	-		0.6	-	-	-
Dolage et al. (2013)		-		0.9	2.65	-	-

Table 2.6 continues...

Reference	Material	Silica content (%)	Particle size		Specific gravity	Bulk density (kg/m ³)	Water absorption (%)
			FM*	Mean size (mm)			
Limeir et al. 2012	Marine sediments	-		<0.25	-	2630	1
Limeira et al. 2011		-		0-4	-	2570-2670	0.8-1.7
Zentar et al. 2009		-		<0.063	-	-	-
Kazi Aoual-Benslafa et al. 2014		55		>0.063	-	2450	-
Ozer-Erdogan et al. 2016		97.5	3.15		-	1222-1385	0.7 – 4.6
Tribout et al. 2011		46.4	-		-	1000	-

Note: FM: Fineness modulus

Presence of fines and poor gradation are common problems in these materials. Gradation of fine aggregates is not strictly monitored due to the limited availability of river sand. However, optimization of fine aggregate gradation can increase the strength up to 39% and preferred gradation can be achieved by incorporating different size fraction of crusher fines with available natural sand (Sabih et al. 2016). Compact packing of aggregates helps in increasing the density and reducing the void ratio, resulting in improved strength. Hence, granulometric corrections are necessary for alternative materials, atleast when intended to be used in structural concrete.

2.4.2 Recycled materials

Unanimously, all the recycled materials need crushing or breaking in to particles of fine aggregate size. This can affect the material properties of such aggregates. Recycled brick aggregates are crushed and segregated based on size for use as coarse or fine aggregate in concrete (Aliabdo et al. 2014). Importantly, water absorption and permeability are higher in brick aggregates due to its porous nature (Debieb and Kenai 2008; Vieira et al. 2016). From Table 2.5, it is clear that water absorption of crushed brick aggregate varies from 14 to 21.8%. Crushed ceramic waste exhibits lower absorption value for wall tiles and sanitary ceramic waste and exceptionally high value for floor tile ceramics (Table 2.7). Presence of crushed aggregates containing impurities like gypsum plaster, organic substance and salts affects the properties of concrete containing these aggregates (Keseđić et al. 2008).

In case of recycled aggregates, the main problem is with the particle density, which is lower than natural sand, as the older mortar present in them are porous and lighter in weight

(Evangelista and de Brito 2007). Recycled aggregates (RA) made using jaw-crusher and vertical axis impactor, followed by attrition techniques, are better end products as the older mortar surrounding the recycled aggregates are removed (Ulsen et al. 2013). Type of crushing process affects the quality of RA and its properties (Fan et al. 2016). Mortar content increases with reduced density and size of aggregate. Similarly, recycled glass aggregates are also crushed before use and material properties highly depend on the speed, duration and type of crushing (Rashad 2014).

Table 2.7 Physical properties of recycled fine aggregate alternatives

Reference	Material		Silica content %	Particle Size		Specific gravity kg/m ³	Bulk density kg/m ³	Water absorption %
	Type	Treated/ Un-treated		FM*	Mean size (mm)			
Aliabdo et al. (2014)	Recycled brick waste	Un-treated	54.2		0.075	-	-	18.3
Debieb and Kenai (2008)			-	3.91		2.49	1010	14.0
Jankovic et al. (2012)			-	-	0 – 4	1.61	1216	21.8
Alves et al. (2014)	Recycled ceramic waste	Un-treated	-	-	-	-	2969	0.2
Binici (2007)			-	2.68	-	2.44	1395	0.71
Elçi (2015)			69.93	-	-	2.05	2619	17.2
			71.89	-	-	2.33	2652	2.75
Medina et al. (2012)			-	-	< 4	2.39	-	0.55
Medina et al. (2013)			19.39	-		2.39	-	0.55
Evangelista and de Brito, (2014)	Recycled concrete	Un-treated	60 – 85	-	<4.75	2.2 – 2.64	1950 – 2560	2 – 14
Akbarnezhad et al. (2011); Purushothaman et al. (2000)		Treated	-	-	-	2.44 – 2.7	2380 – 2500	0.78 – 4.1
Rashad (2014) and Rashad (2015); Tan and Du (2013)	Recycled glass	Un-treated	70 – 90	-	0.15 – 5	2.53	-	0.07
Siddique and Naik (2004); Thomas and Gupta (2016)	Recycled rubber	Un-treated	-	-	0.0075 – 4.75	0.48 – 0.83	-	-

Note: FM: Fineness modulus

Recycled rubbers are available as shredded tires, ground rubber and crumb rubber, of which crumb rubber falls in the size range of 4.75 mm and below. The waste rubber can be converted to crumb rubber by crack mill process, micro-mill process or granular process which is well explained by Heitzmann (1992). These processes involve either tearing or shearing of tire rubber, resulting in irregular and granular rubber particles, respectively. Lighter weight rubber particles result in lower specific gravity and density (Table 2.7).

2.4.3 Industrial by-products

Properties of industrial by-products vary based on the type of industry and the raw materials used. Foundry sand is one such material which is finer than river sand, and contains clay and other chemicals as impurities (Table 2.8). Metal ions from cast-industries gets mixed up with this sand making it impure. Properties of quarry dust vary based on the type of stone quarried and some of them are shown in Table 2.8. Both foundry sand and quarry dust have the problem of excess fines content.

Composition of slags varies based on the source metal processed. For example, steel slag has 97% of Fe_2O_3 as mentioned by Qasrawi et al. (2009). Slags have the potential to be used as cement replacement material, properties like abrasion resistance, soundness and dimensional stability makes them better aggregate materials in concrete production (Gorai et al. 2003). However, some of them like slag obtained from steel industries have very low pozzolanicity that cannot be used for cement replacement (Qasrawi et al. 2009).

Material properties of bottom ash, obtained from thermal power stations, depend on the source and quality of coal used, as reported by various authors (Table 2.8). Pre-treatment may be needed to avoid leaching of chlorides and to adjust the particle size distribution (PSD) of the aggregates. Few studies have been done after pre-treatment of bottom ash. Though PSD of raw bottom ash does not affect the properties of mortar produced, chlorides and unburnt carbon should be taken care of to preserve the durability properties.

Table 2.8 Physical properties of industrial fine aggregate alternatives

Reference	Material		Silica (%)	Particle size		Specific gravity	Bulk density (kg/m ³)	Water absorption (%)
				FM	Mean size (mm)			
Siddique et al. (2015)	Foundry sand		81.5	-	0.15 – 0.6	2.1	-	-
Ganesh Prabhu et al. (2014)			87.48	-	< 2.36	2.24	1576	1.13
Khatib et al. (2013)			87.91	-	< 0.25	-	-	-
Monosi et al. (2013)			-	-		2.04 – 2.26	-	3.3 – 5.4
Basar and Deveci Aksoy (2012)			81.85	-	< 2		2510	0.9
Singh and Siddique (2012)			83.8	1.89	-	2.18	-	-
Siddique et al. (2011)			87.9	1.78	-	2.61	1638	-
Celik and Marar (1996)	Quarry dust				<0.075			
Rai et al. (2014)					< 2.36	2.54	1800	1.4
Naganathan et al. (2012)			69.94	3.3		2.59	1720	-
Raman et al. (2011)			-	-	<5	2.83	-	0.78
Ho et al. (2002)			-	-	-	2.6	-	-
Corinaldesi et al. (2010)	Marble dust		-	-	-	-	2550	-
Gameiro et al. (2014)			-	-	-	-	2684	0.14
Zhao et al. (2014)	Mine tailings	Iron ore tailings	52.06	-	0 – 0.5	-	-	-
Gallala et al. (2017)		barite-fluorspar mine waste	14.33	1.1	-	-	3270	-
Saikia et al. (2008)	Slag	Pb slag	25.7	-	0 – 4	-	-	-
Tripathi et al. (2013)		Pb and Zn slag	17.01	-	0 – 2.36	3.69	-	0.45
Al-Jabri et al. (2009)		Cu slag	33.05		-	3.4	-	0.17
Wu et al. (2010)			31.92	1.78	-	3.66	-	-
Al-Jabri et al. (2011)			33.05	-	-	3.4	-	0.17
Ambily et al. (2015)			25.84	3.43	-	3.37	2080	0.3 – 0.4
Qasrawi et al. (2009)		Steel slag	0.8		0 – 5	3.25	-	0.8
Yüksel et al. (2011)		Blast furnace slag (BFS)	35.1	-	0 – 5	2.08	-	8.3
Aboubakar et al. (2017)			-		0.3 – 5	2.23	2720	1.12

Table 2.8 continues...

Reference	Material	Silica (%)	Particle size		Specific gravity	Bulk density (kg/m ³)	Water absorption (%)
			FM*	Mean size (mm)			
Bilir (2012)	Coal bottom ash	57.90		0 - 5	1.39	632	12.10
Andrade et al. (2009)		56	-	-	1.674	-	-
Kim et al. (2014)		44.2-48	2.34 – 3.81	-	1.91-1.98	-	8.7-10.8
Yüksel et al. (2007)		57.90		-	1.39	620	12.10
Singh and Siddique (2014a)		47.53-56.44	1.37	-	1.39	-	31.58
Siddique et al. (2012)		57.76	1.60	-	1.93	-	-
Kim and Lee (2011)		-	2.36	-	1.8	-	5.45
Kim et al. (2012)		34	2.34	-	1.87	-	5.45

Note: FM: Fineness modulus

2.5 USE OF ALTERNATIVE FINE AGGREGATE IN CEMENTITIOUS SYSTEM

2.5.1 Natural Origin

2.5.1.1 Fresh properties

Materials of natural origin have been studied for their performance in mortar, concrete and in self-compacting concrete (SCC) as presented in Table 2.9. Many studies have given importance for fresh property due to the issues of particle size distribution and presence of excessive fines. Cortes et al. (2008) explained that irregular shape of fine aggregate made of crushed stone results in loosest packing density that needs more paste for better flowability and strength. Other than shape factor, crushed stone has high fines exceeding the limit prescribed in standards which affect the workability of the concrete (Shen et al. 2016). Gradation is a common issue with not only in crushed stone but also with most of the alternative materials, which affect the fresh properties of mortar and concrete (Table 2.6).

Flow was found to improve when the volume of paste exceeds the volume of voids in the mixture. Studies on dune sand and excavated soil show that cement to fine aggregate ratio should be maintained below 2 for better workability and strength (Falade 1991; Falade 1994; Zhang et al. 2006). Ji et al. (2013) derived a mix proportion with minimum paste content concept by using microfines present in the crushed stone to replace some of the cement content. However, this works only when the amount of microfine is moderate. Strength was

found to get reduced when a certain limit is exceeded. The minimum paste theory in crushed stone helps to enhance the volume stability of cement composite. Ding et al. (2016) mentioned that a powder content of 9%, benefits workability by improving the cohesiveness of fresh concrete and long term compressive strength is not affected up to 13% of replacement level. The presence of fines has also been positively used to enhance the flow properties in SCC (Nanthagopalan and Santhanam 2011; Benabed et al. 2012).

Organic matter present in the aggregates adsorbs the calcium ions from hydration products and disturbs the setting time. The intensity depends on the type of organic compound and it is grouped based on its reactivity by Clare and Sherwood (1954). In case of offshore sand and marine sediments, the presence of chloride ion and organic contaminants affects the workability. Setting time with chloride ions in concrete is reduced and needs a suitable retarding agent (Kaushik and Islam 1995). Though clay affects the concrete properties negatively, the presence of fewer clay particles (up to 2.84%) helps in better bonding between the cement and aggregate surface, resulted in lower chloride ion penetration in sea sand mix (Yin et al. 2011). Water-cement ratio of the offshore sand mix should be made lower to make it less permeable for the ingress of chloride ions. All these factors should be considered for designing the mix with natural fine aggregate alternatives.

2.5.1.2 Hardened properties

Presence of fines not only affects fresh concrete properties but also hardened properties by influencing the particle packing. As per standards, fines content i.e., content of particles less than 75 μm size is restricted to a maximum of 7% (ASTM C33, 2016). However, research shows that this limit can be extended up to 15% to improve the packing density in wet condition (Kwan et al. 2014). This could also increase the strength of concrete by 30% and reduces carbonation by reducing the open porosity. On the negative side, this increases the volume change and water demand as the powder content increases with fines (Katz and Baum 2006). However, on the contrary, with an increase in fines content from 7% to 9%, strength reduction and increase in porosity was observed by Cho (2013) which means the particle packing is negatively affected. Presence of fines in dune sand entraps air bubbles leading to higher air content which in-turn reduces the concrete strength further. However, lower fine aggregate to cement ratio (<1.41) gives higher strength which is attributed to the nucleation and pozzolanic effect of fines, thereby enhancing cement hydration (Luo et al. 2013).

Table 2.9 Studies on cementitious system using untreated fine aggregate of natural origin

Author, Year	Material	Problems in raw materials	Fresh properties	Strength properties	Durability properties	Properties with treated material
Kou and Poon (2009); Li et al. (2011); Cepuritis and Mørtzell (2016);	Crushed stone	Angular shape of crushed aggregates.	Angular shape affects the workability. Shape, texture, grading and dust decide the amount of additional water/cement required.	Strength reduced with increase in water content to maintain workability.	Larger particle size and lower surface area reduced drying shrinkage. Slump reduction resulted in poor compaction and looser microstructure.	Slump value improved from 35 mm to 165 mm by washing.
Zhang et al. (2006); Luo et al. (2013); Bédérina et al. (2005); Belferrag et al. (2016)	Dune sand	Fine size and presence of excessive fines.	Maximum grain size is 1.18 mm. Workability is reduced by adsorption of water by excess fines.	When Cement to sand (C/S) ratio > 0.7, compressive strength is comparable with river sand mix. When C/S < 0.7, entrapped air increases and reduces the strength.	-	Granulometric correction with river sand reduced drying shrinkage and improved strength.
Dias et al. (2008); Yin et al. (2011)	Offshore soil	Presence of chlorides, shell.	-	With 0.075% of allowable Cl ⁻ , the properties are similar to control mix.	Improved resistance to Cl ⁻ ion penetration with W/C ratio of 0.45 to 0.6.	Accelerated corrosion performance was satisfactory and similar to chloride free control mix.
Balogun and Adepegba, (1982); Thandavamoorthy, (2014); Kanema et al. (2016)	Excavati-on soil	Presence of clay and fines.	Up to 40% clay could be used for laterized concrete though lower percentages are preferable. Less W/C ratio results in poor compaction.	Compressive strength reduced with increasing percentage of excavation soil.	Results in high shrinkage strains and even cracking during drying.	Granulometry improved granular skeleton and reduced shrinkage. Limed earth helped in stabilizing the clay.
Couvidat et al. (2016); Ozer-Erdogan et al. (2016)	Marine sediment	Presence of >50% excessive fines, shell and other impurities.	Water demand increased with increasing fines content.	Strength reduced drastically.	-	Removal of chlorides and sulphates by washing improved durability.

Use of blended aggregates with dune sand, crushed stone and river sand improved the concrete strength (Bédérina et al. 2005; Benabed et al. 2012 and Khouadjia et al. 2015), which could be the effect of granulometric correction of particle size distribution. Granulometry is adopted by partial replacement of river sand with laterite soil to match the mechanical properties of concrete with 100% river sand. Even untreated soil with 50% clay content performed better when used as a partial replacement (Balogun 1982; Ettu et al. 2013). As concrete is a complex composite system with different type of aggregates, its gradation, fines content and its distribution, and the effect of fines and their acceptable limit may change from case to case.

2.5.1.3 Durability properties

The permeability of concrete is controlled with the use of aggregates containing fines up to 20%. This improves the resistance to chloride ion penetration. However, resistance to freeze-thaw and sulphate attack are negatively affected by the use of aggregates like crushed limestone (Li et al. 2009). When used in cement pavements, the natural rough, angular crushed stone gives good flexural strength and abrasive resistance compared to concrete with river sand (Li et al. 2011). In few cases, crushed stone with larger particles helps in reducing drying shrinkage due to its lower specific surface area (Kou and Poon 2009). This is also achieved by a granulometric correction in concrete with dune sand to adjust the proportion of fines (Belferrag et al. 2016). Shrinkage strain reduces when excavated soil is partially replaced with other alternatives like recycled concrete aggregates or quarry dust (Kanema et al. 2016). However, with 100% laterite soil (excavated soil), creep and shrinkage strains are several times greater than concrete with river sand (Salau and Balogun, 1999). Hence, to utilize a larger volume of such materials, treatment is inevitable.

2.5.2 Recycled Materials

2.5.2.1 Fresh properties

Recycled brick and concrete aggregates show higher water absorption (Table 2.10). This affects the fresh properties by reducing flow or by increasing the water demand. Though ceramic waste does not absorb water, the agglomeration of particles that prevents them from sliding resulted in increased water requirement to maintain workability. In such cases, super plasticizers would help improving the workability (Kanema et al. 2016). The water storage by

this type of aggregates positively affects in internal curing and lowers internal stress, reducing the shrinkage effects (Gonzalez-Corominas and Etxeberria 2014).

In addition to old mortar in recycled aggregate (RA), contaminants such as brick, gypsum or wood contributes to increase in water absorption of RA. Due to this, a large scatter value is observed (Rodrigues et al. 2013). Specific surface area is higher for RA due to the open pores on the surface, which affects the workability. Different mixing procedure and alterations were suggested, such as pre-wetting the RA before mixing, adding pozzolan to the RA and introducing cement at the last stage of mixing. Workability is also affected due to angular shape of RA, however there are also results showing better slump due to the presence of excess fines that acts as lubricant (Evangelista et al. 2015).

Glass aggregate varies in property with its colour. Heat of hydration is higher for glass aggregate incorporated concrete depending on the colour of the glass used. When compared among three common colours (emerald, amber and clear), emerald coloured glass gives the maximum heat of hydration (Poutos et al. 2008). Workability increased with glass aggregate content and is related to the impermeability, smooth surface and lower water absorption of glass aggregate compared to traditional fine aggregate (Terro, 2006). However, there are also studies reporting workability reduction with glass aggregate due to their sharp edge, angularity and texture (Tala and Nounu, 2008).

Density reduced as specific gravity is lower for glass aggregates whereas density increased with cathode ray tube (CRT) glass sand owing to their higher density (Rashad 2015). When it comes to rubber aggregate, in general, it makes the concrete harsh to work. However, incorporation as fine particles results in better workability compared to coarser particles. Non-polar nature of rubber particles entraps air and repels water. It also reduces the unit weight of the mixtures with recycled rubber aggregate (Khatib and Bayomy, 1999).

2.5.2.2 Hardened properties

Crushed brick has been studied for its suitability as aggregates in sub-base (Poon and Chan 2006), coarse aggregates in concrete (Akhtaruzzaman and Hasnat, 1983), brick powder in concrete (Ge et al. 2012) and in brick manufacturing (Sadek, 2012). Compressive strength reduced up to 30% due to higher water absorption. Debieb and Kenai (2008) warns against the usage of brick aggregates in concrete due to adverse effect on strength.

Table 2.10 Studies on cementitious system using untreated recycled materials as fine aggregate

Author, Year	Material	Maximum replacement (%)	Problems in raw materials	Properties of mortar/concrete with fine aggregate as recycled material			
				Without treatment			Improvement after treatment
				Fresh properties	Strength properties	Durability properties	
Debieb and Kenai (2008); Bektas et al. (2009); Alves et al. (2014)	Recycled brick & ceramics	100	Porous nature. High water absorption of brick aggregates. Glazed surface of ceramics.	Flow reduces with increasing brick/ceramic aggregates. Setting delayed due to segregation.	30% reduction in compressive strength.	Shrinkage is 6 times higher than control. Higher permeability and water absorption in cement mortar.	-
Evangelista and de Brito (2014); Tam et al. (2007); Juan and Gutiérrez (2009); Purushothaman et al. (2015)	Recycled concrete	100	Angular shape. High water absorption Presence of impurities like brick, wood, old mortar.	Water absorption reduces. workability Plasticizers lose performance.	Up to 60% strength loss with 100% fine recycled aggregates.	50- 70% increase in shrinkage due to absorbed water and porosity. 30% more pore volume than control.	40% improvement in workability was observed. Compressive Water absorption rates reduced significantly between 7 to 12%.
Rashad (2014, 2015); Naik and Singh, (1991); Rostami et al. (1993)	Recycled glass	100	Sharp edges, angular particles. Properties vary with glass colour.	Workability reduced. Bleeding and segregation.	Weak adhesion between smooth glass and cement (ITZ) results in strength reduction.	Increased permeability and water absorption. Reduced shrinkage due to impermeable nature of glass aggregates. Highly affected by ASR.	-
Rostami et al. (1993); Siddique and Naik (2004); Thomas and Gupta (2016)	Recycled rubber	100	Requires special equipment to shred/ground.	Workability reduced. Lighter rubber results in segregation.	No proper bonding between rubber and cement paste resulting reduced strength.	Increases abrasion resistance by acting like a brush. Non-polar rubber traps air in its surface and increases porosity near ITZ increasing permeability, water absorption and sorption. Shrinkage increased by 95% with 20% inclusion of rubber.	Simple water washing improved the strength by 16%, with chemical treatment the strength increment increased to 57%.

Usage of fine recycled aggregates (FRA) usage in concrete was first studied by Hansen (1992) when 100% waste products like recycled aggregates and fly ash were used to produce concrete without cement. This was suggested to be used in fill and road bases. Bairagi et al. (1990) recommended a mix design procedure for recycled aggregate concrete (RAC) and advised to use 10% extra cement to compensate for the poor quality of recycled aggregates. Strength reduction is noted with FRA concrete, which was improved by increasing the cement content. Use of super plasticizer also helps in reducing the strength loss in FRA concrete (Pereira et al. 2012). Works on rheology of RA concrete throw light on the usage of high performance super plasticizer to offset the negative effect of high water demand and its contribution towards the improvement in strength properties (Cartuxo et al. 2015; Kubissa et al. 2015 and Ait Mohamed Amer et al. 2016). Zhao et al. (2015) incorporated saturated FRA to reduce the water demand, however this reduced the compressive strength by increasing the interfacial transition zone (ITZ). Addition of pozzolanic materials like fly ash and slag recovers strength loss of recycled aggregate concrete by improving aggregate-binder bond strength (Anastasiou et al. 2014).

Strength of mortar and concrete reduced with inclusion of glass aggregates and CRT glass aggregates as their smooth surface resulted in poor bonding and weaker interfacial transition zone (Ling and Poon, 2011; Park et al. 2004). Glass powder, when replaced for cement, affected the mechanical properties negatively, whereas when used together with crushed glass aggregates, enhanced the concrete properties (Afshinnia and Rangaraju 2016). Heavy weight glass aggregate was used in high density concrete by Choi et al. (2017). It was suggested for application in radiation shielding.

Size and shape of the rubber particles affects the strength properties of concrete with crumb rubber (rubcrete). Addition of coarse rubber resulted in higher strength reduction compared to its use as replacement for fine aggregates (Aiello and Leuzzi 2010). However, pre-treatments like water soaking (Mohammadi and Khabbaz 2015), washing, etching with acid improved the strength up to 57%. Addition of silica fume (Güneyisi et al. 2004; Pelisser et al. 2011; Xue and Shinozuka 2013), fly ash (Yilmaz and Degirmenci 2009) enhanced the mechanical properties of rubberized concrete and an optimum replacement of 25% of aggregates with crumb rubber helps to retain the properties of concrete without any strength loss (Güneyisi et al. 2004; Khaloo et al. 2008; Issa and Salem 2013).

2.5.2.3 Durability properties

Brick aggregates are found to be highly alkali silica reactive. Bektas et al. (2009) made an extensive study on alkali silica reaction (ASR) with brick aggregates and concluded that it does not affect the engineering property of concrete, except strength. Durability properties like shrinkage, carbonation, chloride penetration and water absorption are highly affected with the use of brick aggregate compared to ceramic waste aggregate due to the porous structure of the former (Vieira et al. 2016). However in both the cases, replacement of 25% seems to be the optimum value in concrete application to match the properties of control concrete with river sand.

Generally, shrinkage of recycled aggregate concrete (RAC) is higher compared to conventional concrete because of high demand for water to maintain similar workability (Hasaba et al. 1981). This could be taken care by pre-wetting the aggregates and using super-plasticizer (Merlet and Pimienta, 1993). Hanif et al. (2017) reported contradicting results with reduced microcracks of RAC due to lower shrinkage strains. The presence of cracks in older mortar of RA increases the porosity of concrete made of recycled aggregates (Omary et al. 2016). Durability properties are badly affected by the use of RA, particularly fine recycled aggregates (FRA) (Bravo et al. 2015). Addition of pozzolans improves the durability properties of FRA concrete (Singh and Singh, 2016). RA in lime mortar performed better compared to cement mortar and reduced water absorption by reducing the capillary pores (Raeis Samiei et al. 2015).

Rashad (2015) has explained the reason for CRT glass aggregate showing reduced drying shrinkage is due to the low water absorption characteristics of glass. Alkali Silica Reaction (ASR) is another important factor to be considered in case of usage of glass as aggregates. It results in expansion of aggregates and formation of crack between aggregates and cement paste. However, it is suggested that the use of suitable pozzolan like fly ash, slag, silica fume or metakaolin would reduce this effect. Effect of incorporating pozzolan in concrete with glass aggregate has been studied by Cota et al. (2015). It is reported that 7.5% to 15% of glass aggregates can be used in cement composites without compromising the mechanical properties, if 15% of cement is replaced with metakaolin. This also improved the strength and shrinkage properties of glass aggregate concrete at later ages (Bostanci et al. 2016).

In concrete with crump rubber aggregate, durability related properties such as water absorption, carbonation resistance, chloride penetration increases with increasing size of the aggregates (Bravo and De Brito 2012; Thomas and Gupta 2015). However, shrinkage of concrete reduced with incorporation of rubber particles to it. With increasing percentage of replacement, the length and width of shrinkage crack reduces (Raghvan et al. 1998). Post peak response of concrete with rubber is improved and the deformation is elastic unlike normal concrete which shows brittle failure (Tantala et al. 1996). Rubcrete absorbs more energy and can be applied in places where vibration damping is required (Khaloo et al. 2008). Post cracking residual strength and energy absorption improved with the addition of rubber in concrete (Al-Tayeb et al. 2013). Rubber acts like a fibre, bridging the cracks and helps to avoid brittle failure of concrete (Aiello and Leuzzi 2010). Benazzouk et al. (2007) and Wang et al. (2013) produced light weight concrete with shredded rubber wastes that has high resistance to water absorption and improved toughness. Rubcrete can also be employed as sound absorbent and insulating material in various applications (Holmes et al. 2014).

2.5.3 Industrial By-products

2.5.3.1 Fresh properties

Workability of concrete reduces with increasing foundry sand replacement due to the fine size of the particles (Table 2.11) and high absorbing nature of this sand (Ganesh Prabhu et al. 2014). Similarly, Celik and Marar (1996) studied the use of quarry dust as aggregate replacement material and inferred that workability reduced with replacement for the same reason as that of foundry sand. Raman et al. (2011) investigated high strength concrete with rice husk ash combined with quarry dust. Workability improved by incorporating quarry fines. The contradicting results may be due to the differences in the source material and type of processing in different quarries. With the use of marble dust, cohesiveness improved and 10% replacement level was found to work better for SCC mix (Corinaldesi et al. 2010). Marble dust aggregate seems to be a better replacement for granite aggregate (from quarries), than river sand (Gameiro et al. 2014). Zhao et al. (2014) used iron-ore tailing as fine aggregate material in ultra-high performance concrete, and reported that 100% replacement reduced the workability and strength properties. However, 20 to 40% was identified as optimum percentage to achieve comparable results with concrete made of river sand.

With slag as aggregates, water demand of concrete reduced due to lower water absorption of the slag. In addition, smooth glassy surface of the slag reduces the friction and improves workability (Al-Jabri et al. 2009). However, due to lower absorption capacity of aggregate particles, free water content increases resulting in voids and micro-cracks. This restricts the optimum replacement percentage of 40 to 50% (Wu et al. 2010; Al-Jabri et al. 2011). With steel slag, the mix becomes sticky beyond 50% replacement level of aggregates (Qasrawi et al. 2009).

Water demand increased with increasing bottom ash content due to its porous nature (Aggarwal and Siddique 2014). Kim and Lee (2011) and Singh and Siddique (2014a) highlights the reduction of workability of the mix due to water absorbing nature of rough bottom ash. However, Andrade et al. (2009) used bottom ash to prevent plastic shrinkage. The porous aggregate acts as water reservoir which enables water supply after moulding, and the formation of plastic shrinkage cracks is avoided.

2.5.3.2 Hardened properties

Naik et al. (1994) studied the possibility of using used-foundry sand as fine aggregate in concrete and concluded that 25 to 35% replacement gave almost the same property as that of control concrete with river sand. Property enhancement is observed when foundry sand up to 15-20% is used in concrete (Naik et al. 1994; Siddique et al. 2015; Basar and Deveci Aksoy 2012). However, 100% replacement severely affects the concrete properties (Table 2.11). Presence of sawdust, wood particles and clay affect the properties negatively, thereby restricting the percentage of replacement.

There are very few works done which focus on quarry dust as aggregate material in cement composites. When used as fine aggregate in fly ash-based concrete, it exhibited excellent performance is exhibited due to combined effect of pozzolanic reactivity and micro-filling ability (Rai et al. 2014). Strength increased for concrete with up to 10% replacement of river sand, however drying shrinkage increased with increment in quarry dust (Celik and Marar 1996). Self-compacting concrete (SCC) can be produced with quarry dust as fine aggregate. However, high dosage of super plasticizer is needed to obtain the required rheological properties (Ho et al. 2002).

Table 2.11 Studies on cementitious system using untreated industrial by-products as fine aggregates

Author, Year	Material	Maximum replacement (%)	Problems in raw material	Properties of mortar/concrete with fine aggregate as recycled material			
				Without treatment			Improvement after treatment
				Fresh properties	Hardened properties	Durability properties	
Naik et al. (1994); Siddique et al. (2011); Bhardwaj and Kumar (2017)	Foundry sand	100	Mostly <2 mm in size. Presence of foundry binders like clay, oil, cement etc.,	Workability reduces due to clay/impurities present.	Strength is affected by the presence of foundry binders like clay.	Increased permeability and carbonation. Shrinkage is higher than control mix.	River sand substitution with foundry sand can be increased more than 30% without affecting the concrete properties.
Celik and Marar (1996); Rai et al. (2014)	Quarry dust	Up to 50%	D ₅₀ is less than 600 µm.	Workability reduces with increasing replacement percentages.	Strength is positively influenced up to optimum (10%) replacement percentage.	Shrinkage increases if completely replaced as surface area increases with fineness of the material.	-
Zhao et al. (2014); Gallala et al. (2017)	Mine tailing	100	Very fine particles (0.003 – 0.5 mm) Rough and angular shape.	Water demand increased.	Ultra-high strength concrete can be made with mine tailing aggregates Strength is reduced compared to control as stiffness/hardness is poor.	Mine tailing with metal wastes helps in achieving radiation shields. ITZ improved.	-
Al-Jabri et al. (2009); Qasrawi et al. (2009); Tripathi et al. (2013)	Steel slag, Copper slag,	100	Finer material (40% <0.15 mm)	Mix with steel slag is sticky with low slump. Copper slag enhances workability due to smooth surface.	Strength increased due to better bonding between steel aggregates and paste. Strength reduced with copper slag due to excessive free water.	Cavities in slag particles hold water and lowers the rate of self-desiccation shrinkage.	With elimination of particles less than 0.15 mm, concrete increased up to 25%.
Andrade et al. (2009); Kim and Lee (2011)	Bottom ash	100	Porous material with carbon content	Bleeding is observed Workability reduced due to the rough surface of bottom ash	Compressive strength was not much affected whereas flexural strength reduced linearly.	Presence of porous aggregate enables supply of water and prevents plastic shrinkage.	-

Utilization of mine waste in building blocks has been extensively studied for different mine wastes like copper mill tailings, lead-zinc mine waste (Hansen et al. 1968) and hematite tailings (Zhao et al. 2012). Yellishetty et al. (2008) suggested that mine waste of size fraction 12.5 to 4.75 mm can be used for fine aggregate replacement whereas those with less than 4.75 mm fractions can be used as brick making material. This material needs to be studied in detail for use as fine aggregate material due to large availability and has the potential to be an alternative to river sand (Table 2.3).

Mortars with slag aggregates produce better strength with corresponding increase in density. Complete replacement of river sand with slag resulted in strength reduction due to the probable presence of excessive free water that causes microcracking (Ambily et al. 2015). Elimination of particles less than 0.15 mm helps to increase the strength further (Qasrawi et al. 2009). This can be made possible by pre-treating the slag aggregates.

Porous bottom ash particles support crack propagation and thereby reduce the flexural strength of concrete (Kim et al. 2012). However, the capillary pores in bottom ash concrete get reduced with increasing curing age due to its pozzolanicity (Singh and Siddique 2014a). Up to 20% replacement of bottom ash produces concrete without any difference in strength compared to control mortar with river sand.

2.5.3.3 Durability properties

Foundry sand when replaced up to maximum of 20% in concrete shows improvement in abrasion resistance, owing to corresponding strength improvement (Singh and Siddique 2012). It is also evident from the studies of Siddique et al. (2011) that with the incorporation of foundry sand, resistance to aggressive environment improved due to the consumption of calcium hydroxide in concrete and development of additional CSH gel. The properties of foundry sand concrete could be further improved by adding metakaolin to reduce the porosity caused by the foundry sand (Siddique and Kadri 2011). Concrete made of mine waste can be applied for radiation shielding due to their dense structure (Gallala et al. 2017). With such a good application potential and huge availability, mine waste needs further detailed investigation for its better use.

BFS chemically binds chloride due to the formation of pozzolanic products and helps in corrosion prevention (Bilir 2012). Similarly, pozzolanic reaction in the vicinity of bottom

ash aggregates aids chloride binding and develops resistance to chloride ion penetration (Kim et al. 2014; Singh and Siddique 2014a). With increase in porosity and lower stiffness, drying shrinkage increases in concrete with blast furnace slag (BFS) aggregates (Valcuende et al. 2015)

When used in self-compacting concrete (SCC), it shows higher autogenous shrinkage and chemical shrinkage due to higher self-desiccation and reactivity (Valcuende et al. 2015). Concrete with bottom ash exhibited greater dimensional stability with reduced drying shrinkage. This may be due to the internal curing effect of bottom ash (Singh and Siddique 2014b). Performance of bottom ash concrete in high temperature, freeze-thaw environment and drying and wetting cycle is comparable with river sand concrete.

2.6 NEED FOR THE PRESENT STUDY

Several alternative aggregates were studied for their potential to replace river sand in mortar and concrete. Of which, crushed stone is the widely accepted alternative that lies in the category of naturally originated material (USGS 2017). This is again a depleting natural resource. However, not all naturally occurring sources provide aggregates that can be used in concrete production. Most of these materials need further processing before its use as aggregate in concrete. There are specifications to be met for a material to be used as fine aggregate in cementitious systems. However, in many places like coastal regions and deserts, choosing a particular material available is the only option to control cost involved (Elipe and López-Querol, 2014; Ratnayake et al. 2014). These materials are location specific.

Considering the alternative materials from recycling of concrete and brick or the by-products of industries such as foundry sand and bottom ash, it could be noted that each of these materials have their own disadvantages such as presence of deleterious materials like chlorides, clay, carbon, fines, etc., (Dias et al. 2008; Monosi et al. 2013). The particle size distribution of the material could also affect the properties of mortar and concrete made of these alternatives (Katz and Baum 2006). This makes most of the available alternatives suitable for a partial replacement to retain the mortar properties. Most of these alternative sources open up exciting opportunities for undertaking in-depth studies on effectiveness of different treatment methods in making them as value added replacement material for fine aggregate. Researchers also studied the applicability of different treatment methods such as sieving, washing, granulometric corrections, chemical and thermal treatment to improve the properties of the alternatives (Dias et al. 2008; Cepuritis et al. 2015, (Belferrag et al. 2016).

Excavation soil which is composed of rock, sand and clay from earth works like underground constructions, tunnelling, metro developments and mine spoils is available all over the world in huge volume (Magnusson et al. 2015), which has not been well explored for its use as fine aggregate in concrete. Raw earth or soil is widely used in various applications based on the quality of the material. It is mainly applied in backfilling, if the soil does not contain any expansive clay. Rammed earth, stabilized blocks and adobe are made of raw earth; however, poor resistant to moisture restricts their usage (Pacheco-Torgal and Jalali 2012). Plastering with soil as fine aggregate is possible by maintaining the proportion of soil to lime as minimum as possible and the application is restricted to interior walls and needs proper maintenance plan (Stazi et al. 2016). Calcined soil with high clay content are used in alkali activated materials which uses Al-Si as geopolymeric precursors (Li et al. 2016). However, the presence of fines and clay is an important draw back in considering materials like excavation soil as a fine aggregate in mortar applications. Hence, a systematic approach to convert excavation soil as a potential replacement for river sand is essential. Such an effort can help in providing value addition to excavation soils, and thereby serve as a potential alternative to river sand.

CHAPTER 3

OBJECTIVES, SCOPE AND METHODOLOGY OF THE PRESENT STUDY

3.1 GENERAL

Based on the review of literature on alternatives for fine aggregates, the problems associated with their usage as unprocessed material, processing techniques involved and the importance of the treatment methods to mitigate the problem of fines (clay + silt) in excavation soils has been emphasized. However, variation in properties of excavation soil based on its source makes it difficult to generalize the treatment for excavation soils. A specific method cannot be applied for soils from different sources emphasizing the need for different treatment methods. Hence, there is a need to study the relative effectiveness of the available treatment methods such as granulometry, dry and wet sieving, chemical and thermal treatments on soil with varying characteristics. Based on this understanding, objectives, scope and methodology for the present study have been outlined in this section.

3.2 OBJECTIVES AND SCOPE

With the main aim to utilize excavation soil as a complete replacement for fine aggregate in mortar applications, the following objectives are formulated.

- i. To study the untreated excavation soil as fine aggregate in fly ash based geopolymer mortar.
- ii. To understand the effect of stabilizers on raw and dry sieved excavation soil and the performance of stabilized soil in cement mortar
- iii. To explore the efficiency of wet sieved excavation soil as fine aggregate in cement mortar
- iv. To determine the optimum temperature and duration of thermal treatment for excavation soil and its effect in cement mortar properties

The scope is limited to the following with respect to raw materials and methods,

- i. Only the effect of fines (clay + silt) is given importance in this study. The influence of other deleterious materials is not considered.
- ii. Three types of excavation soil from local construction work, viz., low (LP), medium (MP) and high plastic (HP) soil are used and salient properties are given in Section 3.2.1. River sand (RS) is used as fine aggregate in control mortar.
- iii. Shrinkage strain measured at 90th day of exposure is taken for comparing different mixes.
- iv. OPC 53 grade cement is used as binder in cement mortar studies.

3.2.1 Soil Properties

Three types of excavation soil from local construction work has been chosen for this study. The physical and mineralogical properties of three samples of excavated soil are presented in Table 3.1. Free swell which gives the increase in volume of soil without any constraints, express the plastic behaviour of these soils and their mineralogical compositions supports their expansive/non-expansive nature. Mineralogical compositions were identified using Xpert Pro Panalytical X-ray diffraction (XRD) spectrometer with Cu-K α (1.54Å) as the source (Figure 3.1). Slit width of the detector was 6mm with step scan size of 0.02 degrees per second and counting time of 20 seconds. The raw soil samples after complete drying, were pulverized and sieved, and powder finer than 50 μ m was used for testing. The X-ray diffraction patterns were identified with powder diffraction search manual - ICSD database. These soils compose mainly of clay minerals like non-expansive kaolinite, illite and Stilbite, and, highly expansive montmorillonite which give them different level of plasticity. The acquired soils were classified according to their atterberg's limit as silty (Low plastic soil, LP), clayey sand (Medium plastic soil, MP) and fat clay (High plastic soil, HP) based on their Atterberg's limit.

Figure 3.2 indicates the gradation pattern of river sand and different soils used. The quantity of particles less than 75 μ m varies between aggregates. These particles include silt of 2-75 μ m size fraction and clay with particles less than 2 μ m size. Void ratio increases with increasing percentage of particles less than 75 μ m. High porosity and voids resulted in reduced bulk density of soils as in Table 3.1.

Table 3.1 Properties of fine aggregate

Properties	River sand	Soil-1	Soil-2	Soil-3
Major clay minerals	-	Kaolinite	Montmorillonite, Stilbite and illite	Montmorillonite and illite
<i>Atterberg's Limit</i>				
Liquid limit (%)	-	25	37	62
Plastic limit (%)	-	Non-plastic	18	18
Plasticity index (%)	-	Non-plastic	19	44
Free swell (%)	0	0	15	300
<i>Particle size</i>				
<75 μ (%)	1	29	48	58
Clay <2 μ (%)	0	15	25	41
Fines 2-75 μ (%)	0.8	14	23	17
Sand 75 μ -4.75 mm(%)	99.2	71	52	42
Soil type as per ASTM D2487	Well graded sand	Silty sand	Clayey sand	Fat clay
<i>Physical properties</i>				
Specific gravity	2.7	2.69	2.61	2.56
Bulk density (kg/m ³)	1610	1379	1112	1078
Void ratio (%)	36	48	57	61
Surface area of particles <75 μ (m ² /g)	-	220.67	265.38	303
LOI (%)	0	9.7	5.8	2.8
Identification for reference	<i>River sand (RS)</i>	<i>Low plastic soil (LP)</i>	<i>Medium plastic soil (MP)</i>	<i>High plastic soil (HP)</i>

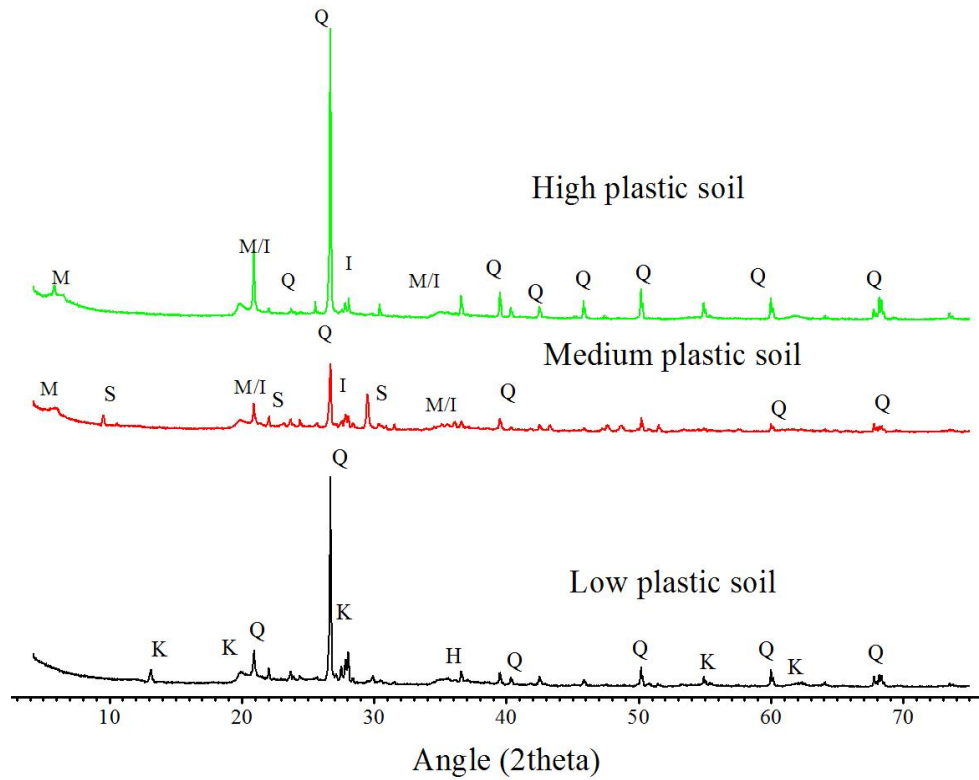


Figure 3.1 XRD plots of soils

(Q- Quartz, M- Montmorillonite, C- Calcite, I- Illite, K- Kaolinite, H- Hematite, S-Stilbite, Mu- Mullite, A-Anhydrite)

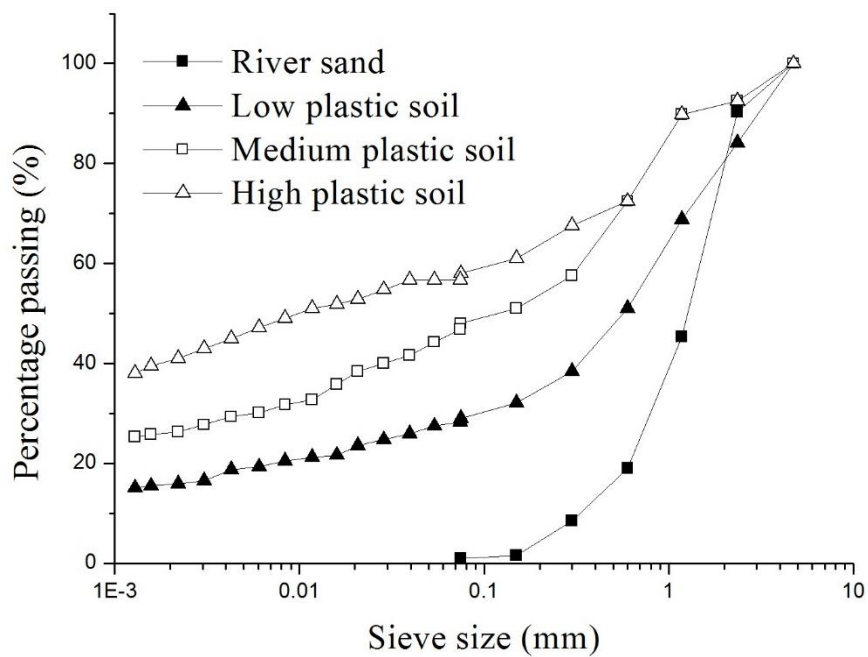


Figure 3.2 Particle size distribution curve

3.3 METHODOLOGY

After preliminary studies, the following methodology has been arrived for utilizing excavation soil as a potential replacement material for fine aggregate in mortar and concrete. Figure 3.3 gives the detailed structure of different treatment methods adopted in the present study to overcome the problem of silt and clay present in excavated soil.

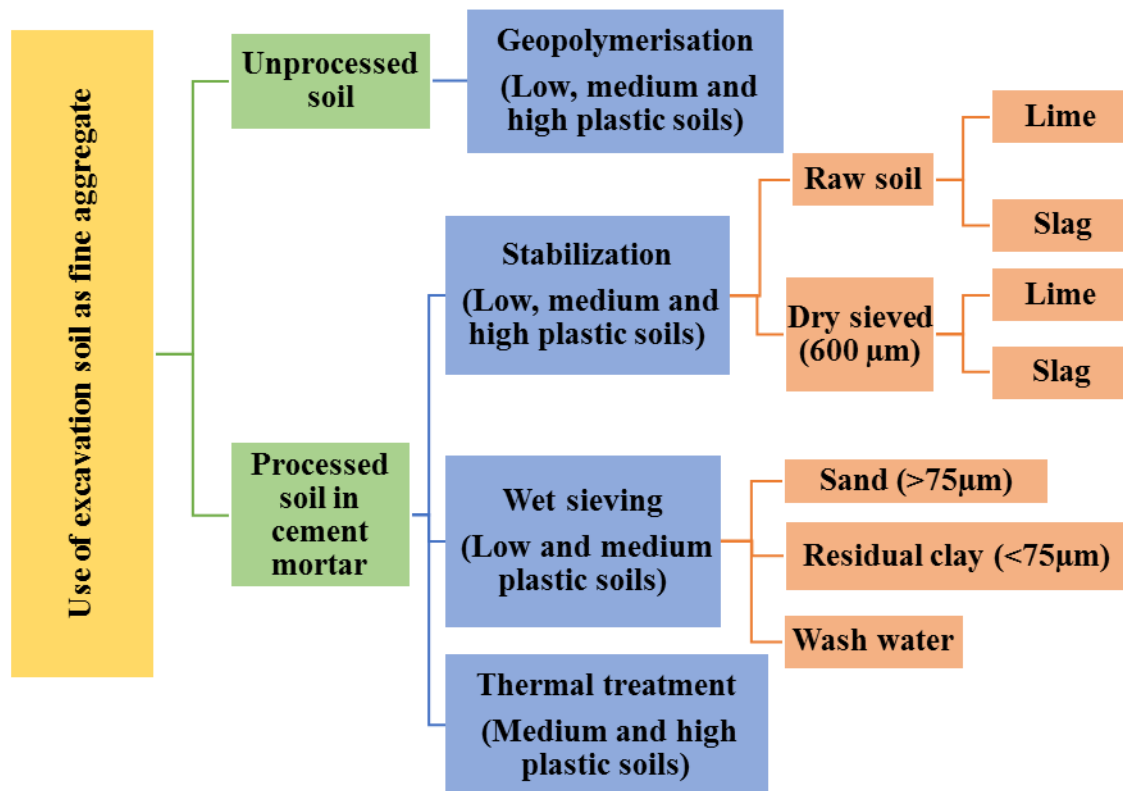


Figure 3.3 Treatment processes adopted

CHAPTER 4

PERFORMANCE OF EXCAVATION SOIL AS FINE AGGREGATE IN GEOPOLYMER MORTAR

4.1 GENERAL

Geopolymerisation is the process of using materials rich in SiO_2 and Al_2O_3 activated by alkali solution to form alumina-silicate inorganic polymers (Xu and Van Deventer, 2000). Concentration of alkali activator plays a major role in leaching of silica and alumina ions from clay particles which helps in improving strength properties (Phetchuay et al. 2016). Alkali-activated excavated clayey fine aggregates incorporating nano alumina-silicates was used by Muñoz et al. (2015) to produce compressed masonry blocks and reported reduced water absorption and increased linear shrinkage due to pore refinement. River sediments with expansive clay were recycled as construction material by geopolymerisation, which is reported to change the structure of clay due to loss of interlayer water (Li et al. 2016). Douiri et al. (2017) could make phosphoric acid geopolymeric material with calcined natural clay rich in illite mineral with dielectric properties similar to metakaolin based geopolymers. It is also possible to reduce 30% of global warming potential with the use of clay-based geopolymers compared to Portland cement-based binders (Heath et al. 2014). Table 4.1 gives an insight of geopolymerisation done on clay bearing materials.

Though some studies have been made to adopt geopolymerisation for clayey soils and use it as fillers in place of river sand, there is a need for systematic investigation on excavated soils of different plasticity to facilitate their application. Three types of plastic soils with varying plasticity and mineralogy have been chosen. The behaviour in geopolymer mortar has been studied for the effects of type of soil, fly ash to fine aggregate (F/A) ratio, curing temperature and molarity of NaOH. The interaction effect of these parameters with four different fine aggregates (river sand, low, medium and high plastic soils) were identified and discussed. Their fresh and hardened properties have been compared with conventional geopolymer mortar made with river sand as filler.

Table 4.1 Literature on production of geopolymer with clay-based materials

Author, Year	Material	Binder	Alkaline solution	Curing temperature	Salient observations
Esaifan et al. (2015)	Kaolinitic clay	-	NaOH	80°C for 24 hours	Curing at 40 to 100°C is needed to initiate the alkali activation. Mixing water just below the plastic limit of clay is used. This converts almost 50% of the clay in to reaction products.
Li et al. (2016)	Milled clayey soil	-	Na ₂ SiO ₃ / NaOH/ Na ₂ CO ₃ / Na ₂ SO ₄	Ambient, 80°C for 24 hours	Activator type and fineness of material affects the properties. Na ₂ SiO ₃ + NaOH give better results.
Ferone et al. (2015)	Calcined clay sediments	GGBS (slag)	Na ₂ SiO ₃ + NaOH	60°C for 72 hours	Calcining the clay sediments from 450 to 700°C activated the clay and dissolution increased with increasing calcination temperature.
Chen et al. (2011)	Calcined reservoir sludge	Meta-kaolin	Na ₂ SiO ₃ + NaOH	45 to 85°C for 2 hours and ambient.	Soluble silica in a material determines the polycondensation efficiency of that material. Dense microstructure is formed with higher Na ₂ O content up to optimum value.

4.2 GEOPOLYMER SYNTHESIS AND TESTING

River sand (RS) was used as fine aggregate in geopolymer mortar specimens to serve as the control. All the three acquired excavation soils, low plastic, medium plastic and high plastic soils were used as fine aggregate in geopolymer synthesis. Fly ash conforming to class-F as per ASTM C618 was used as a geopolymer binder material. The chemical composition of fly ash determined by XRF is presented in Table 4.2. Sodium hydroxide (NaOH) in combination with sodium silicate (Na₂SiO₃) was used as alkali activator. NaOH as pellets of 97% purity was dissolved in distilled water to make solution of specific concentration. Na₂SiO₃ was acquired as solution with 7.5-8.5% of Na₂O and 25-28% of SiO₂.

Table 4.2 Chemical composition of Fly ash

Oxides	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	SO ₃	Na ₂ O	K ₂ O	MgO
Composition (%)	59.32	29.95	4.32	1.28	0.16	0.16	1.44	0.61

The fly ash binder was mixed with fine aggregate in a Hobart mixer for 60 s in slow mode. The activator solution was added and the mixing was continued for 30 s each in slow and fast modes. Ingredients used for the geopolymer studies of this work are shown in Figure 4.1. The workability of fresh mortar was measured through flow table test (ASTM C1437). Flow values are reported as an average of three readings for each mix and the spread was calculated as a percentage of base diameter of the flow cone as shown in Figure 4.2. For each mix, 12 numbers of 50 mm cube specimens (3 for dry density, 3 for water absorption and 6 for compressive strength) and 3 numbers of prisms of size 160 mm x 40 mm x 40 mm for shrinkage measurements were cast. The specimens after a dissolution time of 24 hours in room temperature were oven-cured at specific temperature (60°C to 90°C) for 24 hours before demoulding. Dry density was measured after the specimen was kept in oven at a temperature of $110 \pm 5^\circ\text{C}$ for 24 hours and then allowed to cool to room temperature. Water absorption was determined as per ASTM C1403.

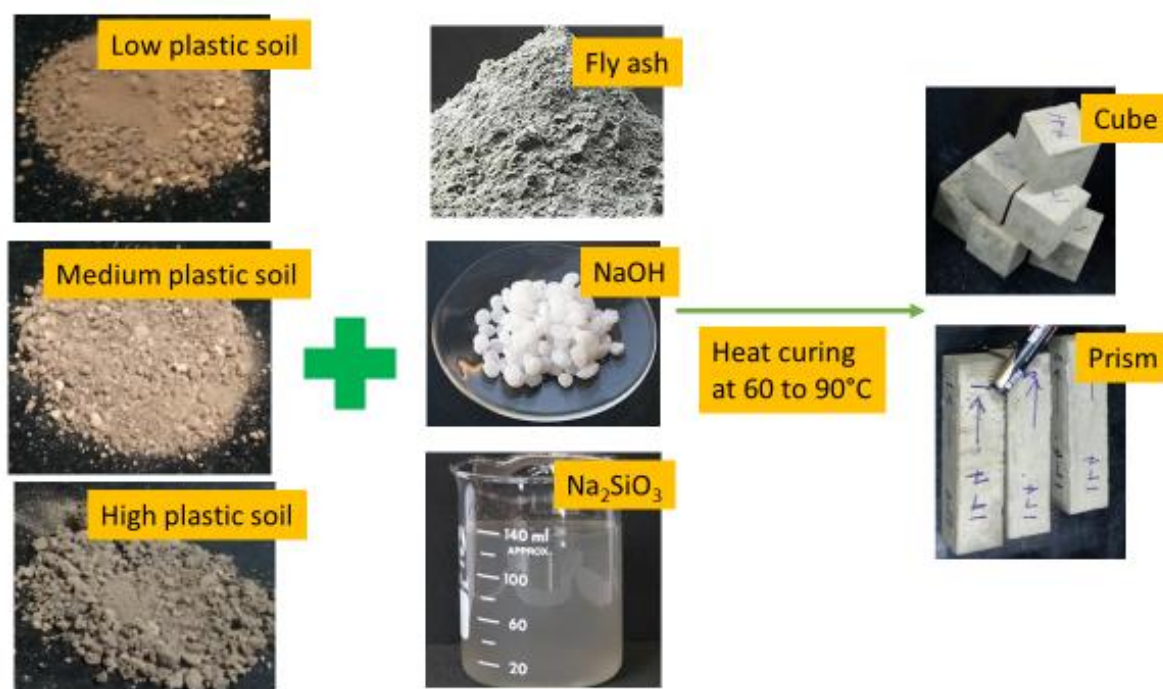


Figure 4.1 Ingredients used in geopolymer synthesis

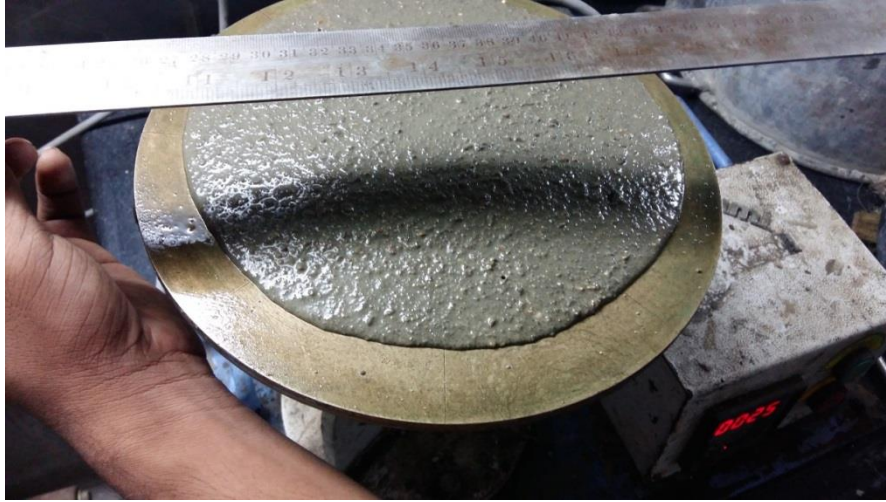


Figure 4.2 Mortar flow measurement in flow table

Shrinkage is the important property that needs to be studied in detail for the materials that involve the presence of clay in it. After oven curing, the prism specimens were kept under water for 3 days to make them saturated. This was adopted to avoid the effect of water evaporation during curing at high temperatures and to know the maximum possible shrinkage that can happen in the specimens. On the fourth day, the specimens were taken out of water, wiped and initial reading (l_1) was taken in the length comparator. The specimens were then kept in a temperature-controlled room at $23 \pm 2^\circ\text{C}$. The shrinkage measurements were made as per ASTM C157 and drying shrinkage in micro strains is calculated as per equation 4.1.

$$\text{Shrinkage strain} = \left(\frac{l_2 - l_1}{L_d} \right) \quad \text{----- (4.1)}$$

where, L_d is the actual initial length of the specimen, l_1 is the initial length reading of the specimen and l_2 is the length of the specimen at different time intervals. The readings were taken at regular intervals of time up to 28 days. Shrinkage value was taken as the average reading of 6 specimens. Based on trials, it was observed that low alkaline to solid ratio makes the mix stiff, whereas segregation and bleeding occurs with higher liquid alkaline to solids ratio. Alkaline liquid to solid (fly ash + aggregate) value increases with increasing fines and clay content for plastic soils. Smaller particles have higher surface area that reduces the workability and increases the demand for alkaline solution. Hence, alkaline liquid to solid ratio which resulted in flow of $110 \pm 5\%$ when molarity of NaOH is 8 and fly ash to fine aggregate ratio is 0.5 was used. Accordingly, alkaline liquid to solid ratio for mixes with river sand, low, medium and high plastic soils was fixed as 0.23, 0.32, 0.35, and 0.38, respectively.

4.3 DESIGN OF EXPERIMENT

4.3.1 Factors and Their Range Considered

Molarity of NaOH, curing temperature and fly ash to fine aggregate ratio are the factors considered for the present study. The upper and lower limit of each parameter has been fixed based on the preliminary studies. When $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio exceeded one, disintegration of specimens with high plastic soil was observed. Though hardened gel was formed and the specimens retained its shape after demolding, the specimen disintegrated when exposed to water. In the present study, sodium silicate/sodium hydroxide ratio was fixed as 1.

4.3.1.1 Molarity of NaOH

A minimum of 6M NaOH is needed for the effective activation of geopolymer precursors. Use of a molarity greater than 10 caused the excess solution to ooze out of the specimens and caused efflorescence in the case of high plastic soil. Hence, the molarity of sodium hydroxide was varied between 6 and 10.

4.3.1.2 Curing temperature and duration

Temperature was varied between 60 to 90°C as in most of the earlier geopolymer studies. The duration of curing has been fixed as 24 hours.

4.3.1.3 Fly ash/ fine aggregate ratio

Use of fly ash to fine aggregate (F/A) ratio greater than one led to disintegration of specimens with high plastic soil when exposed to water. To make the design uniform, the range of ash/aggregate ratio that works for all the four types of soil was varied between 0.5 and 1.

The coded and un-coded values of the parameters are given in Table 4.3.

Table 4.3 Factors in un-coded values for the coded values of different parameters

Notation	Parameter	Unit	Coded values				
			+2	+1	0	-1	-2
			Un-coded values				
X_1	Molarity of NaOH	M	6	7	8	9	10
X_2	Curing temperature	°C	60	67.5	75	82.5	90
X_3	Fly ash/Aggregate ratio (F/A)	-	0.5	0.63	0.75	0.88	1

4.3.2 Experimental data points

To study the influence of each of these three parameters, 20 sets of experiments were designed as given in Table 4.4, using Response Surface Methodology (RSM) of central composite design (Montgomery 2012), for each type of fine aggregates (river sand, low, medium and high plastic soils).

Table 4.4 Experimental design based on central composite design for three selected parameters

S.No	Molarity of NaOH (6M to 10M)		Curing temp (60°C to 90°C)		Fly ash/ fine aggregate ratio (0.5 to 1)	
	Actual	Coded	Actual	Coded	Actual	Coded
1	8.00	0	90	2	0.75	0
2	8.00	0	75	0	0.75	0
3	6.81	-1	66	-1	0.90	1
4	8.00	0	75	0	0.75	0
5	8.00	0	75	0	0.75	0
6	6.81	-1	84	1	0.60	-1
7	9.19	1	66	-1	0.60	-1
8	8.00	0	75	0	0.50	-2
9	8.00	0	75	0	1.00	2
10	8.00	0	60	-2	0.75	0
11	9.19	1	66	-1	0.90	1
12	9.19	1	84	1	0.60	-1
13	6.81	-1	66	-1	0.60	-1
14	8.00	0	75	0	0.75	0
15	6.81	-1	84	1	0.90	1
16	6.00	-2	75	0	0.75	0
17	10.00	2	75	0	0.75	0
18	8.00	0	75	0	0.75	0
19	9.19	1	84	1	0.90	1
20	8.00	0	75	0	0.75	0

4.3.3 Precision and Reliability of Models

Based on the experimental data points, the analysis was carried out using Statistical Analysis Software (SAS) for each type of soil separately, to arrive at the response surface equations (SAS Release 8.02). The statistical model is validated based on R^2 , P-value and F-values in the output data which is presented in Table 4.5. R^2 Value is observed to be greater than 0.9 for most of the responses. This gives confidence in applying the predicted model and can be used for prediction of responses for various combinations of parameters used, within the range studied. The models are also statistically significant as P-value < 0.05.

Table 4.5 Validation of statistical model for geopolymer mortar with different fine aggregate

Type of fine aggregate	Properties	Dry density (kg/m ³)	Compressive strength (MPa)	Water absorption (%)	Drying shrinkage (µstrains)
River sand	R^2	0.9946	0.9913	0.9317	0.8009
	F-Value	206.38	127.02	15.15	4.47
	P-Value	< 0.0001	< 0.0001	0.0001	0.0142
Low plastic soil	R^2	0.9758	0.959	0.89	0.7854
	F-Value	44.85	25.96	8.99	4.07
	P-Value	< 0.0001	< 0.0001	0.001	0.0196
Medium plastic soil	R^2	0.9615	0.9601	0.7755	0.8043
	F-Value	27.78	26.73	3.84	4.57
	P-Value	< 0.0001	< 0.0001	0.0238	0.0132
High plastic soil	R^2	0.9863	0.9306	0.8461	0.8109
	F-Value	79.94	14.89	6.11	4.76
	P-Value	< 0.0001	< 0.0001	0.0046	0.0114

4.4 DISCUSSION OF RESULTS

4.4.1 Workability

The variation in workability of the mix for the influence of molarity of NaOH and fly ash to fine aggregate ratio is presented in Figure 4.3. An increase in sodium hydroxide concentration reduces the workability of geopolymer mix irrespective of the type of fine aggregate used. This is attributed to the increase in viscosity of the alkaline solution with molarity of NaOH. At higher fly ash to fine aggregate (F/A) ratio, i.e., with increase in finer fly ash content, the mix require more alkaline solution for wetting which resulted in linear reduction in workability. At lower molarities of NaOH, geopolymer mortar with clay having higher surface area results in high amount of adsorbed water and less free layer water as compared to mixes with river sand. According to Chuah et al. (2016), separation of particles need free water which gives better workability. Presence of free water helps in better workability of mortar with river sand and low plastic soil compared to medium and high plastic soils.

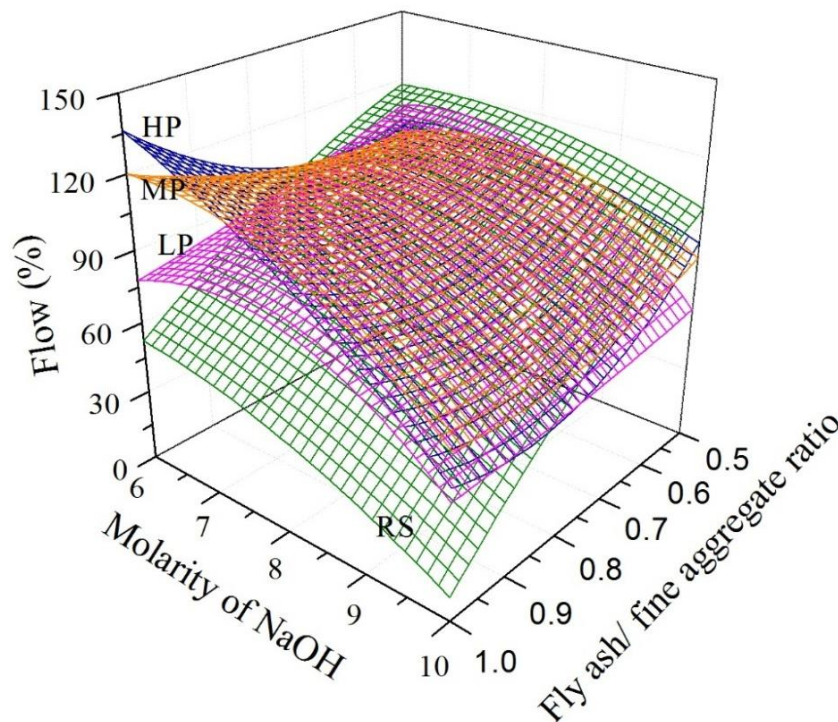


Figure 4.3 Response surface of flow of geopolymer mortar

4.4.2 Dry Density

The response surface equations for dry density of geopolymer mortar are summarised in Table 4.6. The ANOVA represented in Table 4.7 indicates that molarity of NaOH and curing temperature are the main influential factors that affects the dry density of geopolymer mortar, irrespective of type of fine aggregate used. Fly ash to fine aggregate ratio affects the dry density of geopolymer mortar with high plastic soil. The interaction is not significant in all the four types of fine aggregate and quadratic effect of these parameters affects the dry density of geopolymer mortar with river sand. The response surface equations were used to draw response surface plot to study the relative behaviors of different fine aggregates.

Table 4.6 Response surface model equations for dry density

Type of fine aggregate	Dry density in kg/m ³
River sand	$+1661.84035 + (36.86099 * X_1) - (2.46459 * X_2) - (426.37551 * X_3) - (0.35025 * X_1 * X_2) - (7.33977 * X_1 * X_3) + (0.62603 * X_2 * X_3) + (2.43289 * X_1^2) + (0.041607 * X_2^2) + (335.54472 * X_3^2)$
Low plastic soil	$+1004.86784 + 57.19627 * X_1 + 7.01081 * X_2 + 64.44366 * X_3 - 0.22722 * X_1 * X_2 - 3.33754 * X_1 * X_3 + 0.65431 * X_2 * X_3 - 0.81751 * X_1^2 - 0.034378 * X_2^2 - 56.32040 * X_3^2$
Medium plastic soil	$+1323.66010 + (15.87354 * X_1) + (1.55145 * X_2) + (68.80972 * X_3) - (0.049497 * X_1 * X_2) + (1.27279 * X_1 * X_3) - (0.39598 * X_2 * X_3) + (0.27885 * X_1^2) - (1.70942E-003 * X_2^2) - (22.15391 * X_3^2)$
High plastic soil	$+1406.50142 + (11.67468 * X_1) - (0.46445 * X_2) - (52.74648 * X_3) - (0.10607 * X_1 * X_2) + (7.77817 * X_1 * X_3) + (1.79134 * X_2 * X_3) + (0.92859 * X_1^2) + (3.17497E-003 * X_2^2) - (68.57012 * X_3^2)$

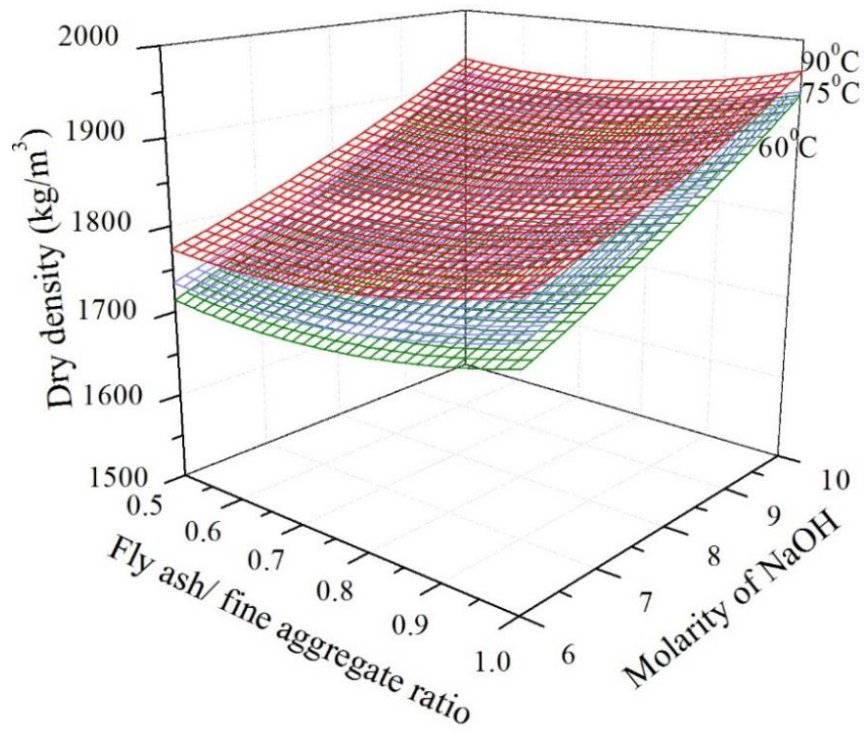
*Note: Molarity -X₁; Temperature-X₂; Fly ash/Fine aggregate - X₃

Table 4.7 ANOVA for dry density

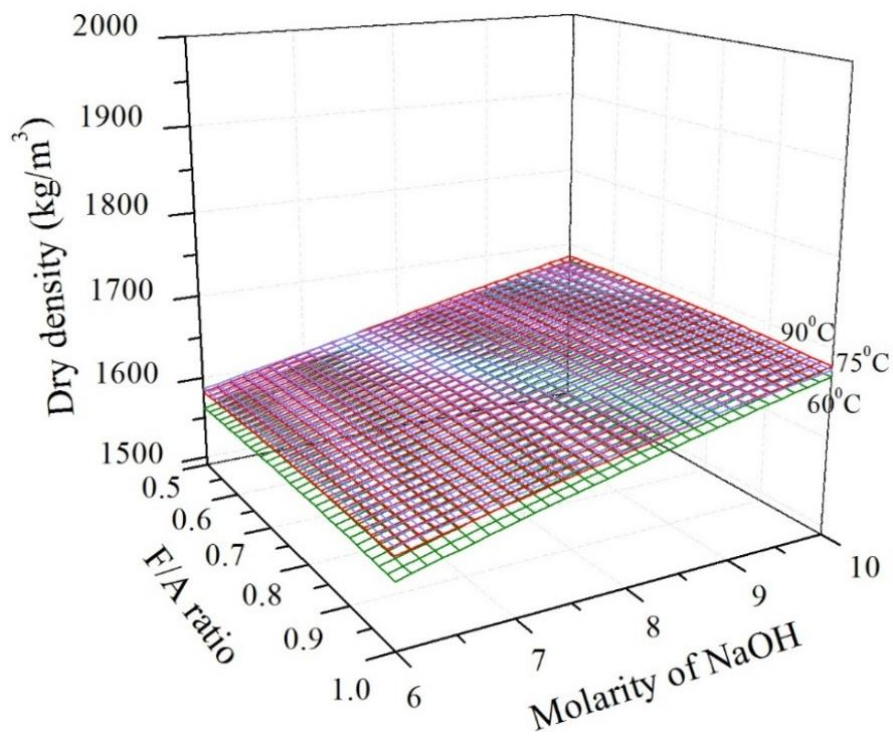
Response	Factor	River sand		Low plastic soil		Medium plastic soil		High plastic soil	
		F-value	P-value	F-value	P-value	F-value	P-value	F-value	P-value
Main effects	X_1^*	1651.89	< 0.0001	387.36	< 0.0001	231.34	< 0.0001	666.14	< 0.0001
	X_2^*	100.01	< 0.0001	10.03	0.0100	15.27	0.0029	16.15	0.0024
	X_3^*	56.60	< 0.0001	0.05	0.8198	3.02	0.1130	29.32	0.0003
Interaction effects	X_1X_2	4.87	0.0517	1.54	0.2424	0.09	0.7760	0.59	0.4616
	X_1X_3	0.59	0.4585	0.09	0.7672	0.02	0.9028	0.88	0.3715
	X_2X_3	0.24	0.6325	0.20	0.6643	0.09	0.7760	2.61	0.1371
Quadratic effects	X_1^2	7.53	0.0207	0.64	0.4423	0.09	0.7742	1.44	0.2580
	X_2^2	6.97	0.0247	3.58	0.0877	0.01	0.9210	0.05	0.8222
	X_3^2	34.98	0.0001	0.74	0.4094	0.13	0.7221	1.92	0.1965

*Note: Molarity - X_1 ; Temperature- X_2 ; Fly ash/Fine aggregate - X_3

The variation in dry density with different curing temperature for geopolymer mortar with river sand and plastic soils are shown in Figure 4.4 (a) to (d). For any molarity and F/A ratio, the effect of curing temperature has marginal effect on dry density i.e., less than 100 kg/m³. For a constant fly ash to fine aggregate ratio, an increase in molarity resulted in linear increase in dry density. This is attributed to the increase in polymerisation with the increase in molarity of NaOH. Consequently, the porosity of the geopolymer matrix is reduced as the polymerisation product of reactive ingredients fills up the pores. For a given molarity of NaOH, variation in F/A ratio does not contribute to appreciable variation in dry density.

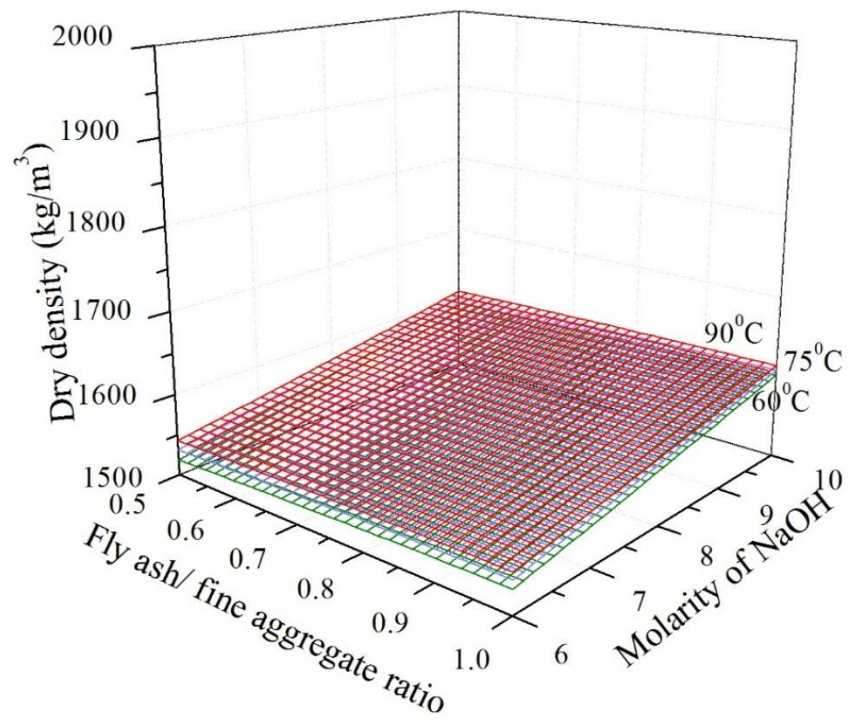


(a) With river sand

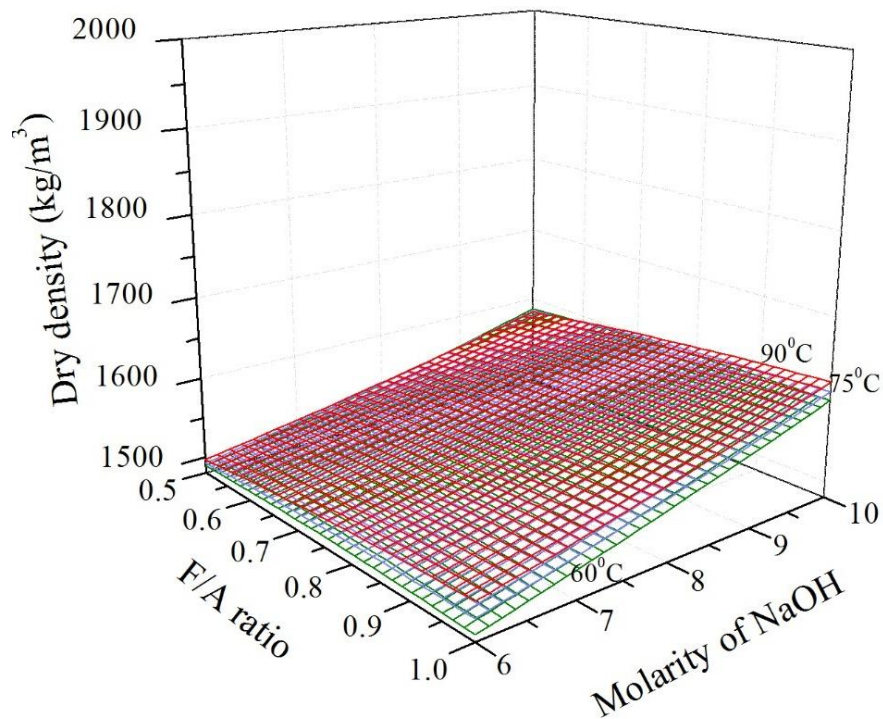


(b) With low plastic soil

Figure 4.4 continued...



(c) With medium plastic soil



(d) With high plastic soil

Figure 4.4 Dry density of geopolymer mortar

For a constant molarity and F/A ratio, Figure 4.5 shows a comparison of dry density of geopolymer mortar with different fine aggregates. The dry density reduces with the increasing plasticity of the soil used and the variation is much higher between geopolymer mortar with river sand and that of plastic soils. This can be attributed to the reduction in bulk density of plastic soils with increasing proportion of fines content (clay + silt). Though these fine particles may help in improving the workability of the mix, it reduces density by poor packing.

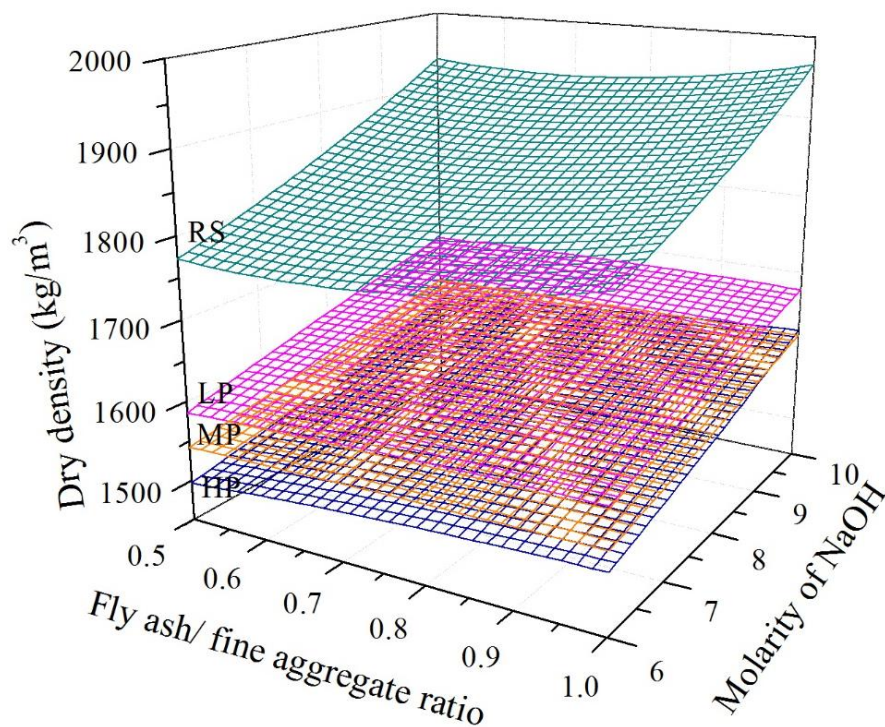


Figure 4.5 Dry density of geopolymer mortar with different fine aggregates cured at 90°C

4.4.3 Compressive Strength

Table 4.8 shows the response surface equation for compressive strength of geopolymer mortar with different fine aggregates. It can be observed from Table 4.9, that all the three parameters, molarity of NaOH, temperature and fly ash to fine aggregate ratio, significantly influence the compressive strength of geopolymer mortar and there is no interaction effect for geopolymer mortar with soil.

Table 4.8 Response surface model equations for compressive strength

Type of fine aggregate	Compressive strength in MPa
River sand	$-132.25735 + (22.18955 * X_1) + (0.73487 * X_2) + (62.33507 * X_3) - (0.071536 * X_1 * X_2) + (0.23335 * X_1 * X_3) + (0.44783 * X_2 * X_3) - (0.80052 * X_1^2) - (3.72039E-003 * X_2^2) - (50.59340 * X_3^2)$
Low plastic soil	$-13.35764 + (5.84751 * X_1) - (0.051292 * X_2) - (4.43398 * X_3) - (0.016971 * X_1 * X_2) - (0.35355 * X_1 * X_3) + (0.22816 * X_2 * X_3) - (0.20675 * X_1^2) + (2.13347E-004 * X_2^2) - (5.23195 * X_3^2)$
Medium plastic soil	$-27.19780 - (0.68989 * X_1) + (0.42925 * X_2) + (17.08891 * X_3) - (0.030406 * X_1 * X_2) + (3.25269 * X_1 * X_3) - (0.23570 * X_2 * X_3) + (0.18577 * X_1^2) + (5.24832E-004 * X_2^2) - (7.79060 * X_3^2)$
High plastic soil	$-11.47386 - (0.87277 * X_1) + (0.36756 * X_2) - (3.07342 * X_3) - (0.047848 * X_1 * X_2) + (2.16375 * X_1 * X_3) + (0.13388 * X_2 * X_3) + (0.26673 * X_1^2) - (6.58090E-004 * X_2^2) - (10.92912 * X_3^2)$

*Note: Molarity - X_1 ; Temperature- X_2 ; Fly ash/Fine aggregate - X_3

Table 4.9 ANOVA for compressive strength

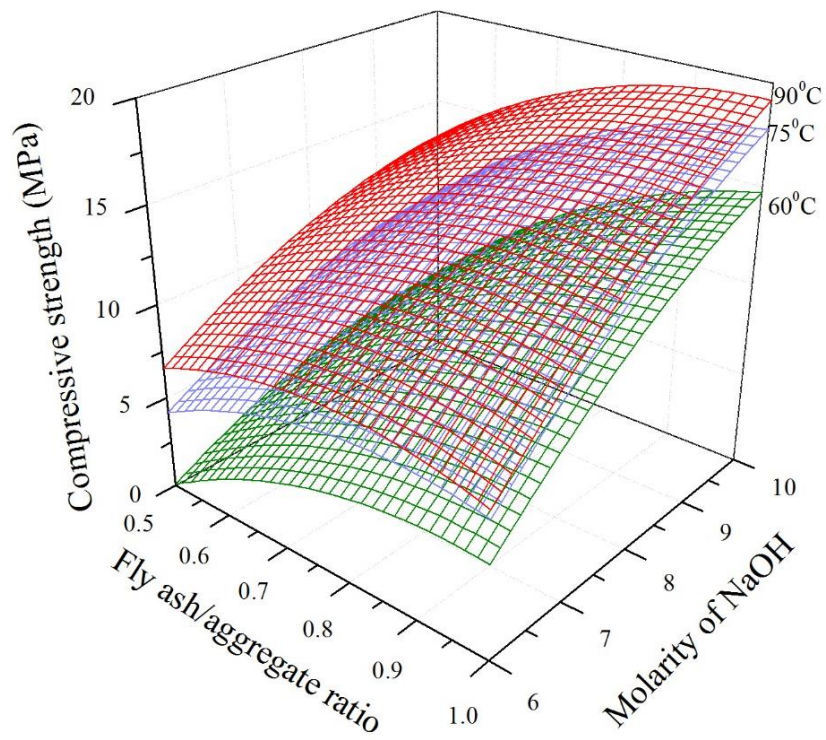
Response	Factor	River sand		Low plastic soil		Medium plastic soil		High plastic soil	
		F-value	P-value	F-value	P-value	F-value	P-value	F-value	P-value
Main effects	X_1^*	557.71	< 0.0001	126.71	< 0.0001	122.54	< 0.0001	68.84	< 0.0001
	X_2^*	257.77	< 0.0001	29.58	0.0003	73.67	< 0.0001	19.65	0.0013
	X_3^*	257.01	< 0.0001	67.17	< 0.0001	40.22	< 0.0001	32.07	0.0002
Interaction effects	X_1X_2	1.51	0.2477	0.73	0.4121	0.08	0.7866	0.09	0.7663
	X_1X_3	12.48	0.0054	2.00	0.1876	2.44	0.1492	0.55	0.4737
	X_2X_3	6.84	0.0258	0.67	0.4314	0.03	0.8708	2.00	0.1881
Quadratic effects	X_1^2	16.59	0.0022	6.53	0.0286	1.24	0.2921	1.78	0.2112
	X_2^2	9.14	0.0128	0.53	0.4853	0.02	0.8820	3.92	0.0759
	X_3^2	32.80	0.0002	0.11	0.7473	0.24	0.6341	4.67	0.0560

*Note: Molarity - X_1 ; Temperature- X_2 ; Fly ash/Fine aggregate - X_3

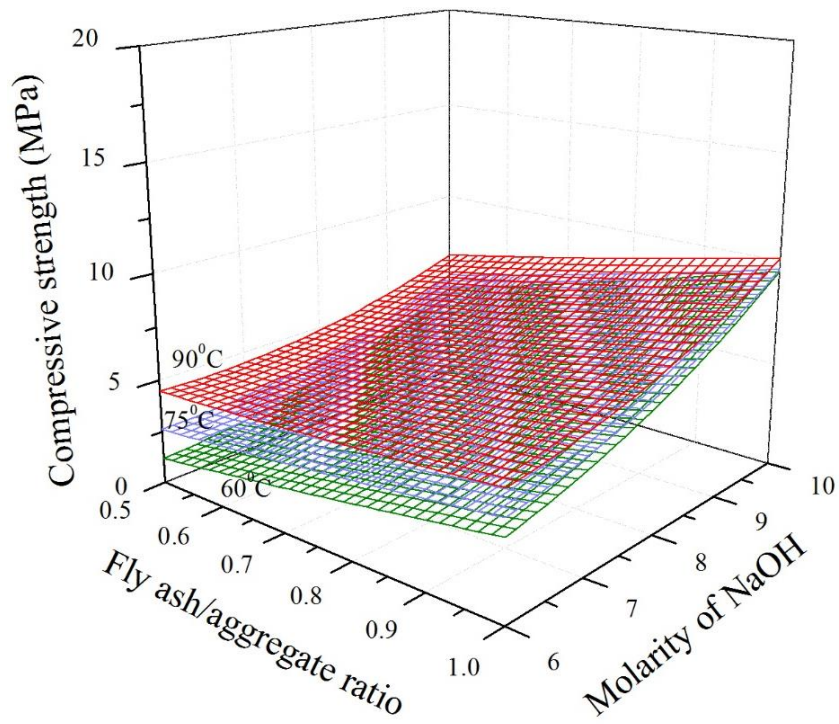
Figure 4.6 (a) to (d) presents the response surface curves of compressive strength of geopolymer mixes with river sand, low, medium and high plastic soils. For any molarity and F/A ratio, increasing curing temperature from 60°C to 90°C increases the compressive strength of the geopolymer mortar considerably, viz., 63, 49, 94 and 47% with river sand, low, medium and high plastic soils respectively.

The effect of curing temperature is highly pronounced in mix with medium plastic soil (Figure 4.6), as its reactive clay combination helps in geopolymerisation at higher temperature and results in continuous strength gain for all combination of F/A ratio, temperature and molarity of NaOH. Due to the presence of stilbite type of clay (Figure 3.1), which participates in geopolymerisation, the higher temperature curing helps in better activation. XRD graph in Figure 3.1 clearly shows the presence of Stilbite and other clay minerals which participated in geopolymerisation and disappeared in XRD graphs of mortar samples shown in Figure 4.7. This is supported by (Xu and Van Deventer 2000), in their studies on 15 different Al-Si minerals; stilbite participated in geopolymerisation process better than other clay minerals.

As the clay is non-reactive in low plastic soil, the effect of temperature at higher molarities was not significant compared to mix with medium plastic soil (Figure 4.6b). In high plastic soil, the layered bentonite clay absorbs much of alkaline solution and the strength improvement is not notable beyond a curing temperature of 75°C (Figure 4.6d). Ekaputri et al. (2014) reported similar absorption of alkali solution by clay flakes resulting in denser paste with less strength. Specific surface area of clay particles affects the strength of geopolymer as it defines the amount of geopolymer binder phase needed to cover the unreacted particles (Dietel et al. 2017). This explains the reduction in strength with increasing specific surface area of high plastic soil compared to low and medium plastic soil geopolymer mortars. The variation in strength between geopolymer mortars with different clays has been discussed separately through a correlation to its density in the following section.

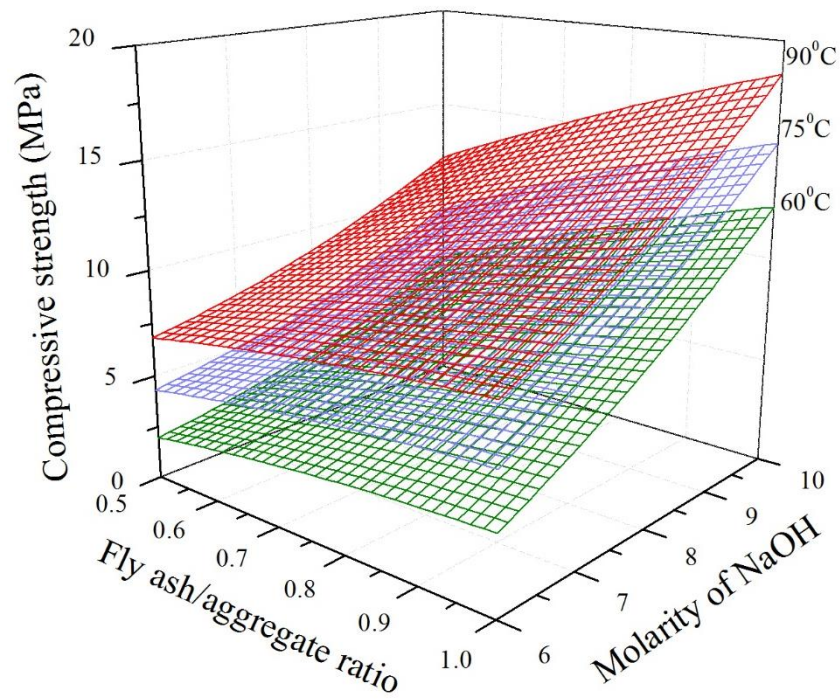


(a) With river sand

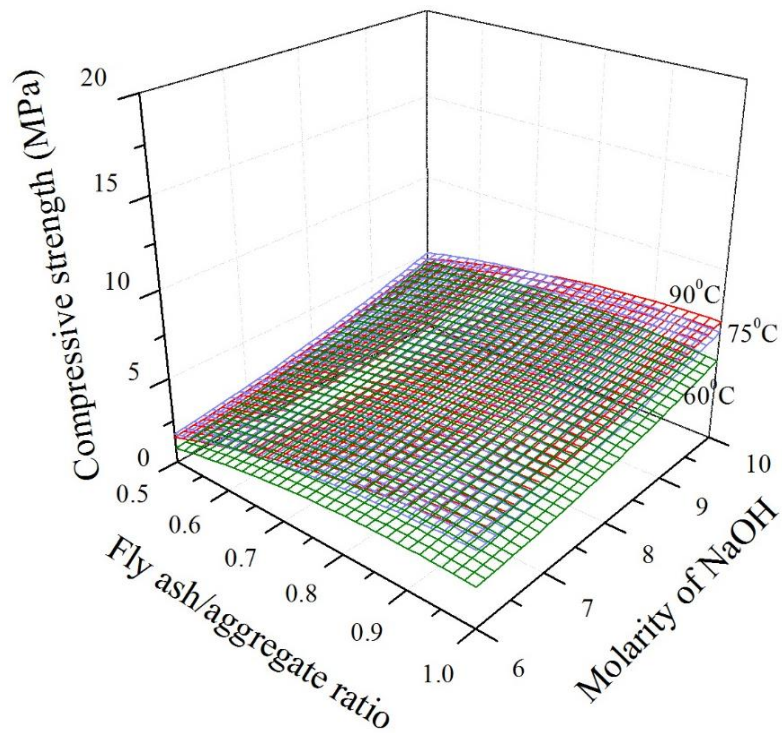


(b) With low plastic soil

Figure 4.6 continued...



(c) With medium plastic soil



(d) With high plastic soil

Figure 4.6 Compressive strength of geopolymer mortar

From Figure 4.6, it can be observed that with a given type of fine aggregate in geopolymer mortar the compressive strength increases linearly with increasing molarity for geopolymer mortar with different fine aggregate. The increase in molarity of NaOH from 6 to 10 resulted in the percentage increase in compressive strength in the range of 78% to 128%. The solubility of alumino-silicate oligomers to form dense Si-O-Al network increases with molarity of NaOH, resulting in a strength increment (Görhan and Kürklü 2014). At any molarity of NaOH and curing temperature, the increase in F/A ratio increases the fly ash content in the mix resulting in strength enhancement (Figure 4.6). The increase in fly ash content that helps in better geopolymerisation which tend to a strength improvement of 34% to 88% for different fine aggregates used (Figure 4.6a and b).

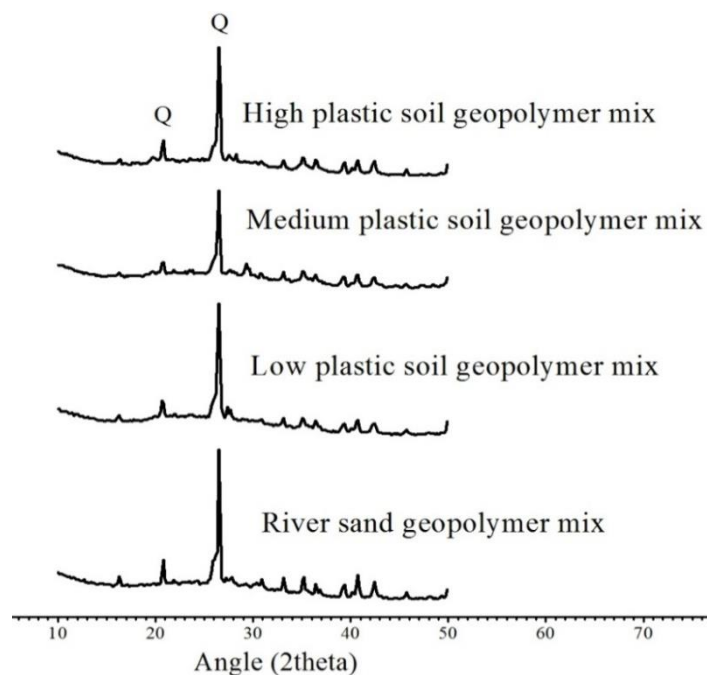


Figure 4.7 XRD plots of geopolymer mortar

4.4.3.1 Influence of type of fine aggregate

Designed experimental data representing variation in compressive strength with dry density of geopolymer mortar is shown in Figure 4.8. For a given strength, use of river sand resulted in higher density compared to geopolymer mortars with plastic soils as fine aggregates. Mixes with clayey fine aggregates helps to achieve better strength at a lower dry density range. i.e., considering a compressive strength of 9 MPa, geopolymer mortar with river sand has a dry density of 1850 kg/m^3 , whereas the dry density of mixes with plastic soils ranges between 1550 to 1650 kg/m^3 (Figure 4.9b).

Geopolymer mortar with river sand, without any clay to absorb the solution, has more solution to react resulting in higher strength (6.5 to 19 MPa) while higher specific gravity of river sand and low fines content leads to higher density. In high pH environment, clay particles acquire net negative charge causing inter-particle repulsion and disperse in the matrix causing reduction in density (Schofield and Samson, 1954). The proper distribution of clay particles also encourages the geopolymerisation reaction improving the mortar strength. Mortar with non-reactive clay in low plastic soil helps in density reduction with the range of 1575 to 1675 kg/m³, however does not participate in geopolymerisation process reaching a maximum strength of only 10 MPa.

Mortar with medium plastic soil, as mentioned earlier, performs better than other geopolymer mortars achieved compressive strength in the range of 9 to 18.5 MPa with lower density value (1545 to 1600kg/m³) compared to the mix with river sand. Mix with high plastic soil with fat clay which was not active in geopolymerisation process compared to clay mineral combination in medium plastic soil, achieved compressive strength in the range of 6.8 to 10 MPa with wide density range of 1510 to 1630 kg/m³.

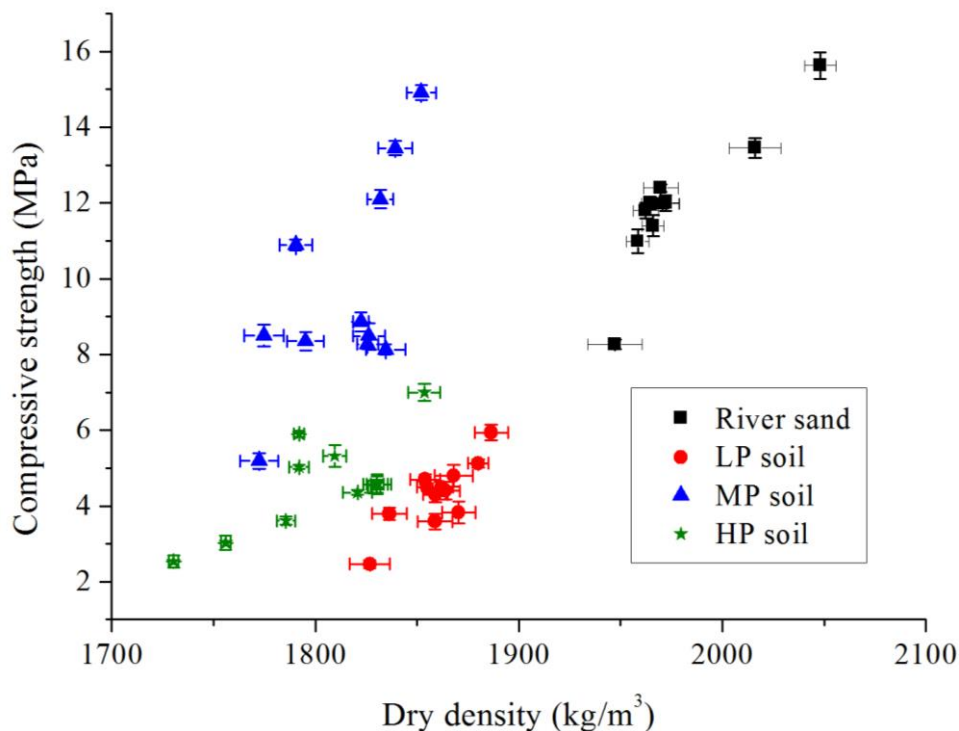
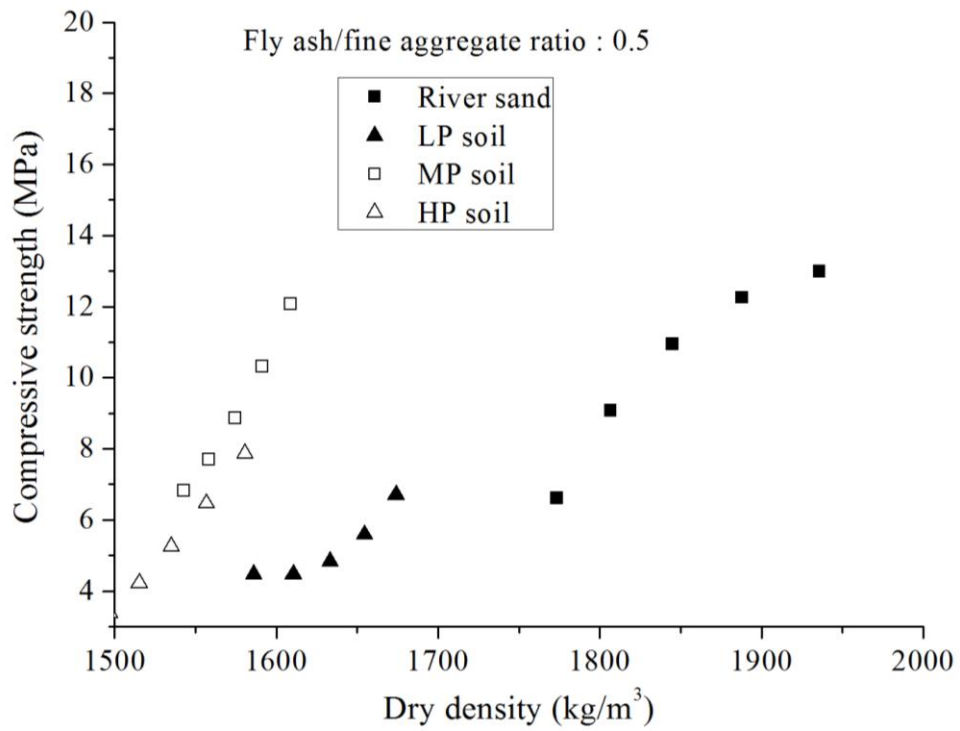
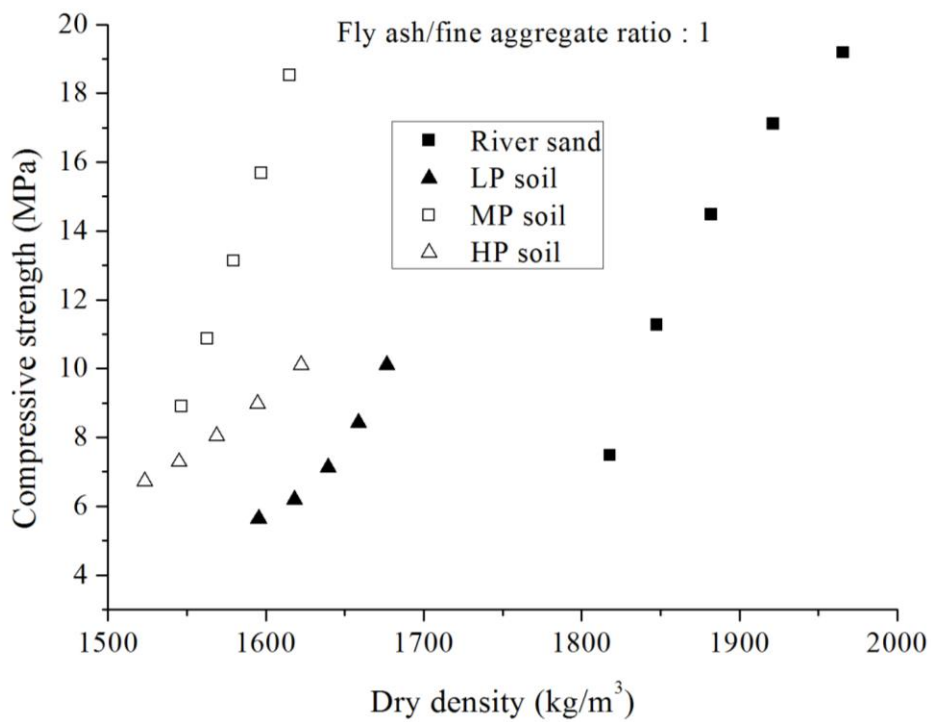


Figure 4.8 Experimental data showing variation in compressive strength with density of geopolymer mortar

(curing temperature: 75°C; F/A ratio: 0.75)



(a) F/A ratio of 0.5



(b) F/A ratio of 1

Figure 4.9 Variation in compressive strength with density of geopolymer mortar at curing temperature of 90°C

4.4.4 Water Absorption

The response surface equation for water absorption of geopolymer mortar with different fine aggregates are summarised in Table 4.10. It could be observed from the ANOVA table (Table 4.11) that, molarity of NaOH is the only factor that affects water absorption of fly ash based geopolymer mortar.

Table 4.10 Response surface model equations for water absorption

Type of fine aggregate	Water absorption in %
River sand	$+85.12103 - (11.60069 * X_1) - (0.28197 * X_2) - (31.82067 * X_3) + (7.07107E-003 * X_1 * X_2) + (0.67882 * X_1 * X_3) - (0.25267 * X_2 * X_3) + (0.65240 * X_1^2) + (1.73150E-003 * X_2^2) + (31.59342 * X_3^2)$
Low plastic soil	$+37.64400 - (4.73893 * X_1) + (0.14397 * X_2) - (9.45789 * X_3) + (2.82843E-003 * X_1 * X_2) - (0.90510 * X_1 * X_3) - (0.092395 * X_2 * X_3) + (90.31762 * X_1^2) - (9.75618E-004 * X_2^2) + (16.24778 * X_3^2)$
Medium plastic soil	$+24.61098 - (1.60885 * X_1) + (0.12250 * X_2) + (2.26504 * X_3) + (1.88562E-003 * X_1 * X_2) - (0.014142 * X_1 * X_3) - (0.056569 * X_2 * X_3) + (0.091074 * X_1^2) - (8.03131E-004 * X_2^2) + (1.10873 * X_3^2)$
High plastic soil	$-3.23857 + (1.82193 * X_1) + (0.63290 * X_2) - (1.91394 * X_3) - (7.30677E-003 * X_1 * X_2) + (0.29698 * X_1 * X_3) - (0.058454 * X_2 * X_3) - (0.10565 * X_1^2) - (3.83386E-003 * X_2^2) + (1.87812 * X_3^2)$

*Note: Molarity - X_1 ; Temperature- X_2 ; Fly ash/Fine aggregate - X_3

Experimental data plot shows that, increase in molarity of NaOH reduces the water absorption linearly for all the mixes with different fine aggregates (Figure 4.10). There is not much effect in water absorption when fly ash to fine aggregate ratio is increased from 0.5 to 1 as represented in response surface plot (Figure 4.11 a and b). The percentage increase in water absorption of mixes with plastic soils with respect to mix with river sand varies from 50% to 80%. With the addition of alkali solution, the clay double layer becomes thicker with Na^+ monovalent ions due to their larger radius and it helps in dispersing the clay in the system (Nyamangara et al. 2007). Though this makes the clay particles easily available for activation, alkali activation with clayey soil is not complete as not all clay particles behave same in alkaline medium.

Table 4.11 ANOVA for water absorption

Response	Factor	River sand		Low plastic soil		Medium plastic soil		High plastic soil	
		F-value	P-value	F-value	P-value	F-value	P-value	F-value	P-value
Main effects	X_1^*	76.89	< 0.0001	26.15	0.0005	21.14	0.0010	23.59	0.0007
	X_2^*	0.86	0.3763	2.96	0.1163	0.25	0.6246	7.96	0.0181
	X_3^*	3.73	0.0824	1.67	0.2255	1.68	0.2246	4.04	0.0720
Interaction effects	X_1X_2	0.13	0.7237	0.07	0.7938	0.10	0.7617	0.53	0.4843
	X_1X_3	0.34	0.5735	2.05	0.1828	0.00	0.9697	0.24	0.6334
	X_2X_3	2.64	0.1354	1.20	0.2988	1.37	0.2696	0.53	0.4843
Quadratic effects	X_1^2	36.06	0.0001	29.09	0.0003	7.26	0.0225	3.53	0.0896
	X_2^2	0.80	0.3911	0.87	0.3733	1.79	0.2111	14.71	0.0033
	X_3^2	20.64	0.0011	18.59	0.0015	0.26	0.6195	0.27	0.6130

*Note: Molarity - X_1 ; Temperature- X_2 ; Fly ash/Fine aggregate - X_3

Clay minerals like kaolinite and montmorillonite are strongly attacked by NaOH solution whereas illite remains unaltered to some extent (Carroll and Starkey 1971). In mixes with plastic soils, presence of unreacted clay particles resulted in weak bonding between the particles and the gel matrix (Soutsos et al. 2016). These unreacted particles will be more in plastic soils as compared to the river sand and increases with increasing plasticity resulting in increased water absorption and reduced density. Alkali treatment of clay particles results in increased specific surface area and water adsorption capacity and these increases with increasing concentration of alkali ions (Sivapullaiah 2005). The surface area to be covered by geopolymer matrix increases with increasing specific surface area of clay. However, high paste content in geopolymer mortar results in strength reduction and increases water permeability.

For a given density, the water absorption of mix with low plastic soil is the least, followed by mixes with medium plastic and high plastic soil (Figure 4.11). Low plastic soil with density in the range of 1575 to 1675 kg/m^3 has minimum water absorption of 18% to 20% compared to mixes with other plastic soils. Whereas, medium and high plastic soils with density range of 1500 to 1650 kg/m^3 have absorption percentages ranging between 19% and 23.5%. As density reduces, the water absorption increases for geopolymer mortars with different fine aggregates.

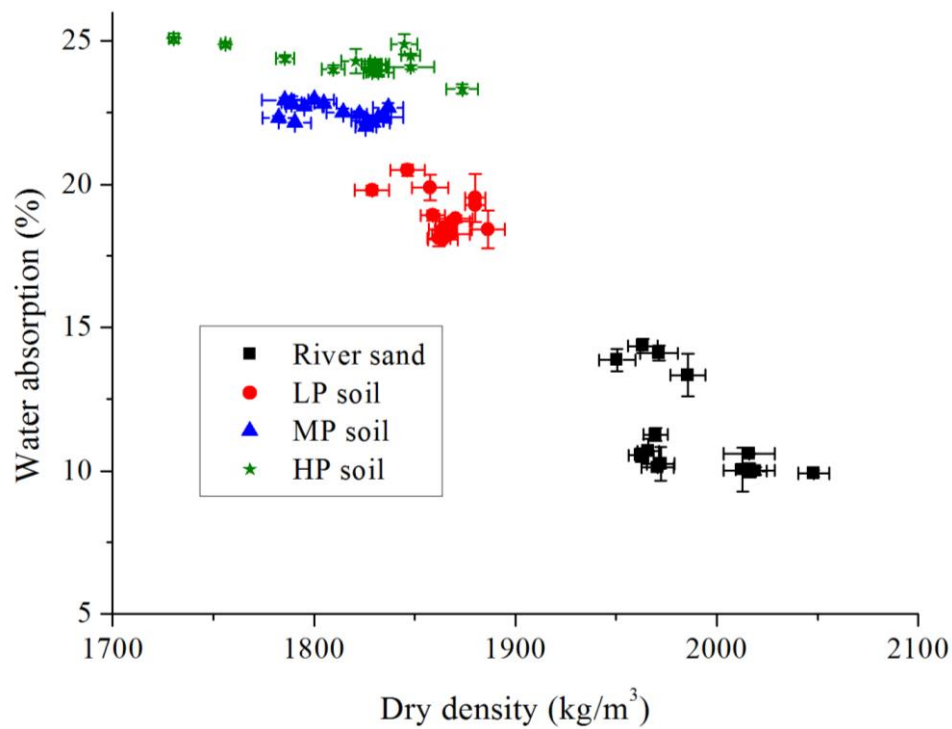
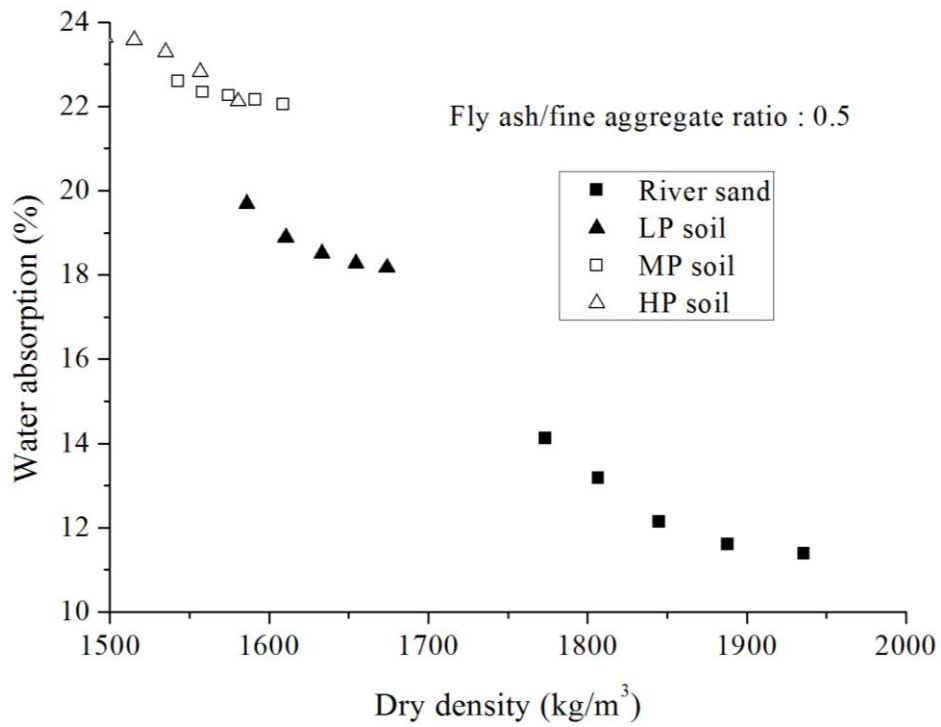
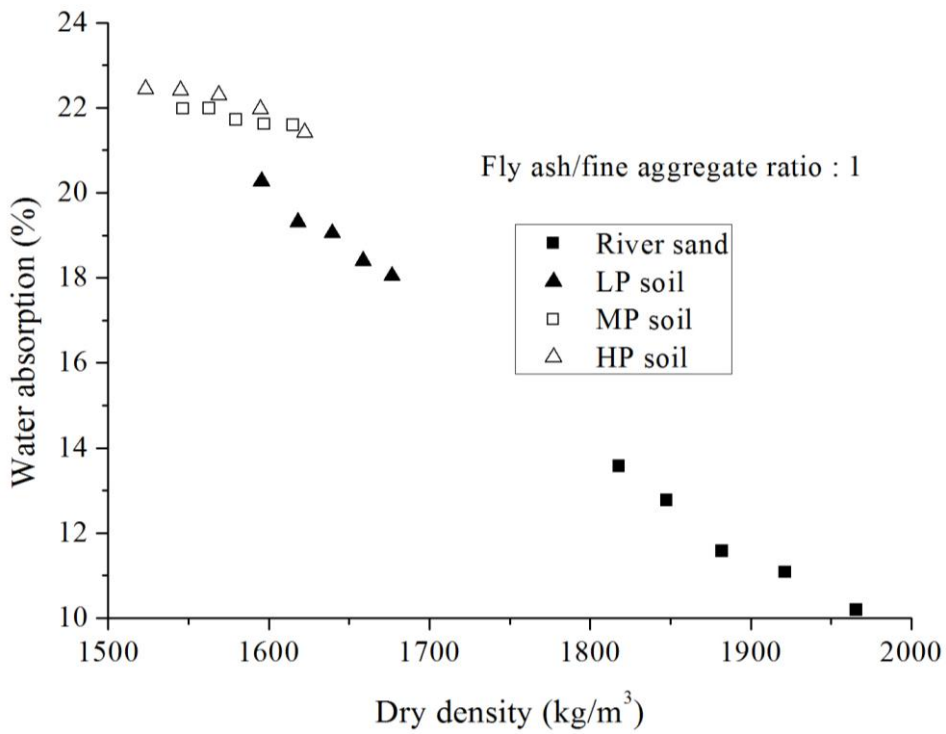


Figure 4.10 Experimental data showing variation in water absorption with density of geopolymer mortar
(curing temperature: 75°C; F/A ratio: 0.75)



(a) F/A ratio of 0.5



(b) F/A ratio of 1

Figure 4.11 Variation in water absorption with density of geopolymer mortar at curing temperature of 90°C

4.4.5 Drying Shrinkage

Changes in soil water content results in swelling/ shrinkage in clayey soils. Drying shrinkage measured from designed set of experiments at different exposure time (Figure 4.12) shows that the least shrinkage is exhibited by mix with river sand. It is a proven fact that plasticity and shrinkage depend on the mineralogy of the soil and relatively high for montmorillonite, moderate for illite and low for kaolinite type of clay minerals. Following that, the shrinkage increases with increasing plasticity of soil. Presence of montmorillonite type of mineral with high surface area greatly affects the shrinkage value of medium and high plastic soil geopolymer mortars and the intensity varies with the quantity of clay present in the soil. The physico-chemical reaction between clay particles mainly depends on the diffuse double layer between the particles. Singh et al. (2016) reported that the shrinkage gets stabilized after 28 days and 89% of shrinkage would be achieved within 28 days for geopolymeric specimens. Hence, 28th day shrinkage value is used for studying the influence of different parameters.

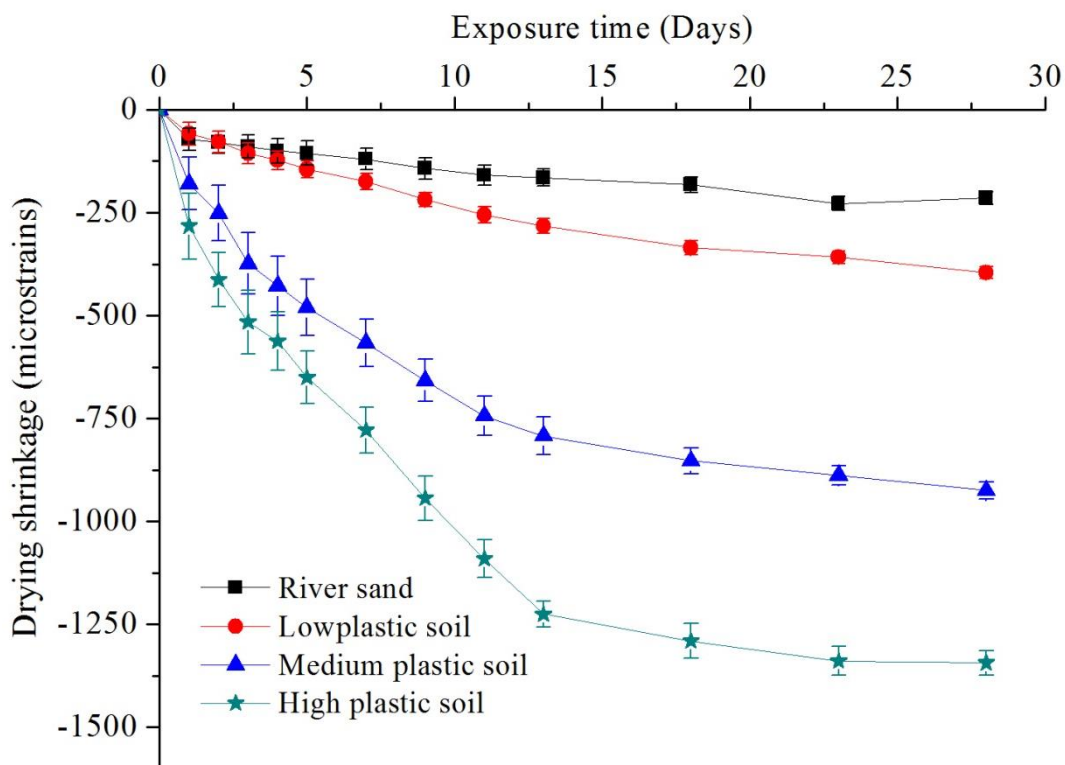


Figure 4.12 Variation in drying shrinkage strain with time for geopolymer mortar using different fine aggregate

(Molarity of NaOH: 8M; Curing temperature: 75°C; F/A ratio: 0.75)

The response surface equation for drying shrinkage of geopolymer mortar with different fine aggregates are summarised in Table 4.12. The ANOVA represented in Table 4.13 shows that, drying shrinkage is influenced by molarity of NaOH for all types of fine aggregate except medium plastic soil. Drying shrinkage of geopolymer mortar with medium plastic soil is affected only by fly ash to fine aggregate ratio. Whereas, drying shrinkage of geopolymer mortar with high plastic soil is influenced by all three parameters. There is no interaction effect between these parameters.

Table 4.12 Response surface model equations for drying shrinkage

Type of fine aggregate	Drying shrinkage in micro-strains
River sand	$+1348.29496 - (237.70489 * X_1) - (5.08183 * X_2) - (259.77360 * X_3) - (0.63050 * X_1 * X_2) + (138.94648 * X_1 * X_3) + (2.40416 * X_2 * X_3) + (14.49266 * X_1^2) + (0.029174 * X_2^2) - (574.97349 * X_3^2)$
Low plastic soil	$+11490.40048 - (1444.59627 * X_1) - (116.60841 * X_2) - (1991.35994 * X_3) + (2.47930 * X_1 * X_2) + (16.52933 * X_1 * X_3) + (4.97332 * X_2 * X_3) + (74.50793 * X_1^2) + (0.61903 * X_2^2) + (978.49179 * X_3^2)$
Medium plastic soil	$+1997.54359 + (295.95990 * X_1) - (29.48261 * X_2) - (2445.68223 * X_3) - (0.35355 * X_1 * X_2) + (18.38478 * X_1 * X_3) + (6.59966 * X_2 * X_3) - (15.22999 * X_1^2) + (0.14702 * X_2^2) + (649.28041 * X_3^2)$
High plastic soil	$+3904.58162 + (400.47368 * X_1) - (78.73980 * X_2) - (1330.40574 * X_3) - (2.11608 * X_1 * X_2) + (4.54316 * X_1 * X_3) + (12.06183 * X_2 * X_3) - (9.75091 * X_1^2) + (0.49247 * X_2^2) - (784.09811 * X_3^2)$

*Note: Molarity - X_1 ; Temperature- X_2 ; Fly ash/Fine aggregate - X_3

For a given molarity and F/A ratio, shrinkage reduces with increasing curing temperature which is shown in Figure 4.13. As increase in curing temperature helps in polymerisation and results in dense structure, the shrinkage is reduced irrespective of the type of fine aggregate used. As curing temperature was increased from 60°C to 90°C, shrinkage reduced in the range of 21% to 48%.

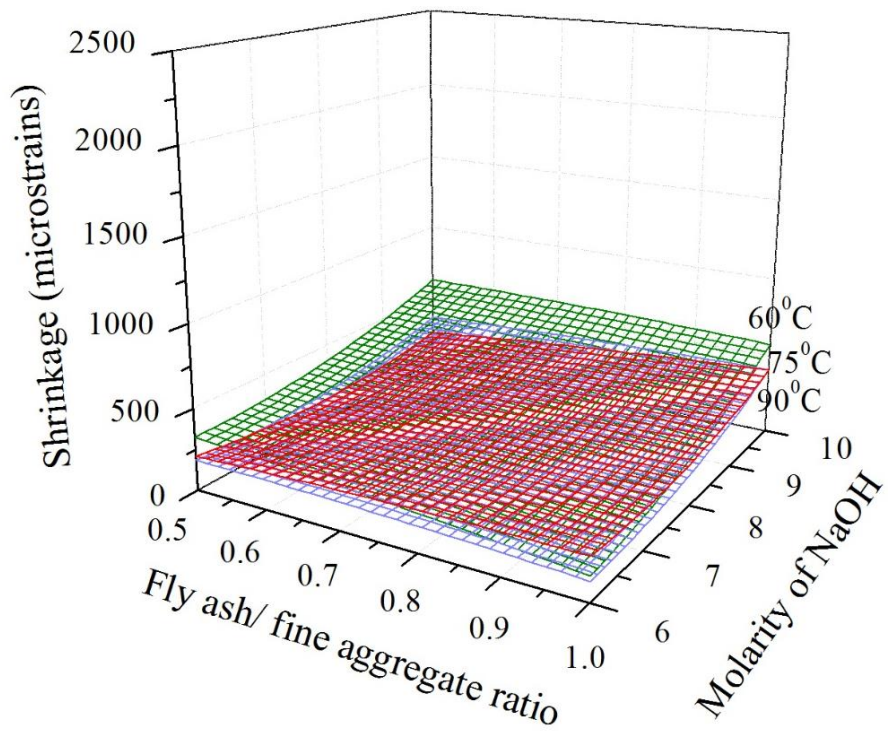
At any curing temperature and F/A ratio, increase in molarity of NaOH reduces the shrinkage of mixes with different fine aggregates in the range of 3% to 38% (Figure 4.14). The shrinkage value decreases as the geopolymer gel becomes denser which results in reduced porosity. For a given molarity, the shrinkage reduces with increasing fly ash to fine

aggregate (F/A) ratio for soil with reactive clay in it (Figure 4.15a and b). This is because of the increasing fly ash content which increases the geopolymerisation and forms stronger gel matrix that can resist shrinkage. When F/A ratio was increased to 1, shrinkage values of mixes with plastic soils reaches closer value comparable to mix with river sand (Figure 4.15b). The reduction in shrinkage of plastic soils with increase in F/A ratio ranges from 5% to 72%.

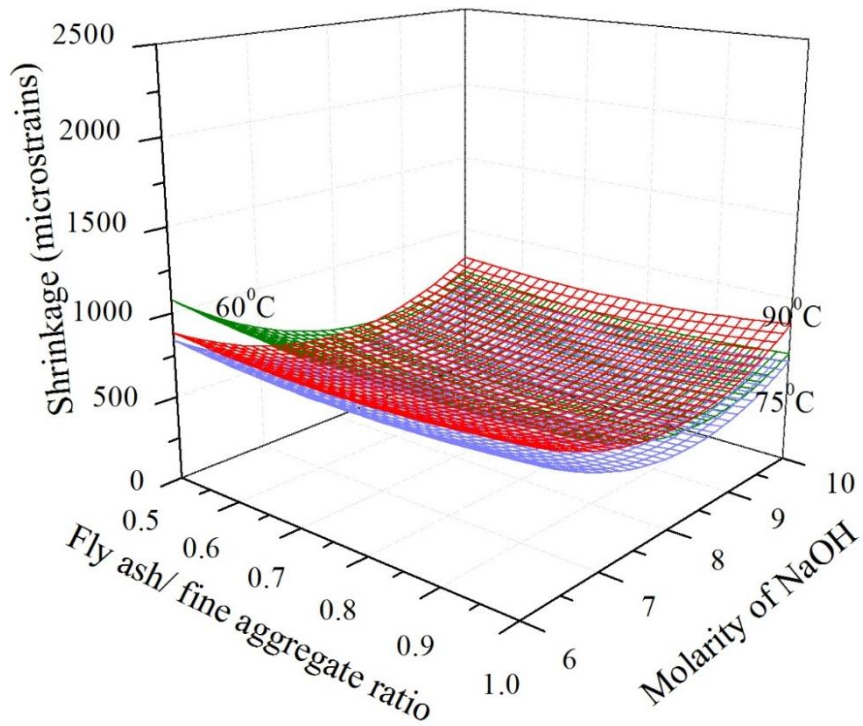
Table 4.13 ANOVA for drying shrinkage at 28th day of exposure

Response	Factor	River sand		Low plastic soil		Medium plastic soil		High plastic soil	
		F-value	P-value	F-value	P-value	F-value	P-value	F-value	P-value
Main effects	X ₁ [*]	22.41	0.0008	8.44	0.0157	4.399431	0.0623	5.94	0.035
	X ₂ [*]	7.52	0.0208	0.01	0.9406	4.456894	0.0609	6.83	0.0259
	X ₃ [*]	3.86	0.0779	0.02	0.9043	30.25026	0.0003	28.61	0.0003
Interaction effects	X ₁ X ₂	0.16	0.6985	0.83	0.3851	0.016381	0.9007	0.16	0.7013
	X ₁ X ₃	2.14	0.1738	0.01	0.9216	0.012304	0.9139	0.00	0.989
	X ₂ X ₃	0.04	0.8531	0.05	0.8244	0.089186	0.7713	0.08	0.7842
Quadratic effects	X ₁ ²	2.69	0.132	23.86	0.0006	0.973474	0.3471	0.11	0.7514
	X ₂ ²	0.03	0.8564	5.21	0.0456	0.287036	0.6038	0.86	0.3768
	X ₃ ²	1.03	0.3333	1.00	0.3398	0.431946	0.5259	0.17	0.6911

*Note: Molarity -X₁; Temperature-X₂; Fly ash/Fine aggregate - X₃

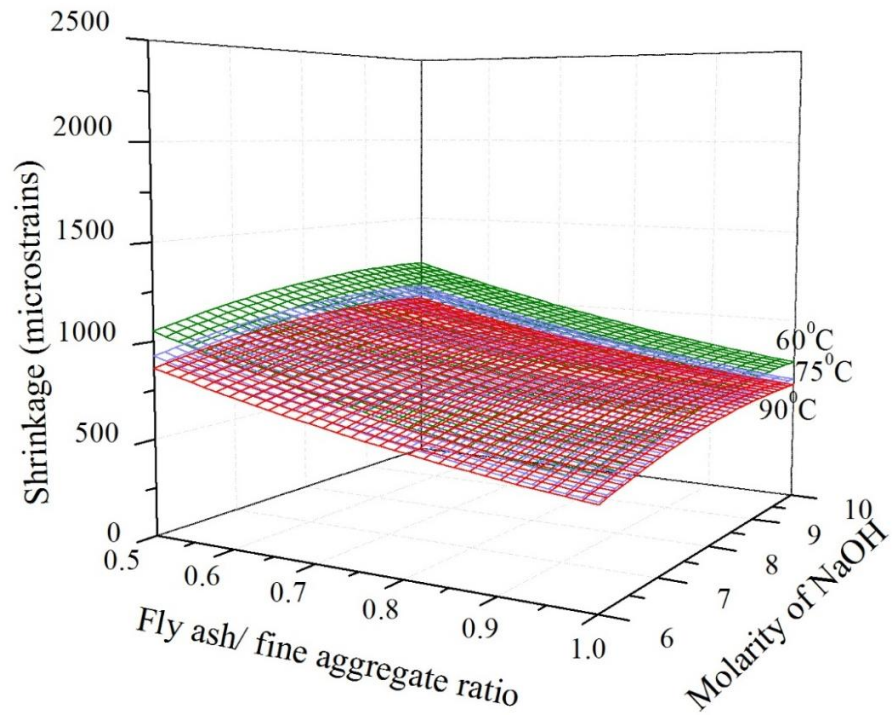


(a) With river sand

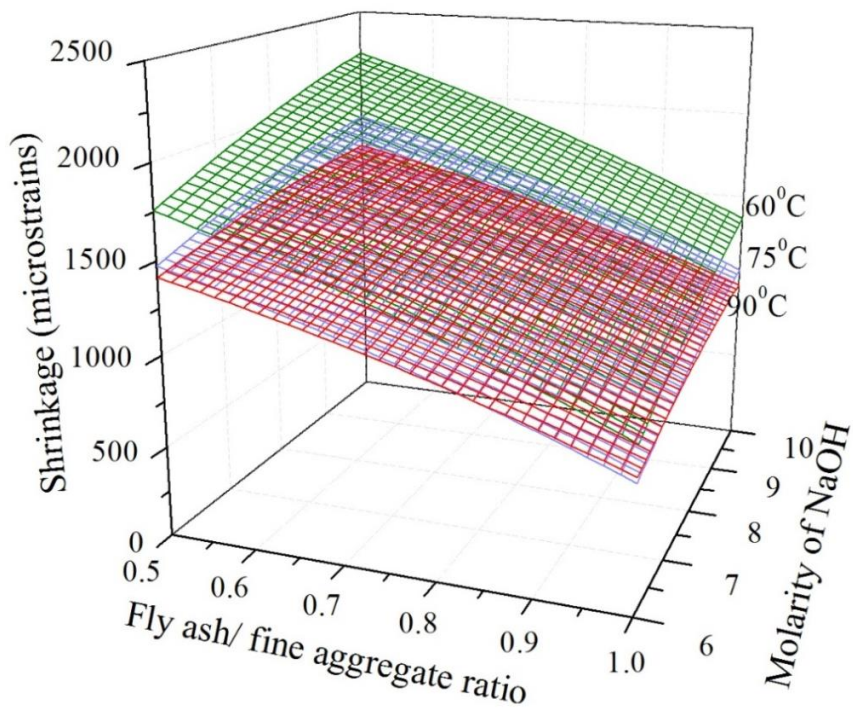


(b) With low plastic soil

Figure 4.13 continued...



(c) With medium plastic soil



(d) With high plastic soil

Figure 4.13 Drying shrinkage of geopolymer mortar

The shrinkage observed in all the types of aggregates is well within the limit prescribed by ACI (2005) (Figure 4.15). River sand mortar mix with density ranging from 1775 to 1925 kg/m^3 resulted in shrinkage of 100 to 400 micro strains. With reduced dry density, the mixes with plastic soil shows hike in shrinkage value compared to the mix with river sand. Geopolymer mortar with low plastic soil with density of 1575 to 1675 kg/m^3 shows shrinkage of 500 to 900 micro strains. Mixes with medium and high plastic soils with the density ranges of 1525 to 1625 and 1500 to 1600 kg/m^3 resulted in shrinkage of 500 to 1200 micro strains and 700 to 1625 micro strains, respectively. High plastic soil with plasticity index 44 (>35) falls under the category of very high shrinkage potential clay (Taylor and Smith, 1986) which could also be used as a fine aggregate material for geopolymer mortar preparation with shrinkage limit acceptable for construction materials.

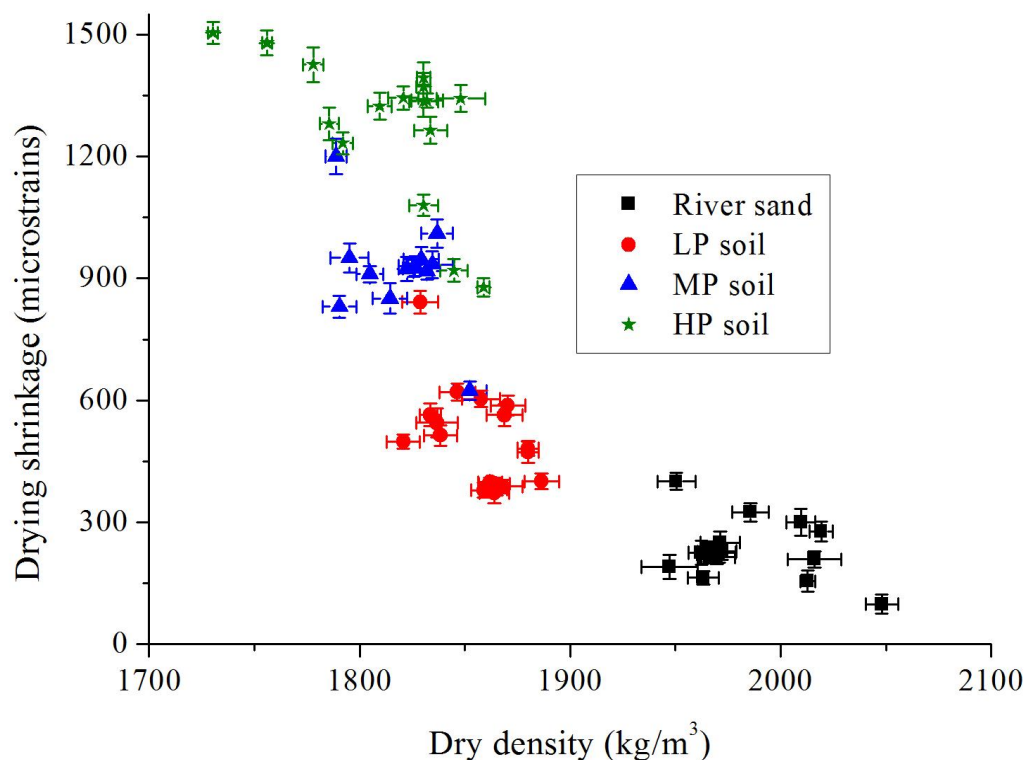
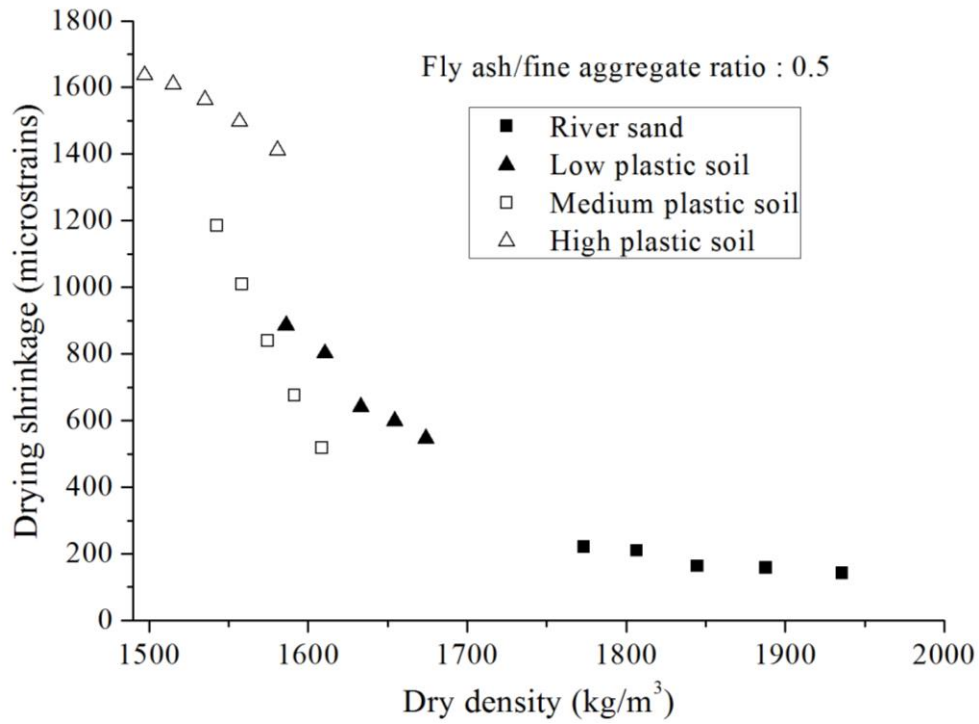
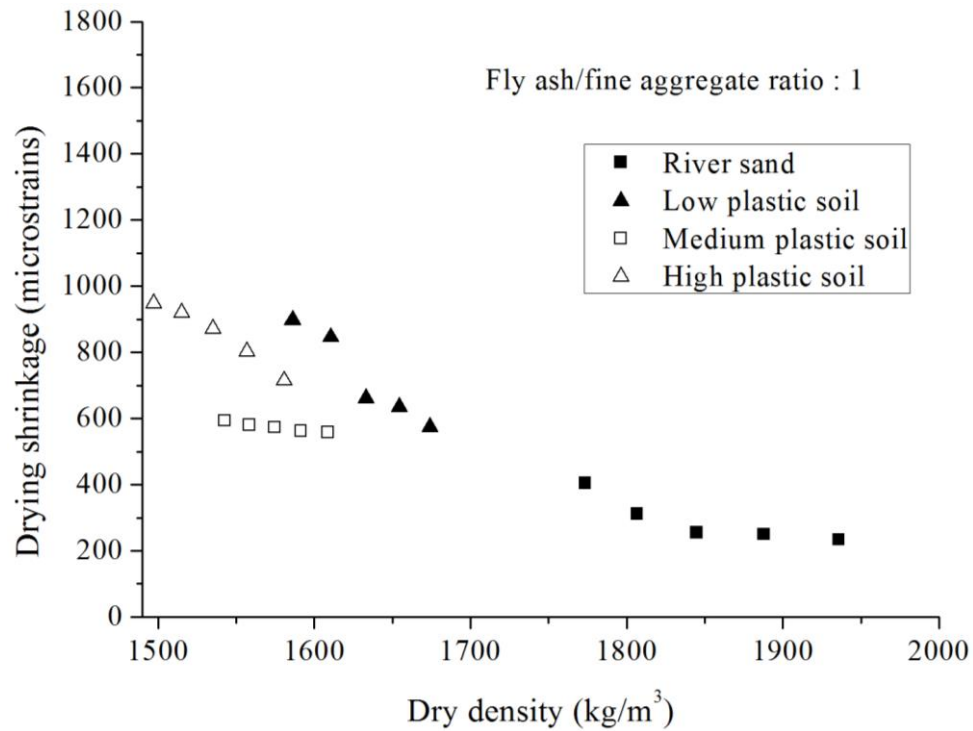


Figure 4.14 Experimental data showing variation in shrinkage with dry density of geopolymer mortar (curing temperature: 75°C; F/A ratio: 0.75)



(a) For F/A ratio 0.5



(b) For F/A ratio 1

Figure 4.15 Variation in shrinkage with density of geopolymer mortar at curing temperature of 90°C

4.5 SUMMARY

Geopolymerisation helps in utilizing even high plastic soil as fine aggregate in construction materials. Soil based geopolymer mortar resulted in lower density range compared to conventional geopolymer of similar strength values. The test results show that strength and shrinkage properties of soil based geopolymer mortar significantly depends on the type of clay present in the soil. Geopolymer mix with each specific soil has an optimum combination of NaOH, curing temperature and binder dosage that helps them achieve the desired properties such as higher compressive strength and lower dry density, water absorption and shrinkage values.

CHAPTER 5

PERFORMANCE OF STABILIZED EXCAVATION SOIL AS FINE AGGREGATE IN CEMENT MORTAR

5.1 GENERAL

Using excavated soil in geopolymer mortar as fine aggregate may be the simplest application for the direct use of untreated excavated soil. Unlike the case of geopolymer mortar, raw excavation soil cannot be directly used in conventional cement mortar. Several treatment methods are used in the beneficiation of aggregate materials, such as stabilization, washing, wet screening, heavy liquid separation, elutriation and magnetic separation which were discussed broadly in Chapter 2.

Stabilization is the common treatment method used for strengthening of poor quality soil for geotechnical engineering applications like road sub-base, embankment, etc. (Petry and Little 2002). Forsman et al. (2013) adopted stabilization to overcome the negative effects of clay such as excessive shrinkage, poor strength in the low quality excavated soils which is not suitable for construction activities if not treated. The soft soil stabilized with fly ash, lime, cement and oil shale ash were used in applications like flood barriers, embankments and landscape construction. Natural pozzolans and industrial wastes which have calcium ions are also adopted as stabilizers in recent times to make the process environmental friendly (Hossain and Mol 2011). Stabilization employed on different soil sources and effect of different stabilizers and their dosages are summarised in Table 5.1. In stabilization, other than cation exchange process that helps in satisfying the charge imbalance of clay layers, calcium in the stabilizer reacts with silica and alumina of clay forming calcium silicate hydrate or calcium alumina-silicate hydrate which strengthens the mixture (Bell 1996).

In this study, three soils of different plasticity as mentioned in Table 3.1 are treated by dry sieving, stabilization and combination of both. Two stabilizers viz., cementitious stabilizer (ground granulated blast furnace slag, GGBS) containing reactive silica alumina with Ca(OH)_2 and a hydraulic stabilizer (lime) with 97% of Ca(OH)_2 have been chosen. Initially, the dosage of GGBS was varied from 5 to 20% and lime was varied from 2 to 10%, for studying its effectiveness in reducing the plastic characteristics of the soil. The treated soils were then used as fine aggregate in cement mortar. The mortar properties such as dry

density, compressive strength, water absorption, and shrinkage strain were compared with control mortar made of river sand.

Table 5.1 Literature on stabilization of soil from different sources

Author, Year	Study on	Stabilizer	Dosage (%)	Salient observations
Glu et al. (1986)	Mine tailing	Lime	5 - 17	Strength properties increased with increasing the lime dosage and curing time.
Choquette et al. (1987)	Marine clay	Lime	0.5 - 10	Neogenic minerals formed and found to be CASH and CSH.
Bell, (1996)	Clayey soil	Lime	2-10	Montmorillonite type of clay minerals responds better than kaolinite minerals.
Rajasekaran and Narasimha Rao, (1997)	Marine clay	Lime	40% injected into clay bed	Results in the aggregation of particles and an overall increase in porosity of soil system.
Di Sante et al. (2014)	Clayey soil	Lime	5	The saturated condition of soil-lime mixes results in more brittle structure and formation of crystalline products.
Horpibulsuk et al. (2010)	Silty clay	Cement	3-45	Cement dosage, water content and curing time are the influencing factors. Water content of 1.2 times of optimum moisture content forms an active zone for stabilization.
Zhang et al. (2014)	Marine clay	Cement Metakaolin (MK)	12-15 1-5	MK improves pore volume distribution and forms products that fill the micro-pores.
Keramatikerman et al. (2016)	Clay	Cement Lime BFS	5-15 2-6 2-6	Addition of slag (BFS) with lime reduces the shrinkage capacity due to the accelerated generation of cementitious products.

5.2 DRY SIEVING PROCESS

Three types of locally available soils covering a range of plasticity such as Low (LP), medium (MP) and high (HP) have been used. Combination of kaolinite, illite and montmorillonite clay minerals were present in these soils, making them vary in their plasticity characteristics. Dry sieving has been done with 600 μm size sieve to remove clay and silt that affects the cement mortar properties. Percentage distribution of sand, silt and clay size particles in low medium and high plastic soils before and after dry sieving process are presented in Figure 5.1. Dry sieving of plastic soil removed 5, 12, 16% of clay ($< 2 \mu\text{m}$) and 5, 8, 8% of silt (2 to 75 μm) in low plastic, medium and high plastic soils, respectively. Even after dry sieving, the percentage particles $< 75 \mu\text{m}$ (silt + clay) present in the low, medium and high plastic soil were 19, 28 and 34%, respectively. This can be attributed to the adhesive nature of clay on to sand particle and hence did not get removed completely with dry sieving. Hence, there is a need to stabilize the sieved soil to treat the clay present in them.

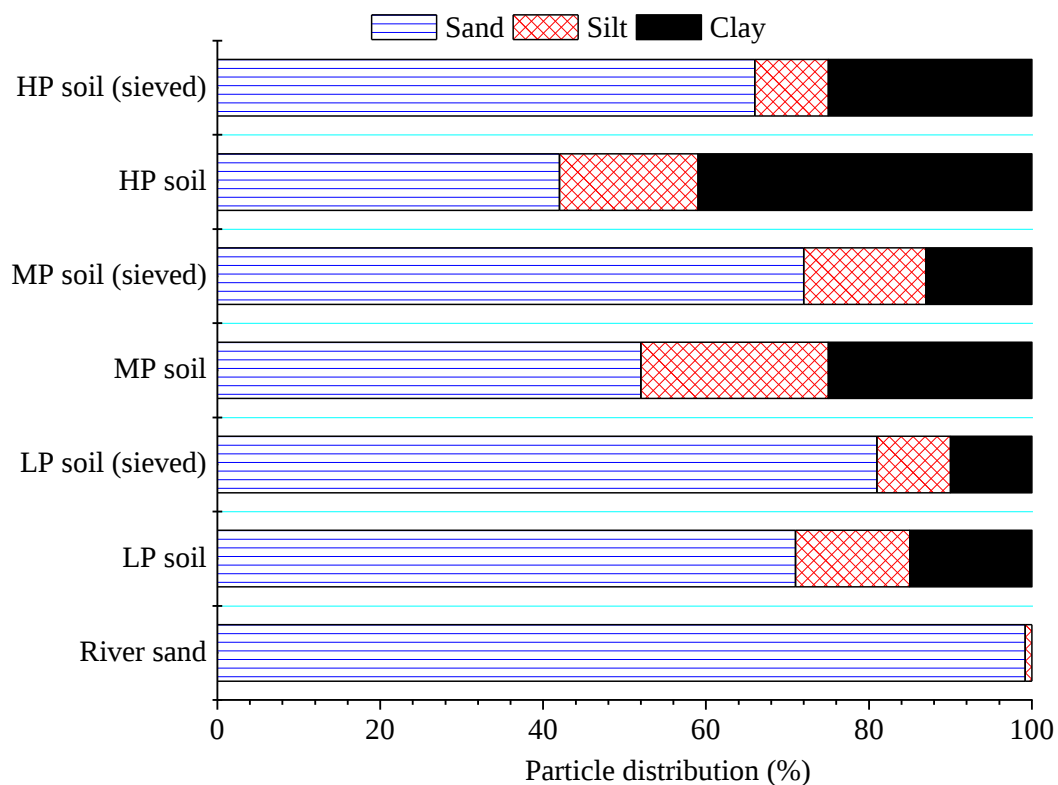


Figure 5.1 Particle distribution of soils before and after dry sieving

5.3 STABILIZATION PROCESS

Soil samples passing 4.75 mm were used in this study. Dry sieving was performed with 600 μm size sieve. The soil samples used for stabilization process are listed in Table 5.2. Lime with 97% Ca(OH)_2 and slag (GGBS) were used as stabilizers. Lime was preferred due to the availability of high calcium content. Slag was chosen as cementitious stabilizer to compare the effectiveness with that of lime. The chemical composition of slag (GGBS) is given in Table 5.3. Preliminary studies indicated that lime up to 10% and slag up to 20% effectively improves the compressive strength and reduces the drying shrinkage strain of cement mortar. Hence, the respective maximum dosages were limited at these levels.

Table 5.2 Types of soil used for stabilization

Processing method	Low plastic soil (LP)	Medium plastic soil (MP)	High plastic soil (HP)
Unprocessed	✓	✓	✓
Sieved (600 μm size)	✓	✓	✓

Table 5.3 Chemical composition of slag

Oxides	SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3	Na_2O	K_2O	MgO	Fineness (m^2/kg)
Composition (%)	32.38	21.06	1.87	31.46	-	0.36	0.88	8.57	430

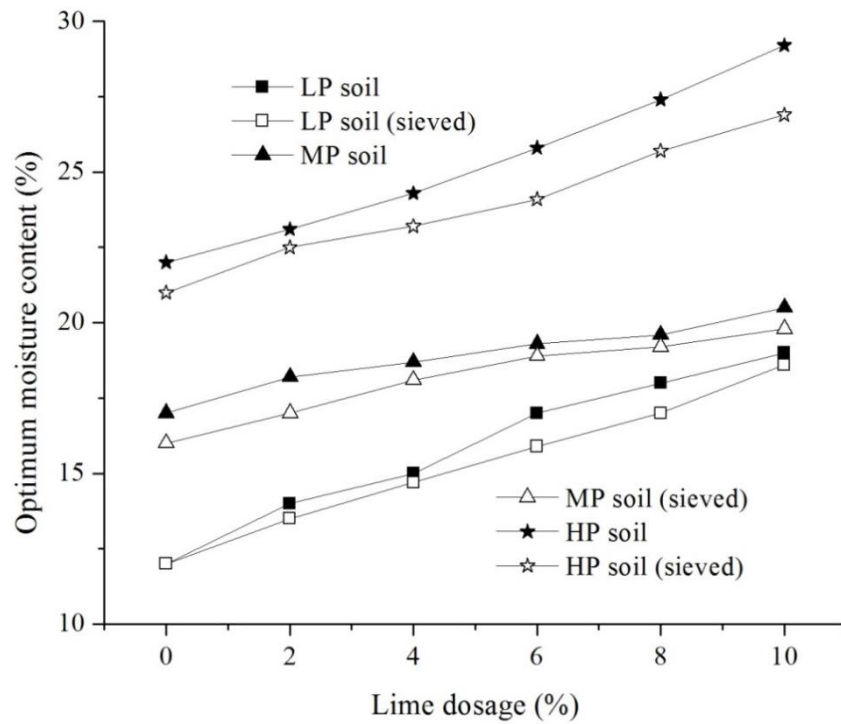
It is reported that for proper hydration of the soil with stabilizer and to form non-reactive crystalline products, 20% additional water content to that of optimum moisture content (OMC) and a curing age of 28 days should be adopted for stabilization (Horpibulsuk et al. 2010; Di Sante et al. 2014). Hence, OMC was first determined for each soil to fix the water content needed for the stabilization process. After dry mixing the soil sample with predefined dosage of lime/slag in a Hobart mixer, water content of OMC + 20% was used to make the slurry of the soil-stabilizer mix. The slurry was allowed for curing and stabilization for 28 days in a closed container to avoid loss of moisture (Figure 5.2). Before proceeding to study the mortar properties with stabilized soils, the index properties of stabilized soils were determined which can be used to assess the relative effectiveness of the stabilizers and its dosages on different types of soil.



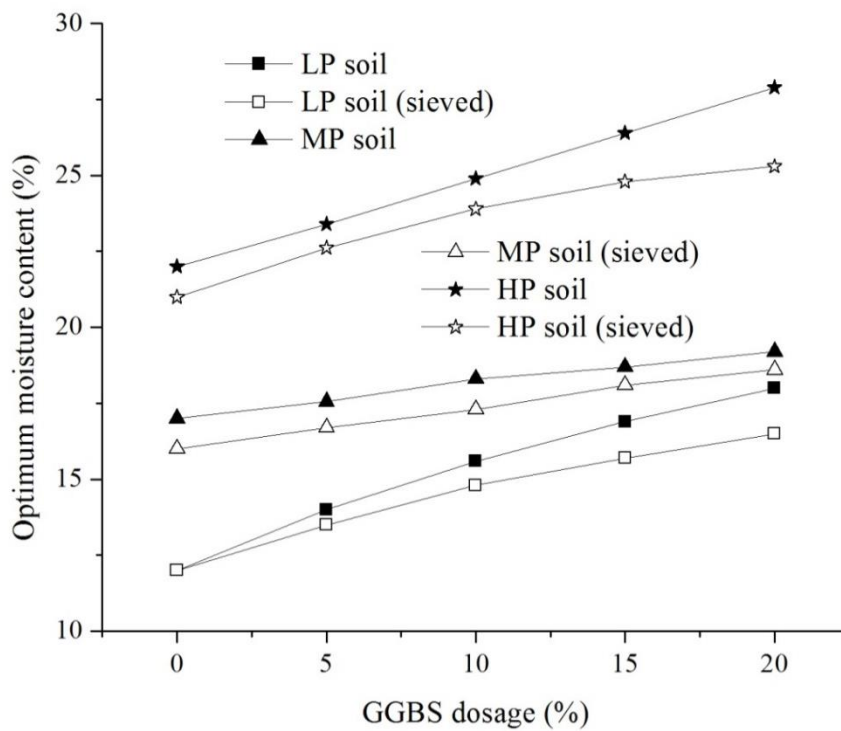
Figure 5.2 Soil stabilization process

5.3.1 Optimum Moisture Content

The Standard Proctor compaction test was performed as per ASTM D698 (2012), to arrive at the OMC of different soil-stabilizer mixtures. The optimum moisture content (OMC) for different soil at various dosages of lime and slag as stabilizers are shown in Figure 5.3 (a) and (b), respectively. Irrespective of the type of soil or stabilizer, OMC increases with stabilizer dosage. Increase in calcium ion concentration with stabilizer dosage results in intercalation of these ions in to clay layers. With increasing calcium content in lime as stabilizer, cation exchange and the affinity of the soil-stabilizer mixture towards water increases the OMC. Hence, at a given stabilizer dosage, say at 10%, lime needs more water to reach OMC compared to slag.



(a) With lime



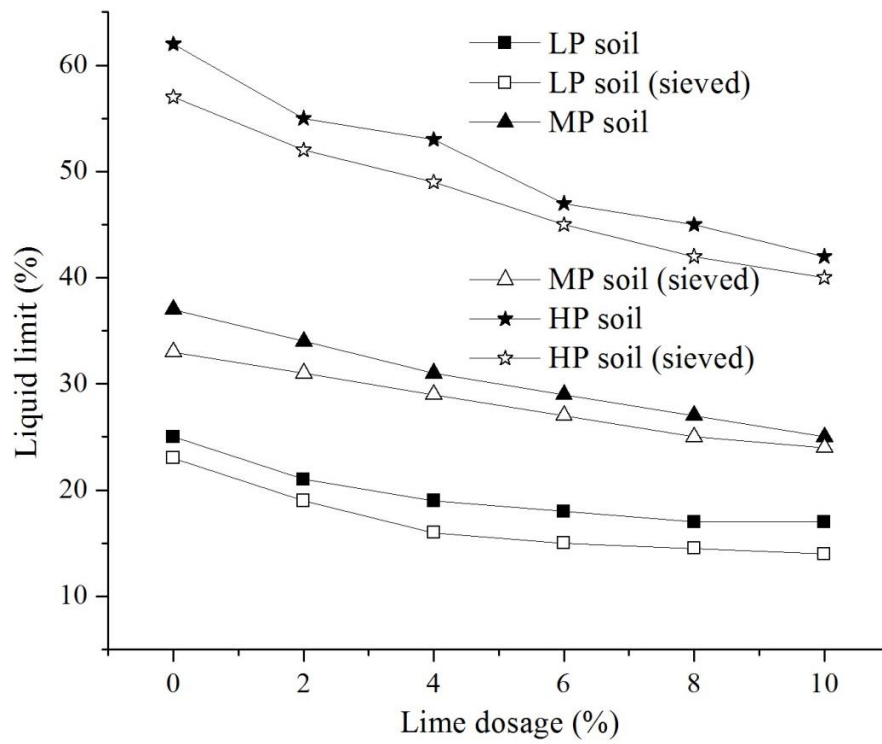
(b) With GGBS

Figure 5.3 Optimum moisture content of plastic soils with different stabilizer dosage

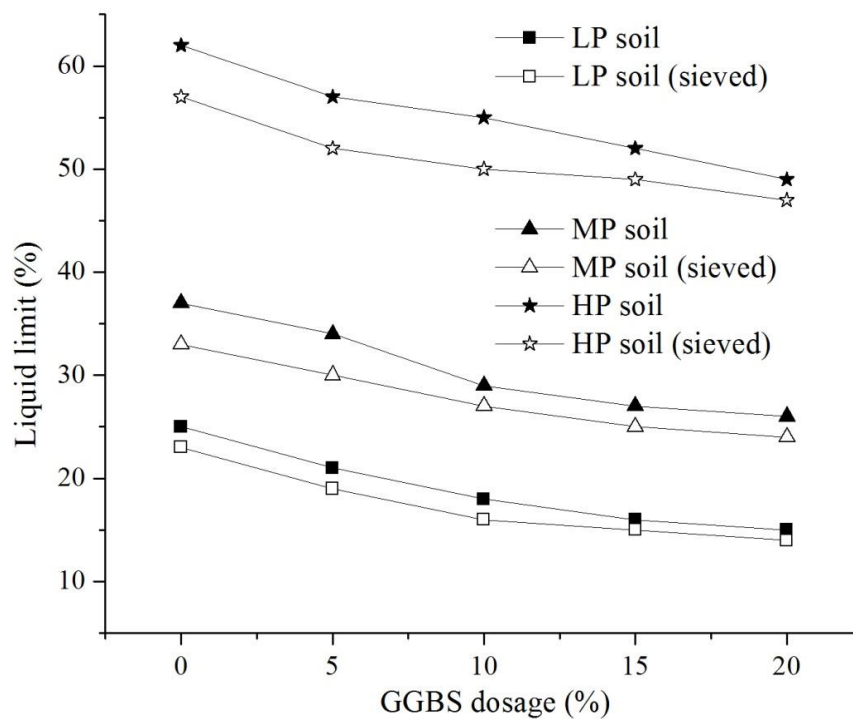
5.3.2 Index Properties of Stabilized Soil

After 28 days curing of stabilized soil (LP, MP and HP) samples, they were completely dried and their index properties were assessed as per ASTM D4318 (2017). Figure 5.4 (a) and (b) indicates that the water content required to make the soil to flow, i.e., liquid limit (LL) reduces with increasing stabilizer dosage. The expansive clay present in the plastic soil reacts with calcium in the stabilizer by cation exchange process and loses its reactivity, thus the clay becomes less sensitive to moisture, leading to lower water requirement. In addition to calcium, the presence of other reactive contents (silica and alumina) in slag increases the water required for flow, hence exhibits relatively high LL. In case of low plastic soil with non-reactive kaolinite clay minerals, there is no appreciable reduction in LL beyond 4% of lime dosage (Figure 5.4b). Irrespective of the type of stabilizer, there is a gradual reduction in LL of medium and high plastic soil with an increasing stabilizer dosage.

Plasticity index (PI) is the range of water content over which a soil behaves plastically and it depends on both liquid limit and plastic limit of the soil and is shown in Figure 5.5. Low plastic soil is not represented in the plasticity index curve as it did not exhibit plasticity. The un-sieved medium plastic soil becomes non-plastic with 6% lime or 15% slag while dry-sieved medium plastic soil could achieve the same level with 4% lime or 10% slag. This clearly shows the effectiveness of stabilization in plastic soils and its improvement with dry sieving. In case of high plastic soil, there is a significant reduction in PI with dry sieving followed by stabilization and it could reach a medium plasticity (PI – 20 to 22%) with 6% lime or 20% slag. The reduction in plasticity provides the necessary encouragement for the use of stabilized soils as fine aggregate in cement mortar or concrete.

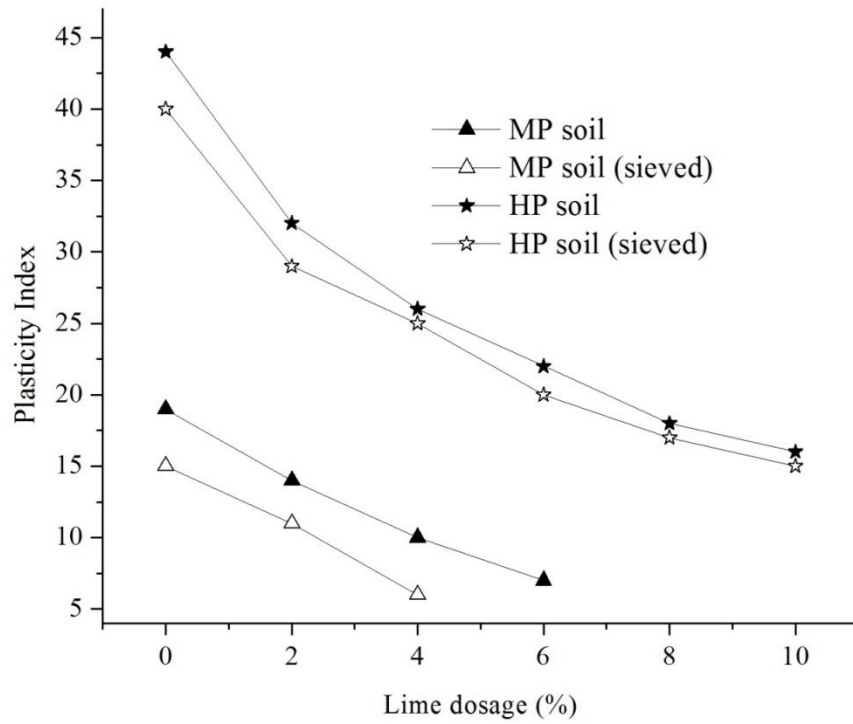


(a) With lime

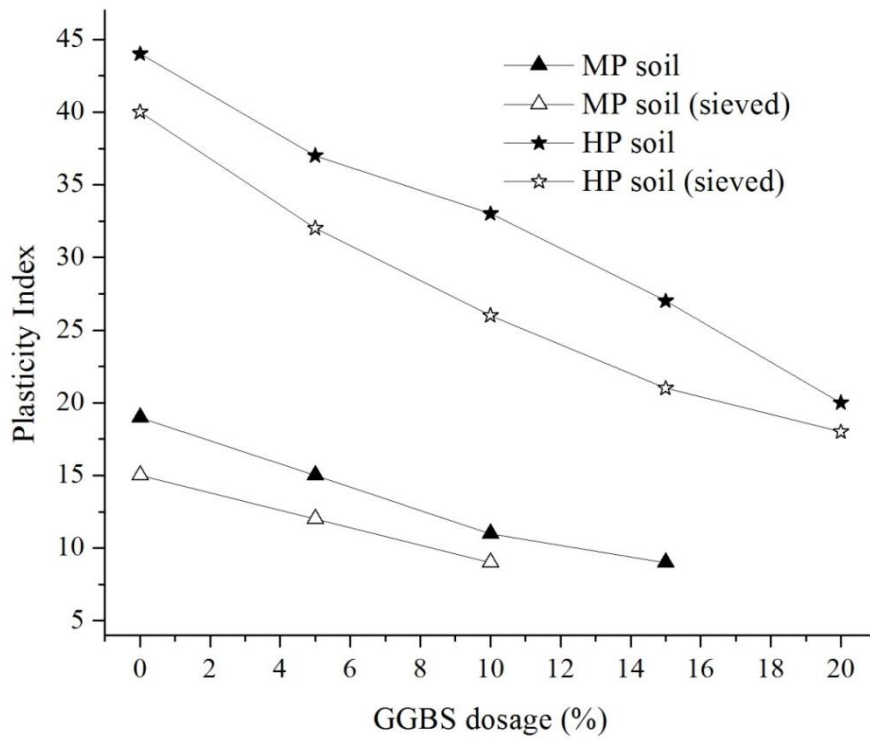


(b) With GGBS

Figure 5.4 Liquid limit (LL) of stabilized plastic soils



(a) With lime



(b) With GGBS

Figure 5.5 Plasticity index (PI) of stabilized plastic soils

5.4 PREPARATION OF CEMENT MORTAR

Well graded river sand (RS) with specific gravity of 2.7, was used as fine aggregate in the control mortar. OPC 53 grade cement conforming to IS 12269 (2013), was used as a binder in mortar preparation. Cement mortar was prepared with a constant fine aggregate to cement ratio of 3.

Cement was dry mixed with fine aggregate in a Hobart mixture for 60 s. Water content required for achieving a constant workability, measured in terms of flow of $110 \pm 5\%$, was added and mixed for another 120 s. Mortar flow percentage was determined in three trials for each of the mix as per ASTM C1437. For each mix, 12 numbers of 50 mm cube specimens (3 for dry density, 3 for water absorption and 6 for compressive strength determination) and 3 numbers of prisms of size 160 mm x 40 mm x 40 mm for shrinkage measurements were cast. Dry density was measured after the specimens were kept at $110 \pm 5^\circ\text{C}$ in an oven for 24 hours and then allowed to cool to room temperature. Water absorption was determined as per ASTM C1403. Shrinkage specimens were cured under water for seven days. The shrinkage readings were taken using length comparator at specific intervals as per ASTM C157 until 90 days of exposure.

5.5 PROPERTIES OF CEMENT MORTAR WITH DRY SIEVED SOIL

Effect of use of dry sieved soil as fine aggregate on cement mortar properties are shown in Figure 5.6 (a) to (d). For maintaining constant workability, cement mortar with unprocessed low, medium and high plastic soil requires 44, 62 and 72.5% higher water content than that of mortar with river sand (Figure 5.6a). Affinity of clay particles towards water is the main reason behind this behaviour. Kaolinite mineral in the low plastic soil is charge balanced, whereas montmorillonite in medium and high plastic soil has affinity towards H^+ ions present in water molecules and adsorbs them in their interlayer spaces. This results in demand for excess water to maintain the workability. Such higher water content is undesirable as it adversely affect the properties of hardened mortar. Though it is only the clay particles that have affinity towards water, silt particles also contribute to the increases in water demand due to the increase in surface area to be wetted.

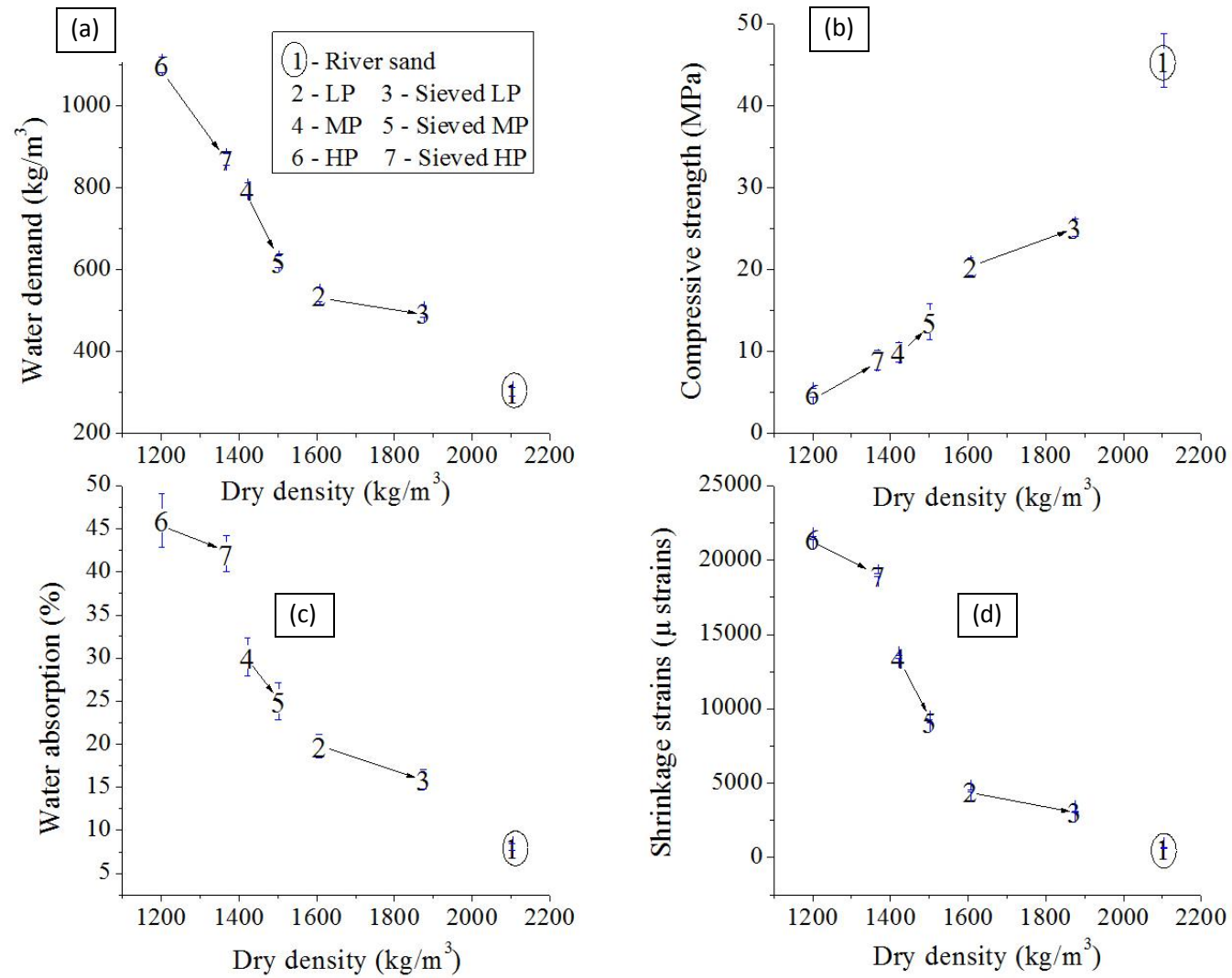
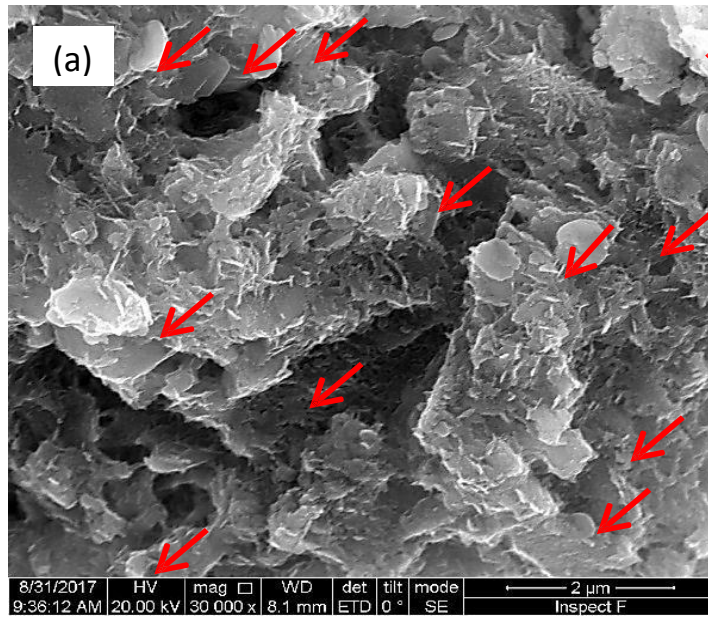


Figure 5.6 Cement mortar properties with dry sieved soil

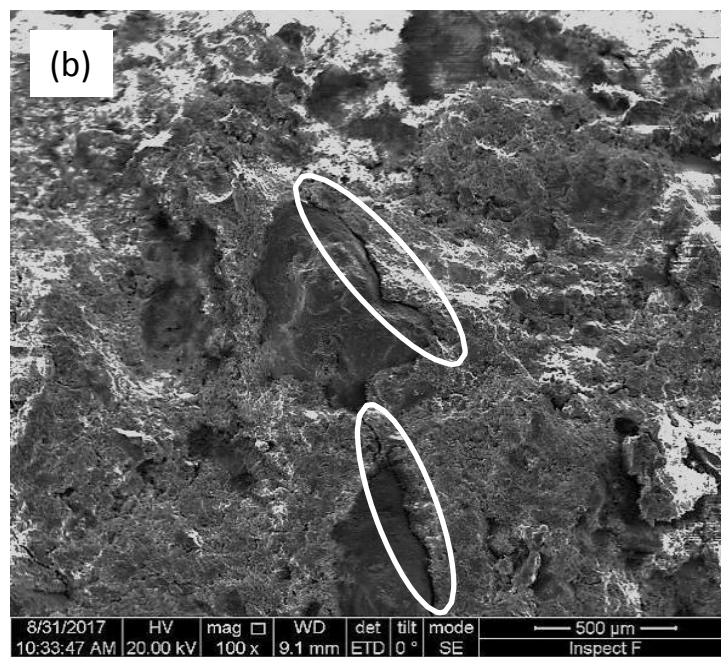
(a) water demand, (b) dry density, (c) water absorption and (d) drying shrinkage

Removal of some of these particles by dry sieving reduced the water demand by 8, 22, and 19% for mortar with low, medium and high plastic soils, respectively, as indicated in Figure 5.6 (a). This can be related to the reduction in liquid limit and plasticity index of soil samples (Figure 5.4 and Figure 5.5). With dry sieving, compressive strength of cement mortar with unprocessed soil varies from 5 MPa to 21 MPa while control mortar with river sand shows a compressive strength of 46 MPa with a corresponding dry density of 2105 kg/m³ (Figure 5.6b). In mortar with unprocessed soil, the clay remains in un-stabilized layers (Figure 5.7a) which agglomerates, shrinks and forms weak, porous reaction products in cementitious composites. Those reaction products near the interfacial transition zone (ITZ) of aggregates results in poor bonding and crack formation, reducing the density and strength drastically (Figure 5.7b). With increasing soil plasticity, water demand and invariably the porosity and permeability of the mortar mix increases, resulting in reduced density and strength properties (Ouellet-Plamondon and Habert, 2016). The reduction in water demand with dry sieving of soils led to an increase in the dry density of mortar by 100 to 200 kg/m³ (Figure 5.6a) and a corresponding improvement in compressive strength of 23, 37, and 80% for mortar with low, medium and high plastic soil, respectively (Figure 5.6b). Similar trend is observed in water absorption with a reduction of 3-4% when dry sieved soils are used as fine aggregates (Figure 5.6c).

Figure 5.6 (d) shows that the shrinkage strain is reduced by 30% in both mortar with low and medium plastic soils. However, high plastic soil with almost 60% of clay and fines did not result in significant reduction in drying shrinkage after dry sieving as considerable amount of highly reactive clay particles were retained in the sieved high plastic soil. Though the increase in shrinkage strains can be related to the increase in water demand of the mortar with clayey aggregates, it has been proved that the addition of extra water does not affect the overall shrinkage in cement system with clay content (Muñoz et al. 2010). This is attributed to the fact that this extra water helps in the improved compaction of such mixture containing clay and hence reduces the shrinkage rate. Hence, the shrinkage caused is mainly due to the action of clay mineral to the exposed environment rather than the amount of water content. As the presence of bound clay in dry sieved soils affects the mortar properties at varying degree, as a next step, stabilization was adopted for both unprocessed as well as dry sieved soils to assess the performance of these soils in cement mortar.



(a) Unreacted montmorillonite clay minerals and reaction products



(b) Poor ITZ with raw high plastic soil

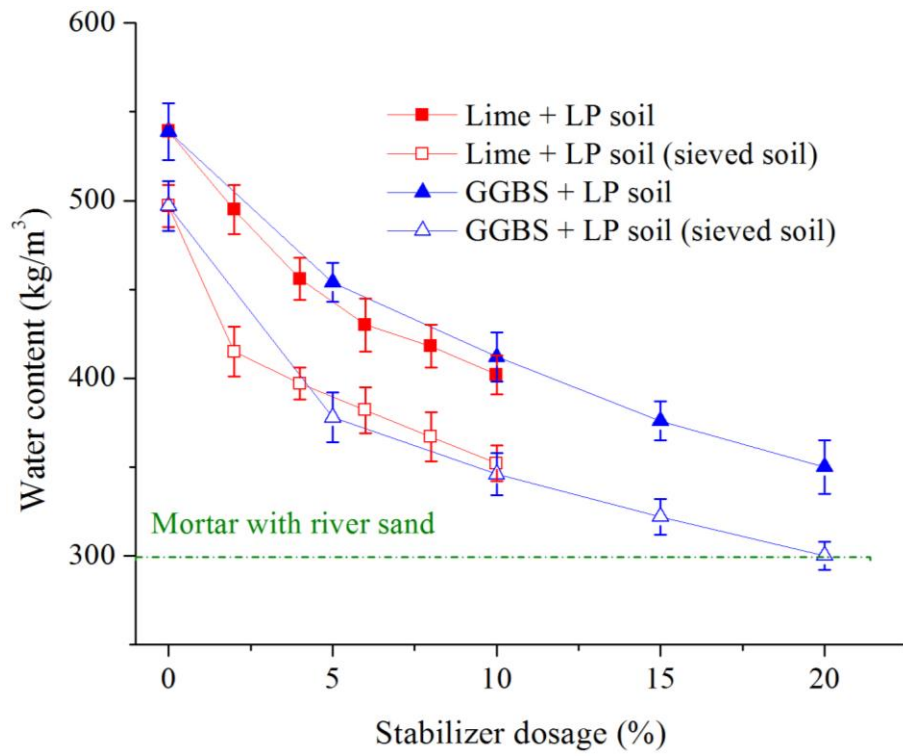
Figure 5.7 Microstructure of cement mortar with high plastic soil under different magnification

5.6 PROPERTIES OF CEMENT MORTAR WITH STABILIZED SOIL

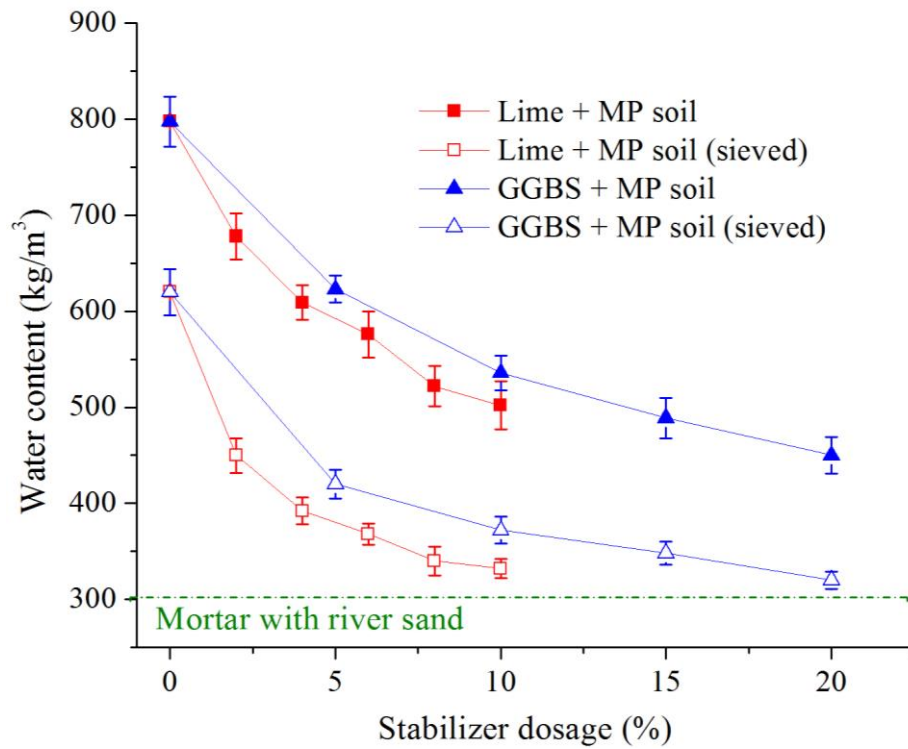
5.6.1 Water Demand for Constant Workability

The variation in water demand of cement mortar with stabilized soil as fine aggregate is presented in Figure 5.8. For a given dosage of stabilizer, mortar with lime or slag stabilized LP soil demands almost the same water content, even though the amount of calcium available for the reaction is higher in lime compared to slag (Figure 5.8a). Similar behaviour is observed in cement mortar with sieved low plastic soil with an average reduction in water demand of 34%. Low plastic soil with charge balanced kaolinite clay mineral is comparatively not an active participant of stabilization reactions. However, at higher stabilizer dosage, mortar with sieved low plastic soil could achieve a desired workability at lower water content comparable to cement mortar with river sand. This improvement in workability must be due to the dilution effect in the clay particles with lime/slag that reduces the water demand.

For achieving constant workability, mortar with medium plastic soil could reach a water demand closer to that of control mortar at a dosage of 20% of slag or at 10% of lime as stabilizer (Figure 5.8b). Though reduction in water demand is significant in mortar with high plastic soil, it could not reach the value equivalent to the control mortar (Figure 5.8c). Unlike, low plastic soil the appreciable difference in the effectiveness of type of stabilizer in medium and high plastic soil can be attributed to the amount of calcium available in lime/slag. In the presence of adequate moisture, Ca^{2+} and OH^- dissociates from $\text{Ca}(\text{OH})_2$ during the stabilization process. It is well established that the OH^- ions increase the pH of the soil and favour the exchange of negatively charged monovalent ions in clay minerals by divalent Ca^{2+} ions. This reduces the thickness of diffuse double layer resulting in flocculation and agglomeration of clay particles and reduces the water demand of the stabilized clayey soils (Bell, 1996). Presence of reactive silica and alumina in the medium and high plastic soil makes use of the available calcium in lime for cation exchange and pozzolanic reaction.

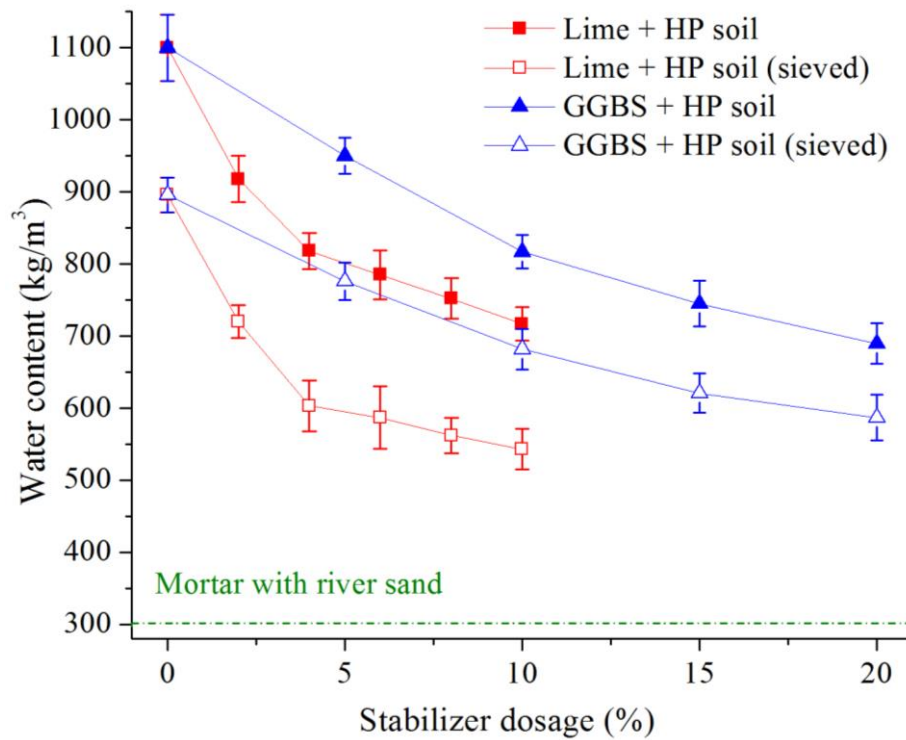


(a) Low plastic soil



(b) Medium plastic soil

Figure 5.8 continued...



(c) High plastic soil

Figure 5.8 Water demand of cement mortar with dry sieved and stabilized soil

On the other hand, formation of porous hydration products in stabilization results in increase in surface area to be wetted (Figure 5.7a). This is the reason that even after stabilization, the water demand of mortar with high plastic soil is higher compared to the control mix with river sand. This has been brought down to some extent by dry sieving. Compared to mortar with unsieved soil, an average additional reduction of 4 to 11% in water demand is observed in mortar with sieved and stabilized medium and high plastic soil.

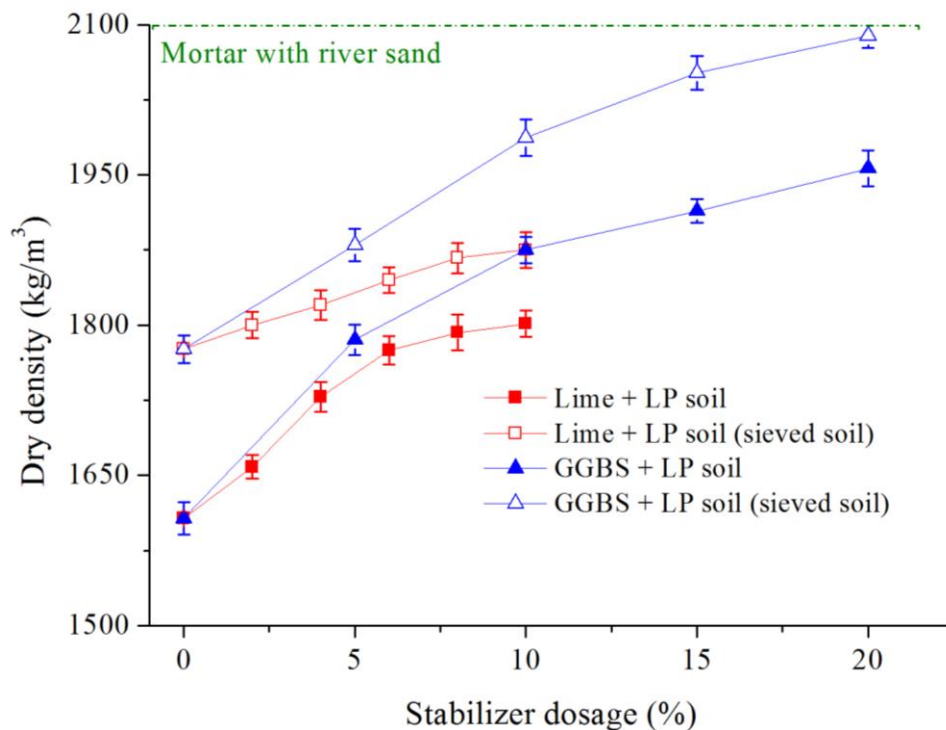
5.6.2 Dry Density

Dry density of cement mortar with stabilized soils as fine aggregate are shown in Figure 5.9(a) to (c). As the water requirement for constant workability reduces with stabilizer dosage, the dry density increases irrespective of type of soil and stabilizer. For a given stabilizer dosage, mortar with slag stabilized LP soil shows higher density increment compared to mortar with lime stabilized LP soil. Similar trend is followed in mortar with sieved and stabilized low plastic soil (Figure 5.9a).

The reactive silica and alumina together with Ca(OH)_2 in slag contributes to the formation of dense hydration products during the stabilization process. This seals the non-

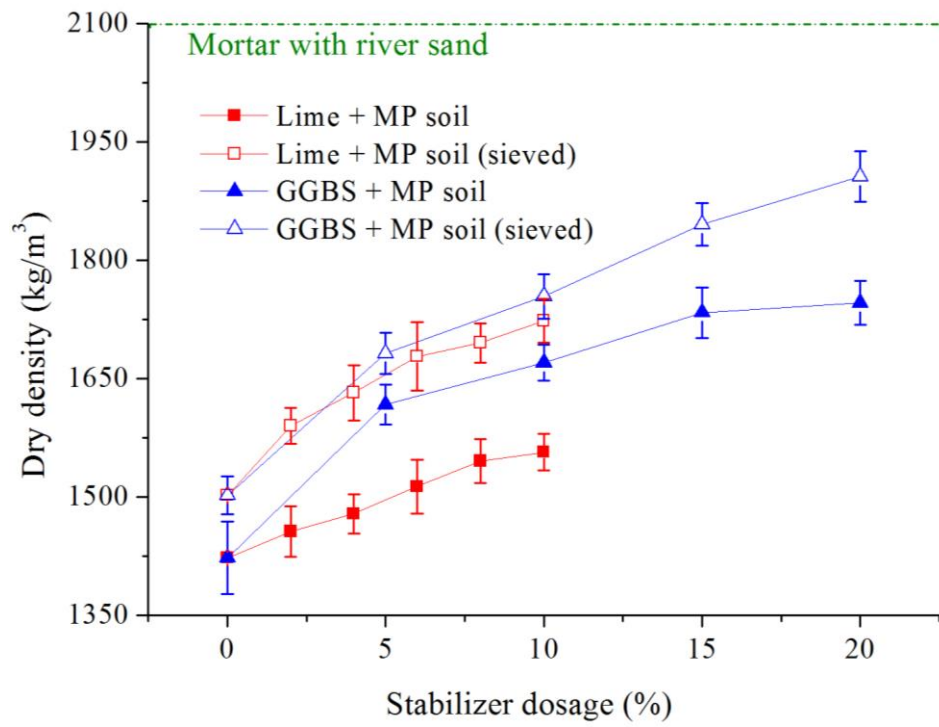
reactive clay and fine particles resulting in increase in the dry density (Figure 5.10a). At a dosage of 20% slag, mortar with sieved and stabilized low plastic soil could reach a dry density of 2050 kg/m³ which is almost equal to that of control mortar.

Mortar with stabilized medium plastic soil exhibits similar trend as that of mortar with low plastic soil. However, the variation in mortar density is marginal with sieved and stabilized MP soil (Figure 5.9b). With sieved MP soil, reduction in clay content must have resulted in improved dispersion and availability of stabilizers. Hence, the effects of slag and lime as stabilizers lead to comparable density. Mortar with stabilized high plastic soil shows a reverse trend with lime, performing better than the slag as stabilizer. The variation in density of mortar is marginal with sieved and stabilized HP soil between lime and slag (Figure 5.9c). Slag mainly binds the reactive clay (Figure 5.10b) whereas lime diffuses through the clay and forms reaction products (Figure 5.10c). Effect of lime diffusion is reported to play an important role in the expandable lattices of montmorillonite minerals present in the HP soil which could easily accommodate the calcium ions in their lattice space and densify (Bell, 1996). This mainly contributes to the increase in dry density, compared to the binding effect of slag.

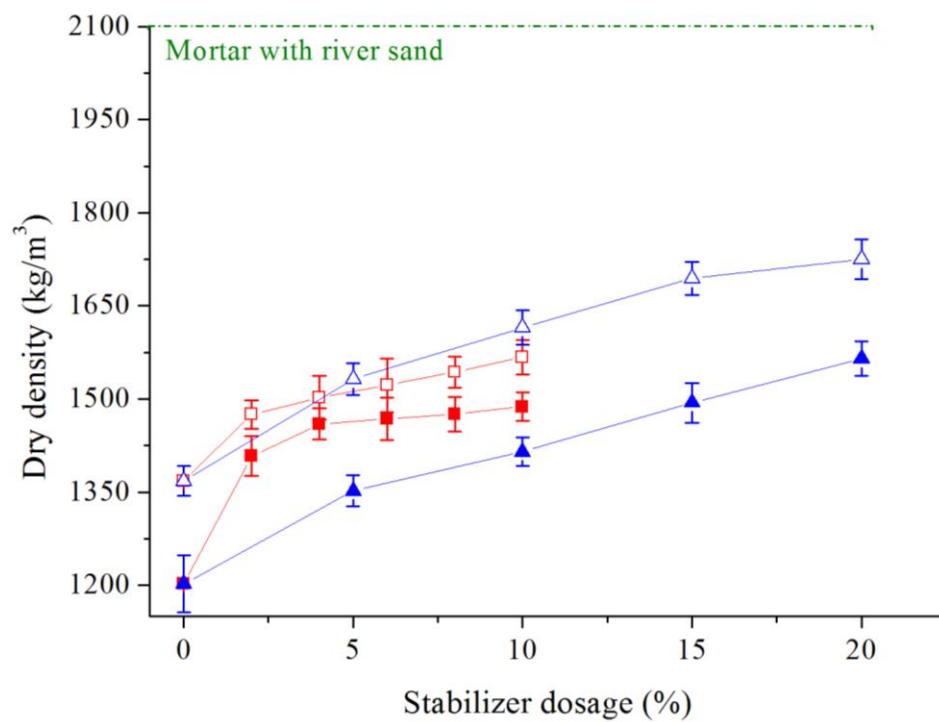


(a) Low plastic soil

Figure 5.9 continued...

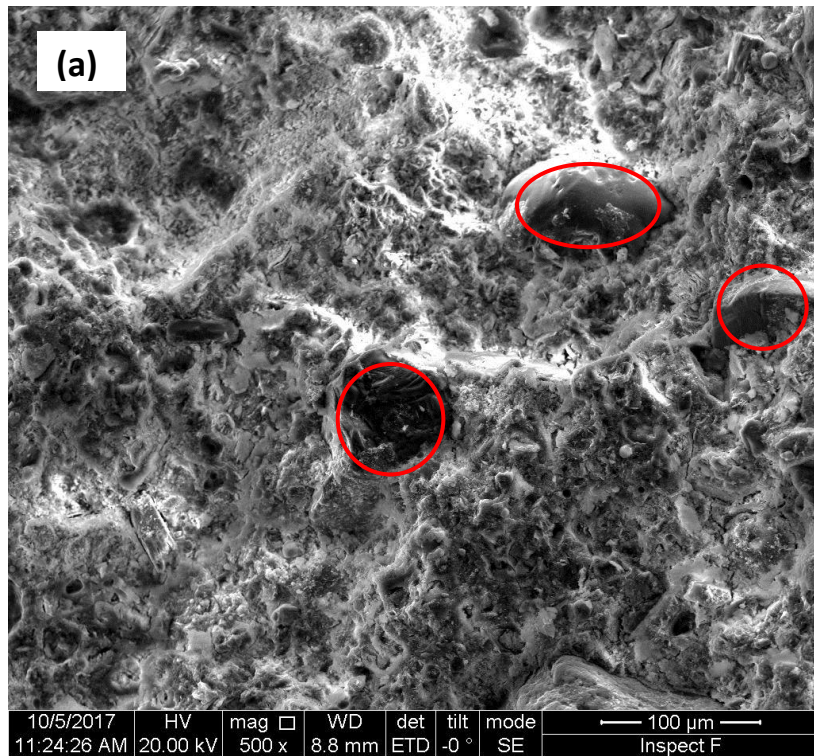


(b) Medium plastic soil

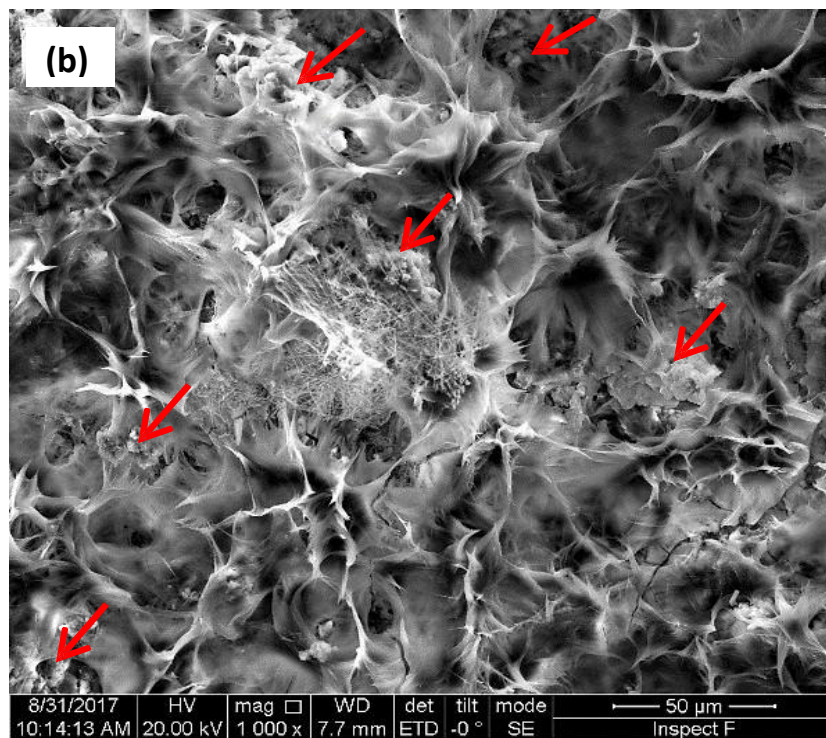


(c) High plastic soil

Figure 5.9 Dry density of cement mortar with dry sieved and stabilized soil

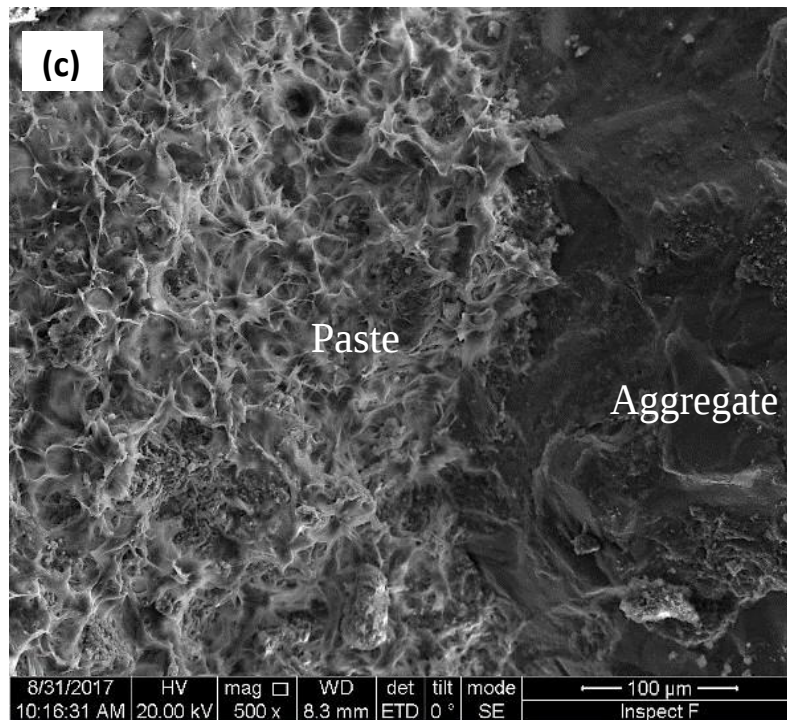


(a) LP soil showing non – reactive fines sealed within reaction products



(b) Slag stabilized high plastic soil showing agglomerated clay particles within reaction products

Figure 5.10 continued...



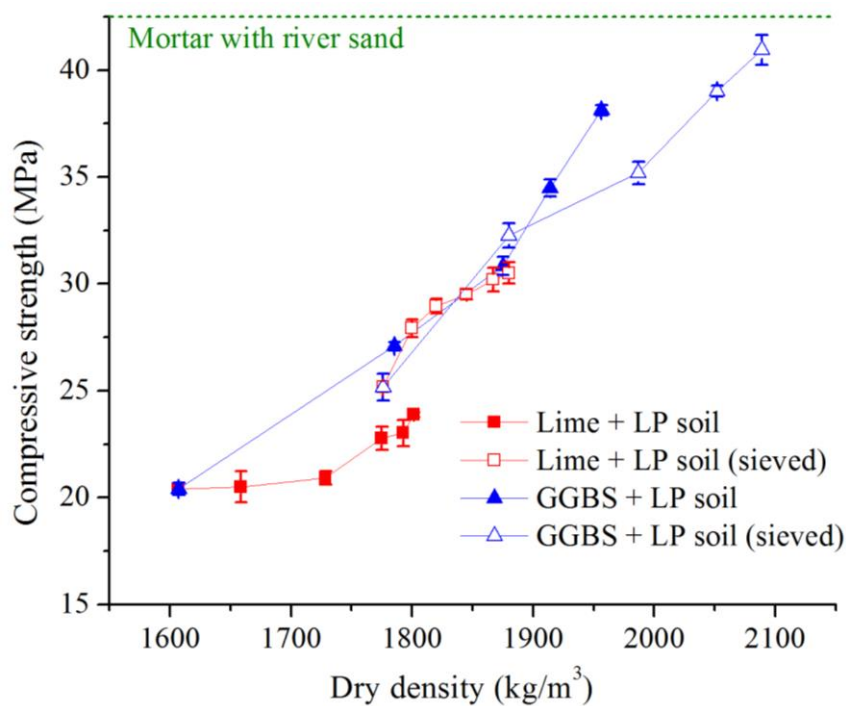
(c) Lime stabilized high plastic soil showing improved ITZ

Figure 5.10 SEM images of cement mortar with stabilized soils

Though both lime and slag results in the flocculation and agglomeration of soil particles, it can be inferred that slag performs well in stabilizing soil of low and medium plasticity whereas lime is an effective stabilizer for high plastic soil. With non-reactive clay in low plastic soil, slag as a cementitious stabilizer could provide some reactive silica and alumina to improve the density. In case of reactive clay, lime performs better by providing calcium ions to act on the clay layers, making them denser. Stabilization improves strength of soil by acting upon clay layers whereas dry sieving of soil results in granulometric correction. Together stabilization and dry sieving increases the dry density of cement mortar, irrespective of the type of soil.

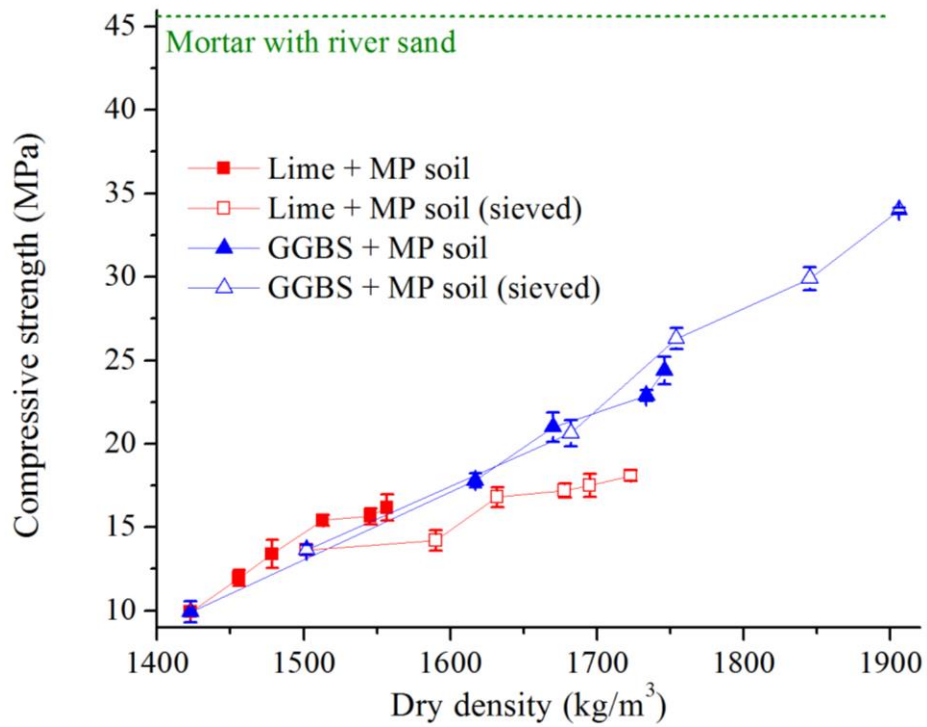
5.6.3 Compressive Strength

Compressive strength of mortar with lime and slag stabilized soil as fine aggregate is presented in Figure 5.11. The compressive strength of mortar with stabilized soil increases with stabilizer dosage. In mortar with LP soil, lime stabilization does not contribute to significant increase in strength and density improvement beyond 6% dosage whereas, slag stabilization shows linear improvement up to a dosage of 20% (Figure 5.11a). With dry sieving and stabilization, the increase in compressive strength of mortar with LP soil reached 41 MPa with 20% slag which is marginally lower than the control mortar (Figure 5.11a). With slag, there is a dense reaction product formation resulting in reduction in pore size and increase in strength (Figure 5.10a). This is substantiated by Keramatikerman et al. (2016) that presence of slag results in accelerated generation of cementitious products and pore size reduction.

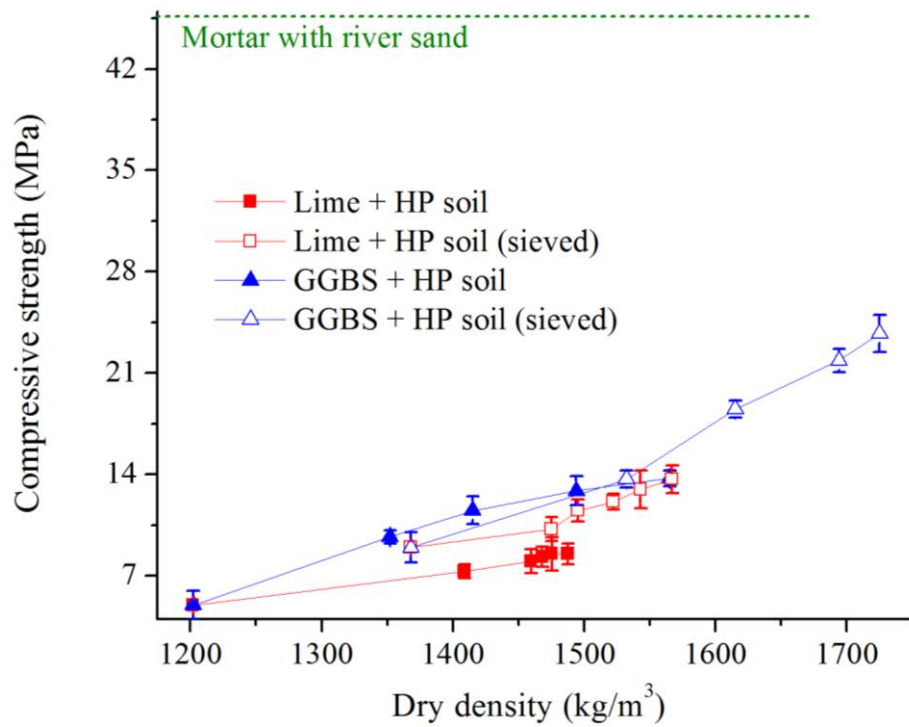


(a) Low plastic soil

Figure 5.11 continued...



(b) Medium plastic soil



(c) High plastic soil

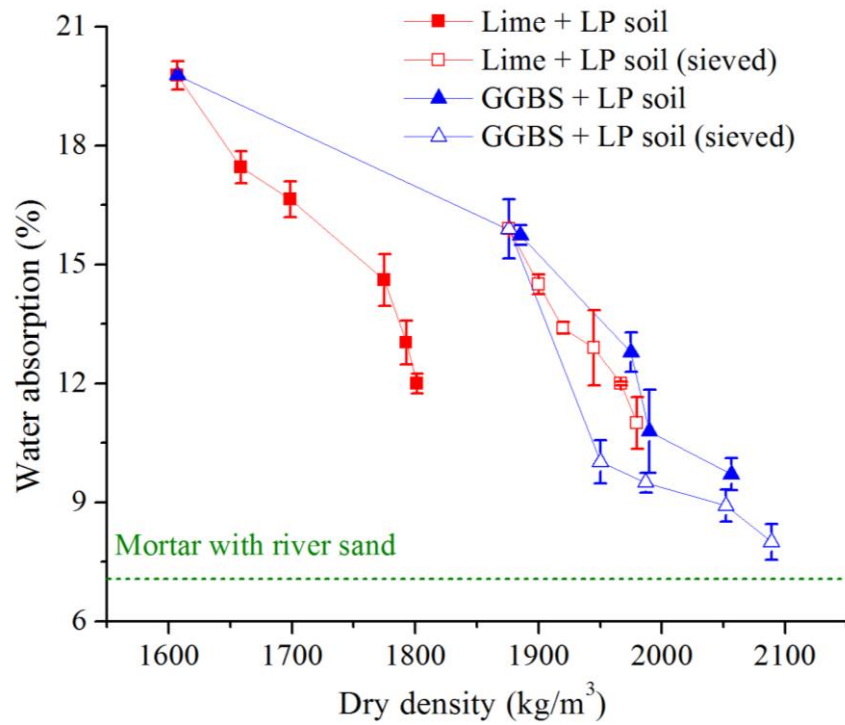
Figure 5.11 Variation in compressive strength of cement mortar with dry sieved and stabilized soil

For a given stabilizer dosage, mortar with medium and high plastic soil shows higher compressive strength with slag stabilization compared to that of lime stabilization (Figure 5.11b and c). This can be related to the density increment with slag due to the formation of dense microstructure (Figure 5.10b). The percentage improvement in compressive strength of mortar with high plastic soil is 50 – 100% higher compared to that of mortar with low and medium plastic soil. This is attributed to the presence of immense amount of desirable clay minerals (montmorillonite) in the HP soil to react with the stabilizer. However, the maximum strength achieved by mortar with stabilized HP soil is 48% less than the control mortar with river sand (Figure 5.11). This is due to the higher water demand that contributes to the density and strength reduction (Figure 5.8c).

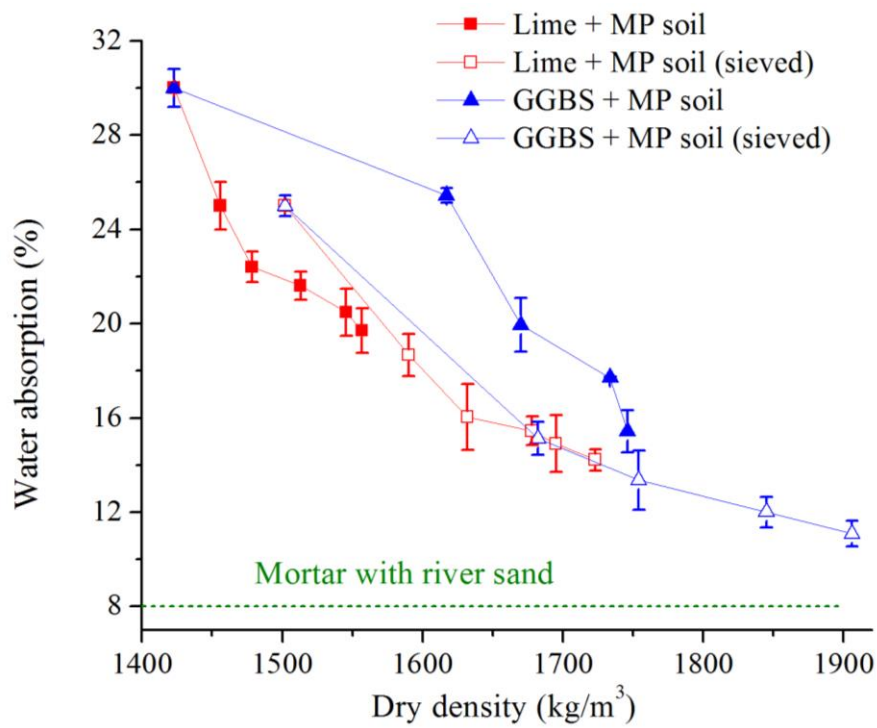
The percentage increase in compressive strength of mortar with sieved and stabilized soil is almost twice the value of mortar with unsieved soil. This is due to the granulometric improvement in addition to the stabilization effect. Interestingly, mortar with sieved and stabilized high plastic soil could achieve a compressive strength in the range of 9 to 24 MPa only for a density range of 1400 to 1700 kg/m³ (Figure 5.11c). This could be a potential light weight material with good strength range for many applications.

5.6.4 Water Absorption

Water absorption of cement mortars with stabilized soils is shown in Figure 5.12. For a constant stabilizer dosage, the reduction in water absorption is higher for slag compared to lime as stabilizer in mortar with stabilized low plastic soil, (Figure 5.12a). This can also be related to the increased strength and density of the mortar with slag stabilization. With the combination of sieving and stabilization, the mortar with LP soil could reach a water absorption value equal to control mortar at 20% dosage level of slag (Figure 5.12a). Stabilizers with cementitious property bind the silt and clay particles, which reduces the water absorption and increases the density of the system. In case of lime stabilization of LP soil, non-reactive fines (Figure 5.13a) do not consume the entire stabilizer and excess lime remains as free lime in the soil system (Figure 5.13b). This leads to the relatively higher water absorption. Mortar with MP soil shows almost similar trend as LP soil in both the cases of lime and slag stabilization (Figure 5.12b). Stabilization without dry sieving would be sufficient for producing mortar with LP and MP soil to bring down the water absorption in the range of 10 - 12%.

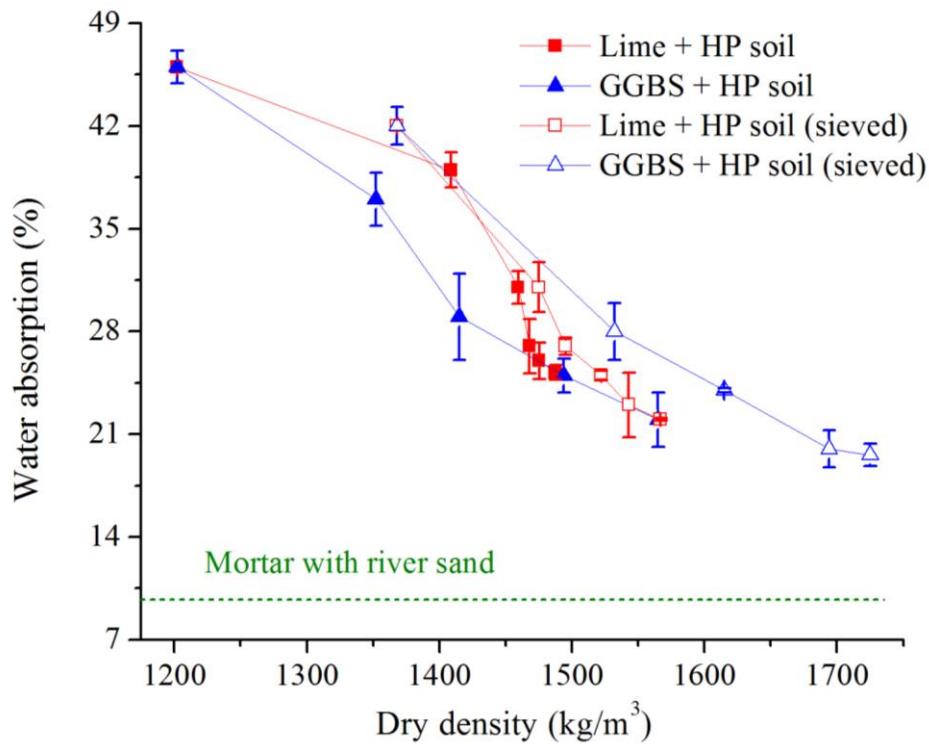


(a) Low plastic soil



(b) Medium plastic soil

Figure 5.12 continued...



(c) High plastic soil

Figure 5.12 Variation in water absorption of cement mortar with dry sieved and stabilized soil.

Mortar with HP soil shows relatively better effect with lime stabilization (Figure 5.12c). For a given dosage of stabilizer, efficiency of lime stabilization increases with increasing soil plasticity and reaches its maximum for high plastic soil. Mortar with high plastic soil shows a water absorption value of less than 20% with dry sieving and stabilization. Unlike LP and MP soil, high plastic soil needs dry sieving before stabilization to remove some of the reactive clay and adjust for its granulometry to achieve comparable results. Higher water absorption of mortar with high plastic soil can be related to its lower density range of 1400 to 1700 kg/m³ equivalent to light weight concrete reported by Nambiar and Ramamurthy (2007).

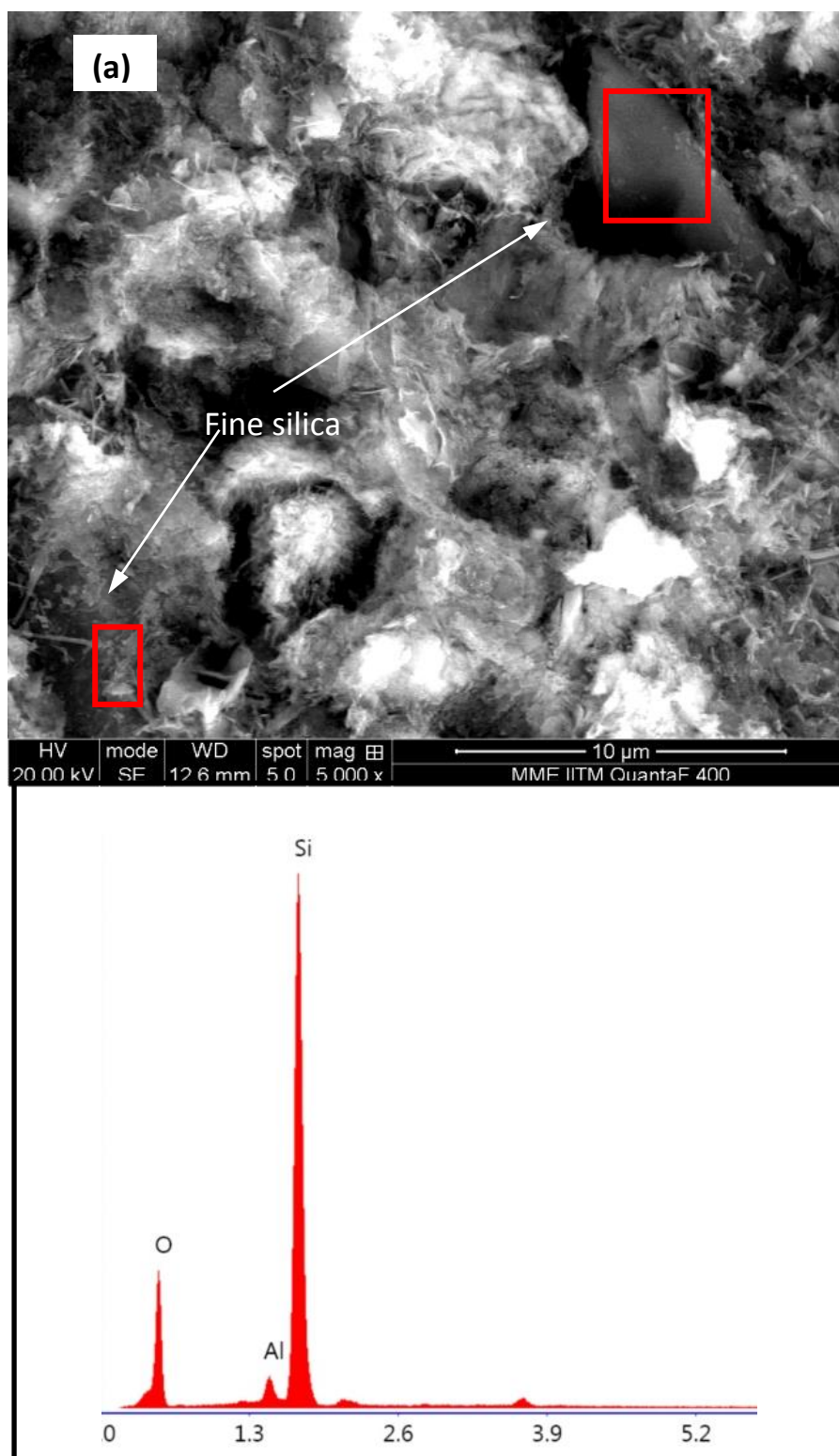


Figure 5.13 continued...

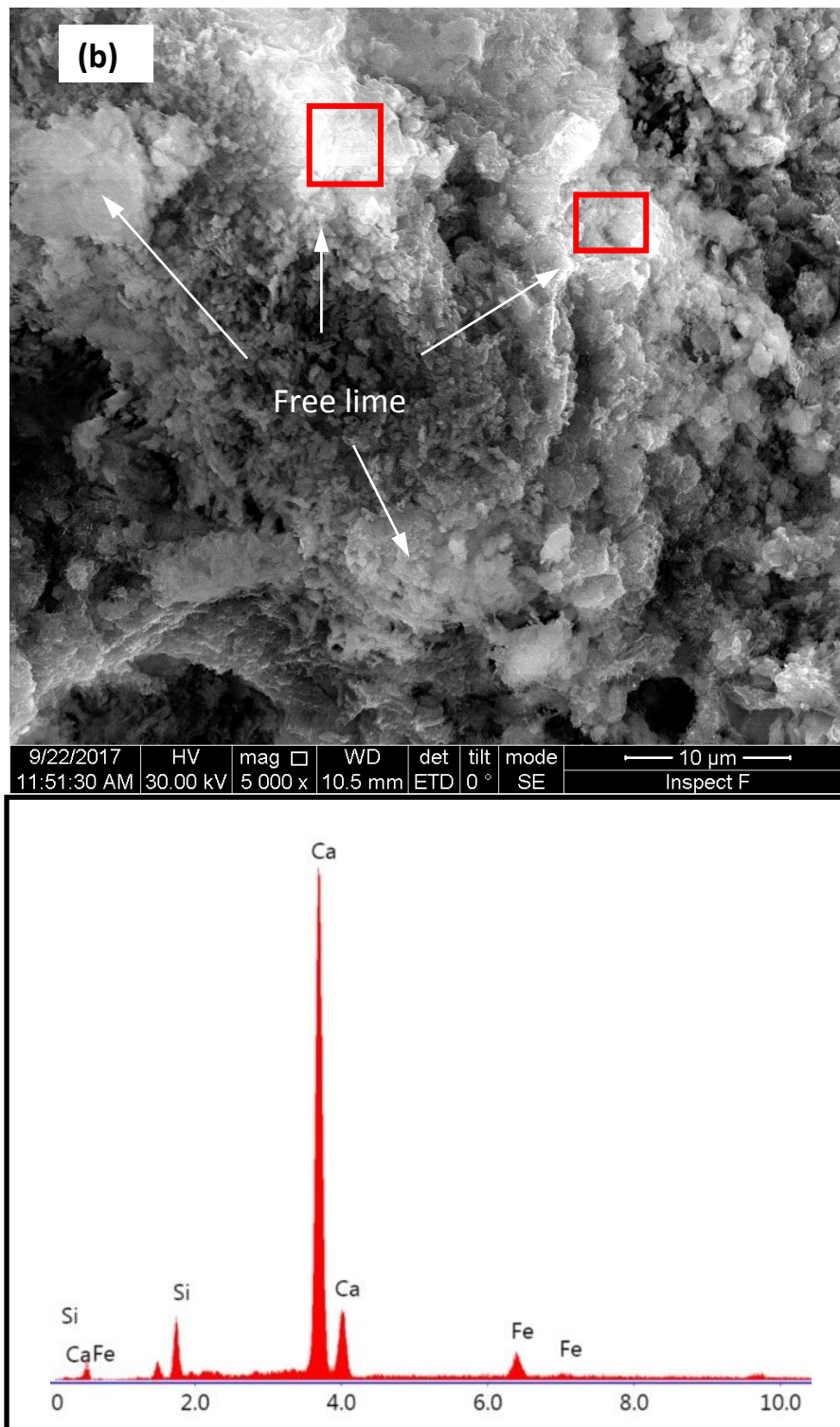


Figure 5.13 Microstructures showing low plastic soil before (a) and after (b) lime stabilization.

5.6.5 Drying Shrinkage Strains

Shrinkage is the most important property which affects the dimensional stability of the mortar and concrete. Shrinkage strain of cement mortar made with soils as fine aggregate highly depends on the plasticity, clay mineralogy and proportion of reactive clay present. Figure 5.14 shows the shrinkage strain values of cement mortar with river sand and plastic soils at different ages. With increasing plasticity, shrinkage strain reaches more than 20000 micro strains for mortar with high plastic soil which is four times higher than the mortar with low plastic soil.

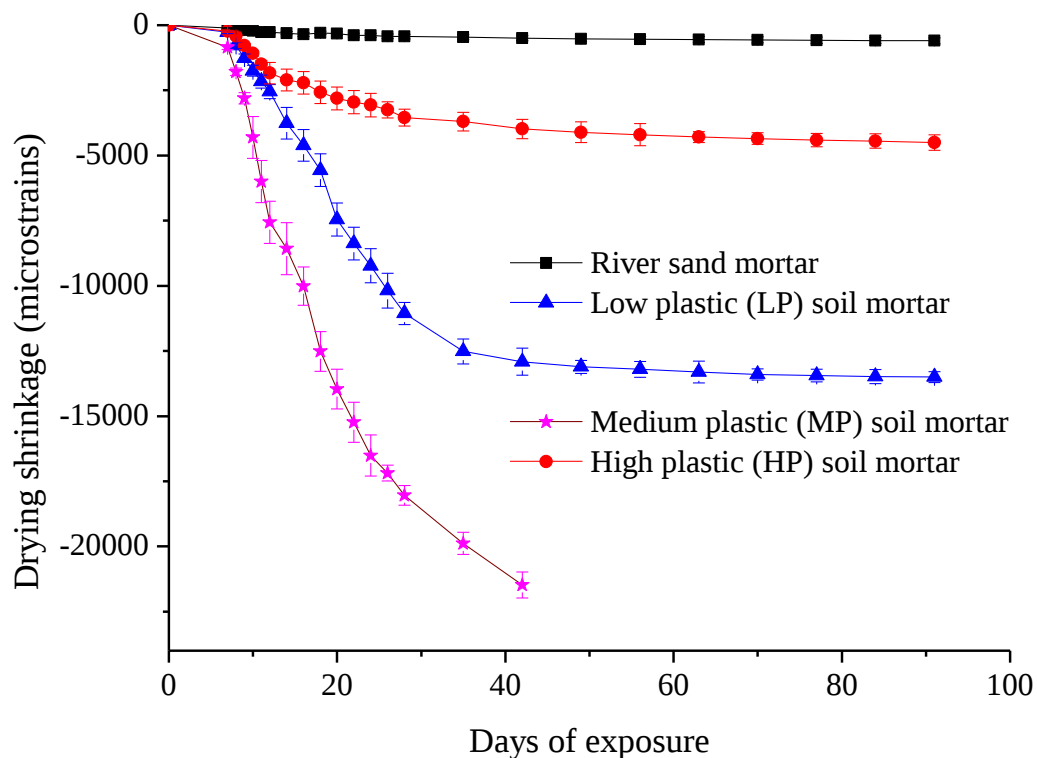


Figure 5.14 Shrinkage strains of cement mortar with different type of plastic soils

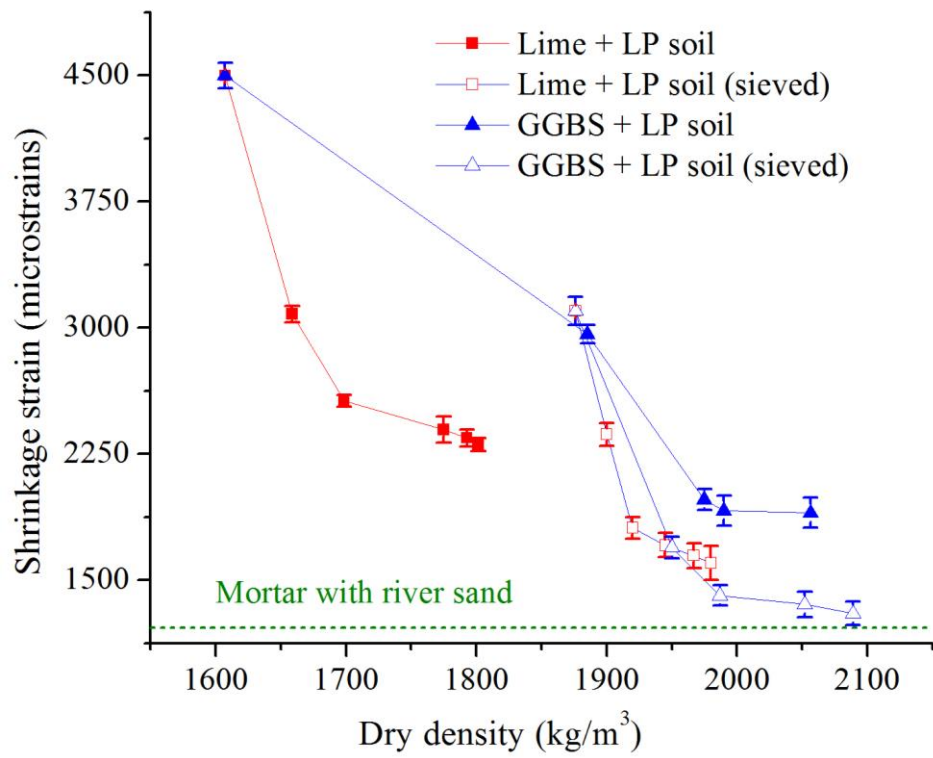
Low plastic soil mortar itself is ten times on higher side compared to control mortar with river sand. This show how intense is the problem of the presence of clay in cement system. In fact, cement mortar with high plastic soil collapsed after 40 days and the readings could not be continued. Kaolinite in low plastic soil has T-O layer which is charge balanced with a strong hydrogen bond between inter-layer which resists water entry, hence lower shrinkage strains comparatively. Whereas, montmorillonite in medium and high plastic soil with T-O-T structure with larger inter-layer space and weak Van der Waal's force, readily allows volume change by cation exchange (Young et al. 1998; Mitchell and Soga, 2005).

Smectites (montmorillonite) are the key ingredient causing volumetric changes and the intensity depends on the nature of the adsorbed ions and molecules in inter-layer sheets of clay (Shah et al. 2017). Intercalation of water molecules between the inter-planar space causes swelling of soil. Drying of soil removes inter-planar water causing desiccation, rearrangement of particles and finally cracking.

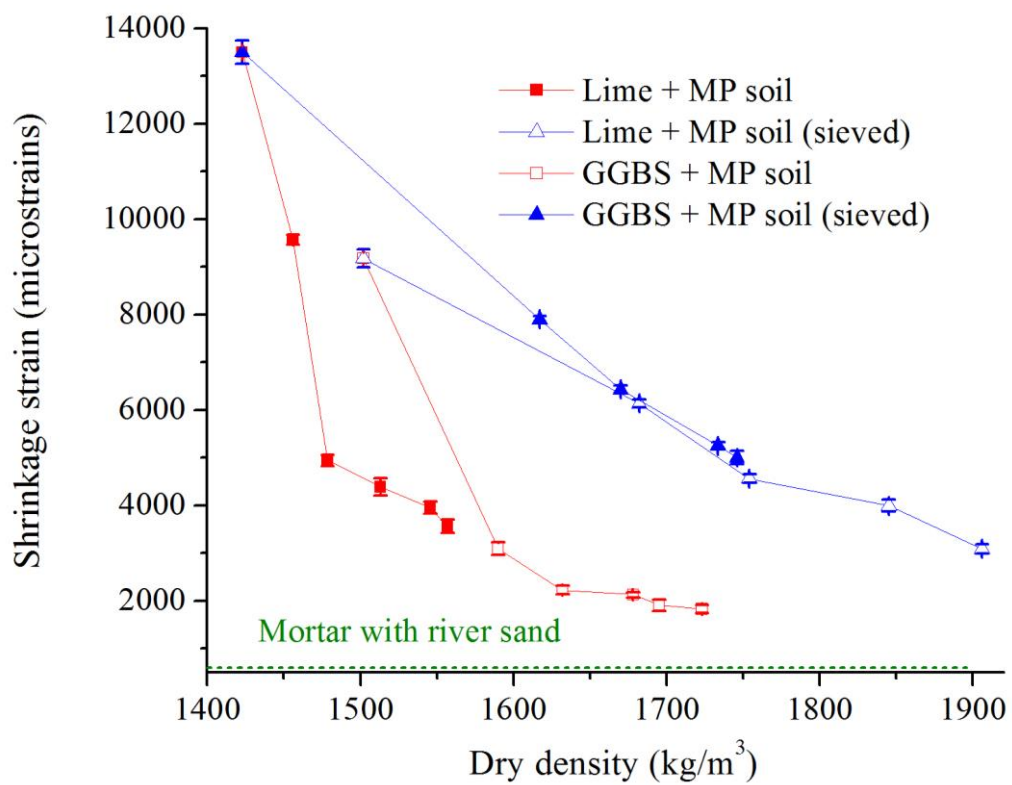
This occurs in four stages; structural shrinkage, normal phase, residual phase and zero shrinkage. Removal of water from inter-particle space (between clay particles) does not cause any volumetric change and is termed as structural shrinkage (Chertkov, 2003). In normal phase, amount of volumetric change is proportional to the quantity of water extracted from inter-layer porosity (within clay particle) and this phase is responsible for 80% of volume change (Tripathy et al. 2002). Water from micro pores of clay layers is removed in residual shrinkage phase. Zero shrinkage is the stage beyond which the clay can no more shrink. The time required to reach zero shrinkage varies with clay mineralogy and initial moisture content (Gapak et al. 2017). Though the increase in shrinkage strains can be related to the increase in water content of the mortar with plastic soils, it has been proved that with the addition of extra water in cement system containing clay does not affect the overall shrinkage (Muñoz et al. 2010). This is attributed to the fact that this extra water helps in the improved consolidation of the mixture with clay, thereby reducing the shrinkage rate. Hence, the shrinkage caused is mainly due to the action of clay to the exposed environment.

5.6.5.1 Influence of stabilizer dosage

Influence of stabilizer type and dosage on shrinkage of cement mortar with low, medium and high plastic soils is shown in Figure 5.15. Irrespective of type of stabilizer, an increase in stabilizer dosage reduces the drying shrinkage strain. Mortar with stabilized low plastic soil does not exhibit significant reduction in shrinkage strains beyond the dosage of 4% lime and 10% slag. For a constant dosage of stabilizer, mortar with low plastic soil shows a 10% higher reduction in shrinkage strain with slag stabilization compared to lime (Figure 5.15a). This is attributed to the physico-chemical phenomenon exhibited by the slag stabilization of soil.



(a) Low plastic soil



(b) Medium plastic soil

Figure 5.15 continued...

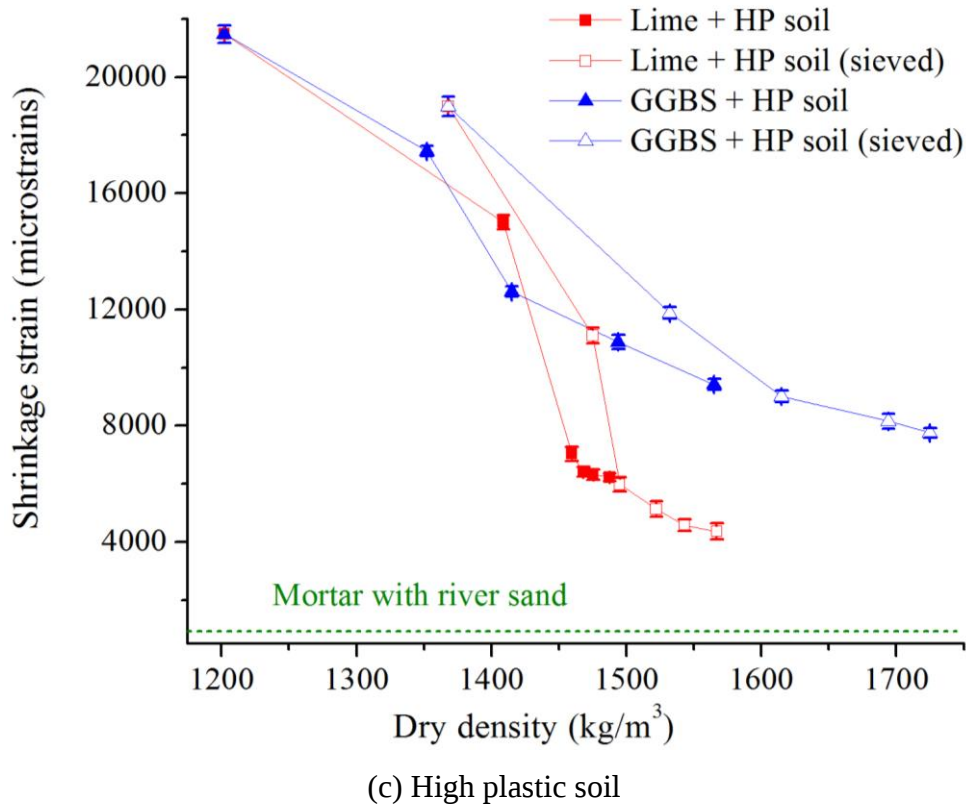


Figure 5.15 Variation in drying shrinkage of cement mortar with dry sieved and stabilized soil

Mortar with slag stabilized LP soil shows a dry density value of 2050 kg/m^3 and a corresponding drying shrinkage of 1300 micro strains. Mortar with lime stabilized low plastic soil with the dry density of 1800 kg/m^3 could reach a shrinkage value of 2300 micro strains without sieving process (Figure 5.15 (a)). This value of density and drying shrinkage is similar to the foam concrete produced by Jones and McCarthy (2005) and can be related to the formation of porous hydration products with lime stabilized soils. However, stabilization together with sieving could improve this and shrinkage can be reduced below 1750 micro strains for a density of 2000 kg/m^3 .

With increasing stabilizer dosage, there is a notable drop in shrinkage strain up to 4% of lime and 10% of slag, further which the decrease is not significant in mortar with medium and high plastic soil. The efficiency of lime stabilization is substantial in mortar with MP and HP soil on reducing the drying shrinkage strains (Figure 5.15b and c). The higher the percentage of reactive clay, hydraulic stabilizer with diffusion effect (lime) works better than the cementitious stabilizer (slag) with binding effect. The reaction of cementitious stabilizers like slag or cement is very quick and hardens fast which restricts the reaction time and is

related to the clay-size fraction (Noble and Plaster, 1970). However, increasing clay content and soil plasticity reduces the effectiveness of such stabilizers (Woo, 1971).

The microstructure of unprocessed high plastic soil shows sheets of highly expansive montmorillonite clay minerals (Figure 5.16a) responsible for the huge shrinkage values. After 28 days of lime stabilization, the continuous reaction with calcium provided by lime resulted in denser reaction products (CSH) and pore size range that helps in reducing shrinkage (Figure 5.16b). However, the drying shrinkage could not reach the value of river sand mortar (control) owing to the presence of superfluous fines content and reaction products. With sieving and stabilization, the percentage reduction in shrinkage strain increased further and it is to be noted that only after dry sieving, medium plastic soil with 10% of slag reaches a strain value less than 2000 micro strains which could be acceptable for mortar production (ACI 209, 2005).

5.6.5.2 Influence of mortar strength

The relation between strength and shrinkage strains of cement mortar with different fine aggregates is shown in Figure 5.17 (a) and (b). Mortar with lime stabilized low plastic soil with strength range of 20 to 24 MPa shows a shrinkage strain in the range of 4000 to 5000 micro strains. Increment in mortar strength with lime stabilization in medium and high plastic soil shows corresponding reduction in shrinkage strain (Figure 5.17a). Dry sieving and stabilization improves the strength further and thereby reduced the shrinkage strain irrespective of the type of soil used.

GGBS (slag) being a pozzolanic material; strength shows drastic improvement as expected. However, the corresponding reduction in shrinkage strain is not comparable to that of mortar with lime stabilized soils (Figure 5.17b). As stated earlier, the presence of micropores due to pore refinement with cementing stabilizers is the reason for such behaviour. Hence the stabilizers should be chosen wisely based on the application.

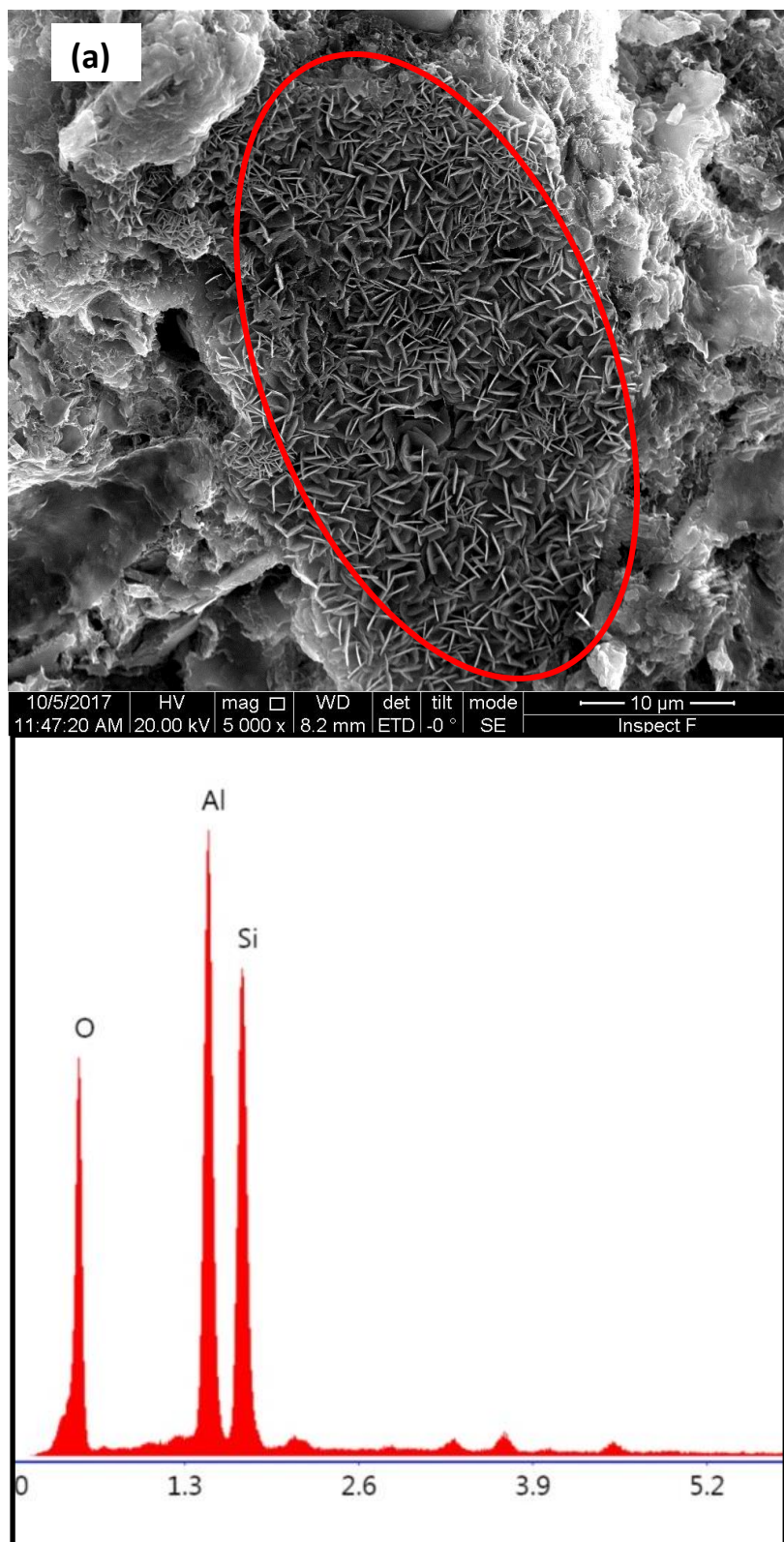


Figure 5.16 continued...

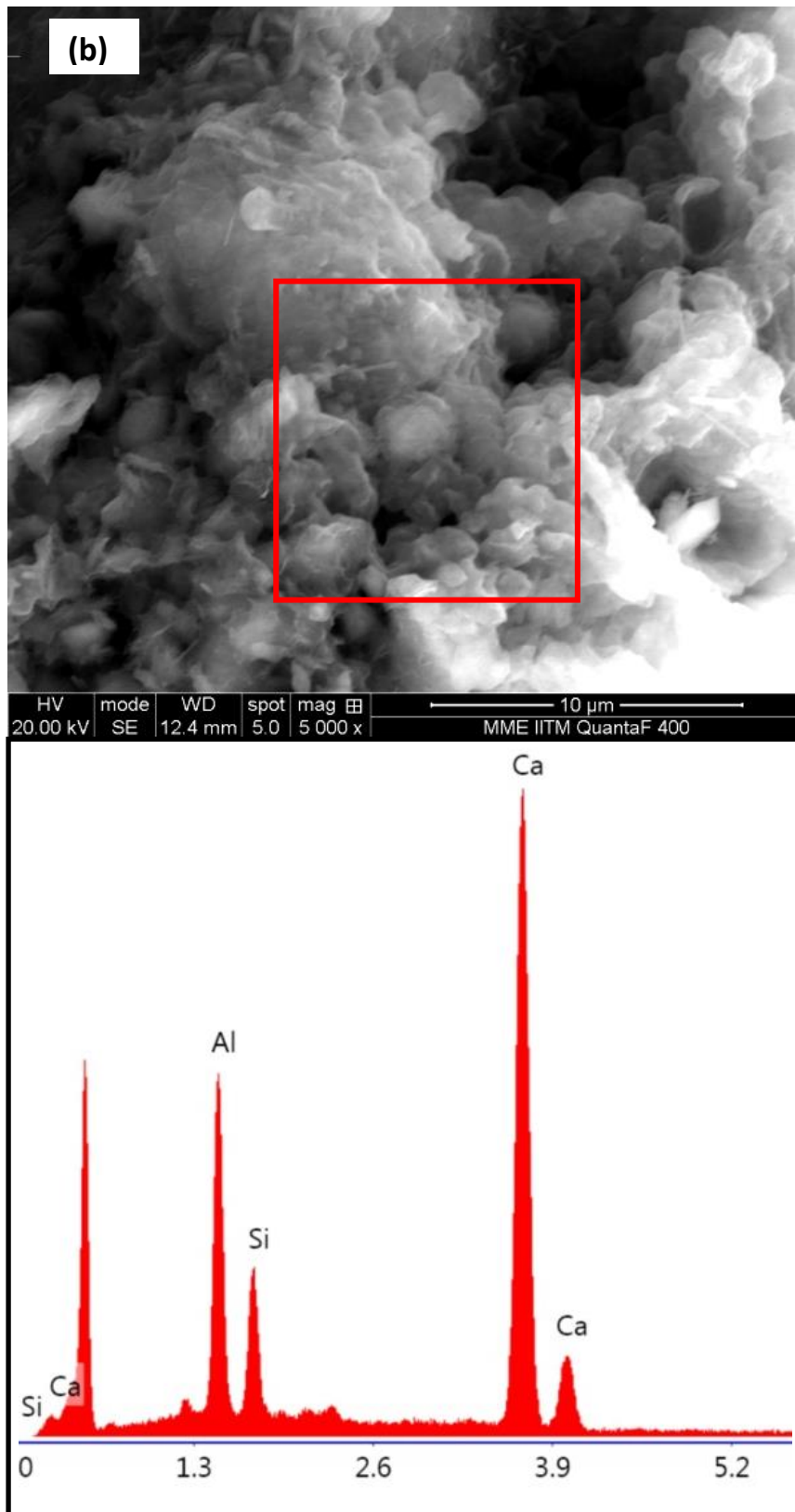
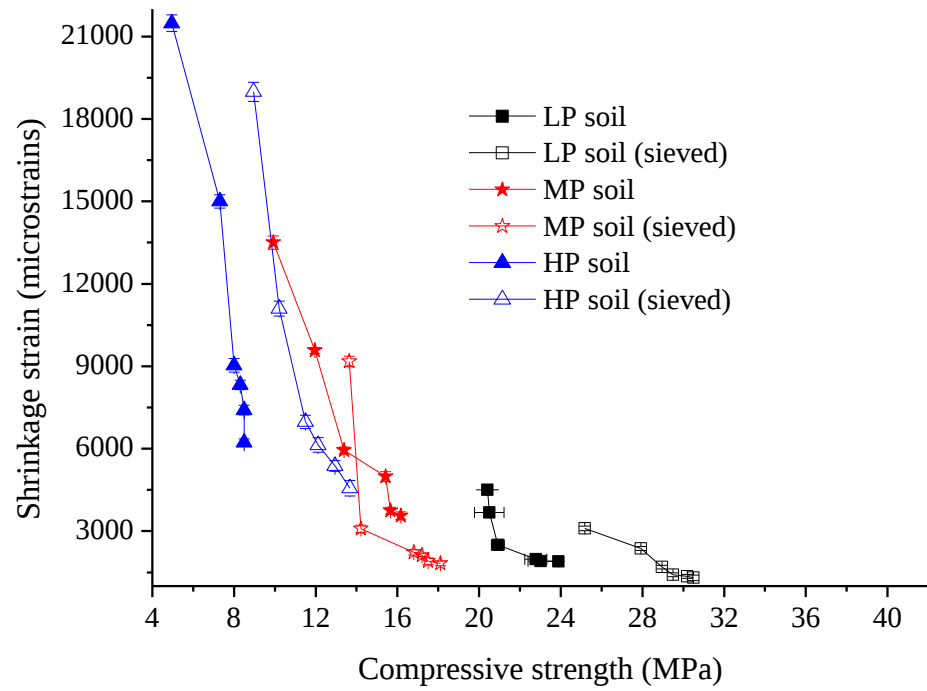
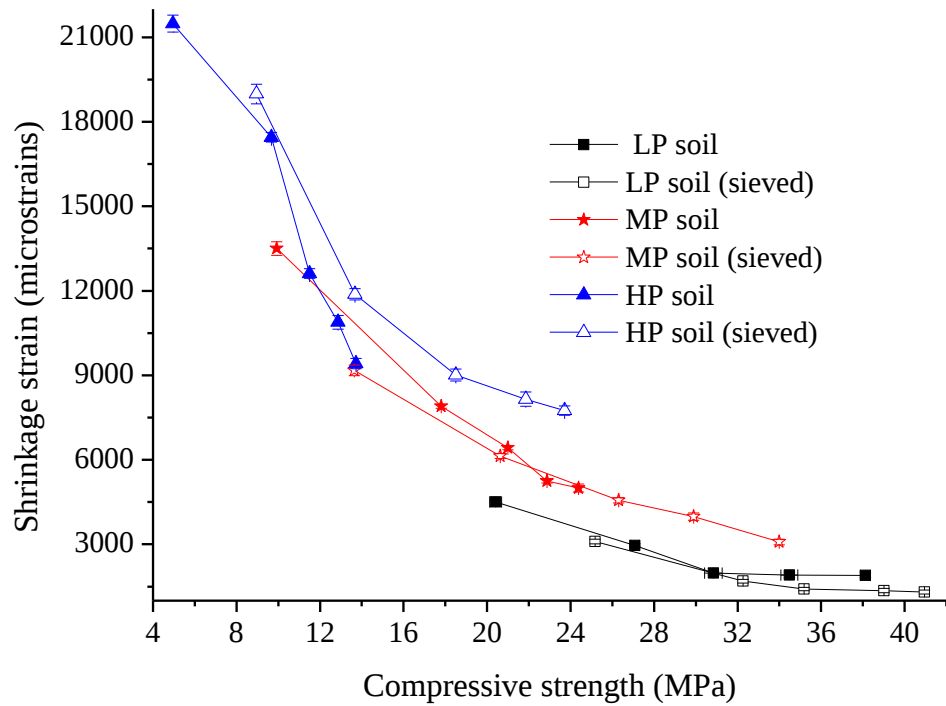


Figure 5.16 Microstructures showing high plastic soil before (a) and after (b) lime stabilization



(a) With lime



(b) With GGBS

Figure 5.17 Drying shrinkage strains of cement mortar with stabilized soil

5.7 SUMMARY

Three different soils with low, medium and high plasticity (with different proportion and type of clay) were used as fine aggregates to produce cement mortar. The same soil samples were treated by dry sieving through 600 μm size sieve and/or stabilized with calcium bearing stabilizers viz., lime and slag, to improve the mortar properties. Different dosages of stabilizers (up to 20%) have been tried to stabilize the clay present. Treated plastic soils (dry sieved, stabilized, dry sieved and stabilized) were then used as fine aggregate in cement mortar. Mortar properties such as strength and shrinkage were tested and compared with control mortar made of river sand. Results showed that it is possible to use low plastic soil as fine aggregate by employing simple dry sieving without compromising mortar properties. Medium plastic soil needs stabilization together with dry sieving to reach an acceptable limit of shrinkage strains. Though mortar with high plastic soil shows improvement in properties, shrinkage strains are not controlled with these treatment methods.

CHAPTER 6

PERFORMANCE OF WET – SIEVED EXCAVATION SOIL AS FINE AGGREGATE IN CEMENT MORTAR

6.1 GENERAL

Wet sieving or washing aggregates is a method adopted to remove particles of poor quality which affects concrete properties as mentioned in Chapter 2. Details of the washing process employed in different fine aggregate alternatives are presented in Table 6.1. Al-Ansary et al. (2012) used washing technique to remove excess ions, clay and fines present in dune sand and studied for its suitability in mortar/concrete applications. Olanitori (2006) reported that to overcome the strength reduction due to higher clay and fines content, the proportion of cement should be increased, if not washing should be adopted to reduce cement consumption and make the process cost effective. Washing is reported to be one of the common and simplest methods adopted for different materials that have siliceous content in them (Al-Ansary et al. 2012; Monosi et al. 2013; Sierra et al. 2011; Ulsen et al. 2013). Cepuritis and Mørtzell (2016) reported that washing of manufactured sand to remove fines resulted in better properties than untreated manufactured sand (Cepuritis and Mørtzell 2016). Rain water is used for washing the offshore sand by dumping them in free space, to remove chloride ions (Dias et al. 2008; Ratnayake et al. 2014). Desalination by washing was also adopted for marine sediments, followed by dewatering and oven drying to improve their quality in cementitious systems (Ozer-Erdogan et al. 2016). In a study by Modolo et al. (2013), industrial washing plant was used to remove chlorides from bottom ash to produce 10 ton/h of the treated material and the wash water was reused for further washing after sedimentation. Separability studies were carried out by Ulsen et al. (2013) in recycled sand through wet screening, heavy liquid separation, elutriation and magnetic separation to remove the patches of adhered cement paste.

Katz and Baum (2006) explained that fines content, though demands increased water content to maintain workability, helps in improving the strength and durability properties when admixtures are used. However, presence of fines beyond a certain limit does not help in filling the voids rather increases it (Cho 2013). Al-Ansary et al. (2012) used washing technique to remove excess ions, clay and fines present in dune sand and studied for its suitability in mortar/concrete applications.

Table 6.1 Literature on wet sieving/washing of alternative fine aggregate materials

Author, Year	Material	Salient observations
Cepuritis and Mørtzell (2016)	Manufactured sand	Washed the particles in the size range of 30 to 250 μm . Particles < 40 μm reduced from 28% to 4.5%. Workability of concrete improved with washed sand.
Ratnayake et al. (2014)	Offshore sand	Natural washing by rainfall removed chlorides from the offshore sand.
Dias et al. (2008)		Fresh water sprinkled from top to stimulate rainfall. The mesh at the bottom of the container allowed drainage of wash water with chlorides.
Olin-Estes and Palermo (2001)	Marine sediments	Most contaminated fraction is removed before utilizing the sediments in different applications. Screens of 75 μm and hydro-cyclones are used. Density separation is also employed if density difference is sufficient. Dewatering helped in reducing the volume of material to handle.
Ozer-Erdogan et al. (2016)		Washed to remove chlorides, dewatered by filter-press, oven-dried in 105°C and sieved in 63 μm sieve.
Monosi et al. (2013)	Foundry sand	Mixed with tap water in a mechanical mixer for 24 h and washed with a sieve size of 75 μm . Wash water is used in concrete production.
Modolo et al. (2013)	Bottom ash	Commercial washing plant of 10 ton/h capacity is used to remove soluble salts. Wash water is treated and reused for further washing.

Though it is mandatory to remove the silt and clay from soil to use them in cementitious systems, the residual sludge has its own applications. Light weight aggregates made of washing residual sludge could be possibly applied for insulation and geotechnical applications (González-Corrochano et al. 2012) and could also be a potential geopolymer precursor (Lampris et al. 2009). When thermally treated in the temperature range of 750°C to 950°C (calcined), dredged dam sediments (mud) proved to be a pozzolanic material in cementitious systems (Laoufi et al. 2016). Souza and Dal Molin (2005) showed that the

calcination of clay between 550°C to 950°C could activate it by distorting the structure of comprised clay minerals and improve its pozzolanic reactivity.

In this study, the wet sieving method is adopted to improve the quality of excavation soil for its application as fine aggregate in cementitious systems. This treatment of excavation soil results in three products namely, sand ($>75\ \mu\text{m}$), wash water and fines (silt and clay sized particles) with particle size less than $75\ \mu\text{m}$. This study aims to provide a total solution for excavation soil by using these three products of wet sieving in construction applications. The relative performance of wet sieved sand and river sand as fine aggregate in cement mortar are evaluated and compared. Though the main aim of the study is to identify an alternate for fine aggregate in cementitious system, the by-products of wet sieving viz., wash water and fines (residual clay) are studied for its constructive utilization to make the process sustainable. The wash water was investigated for its application as mixing water in cement mortar production. Calcined residual clay was tested for its use as pozzolan in cement mortar. Calcination is adopted to treat residual clay, as it could be applied as supplementary cementing material (SCM) in cement mortar which is more related to the present study than other applications like light weight aggregate or geopolymer.

6.2 WET SIEVING PROCESS

Low (LP) and medium plastic (MP) soil were used in wet sieving process and represented as raw soil-1 and soil-2, respectively, in this chapter. High plastic soil with more than 50% of particles were less than $75\ \mu\text{m}$ in size and with 41% of clay (mostly swelling type which clogs the sieve) makes it difficult to treat in wet sieving method. Particle size distribution of raw and treated soils is compared with that of river sand in Figure 6.1. Raw excavated soils were finer than river sand. In particular, soil-2 was finer than soil-1. As per ASTM C33 (2016), up to 3% of particles less than $75\ \mu\text{m}$ are allowed to be present with aggregate that is used in concrete production. The particles finer than $75\ \mu\text{m}$ in soil-1 and soil-2 were 29% and 48%, respectively (Table 3.1). These particles include clay and fines that are deleterious to concrete and hence require processing/treatment to make them suitable for fine aggregate in mortar/concrete.

Wet sieving has been used to separate residual clay (which is less than $75\ \mu\text{m}$ size) from sand particles. The procedure as given in ASTM C142 (2017) was adapted to perform wet-sieving of raw soil. Raw soil sample was first mixed with tap water to produce slurry. The

slurry was poured in to 1.18 mm size sieve allowing the water along with particles to pass through. Material retained (>1.18 mm) was then washed well in running water until the water looked clear. Retained particles were collected separately in a bin. Particles less than 1.18 mm size were collected in a container placed below the sieve. The material passing 1.18 mm was further washed following a similar procedure in a $75\text{ }\mu\text{m}$ size sieve. The sand particles that were retained on the $75\text{ }\mu\text{m}$ sieve were stored together with 1.18 mm retained materials. The wet sieved sand ($>75\text{ }\mu\text{m}$) was sun-dried for two days and used as fine aggregate in mortar preparation.

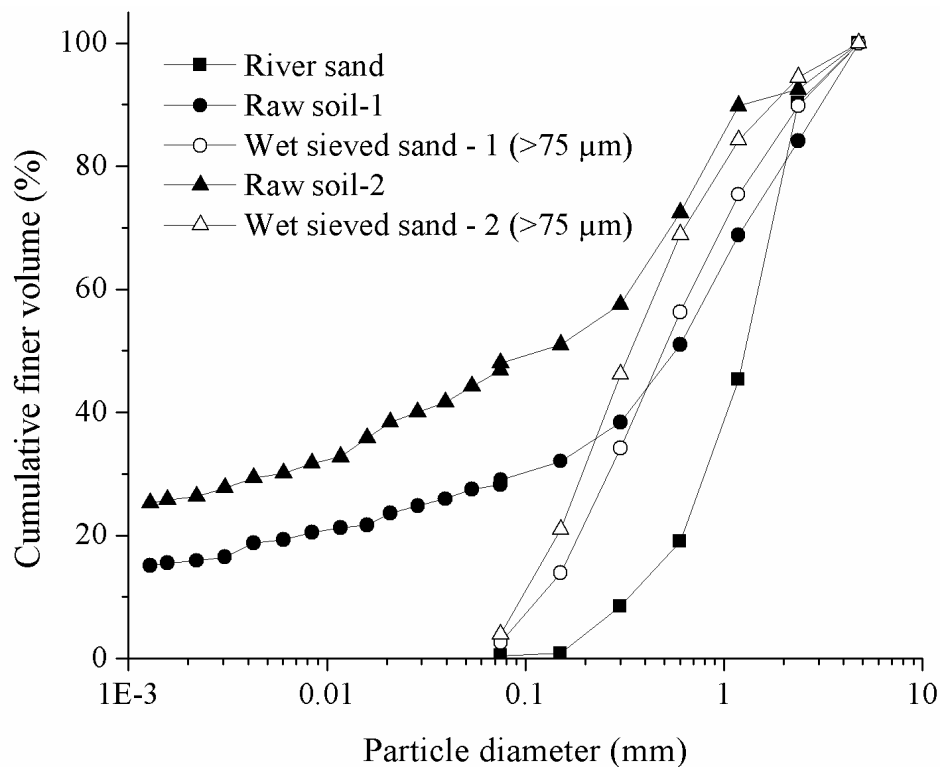
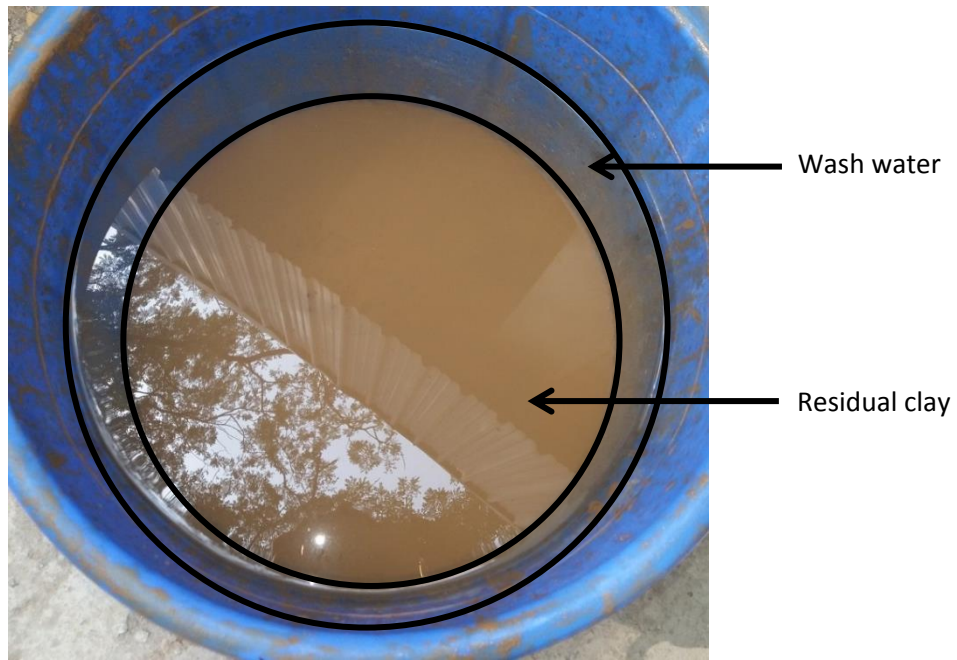


Figure 6.1 Particle size distribution curve

The residual slurry (referred as residual clay in further discussions) with fines and clay that are less than $75\text{ }\mu\text{m}$ was kept undisturbed for 24 hours, allowing the particles to settle down completely. Later, the crystal clear wash water was siphoned out from the mixture leaving behind the fines and clay particles (Figure 6.2a). The residual clay, after removing the water, was sun dried, pulverised and kept separately for further processing.

The three main outcomes (Figure 6.2) of this process are,

- Wet sieved sand (Figure 6.2b) with particle size greater than $75\text{ }\mu\text{m}$
- Wash water and
- Residual clay (Figure 6.2c) with particle size less than $75\text{ }\mu\text{m}$



(a) Residual clay and wash water



(b) Retained sand particles

(c) Sun dried residual clay

Figure 6.2 Products of wet sieving of soil

6.2.1 Properties of Wet Sieved Sand

By wet sieving 71% and 52% of sand were recovered from the raw excavation soil-1 and 2, respectively. They were characterised by means of bulk density, void ratio and specific gravity and presented in Table 6.2. Particle size distributions of wet sieved sand are compared with that of river sand in Figure 6.1. Both wet sieved sands were finer than the river sand, which was used as aggregate for the control mix. A comparison of raw soil and wet sieved sand (Table 3.1 and Table 6.2) shows that removal of fines aided in improving the bulk

density and reducing the void ratio. In wet sieved sand-2, being finer than wet sieved sand-1, the bulk density got reduced due to the difference in particle packing. Specific gravity of wet sieved sands has been found to be close to that of river sand.

Table 6.2 Physical properties of river sand and wet sieved sands

Properties	River sand	Wet sieved sand-1 (particles >75 μm)	Wet sieved sand-2 (particles >75 μm)
Bulk density (kg/m^3)	1680	1675	1603
Void ratio (%)	36	35	40
Specific gravity	2.62	2.58	2.67

The effectiveness of wet sieving method of soil processing was studied through the properties of cement mortar cast using river sand, raw soil- 1 and 2 and wet sieved sand 1 and 2 (after processing) as fine aggregate. Commercially available Ordinary Portland 53 grade cement conforming to IS 12269 (2013) was used. Fine aggregate to cement (A/C) ratio of the mortar mix was varied from 3 to 5 by weight. Workability, dry density, compressive strength (at 28 days), water absorption and shrinkage properties of the mortar were studied.

Mortar flow test was performed thrice for each mix with flow table (ASTM C1437, 2015). The water content required to maintain a constant workability of $110 \pm 5\%$ was measured. For each mix, 12 numbers of 50 mm cube specimens (3 for dry density, 3 for water absorption and 6 for compressive strength) and 3 numbers of prisms of size 160 mm $\times$ 40 mm $\times$ 40 mm for shrinkage measurements were cast. Dry density was measured after the specimen was kept in oven for 24 hours at temperature of $110 \pm 5^\circ\text{C}$ and then allowed to cool to room temperature. Water absorption was determined as per ASTM C1403 (2015). Shrinkage specimens were initially cured under water for seven days after demoulding. Then, the specimens were taken out, wiped off water and initial reading was taken using a length comparator. Later, shrinkage readings were taken at specific intervals as per ASTM C157 (2017).

Mercury Intrusion Porosimetry (MIP) was used to study the porosity in different mortar specimens. Thermo Scientific Pascal 440 instrument with a maximum pressure of 440 MPa which can sense pores as small as 3 nm was used for this study. Small chunk of mortar samples was drilled from 100 mm cube specimens. Mercury was then forced in to vacuum dried samples. With raising pressure, mercury enters smaller pores. The porosity is expressed using the Washburn-Laplace law which gives the relation between diameter (D) of the mercury intruded pores, the surface tension of mercury (γ) and the cosine of the contact angle (θ) and the corresponding pressure (P). Hence the applied pressure on mercury can be used to estimate the radius of pores present in the mortar samples from the Washburn equation (Washburn, 1921) as in equation 6.1,

$$D = \left(\frac{-4\gamma \cos\theta}{P} \right) \quad \text{----- (6.1)}$$

6.3 USE OF WET SIEVED SAND AS FINE AGGREGATE IN MORTAR

6.3.1 Water Demand for Constant Workability

The water demand of cement mortar achieved for constant workability is presented in Figure 6.3. The use of raw excavated soil in mortar increases the water demand by up to 160%. Compared to river sand, soil-1 has 29% and soil-2 has 48% particles of size less than 75 μm . Presence of finer particle fraction in excavated soil increases the water demand as the surface area to be wetted is larger. In addition, the presence of clay in raw excavated soil increases the water demand further for maintaining the workability. Due to its layered structure, clay has the ability to adsorb water in to its microstructure, especially the montmorillonite type of clay (Fernandes et al. 2007).

For a given type of fine aggregate, an increase in fine aggregate to cement (A/C) ratio increases the water demand marginally. After removing the particles less than 75 μm by wet sieving, the water demand was almost close to that required for the control mortar. Removal of clay particles makes the wet sieved sand inert, reducing the water consumption. However, water demand for mortar with wet sieved sand-2 is higher by 27% compared to the control mortar. This can be explained by its particle size distribution (Figure 6.1). Wet sieved sand-2 is the finest compared to river sand and wet sieved sand-1 with poor particle packing shown by its higher void ratio in Table 6.2.

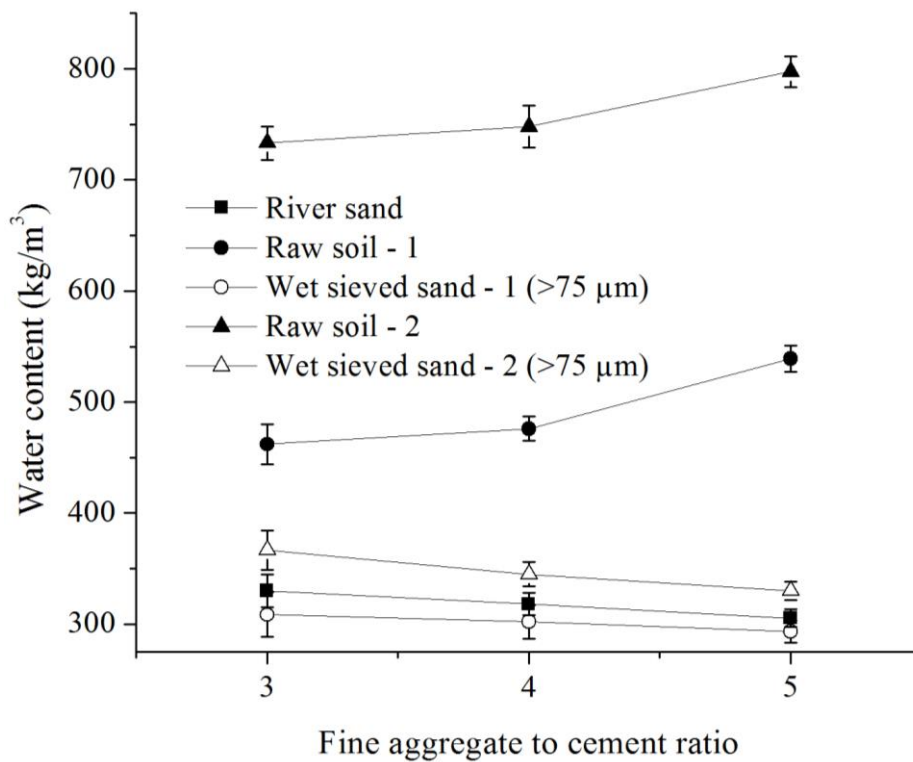


Figure 6.3 Variation in water content for different mortar mixtures

6.3.2 Dry Density

The dry densities of cement mortar cubes with different types of fine aggregates are presented in Figure 6.4. Mortar prepared with excavated soil shows reduced dry density in the range of 1425 to 1775 kg/m³ compared to the control mix. This can be related to the highest bulk density of river sand followed by raw excavated soil-1 and 2 (Figure 3.1). Also, the lowest dry density was noted for mortar with soil-2 containing 25% clay followed by mortar with soil-1 containing 15% of clay.

After wet sieving, dry density increased to a range of 1925 to 2150 kg/m³ which is closer to that of the control mortar. From Table 6.2, it is noted that there is considerable decrease in void ratio and increase in bulk density of wet sieved sands which could be the possible reason for this increase in dry density. However, void ratio of wet sieved sand-2 is higher (40%) than for wet sieved sand-1 (33%) and river sand (36%) resulting in a drop in dry density of its mortar. There is no appreciable variation in dry density when fine aggregate to cement (A/C) ratio is varied from 3 to 5.

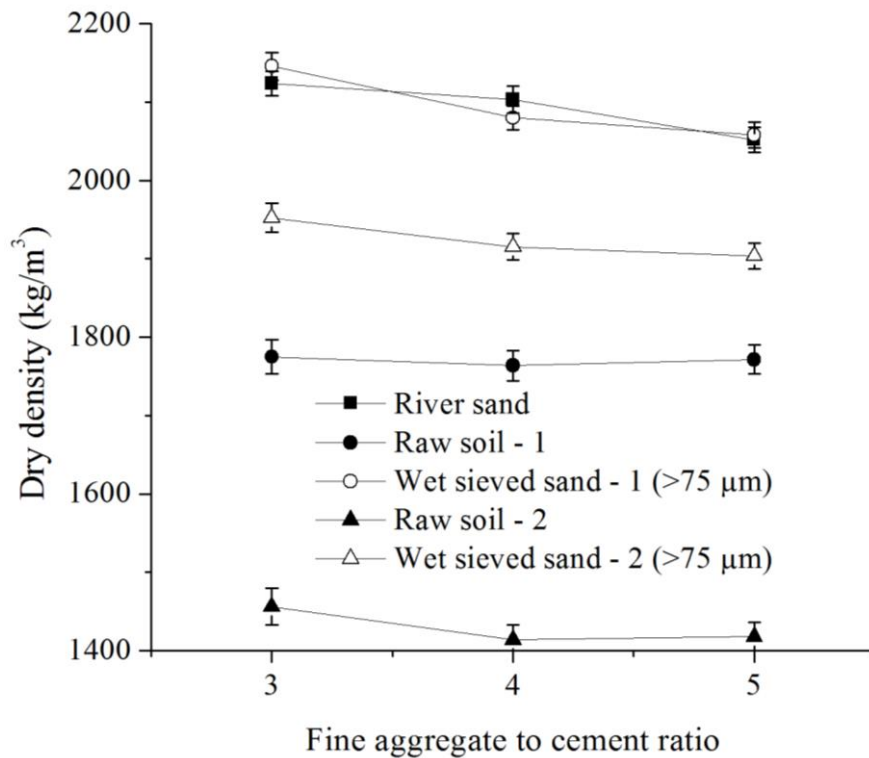


Figure 6.4 Dry density of cement mortar with soil, before and after processing

6.3.3 Compressive Strength

The 28-day compressive strength values of mortar specimens incorporating excavated soils before and after processing are shown in Figure 6.5. Compressive strength of mortar with untreated excavated soil reduces with increasing clay content. This is due to the loosening effect of clay particles between cement and aggregate (Monosi et al. 2013). The presence of clay/fines increases the surface area to be covered by cement paste making weak links in the network when the cement paste is insufficient. Hence, mortar with raw excavation soil-1 and 2 shows strength reduction of 55% and 78%, respectively compared to the control mortar.

Improvement in compressive strength with wet sieving was 107% and 161% for wet sieved sand-1 and wet sieved sand-2 compared to raw excavation soil-1 and soil-2, respectively. However, there is reduction in strength of mortar with wet sieved sand-2 compared to the control mix. This is attributed to the fineness and particle size distribution of the wet sieved sand compared to river sand (Figure 6.1). This shows that the properties of cement mortar with treated excavated soils also depend on the source.

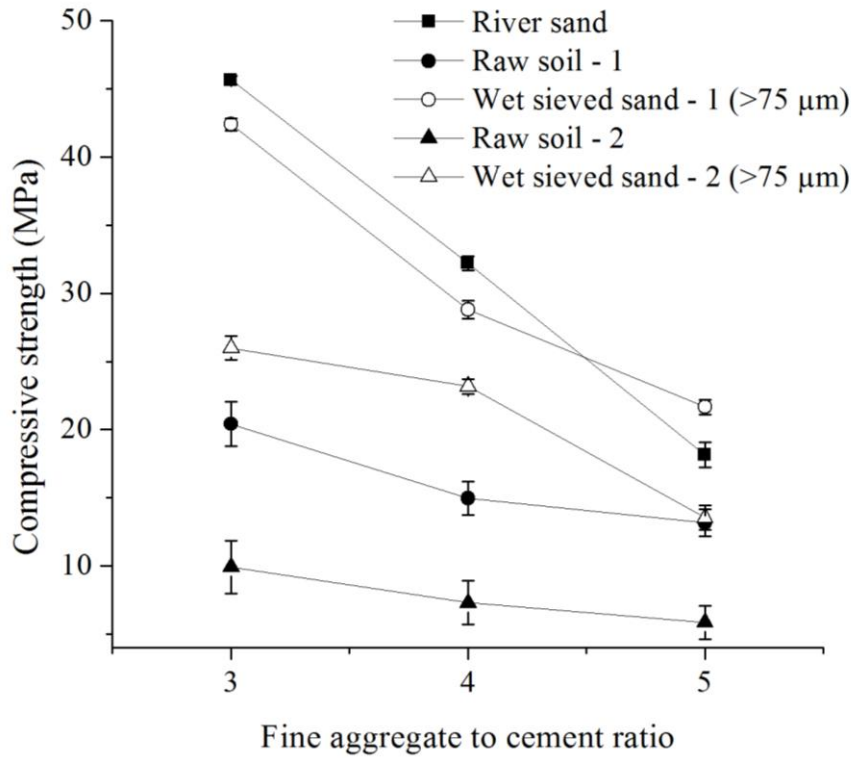


Figure 6.5 Compressive strength of cement mortar with different fine aggregates

When the fine aggregate to cement (A/C) ratio increased, the compressive strength decreased. The subsequent reduction in cement content with increasing A/C ratio is the possible reason for such strength drop. Finer the aggregate, lesser is the strength variation with increasing aggregate proportion (Figure 6.5). However, mortar with raw soil shows less strength variation compared to the control mix. The increase in cement content does not contribute to the proportionate increase in strength for mortar made with raw excavated soils.

6.3.4 Water Absorption

The percentage of water absorption of different mortar mixtures incorporating river sand, excavated soils and treated excavated soils is shown in Figure 6.6. Soil-2 shows the highest absorption followed by soil-1. Excavated soil with clay content and fine particles resulted in higher percentages of water absorption of 20 to 30%. This has been reduced by more than half the value after the wet sieving process to 9 to 14%.

Mortar mix with wet sieved sand-1 reaches a value close to the control mix. The variation of water absorption between wet sieved sand-1 and wet sieved sand-2 can be well explained by the pore size distribution of mortar mixes. To know the relative pore size distribution that causes variation between the properties of wet sieved sands 1 and 2, Mercury

Intrusion Porosimetry (MIP) study was performed on mortar samples with fine aggregate to cement ratio of 3. Figure 6.7 shows the plots of cumulative intrusion with the corresponding pore radius for different mortar samples with river sand, wet sieved sand-1 and soil-2. This explains the variation in mortar properties when wet sieved sands from two different sources were incorporated as fine aggregate.

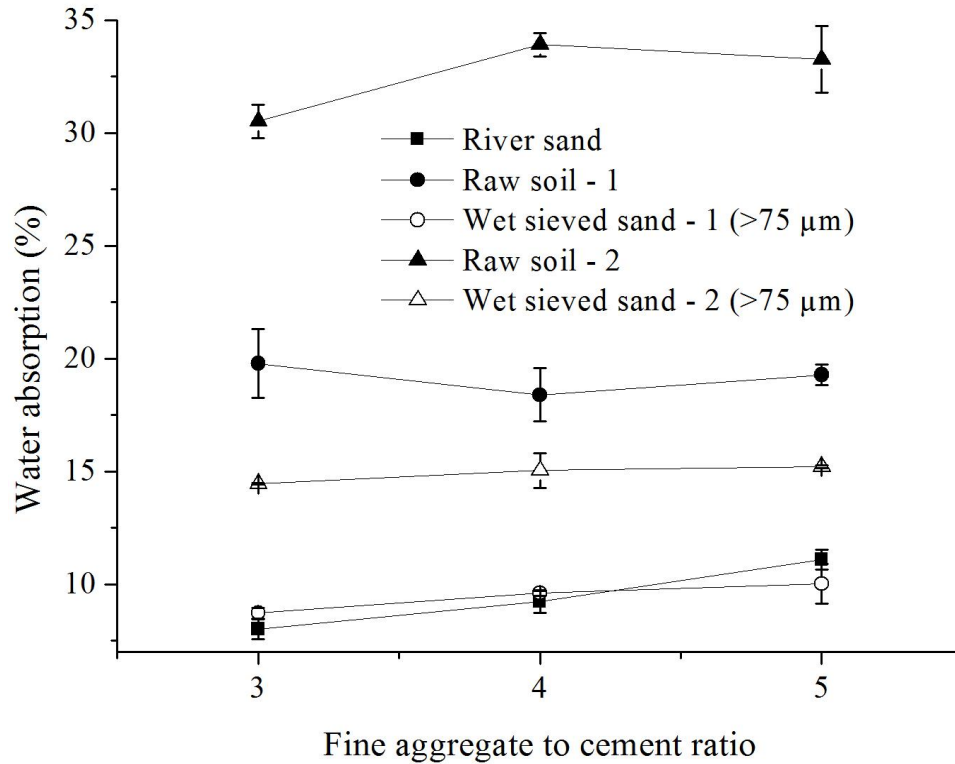


Figure 6.6 Water absorption of cement mortar with different fine aggregates

The threshold diameter is defined as the minimum pore diameter that forms continuous network of permeability inside the mortar. Thus, threshold diameter represents the critical pore diameter of interconnected pores that allows rapid entry of water thereby increasing the total intrusion volume. The threshold diameter of all the three mortar mixtures was less than 1 μm with negligible variation. However, the cumulative volume is higher for mortar with wet sieved sand-2 compared to the control mortar and mortar with wet sieved sand-1. This clarifies that the former has higher volume of connected pores which resulted in increased water absorption value and reduced strength as explained in the previous sections.

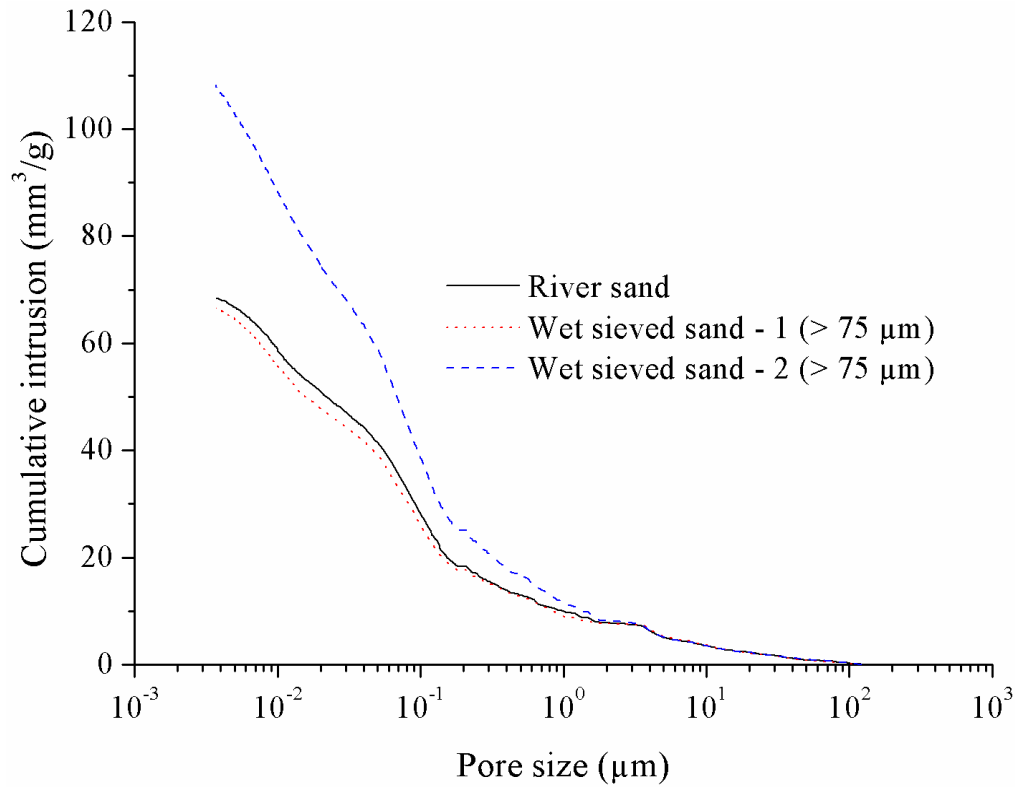


Figure 6.7 Cumulative intrusion from MIP of cements mortar (1:3) with river sand and wet sieved sand

6.3.5 Drying Shrinkage

The drying shrinkage of the mortar samples with different fine aggregates was determined at different ages and presented in Figure 6.8. The shrinkage strain of mortar with raw soil-1 at 28 days of exposure was 7 times higher than that of the control mortar and mortar with raw soil-2 was 20 times higher than the control mortar. The higher shrinkage value of mortar with raw soil-2 is due to the expansive montmorillonite type of clay present in the soil. Drying shrinkage of mortar specimens are highly related to the type of fine aggregate used (Monosi et al. 2013). The presence of clay increases the drying shrinkage and the magnitude of shrinkage depends on the type of clay mineral (Nehdi 2014).

After the removal of the shrinkage causing clay particles by wet sieving, the strain due to shrinkage reduces drastically to 86% of their raw counterparts, for both the excavation soils (Figure 6.8). However, there is a slight increase in shrinkage strains of mortar with soil-2 even after wet sieving. This is attributed to the fact that shrinkage strain depends on size of the aggregate used and Soil-2 with finer particles has less confinement to tensile strain than

river sand/soil-1. Restraining effect in mortar increases with increasing size of aggregate resulting in reduced strains (Rao 2001).

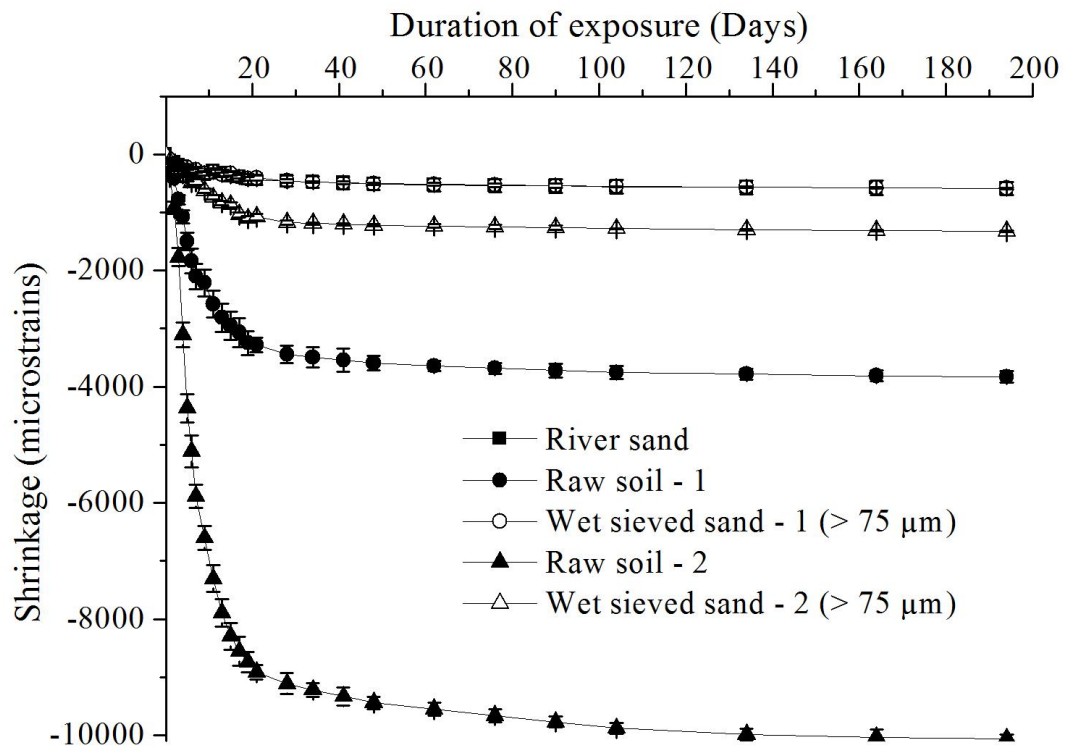


Figure 6.8 Drying shrinkage of cement mortar (1:3) with different fine aggregates

The size of the pores within mortar specimens also contributes to the shrinkage strains. This can be well explained with the help of critical pore diameters from Mercury Intrusion Porosimetry (MIP). The critical pore diameters are peaks in the differential curves which gives the rate of intrusion with pressure variation. There is a slight shift in the critical pore diameter to lower value for mortar with wet sieved sand-2 compared to mortar with wet sieved sand-1 and control mix (Figure 6.9). This means that mortar with wet sieved sand-2 has a large number of smaller pores resulting in higher intrusion rate and volume. Finer pore structure is expected to get higher shrinkage since smaller the capillaries greater will be the tensile stress generated by the water menisci inside capillaries (Valcuende et al. 2015). This is the case with mortar containing wet sieved sand-2 showing higher shrinkage strains compared to the other two mortar mixtures. Clearly, performance of excavated soils after treatment depends on the source from which the soil is extracted.

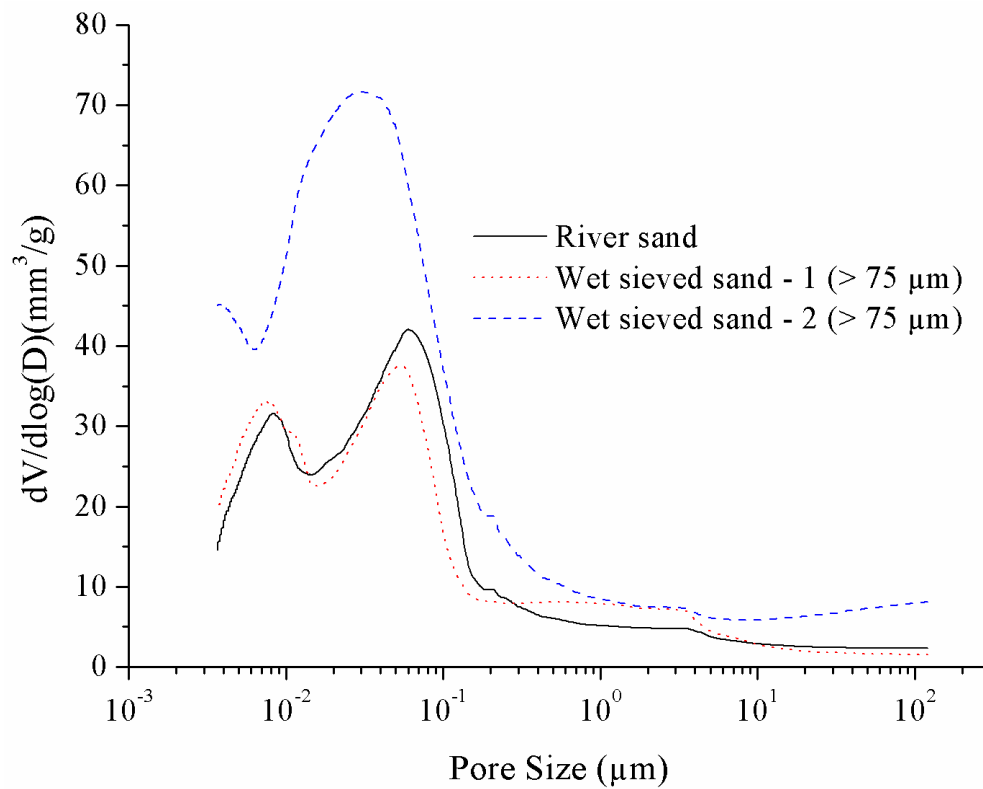


Figure 6.9 MIP curves for mortar (1:3) with different fine aggregates

When the A/C ratio increases, mortar mixtures with raw excavated soils show increase in shrinkage strain with increasing aggregate content (Figure 6.10). This is associated with the increase of clay content with the increasing aggregate content resulting in higher shrinkage strain (Boivin et al. 2004; Nehdi 2014), whereas, there is a reverse behaviour in shrinkage strain of mortar with wet sieved sand (Figure 6.11).

With the increasing aggregate content, shrinkage strain reduces for both types of excavation soil after processing, as shown in Figure 6.11. Increasing A/C ratio reduces the paste volume and drying shrinkage as the latter is proportional to the volume of the paste (Rozière et al. 2007). With increasing cement content, water content of the mortar mix was increased to maintain the constant workability (Figure 6.3) which would also contribute to the increased strain values.

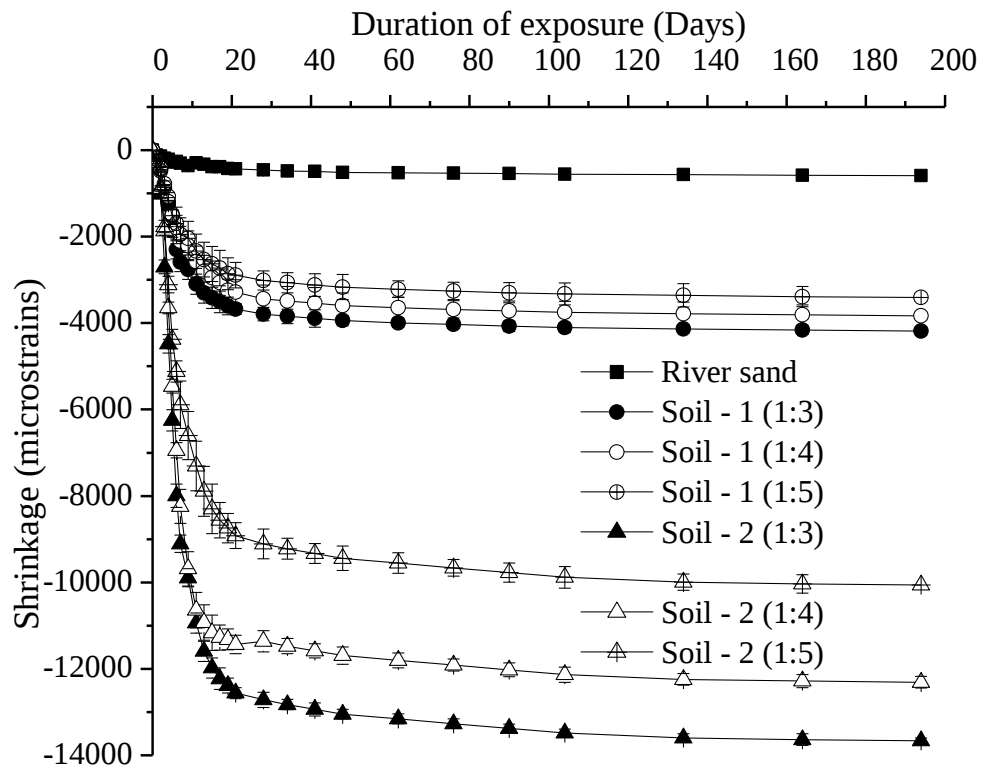


Figure 6.10 Drying shrinkage of raw soil-cement mortar with different A/C ratio

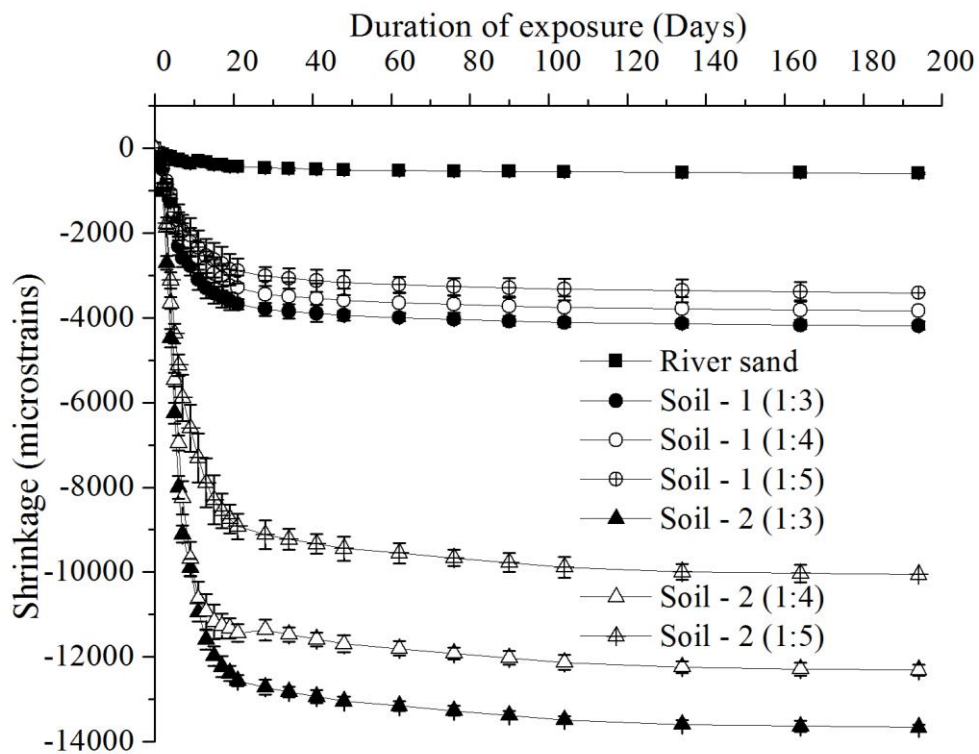


Figure 6.11 Drying shrinkage of wet sieved sand-cement mortar with different A/C ratio

6.3.6 Granulometric Adjustment

As poor performance of wet sieved sand-2 is attributed to the particle size distribution and fineness of the sand particles present, an attempt is made to adjust the granulometry of the material to further improve its properties. Granulometric corrections can be achieved by either including coarse sized particles to the finer material or by removing finer fraction from the source material. Latter method was adopted here to avoid complication of adding a new material and to make the resultant sand a self-sustainable material. Hence, instead of using 75 μm size sieve, 150 μm size sieve was used to alter the particle size distribution which is given in Figure 6.12. It can be noted that wet sieved sand – 2 shifted to coarser side with removal of particles less than 150 μm size and the curve lies between river sand and wet sieved sand – 1. This wet sieved sand – 2 ($>150 \mu\text{m}$) is used as fine aggregate to compare its mortar properties with river sand, wet sieved sand – 2 ($>75 \mu\text{m}$). Fly ash to fine aggregate ratio is fixed as 3 for this study.

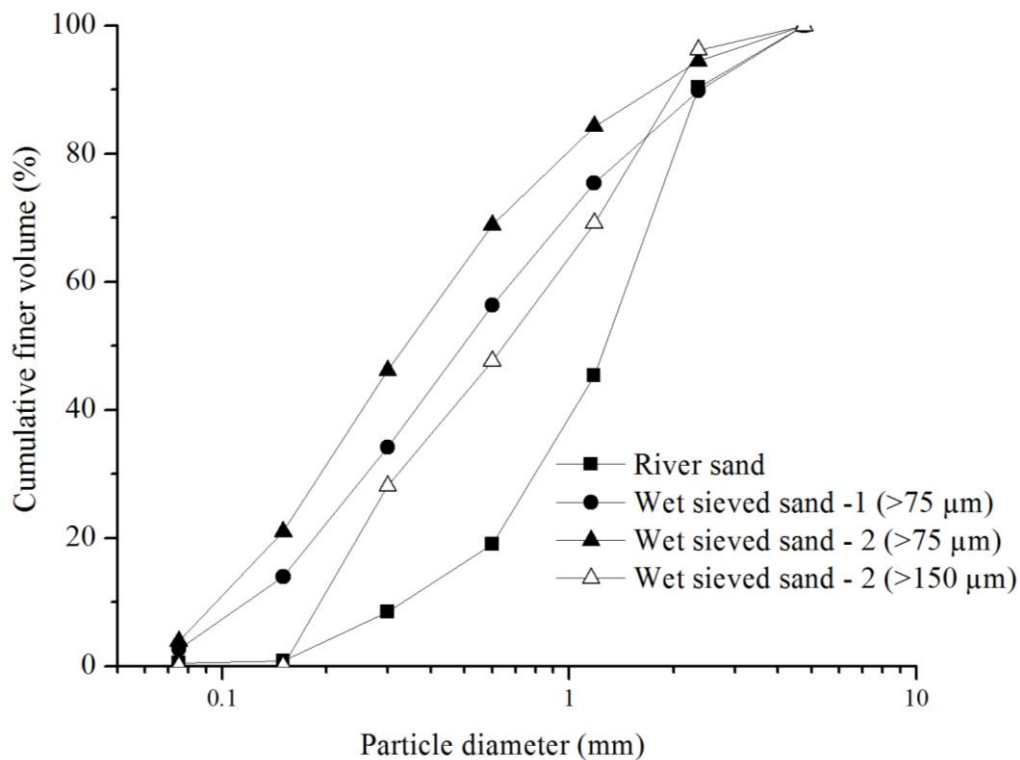


Figure 6.12 Particle size distribution curve after granulometric correction

Cement mortar properties using river sand, wet sieved sand – 1 and 2 are compared to that of mortar properties with wet sieved sand – 2 passing 150 μm in Table 6.3. By changing the sieve size from 75 to 150 μm , dry density of mortar with wet sieved sand – 2 could reach

a value equivalent to mortar with river sand. Compressive strength improves by 46% after granulometric correction compared to initial compressive strength of mortar with wet sieved sand – 2. Similar to density and strength, water absorption reduced from 14 to 9% with improved particle size distribution of fine aggregates. Drying shrinkage strain of cement mortar with wet sieved sand – 2 was three times higher than that of mortar with wet sieved sand – 1. With removal of finer portion of wet sieved sand – 2, this could be brought to a value equivalent to mortar with wet sieved sand –1 (Figure 6.13). This can be attributed to the corresponding reduction in water demand of mortar with wet sieved sand – 2 after granulometric corrections (Table 6.3).

Table 6.3 Mortar properties of wet sieved sand – 2 after granulometric correction

Mortar properties	River sand	Wet sieved sand – 1 (>75 μm)	Wet sieved sand – 2 (>75 μm)	Wet sieved sand – 2 (>150 μm)
Water demand (kg/m^3)	310	330	370	340
Dry density (kg/m^3)	2120	2150	1960	2100
Compressive strength (MPa)	47	43	26	38
Water absorption (%)	6	8	14	9

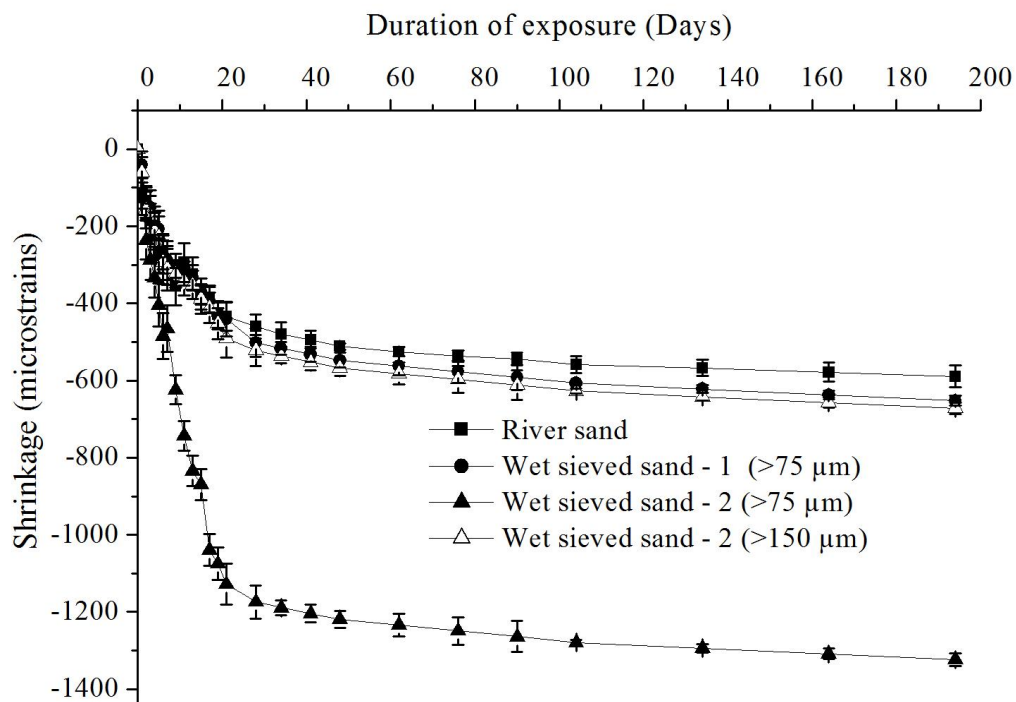


Figure 6.13 Drying shrinkage of wet sieved sand-2 after granulometric correction

6.4 USE OF WASH WATER IN CEMENT MORTAR

6.4.1 Properties of Wash Water

The wash waters from wet sieving of soil-1 and 2 were chemically analysed by Ion Chromatography and Inductively Coupled Plasma spectroscopy in order to assess the presence of ions. This was compared with the ions present in tap water. A high concentration of cation (117.763 mg/l) is observed in tap water (Table 6.4). Decrease in concentration of specific ions like K^+ , Al^{3+} , Zn^{2+} is noted in wash waters (Table 6.4). This may be attributed to the retention of ions by soil while wet sieving. However, the total ion concentration has increased to 124.654 and 137.331 mg/l in wash water-1 and 2 respectively. There are also equal chances of release of ions from clay particles resulting in an increase in concentration of ions (Mg^{2+} , Na^+). In similar studies on wash water from foundry sand, (Monosi et al. 2013) reported that the presence of high concentration of soluble alkaline ions can accelerate the cement hydration. As the individual ions as well as total ion concentration in wash water varies with the characteristics of soil, the influence of use of wash water in mortar can best be established through heat of hydration studies.

Table 6.4 Cation concentration in tap water and wash water

Cation Concentration	Tap water (mg/l)	Wash water from wet sieving of soil-1 (mg/l)	Wash water from wet sieving of soil-2 (mg/l)
Ca^{2+}	40.700	35.890	47.880
K^+	4.264	2.391	1.178
Mg^{2+}	16.960	20.580	25.180
Na^+	55.460	65.770	63.070
Mn^{2+}	0.003	0.003	0.004
Al^{3+}	0.075	0.015	0.017
Zn^{2+}	0.301	0.005	0.002

Heat of hydration of cement pastes made with tap water and wash water was determined as per EN 196-9 (2010), using semi adiabatic calorimeter. Cement and water contents were kept constant in order to assess the relative performance of different types of mix water. The exothermic rise in temperature was measured up to 48 hours from the time of mixing. As a next step, mortar specimens were cast using wash water-1 and wash water-2,

respectively, with wet sieved sand-1 and 2. This has been compared with cement mortar made with both the soils using tap water. This study was restricted to cement to fine aggregate ratio of 1:3 only. Specimens were water cured for 28 days before testing for dry density, water absorption and compressive strength. Shrinkage specimens were water cured for 7 days and strain readings were measured on specific intervals.

6.4.2 Effect of Wash Water on Cement Hydration

Figure 6.14, gives a comparison of rise in temperature for mortar mixtures made with tap water and wash water 1 and 2. From this, it is inferred that all the three paste samples reached peak value at the same time and the peak temperature was also practically the same. The variation in ion concentration of tap water before and water washing process does not affect the hydration process of cement. This gives the confidence to use the wash water of the excavated soil to produce respective mortar samples. However, to know the effect of its usage in mortar properties, tests were carried out with mortars of cement to fine aggregate ratio of 1:3 using the wash water.

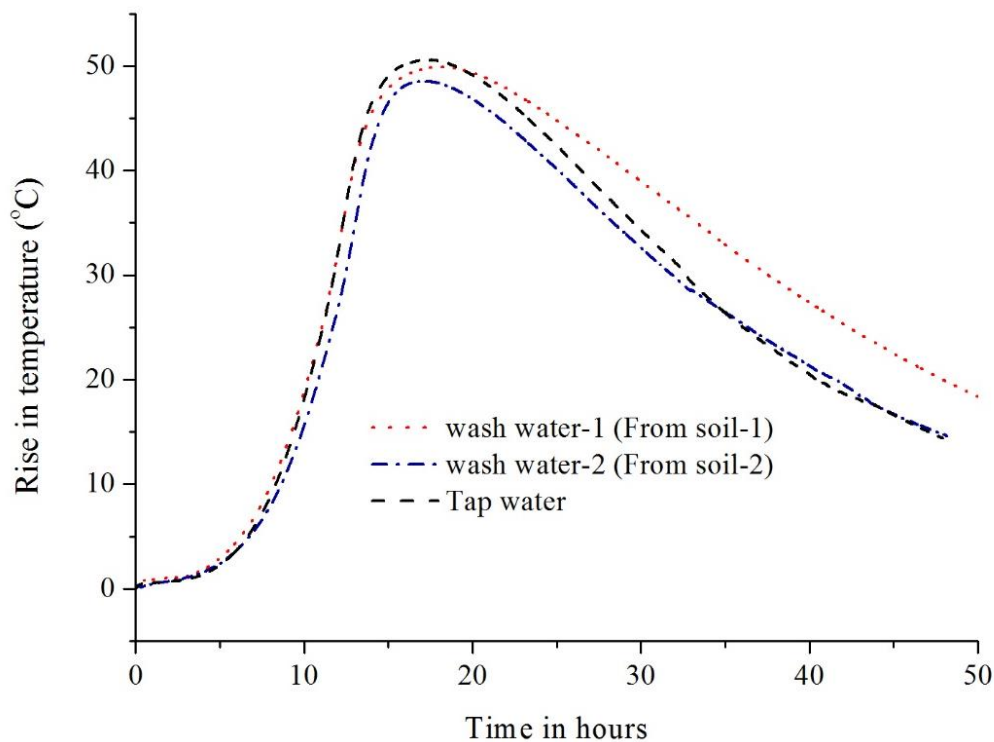


Figure 6.14 Hydration curves of cement paste with wash water

6.4.3 Effect of Wash Water on Properties of Cement Mortar

Wash water from wet sieving of respective excavated soil-1 and soil-2 was used to produce mortars of wet sieved sand-1 and soil-2, respectively. The properties of mortar specimens produced with tap water and wash water are compared in Table 6.5. Dry density, water absorption and compressive strength shows similar values for mortars made of wash water and tap water. This confirms the conclusion made with the hydration study and satisfies the requirement as per ASTM C1602 (2012) for concrete mixing water.

Shrinkage is an important property to study for the case of involvement of ions. Here, the main variation considered between tap water and wash waters was ion content. Hence, shrinkage was also studied and compared in Figure 6.15. The average difference in shrinkage strain of about 5% with wash waters is due to the experimental variations and the effect of wash water on the shrinkage can be considered as negligible.

Table 6.5 Comparison of properties of mortar made with tap water and wash water

Properties	Wet sieved sand-1 (>75 μm)		Wet sieved sand-2 (>75 μm)	
	Tap water	Wash water	Tap water	Wash water
Dry density (kg/m^3)	2145.8	2194.8	1952.6	2015.8
Water absorption (%)	8.72	8.00	14.50	13.30
Compressive strength (MPa)	42.4	42.0	26.0	27.0

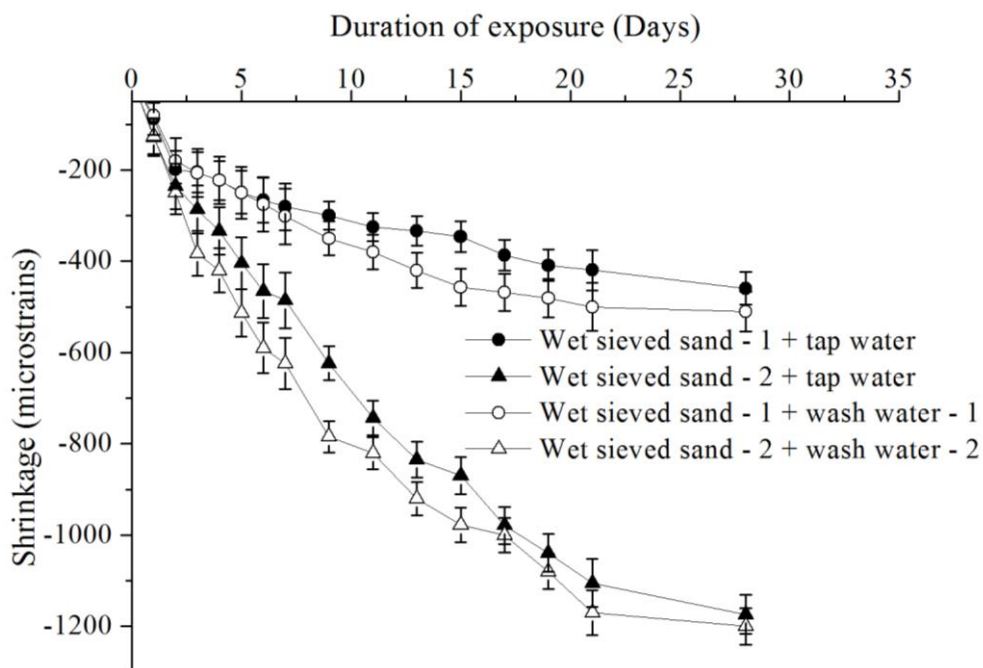


Figure 6.15 Shrinkage of mortar mixes with tap water and wash water

6.5 USE OF RESIDUAL CLAY IN CEMENT MORTAR

6.5.1 Properties of Thermally Activated Residual Clay

The 29% and 48% of silt and clay particles present in raw excavated soil 1 and 2 were separated as residual clay through wet sieving. This part of the soil is responsible for higher shrinkage strains and poor strength properties of mortar incorporated with raw excavated soil. Particle size distribution and D_{50} values of residual clay-1 and 2 were determined using laser diffraction analyser and compared in Figure 6.16. From D_{50} values, it can be stated that residual clay-2 is finer than residual clay-1, which is reinforced by specific surface area found by Blaine's air permeability (Table 6.6).

Table 6.6 Properties of residual clay

Properties	Residual clay-1	Residual clay-2
Major clay mineral	Kaolinite	Montmorillonite
Specific surface area (m^2/kg)	220.67	265.38
D_{50} (μm)	17.76	16.04

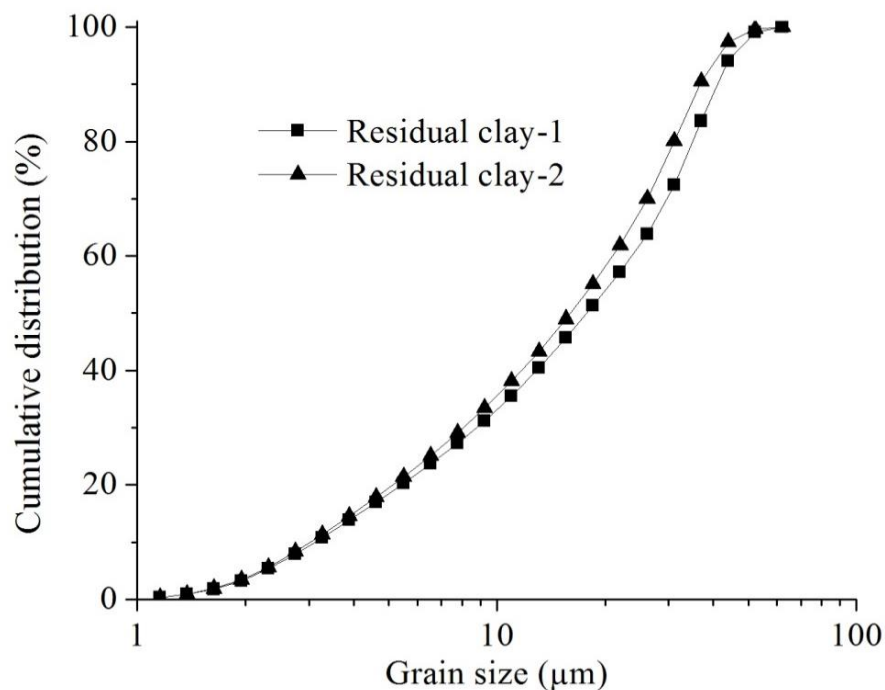


Figure 6.16 Particle size distribution of residual clay

Thermal activation/calcination of pulverised residual clay 1 and clay 2 was performed in a muffle furnace at 400°, 600°, 800° and 1000°C for 1 hour. Calcined residual clay samples were then tested for their pozzolanic performance in cement mortar using the Strength Activity Index (SAI) test as suggested in ASTM C311 (2017). SAI was calculated as per the equation 6.2 given in ASTM C311.

$$SAI = \left(\frac{A}{B} \times 100 \right) \quad \text{----- (6.2)}$$

where, A = average compressive strength of cement-pozzolan mortar (MPa), B = average compressive strength of control cement mortar (MPa). This is performed by replacing 20% of the cement by calcined residual clay in the control mortar. Six 50 mm cube specimens were cast for each type of clay calcined at four different temperatures. The specimens were stored in saturated lime water for curing and tested for compressive strength on the 28th day. According to ASTM standard C618, a material to be accepted as pozzolan should achieve 75% as SAI.

6.5.2 Performance of Calcined Clay as Pozzolan Material in Mortar

Thermally activated (calcined) residual clay 1 and 2 were used as pozzolan to perform strength activity index (SAI) test of cement-pozzolan mortar. Calcined clay is widely used as pozzolan in countries like India, Brazil which are deficient in supply of common supplementary cementitious materials (Scrivener 2014). This test helps in identifying the suitability of a material to be used as pozzolan in cementitious systems. The calcination of residual clay has been performed from 400° to 1000°C with 200°C increments. ASTM C595 (2018) specifies that SAI of a material should exceed 75% to accept it as a pozzolan. The residual clay-1 satisfied the requirement at a calcination temperature of 600°C whereas residual clay-2 calcined at 800°C reached 75% of SAI (Figure 6.17). This variation is attributed to the major type of clay mineral present in the residual clay. Residual clay-1 has major proportion of kaolinite whereas residual clay-2 has montmorillonite type of clay mineral.

Dehydroxylation of kaolinite mineral starts at 550°C and exposes the aluminium ions at the surface accelerating the reaction (He et al. 2000 and Fernandez et al. 2011). He et al. (2000) confirmed in their study that kaolinite attains its amorphous state when heated at 550°C for 100 minutes. This reactivity falls when heated further to 950°C. They also

concluded that kaolin treated at 550°C to 650°C has the fastest reactivity. The decomposition of montmorillonite is completely different from that of kaolinite and it is more a reorganisation of crystallinity. The structural disorder of montmorillonite contributing to the pozzolanic reactivity happens at a calcination temperature around 600° to 800°C (Alujas et al. 2015). This increases SAI value, reaches maximum at 800°C and satisfies the requirement.

Kaolin is the most potential clay for activation due to its structure that favours it, whereas illite and montmorillonite type of clay does not allow major structural disintegration even after calcination (Fernandez et al. 2011). Also, thermal activation makes the material with kaolinite more reactive and influences the SAI. The residual clay-1 with major portion of non-expansive kaolinite has high SAI value compared to residual clay-2 with expansive montmorillonite mineral. Nevertheless, thermal treatment tends to agglomerate montmorillonite particles and reduces the specific surface area which negatively affects the pozzolanic reactivity (Alujas et al. 2015).

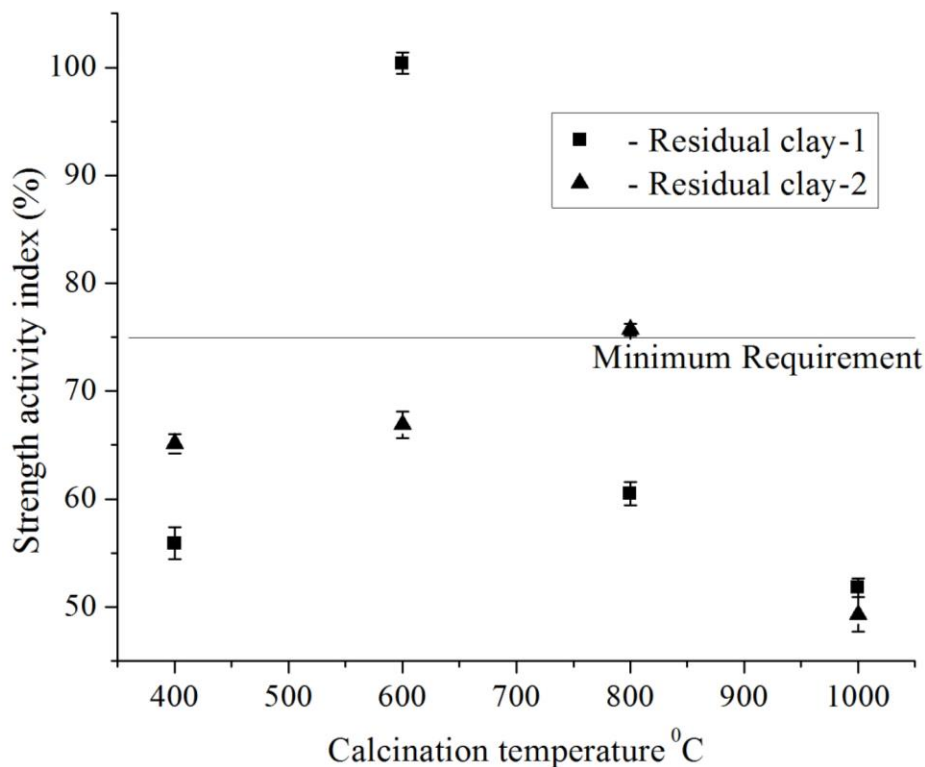


Figure 6.17 Strength activity index of thermally activated residual clay mortar

6.6 SUMMARY

Wet sieving technique was very effective in removing the clay and fines smaller than 75 μm . The difference in properties of excavated soils in mortar was mainly due to the particle size distribution and pore formation inside the mortar samples. Removal of fines improved the mortar properties significantly. Water demand reduced with this treatment which enhanced other properties of mortar such as compressive strength, water absorption by reducing the pore size distribution. Shrinkage strain got reduced with the removal of the clay content from the soil and reached a value equivalent to mortar with river sand. Mortar properties incorporating the wet sieved sand mainly depends on the granulometry of the source material. This could be corrected by adjusting the particle size distribution which further improves the properties of resultant mortar. Wash water from wet sieving of the identified excavation soil was proved to be reusable in concrete production. Pozzolans from calcination of residual could be utilized in cementitious system without compromising the strength of concrete.

CHAPTER 7

PERFORMANCE OF THERMALLY TREATED EXCAVATION SOIL AS FINE AGGREGATE IN CEMENT MORTAR

7.1 GENERAL

Washing and sieving is effective in case of low and medium plastic soil, for eliminating finer fraction. However, it could not be employed for materials with high clay content like high plastic soil with 58% of fines and also results in retrieval of only 42% of fine aggregate material. Hence, there is a need for identifying treatment technique for soil containing expansive clay in larger proportions.

Thermal treatment is an age-old ground improvement technique to avoid differential settlement of weak clayey soils and produces immediate results unlike other methods (Litvinov, 1960; Wang et al., 2008). Temperature $>100^{\circ}\text{C}$ was employed to drive away the moisture content in the soil that improves the soil strength. However, effect of low temperature treatment is a temporary solution. Granular materials are produced from clayey soil with treatment temperature above 900°C which retain its strength under wetting and drying conditions. This was suggested as an economic substitute for gravel and crushed rock in places where construction materials are imported for excessive costs (Joshi et al. 1994). The Pozzolanicity of clay minerals such as kaolinite, illite bentonite were enhanced with heat treatment and improved the chemical interaction of soil aggregates and binder (Joshi et al. 1994).

With this background, medium and high plastic soils were thermally treated at a temperature range of 200 to 1000°C for duration ranging from 30 to 180 minutes. Thermogravimetric analysis and X-ray diffraction spectroscopy are used to understand the structural and mineralogical changes in soil due to the effect of temperature. Statistically designed experiments have been undertaken to study the relative mortar properties of thermally treated excavation soils with respect to dry density, strength and drying shrinkage. Mortar properties with thermally treated soils are compared to mortar with untreated soil and control mortar with river sand. Change in porosity and pore refinement due to thermal

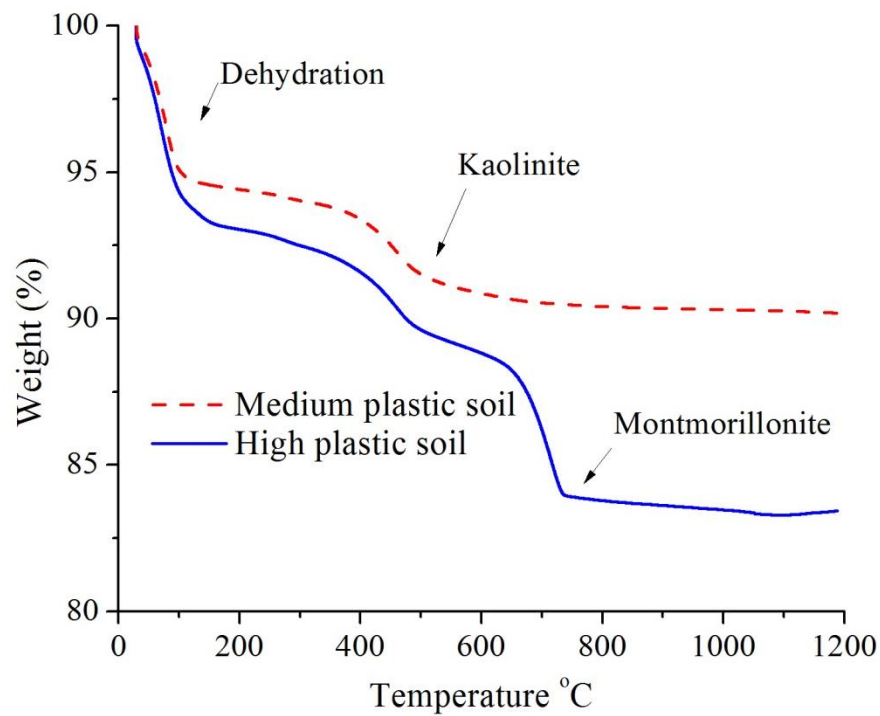
treatment is established with Mercury Intrusion Porosimetry which explains the enhancement in properties of mortar with thermally treated soils.

7.2 PROPERTIES OF THERMALLY TREATED SOIL

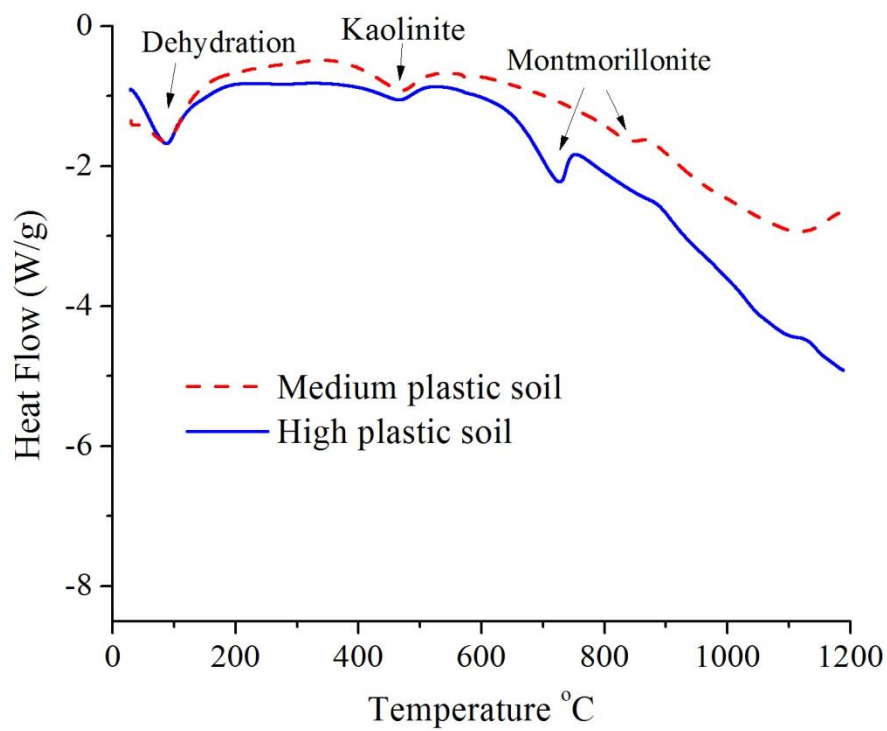
7.2.1 Thermo Gravimetric Analysis (TGA)

SDT Q600 thermo-gravimetric analyzer was used to characterize the thermal behaviour of medium and high plastic soil with 48% and 58% of fines (silt + clay) content respectively. The samples were heated at 20°C per minute and temperature was ramped from ambient to 1200°C in a nitrogen environment. Thermo gravimetric analysis was carried out in soil sample passing 75 µm size sieve. The mass loss and heat flow curves of medium and high plastic soils obtained through TGA is presented in Figure 7.1 (a) and (b), respectively. Thermo-gravimetric curves depend on the change in mass with increase in temperature (Figure 7.1a). The mass change may be due to dehydration, decomposition and oxidation of the sample. The mass loss in the temperature range of 50 to 200°C indicates the dehydration due to loss of free water between the clay layers. Dehydration loss is increasing with plasticity of the soil owing to the amount of clay. High plastic (HP) soil with montmorillonite shows highest mass loss at this temperature range compared to medium plastic soil (Figure 7.1b).

Dehydroxylation loss is due to the removal of structural water from the clay mineral. For kaolinite, dehydroxylation occurs at the temperature range of 400 to 600°C which can be clearly observed in both the soils with a loss in mass at this temperature range (Figure 7.1a). Also, dehydroxylation in this case refers to the loss of crystallinity creating hydrogen bonds between interlayer spaces (Fernandez et al. 2011). Dehydroxylation of montmorillonite occurs at a temperature range of 600 to 800°C and this can be noted with corresponding mass loss in this temperature range for high plastic soil (Figure 7.1a). Though medium plastic (MP) soil does not show a prominent reduction in mass for the presence of montmorillonite, there is a dip in heat flow curve at 800°C indicating the presence of illite/montmorillonite in this soil (Figure 7.1b).



(a) Mass loss



(b) Heat flow

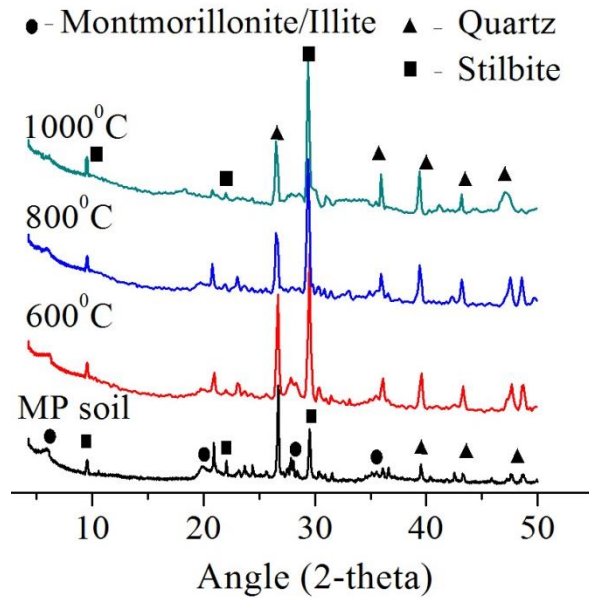
Figure 7.1 TGA curves of soil samples

7.2.2 Effect of Temperature on Mineralogical Characteristics

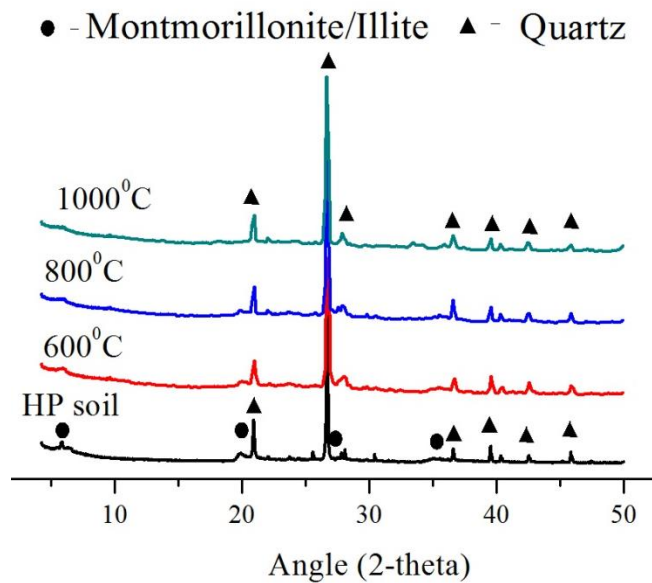
From TGA, it can be observed that there is no mass loss beyond 1000°C, hence the maximum temperature for XRD studies are restricted to 1000°C. The acquired soil samples were thermally treated in a muffle furnace at temperatures of 200, 400, 600, 800 and 1000°C. Soil sample was placed in the muffle furnace at room temperature and the temperature was increased at a rate of 5°C/min until the desired temperature was reached. Once the desired temperature was reached, it was held for 180 minutes and the furnace was turned off. The samples were allowed to cool and used for further characterization.

Treated samples were characterized for their mineralogical changes using PANalytical X'Pert Pro x-ray diffraction (XRD) spectrometer with Cu-K α as the source. Slit width of 6 mm with step scan size of 0.02 degrees per second and counting time of 20 s was adopted. The completely dried raw soil and thermally treated soils were sieved through 50 μ m sieve and the powder samples were used for testing. The X-ray diffraction patterns were identified with powder diffraction search manual – ICSD database. The effect of heat treatment on the mineralogical composition of medium and high plastic soils can be explained with the help of XRD curves as shown in Figure 7.2 (a) and (b). From the XRD analysis, it is evident that medium plastic soil mainly composed of montmorillonite, illite and stilbite (Figure 7.2a), and high plastic soil has majority of montmorillonite and illite (Figure 7.2b).

Medium and high plastic soil shows montmorillonite/illite peaks at 600°C indicating that dehydroxylation is not complete at this temperature. The decomposition of structure of these clay minerals is clearly visible by disappearance of its peak at 1000°C, though TGA indicates that dehydroxylation completes at 800°C. Medium plastic soil, in addition to montmorillonite/ illite show peaks of stilbite clay mineral. The stilbite mineral is stable in thermal treatment without any mineralogical changes and remains crystalline even at 1000°C



(a) Medium plastic soil



(b) High plastic soil

Figure 7.2 XRD pattern of untreated and thermally treated soils

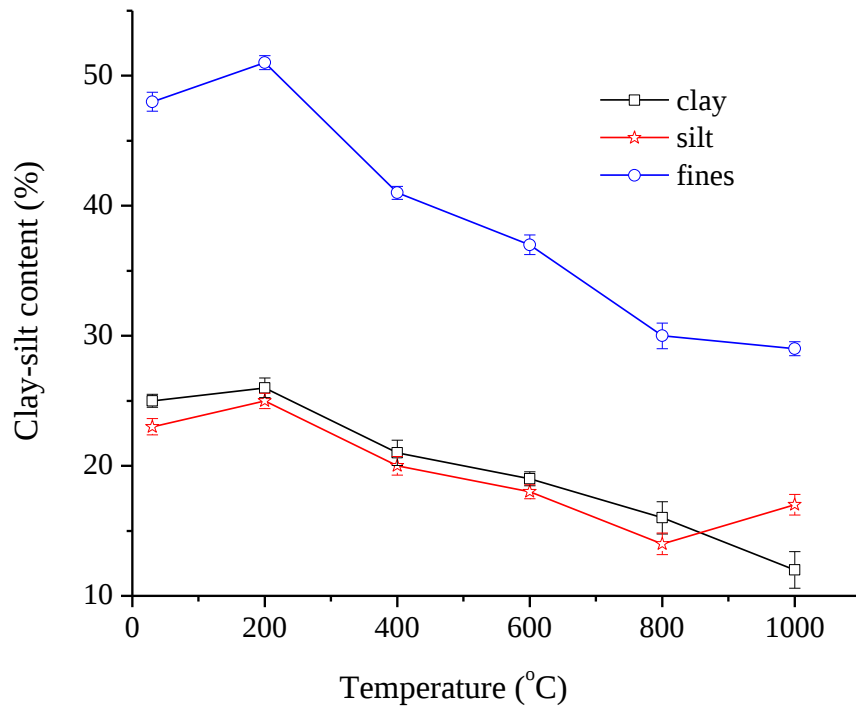
7.2.3 Effect of Thermal Treatment on Particle Size Distribution

Quantitative determination of the particle size distribution of finer portion of soils can be determined using sedimentation or hydrometer method as per ASTM D7928 (2017). As it is the portion of soil that is passing 75 μm (clay and silt) is mainly causing problem when used in cement mortar, it is important to understand the effect of temperature on distribution of

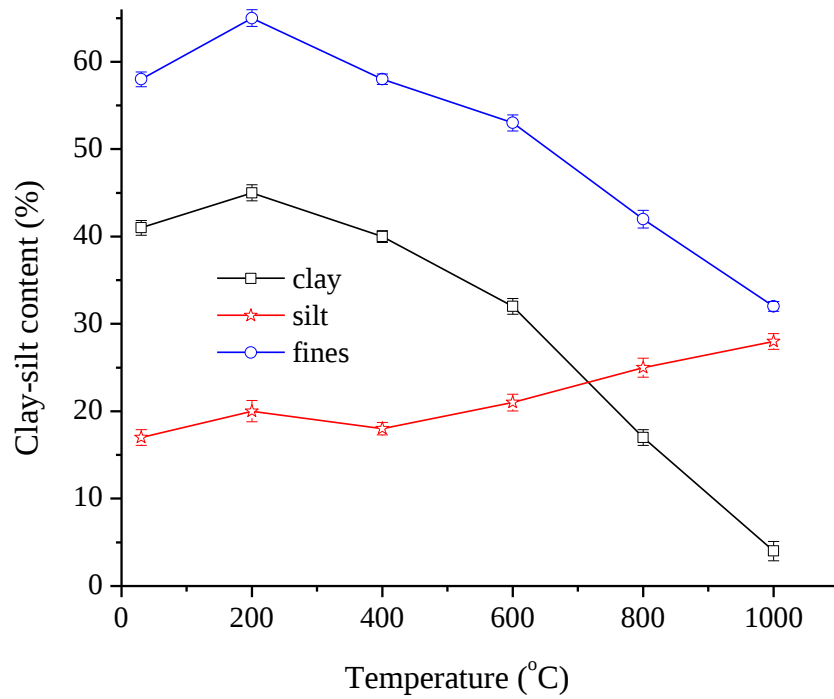
these particles. Thermally treated medium and high plastic soil at different temperature ranging from 200 to 1000°C at an interval of 200°C, were analysed with hydrometer to understand the transition of clay and silt size particles. The thermally treated soil was washed thoroughly through 75 µm size sieve and 50 g of the sample passing 75 µm was used for the study. The oven dried sample was then dispersed using combination of sodium hexametaphosphate (5 g) and sodium carbonate as dispersion agent in distilled water of 1000 ml. Hydrometer reading was taken at subsequent time intervals and measurement at elapsed time is used to calculate the percentage of particles finer than the diameter given by stoke's law. Hydrometer study was repeated 3 times for each of the sample and the mean value with the variation of the test values are represented in Figure 7.3.

Figure 7.3 shows the size transition of clay and silt size particles in medium and high plastic soils. It can be noted that heating soil samples to 200°C increases the clay sized particles irrespective of type of soil. This can be attributed to the reduction in grain size by shrinkage due to dehydration as mentioned in TGA (Section 7.2.1). Similar results are observed by Wang et al. (1990) when dry kaolinite and bentonite clay samples were subjected to heat treatment at different temperature ranging from 100 to 400°C. Further increase in treatment temperature beyond 200 °C reduces both silt and clay size particles pertaining to the growth of the these particles. This can also be noted in the total reduction in percentage of fines content from 48 to 30% in medium plastic soil (Figure 7.3a). However, there is a sudden 4% increase in silt size particles with corresponding reduction in clay size particles between 800 to 1000°C. This can be related to the sintering effect of clay sized particles at high temperature treatments (Bhatnagar and Goel 2002).

In case of high plastic soil, the initial increase in clay size fraction at 200°C is high (4%) compared to medium plastic soil (Figure 7.3b). Since, montmorillonite clay with adsorbed water results in higher reduction and the amount of clay present in high plastic soil is also higher. Though there is a 18% overall reduction in the fines content, it can be noted that in-spite of reduction in clay sized particles, there is also 10% increase in silt size particles. This behaviour is attributed by Yilmaz, (2003) and Wang et al. (2008) to the removal of adsorbed water around the clay particles that brings them together, favors the growth in particle size and reaches the size of silts.



(a) Medium plastic soil



(b) High plastic soil

Figure 7.3 Clay-silt transition with thermal treatment of soil samples

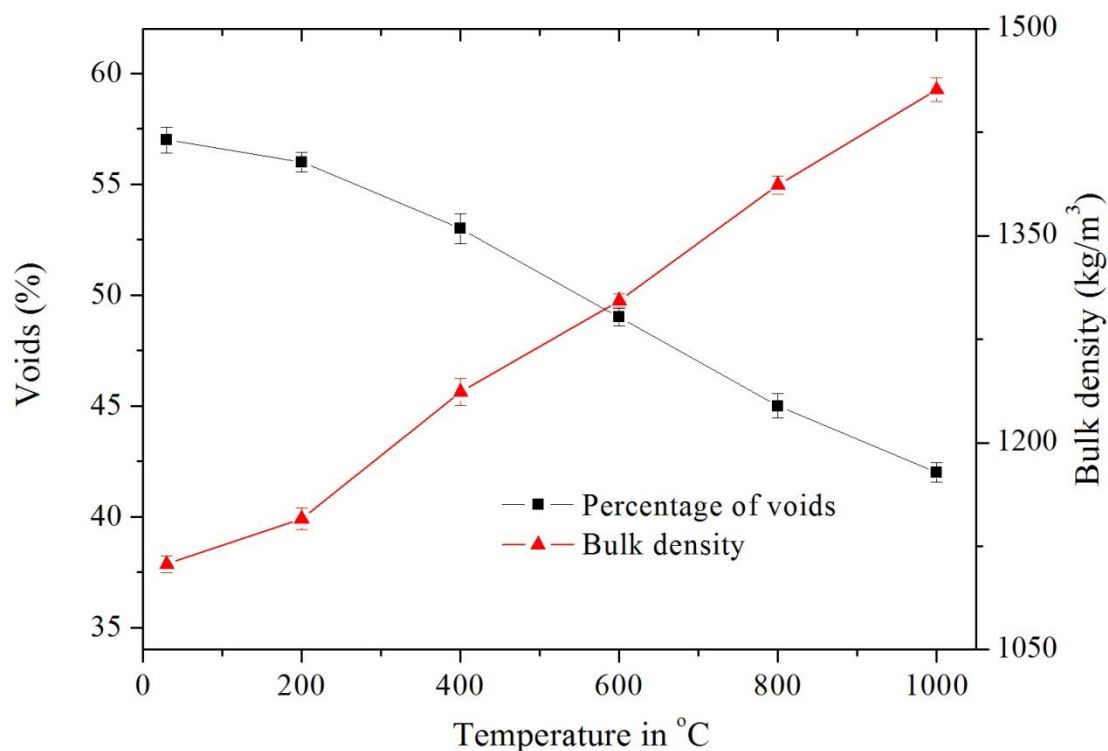
7.2.4 Density and Percentage of Voids

Bulk density (mass/volume) of the thermally treated soil was calculated with a container of known volume and the percentage of voids was calculated from the following relation (equation 7.1),

$$\text{Percentage of voids} = \left(\frac{G_s - \gamma}{G_s} \times 100 \right) \quad \text{----- (7.1)}$$

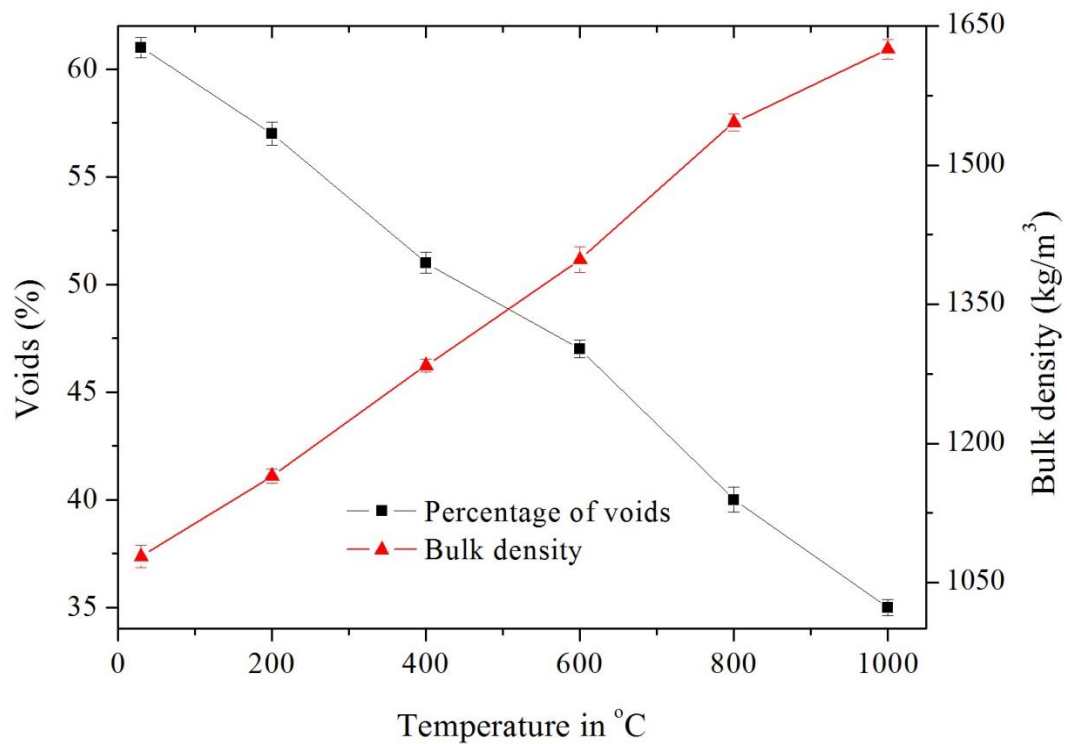
Where, G_s is the specific gravity of soil and γ is the bulk density in kg/litre.

Figure 7.4 shows that the voids percentage reduces with an increase in treatment temperature and consequently increases its bulk density, irrespective of the type of soil. This can be related to the growth of clay size particles which altered the particle size distribution (Figure 7.3) and helps in better packing. It can be noted that bulk density is higher for high plastic soil indicating the efficiency of thermal treatment in soil with high clay content (Figure 7.4b).



(a) Medium plastic soil

Figure 7.4 continued...



(b) High plastic soil

Figure 7.4 Variation in bulk density and voids with thermal treatment of soil samples

7.3 CEMENT MORTAR PROPERTIES

7.3.1 Mortar Preparation and Testing Regime

Fine aggregate to cement ratio has been fixed as 3 (by weight). 53 grade Ordinary Portland cement, conforming to IS 12269 was used as binder. Well graded river sand was used as fine aggregate in control mortar specimens. Initially, cement mortar was cast with untreated soils and river sand to serve as benchmark to evaluate the relative performance of treated soil on mortar properties. To study the influence of temperature and duration of thermal treatment on different soil, experiments were designed using central composite design (Montgomery 2012) of Response Surface Methodology (RSM) with 13 sets of experiments (for each medium and high plastic soil). Each parameter has five levels within the fixed range, i.e., 15 minutes to 180 minutes of duration and 200 to 1000°C for temperature of treatment. The coded and uncoded values of the parameters are given in Table 7.1.

Mortar flow percentage was determined in three trials for each of the mix as per ASTM C1437. For each of the 13 sets of designed mix, 12 numbers of 50 mm cube specimens (3 for dry density, 3 for water absorption and 6 for compressive strength determination) and 3 numbers of prisms of size 160 mm x 40 mm x 40 mm for shrinkage measurements were cast.

Table 7.1 Factors in un-coded values for coded values of the parameters studied

Notation	Parameter	Unit	Coded Values				
			+1.414	+1	0	-1	-1.414
			Uncoded values				
A	Temperature	°C	200	317	600	882	1000
B	Duration	minutes	30	51	105	158	180

In RSM problems, the form of relationship between the response and the independent variables can be fitted by a polynomial model. To obtain a more accurate value, quadratic model was selected. The analysis of experimental data was carried out using Statistical Analysis Software (SAS Release 8.02) and the regression models for medium and high plastic soil based on the designed experiments are given in Table 7.2 and Table 7.3 respectively. The regression models contain terms that are statically significant (i.e., those terms that had a T-statistics greater than that of chosen significance level of 0.05) have been used for obtaining the response surface. The statistical model is validated based on R^2 , P-value and F-values in the output data. R^2 value is found to be > 0.9 for most of the responses which gives confidence in applying the predicted model and can be used for analyzing the responses for different combination of parameters within the mentioned range. Models are found to be statically significant as P-value lies within 0.05.

Table 7.2 Response surface model equations for mortar with medium plastic soil

Mortar property	Response model	R^2	F-Value	P-Value
Water content (kg/m ³)	$907.80592 - (0.39613 * A) - (0.59622 * B) - (0.000727273 * A * B) - (0.0000182099 * A^2) + (0.00375574 * B^2)$	0.9707	46.31	<0.0001
Dry density (kg/m ³)	$1137.42403 + (1.2145 * A) + (1.86739 * B) + (0.00190545 * A * B) - (0.000705099 * A^2) - (0.011268 * B^2)$	0.9506	26.95	0.0002
Compressive strength (MPa)	$-5.1275 + (0.060967 * A) + (0.076691 * B) + (0.0000642424 * A * B) - (0.0000392778 * A^2) - (0.000441322 * B^2)$	0.9668	40.75	<0.0001

Table 7.2 continues,

Mortar property	Response model	R ²	F-Value	P-Value
Water absorption (%)	$42.54775 - (0.047883 * A) - (0.11247 * B) + (0.0000184512 * A * B) + (0.000025679 * A^2) + (0.000448852 * B^2)$	0.9642	37.71	<0.0001
Drying shrinkage (µstrain)	$21816.90097 - (48.64763 * A) - (27.72649 * B) - (0.0153 * A * B) + (0.030791 * A^2) + (0.17787 * B^2)$	0.9281	18.08	0.0007
Permeable pore volume (%)	$57.05337 - (0.056118 * A) - (0.10276 * B) - (0.0000538721 * A * B) + (0.0000253086 * A^2) + (0.000532599 * B^2)$	0.9444	23.77	0.0003

Note: A-temperature, B-duration

Table 7.3 Response surface model equations for mortar with high plastic soil

Mortar property	Response surface model equations	R ²	F-Value	P-Value
Water content (kg/m ³)	$1519.04593 - (1.5229 * A) - (2.43772 * B) - (0.000215488 * A * B) + (0.000483951 * A^2) + (0.00940312 * B^2)$	0.9737	51.82	<0.0001
Dry density (kg/m ³)	$633.22158 + (2.29589 * A) + (4.07401 * B) + (0.00209455 * A * B) - (0.00136531 * A^2) - (0.020266 * B^2)$	0.9824	78.36	<0.0001
Compressive strength (MPa)	$-3.27457 + (0.042357 * A) + (0.078419 * B) + (0.000119192 * A * B) - (0.0000154074 * A^2) - (0.000368044 * B^2)$	0.9631	36.51	<0.0001
Water absorption (%)	$75.51209 - (0.12049 * A) - (0.1906 * B) - (0.000158923 * A * B) + (0.0000776667 * A^2) + (0.00125877 * B^2)$	0.8830	10.56	0.0037
Drying shrinkage (µstrain)	$31681.54612 - (68.33879 * A) - (41.74744 * B) + (0.014074 * A * B) + (0.040299 * A^2) + (0.097502 * B^2)$	0.9727	49.83	<0.0001
Permeable pore volume (%)	$80.74274 - (0.070478 * A) - (0.18191 * B) - (0.000040404 * A * B) + (0.0000203704 * A^2) + (0.000606061 * B^2)$	0.9629	36.20	<0.0001

Note: A-temperature, B-duration

The ANOVA results of water demand, dry density, compressive strength, water absorption, drying shrinkage and permeable pore volume are summarized in Table 7.4 and Table 7.5 for medium and high plastic soil, respectively. The factors influencing the mortar properties can be identified to be significant if P-value < 0.05. The interaction effect of these factors is not significant in properties of mortar with thermally treated soil. Response curves for water demand, dry density, compressive strength, water absorption, drying shrinkage and pore volume have been plotted by substituting upper (180 minutes) and lower limits (30 minutes) of duration of treatment in the response surface model. For discussion of results, actual experimental data from 13 sets of experiments are plotted along with response surface curves, with the average value and the experimental variations of total number of specimens tested for each parameter.

Table 7.4 ANOVA for mortar properties of medium plastic soil

Responses		Main effects		Interaction effects	Quadratic effects	
		A – Temperature (°C)	B – Duration (minutes)	AB	A ²	B ²
Water content (kg/m ³)	F-Value	226.99	2.24	0.86	0.03	1.34
	P-Value	< 0.0001	0.1782	0.3843	0.872	0.2846
Dry density (kg/m ³)	F-Value	113.12	5.02	1.79	12.69	3.66
	P-Value	< 0.0001	0.0599	0.2227	0.0092	0.0973
Compressive strength (MPa)	F-Value	156.57	6.15	1.9	36.84	5.25
	P-Value	< 0.0001	0.0522	0.21	0.0005	0.0556
Water absorption (%)	F-Value	107.61	6.49	0.2	22.57	2.7
	P-Value	< 0.0001	0.0383	0.6714	0.0021	0.144
Drying shrinkage (µstrain)	F-Value	68.58	0.02	0.1	21.63	0.82
	P-Value	< 0.0001	0.8957	0.7574	0.0023	0.3966
Permeable pore volume (%)	F-Value	108.46	2.64	0.48	5.44	2.72
	P-Value	< 0.0001	0.1483	0.5123	0.0524	0.143

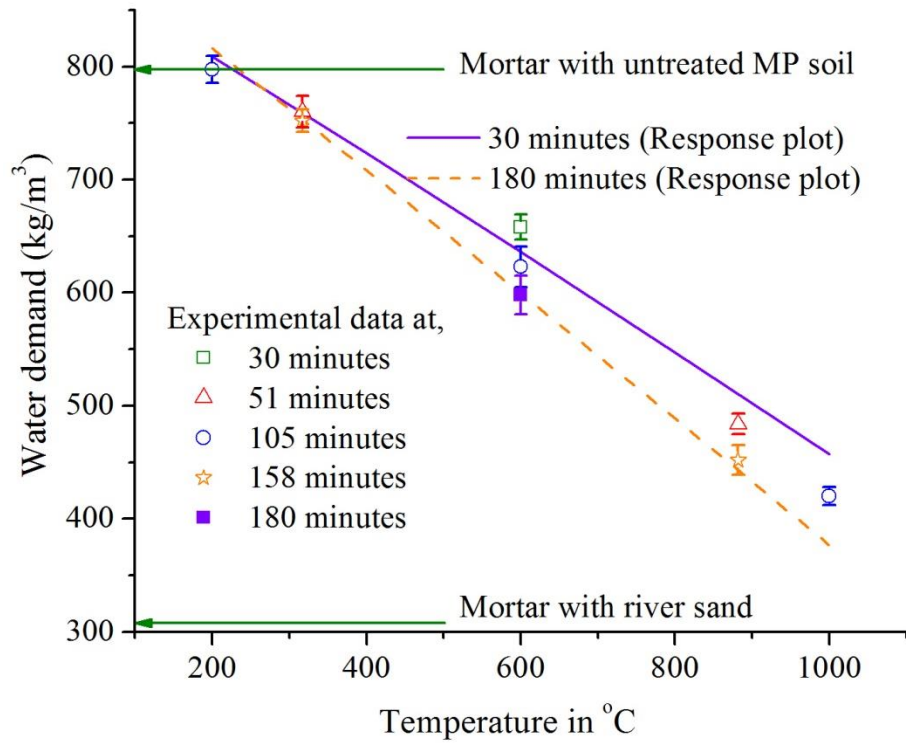
Table 7.5 ANOVA for mortar properties of high plastic soil

Responses		Main effects		Interaction effects	Quadratic effects	
		A – Temperature (°C)	B – Duration (minutes)	AB	A ²	B ²
Water content (kg/m ³)	F-Value	248.42	4.26	0.02	5.01	2.14
	P-Value	< 0.0001	0.0779	0.8937	0.0603	0.1874
Dry density (kg/m ³)	F-Value	300.6	47.46	0.3	37.35	10.55
	P-Value	< 0.0001	0.0002	0.6024	0.0005	0.0141
Compressive strength (MPa)	F-Value	156.33	19.98	2.76	2.38	1.54
	P-Value	< 0.0001	0.0029	0.1409	0.1666	0.2551
Water absorption (%)	F-Value	41.42	0.58	0.7	8.61	2.55
	P-Value	0.0004	0.0352	0.4315	0.0219	0.154
Drying shrinkage (µstrain)	F-Value	192.39	2.84	0.13	53.68	0.35
	P-Value	< 0.0001	0.136	0.7325	0.0002	0.5701
Permeable pore volume (%)	F-Value	162.52	14.92	0.17	2.19	2.19
	P-Value	< 0.0001	0.0062	0.6951	0.182	0.182

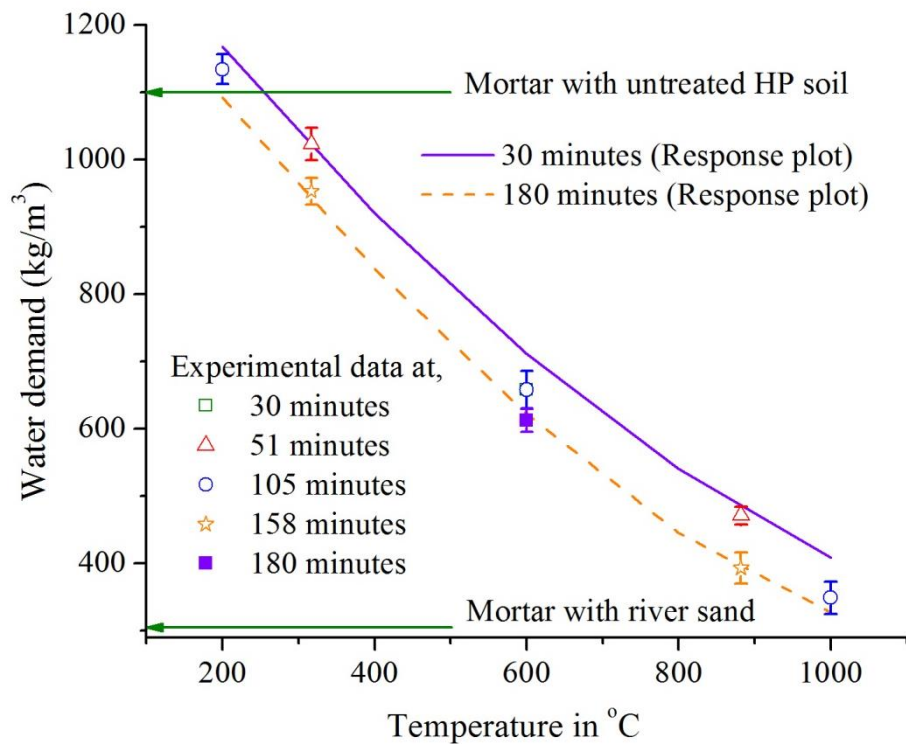
7.4 CEMENT MORTAR PROPERTIES

7.4.1 Water Demand for Constant Workability

Influence of duration and temperature of treatment on water demand for constant workability of cement mortar with medium and high plastic soils are presented in Figure 7.5. ANOVA table indicates that water demand for constant workability is affected only by treatment temperature for mortar with MP and HP soil (Table 7.4 and Table 7.5). With increasing temperature, water demand reduces for cement mortar with soil, irrespective of the type of soil (Figure 7.5a and b). This is attributed to the growth in particle size of clay with increasing treatment temperature which demands less water content for workability. This effect is higher in HP soil with high clay content (Figure 7.5a) and can be attributed to the reduction in the interlayer space between the clay particles which aids in sintering and particle growth (Warshaw et al. 1960). It can be noted that high plastic soil with 41% of expansive clay could reach a water demand equivalent to river sand at 1000°C and this can be related to the reduction in clay content from 41% to 4% at this temperature (Figure 7.3b). This shows that effect of thermal treatment in reducing the reactivity of clayey soil with high plasticity is significant in soil with montmorillonite clay.



(a) Medium plastic soil



(b) High plastic soil

Figure 7.5 Water demand in cement mortar with thermally treated soils

7.4.2 Dry Density

Dry density of cement mortar with thermally treated medium and high plastic soils are shown in Figure 7.6. Treatment temperature is an influential parameter affecting the dry density of mortar with MP soil, whereas, the factors, duration and temperature of thermal treatment contributes to the dry density of the mortar with HP soil.

At a given duration, dry density of mortar with thermally treated soil increases with treatment temperature. When the treatment temperature is raised from 200 to 1000°C, 35 and 68% percentage increase in dry density of mortar with medium and high plastic soil, respectively is noted (Figure 7.6). The particle growth of clay size fraction increases with plasticity which also resulted in reduction in water demand and together, it is depicted here in the density increase. This can also be related to the reduction in void ratio and increase in bulk density with treatment temperature for the thermally treated soil samples (Figure 7.4). At a treatment temperature of about 800°C and duration of 180 minutes, dry density of mortar with thermally treated both soil specimens reaches a value of $1950 \pm 50 \text{ kg/m}^3$.

At a constant temperature, dry density of mortar with thermally treated HP soil increases by an average of 10% with increasing duration of treatment from 30 to 180 minutes. The effect of duration substantially increases with treatment temperature and reaches its maximum at 1000°C. This can be attributed to the fact that montmorillonite clay mineral in HP soil requires high temperature (>600°C) for the complete decomposition, though the reduction in clay lamellar space at lower temperatures also have some effect (Satikaya et al., 2000).

7.4.3 Compressive Strength

Compressive strength of cement mortar with thermally treated MP and HP soil are shown in Figure 7.7. ANOVA results indicate that temperature is the only parameter contributes to the compressive strength of the mortar with MP soil (Table 7.4), whereas compressive strength of mortar with high plastic soil is influenced by the parameters, duration and the temperature of thermal treatment (Table 7.5).

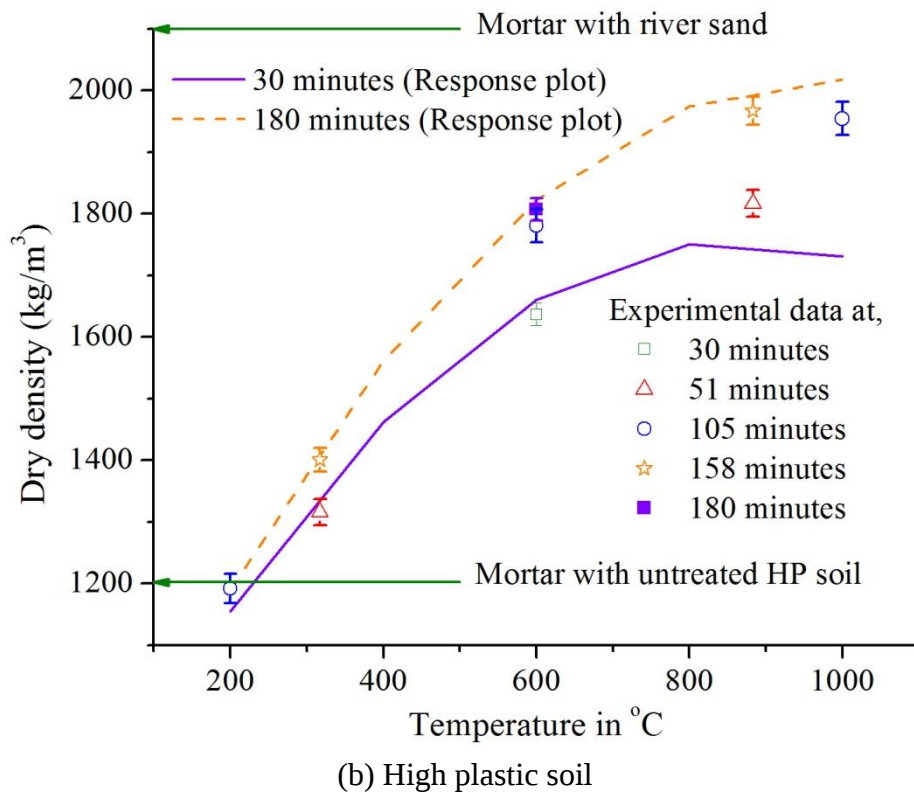
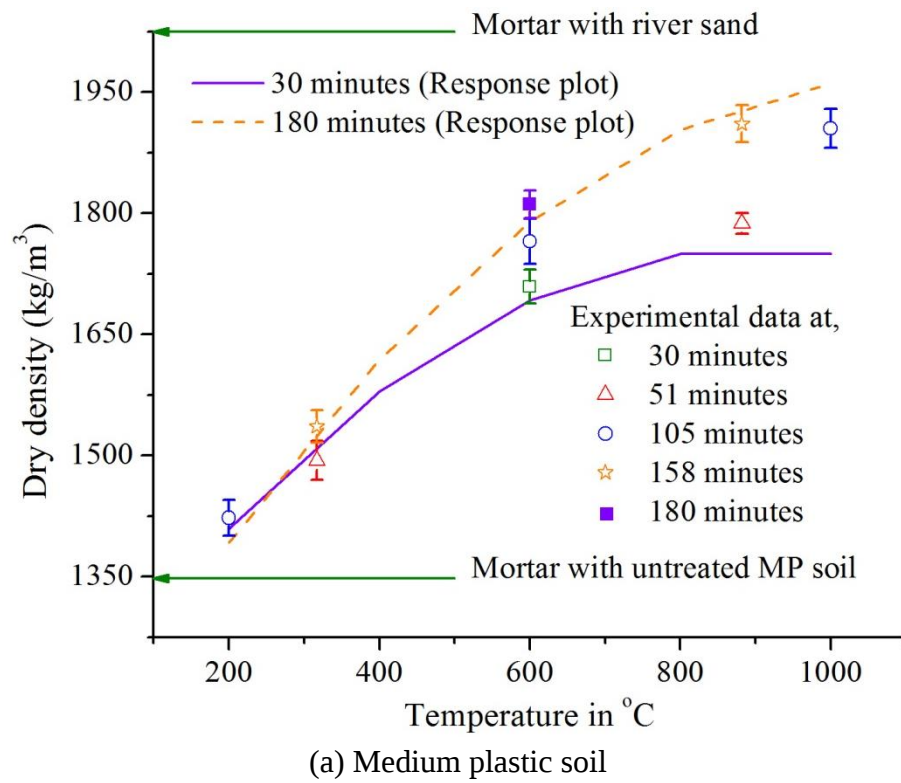
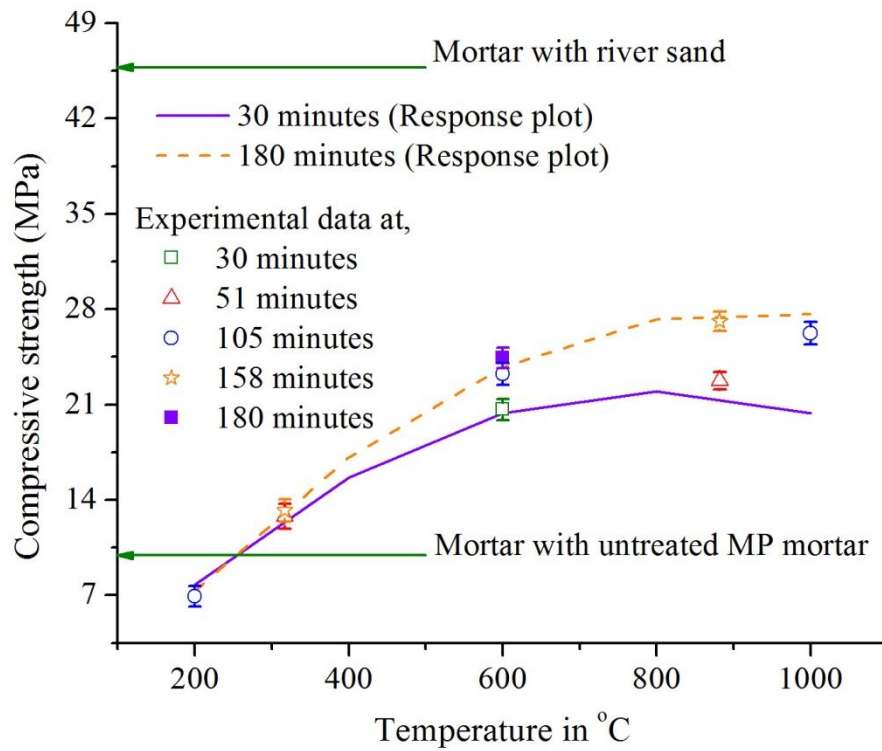
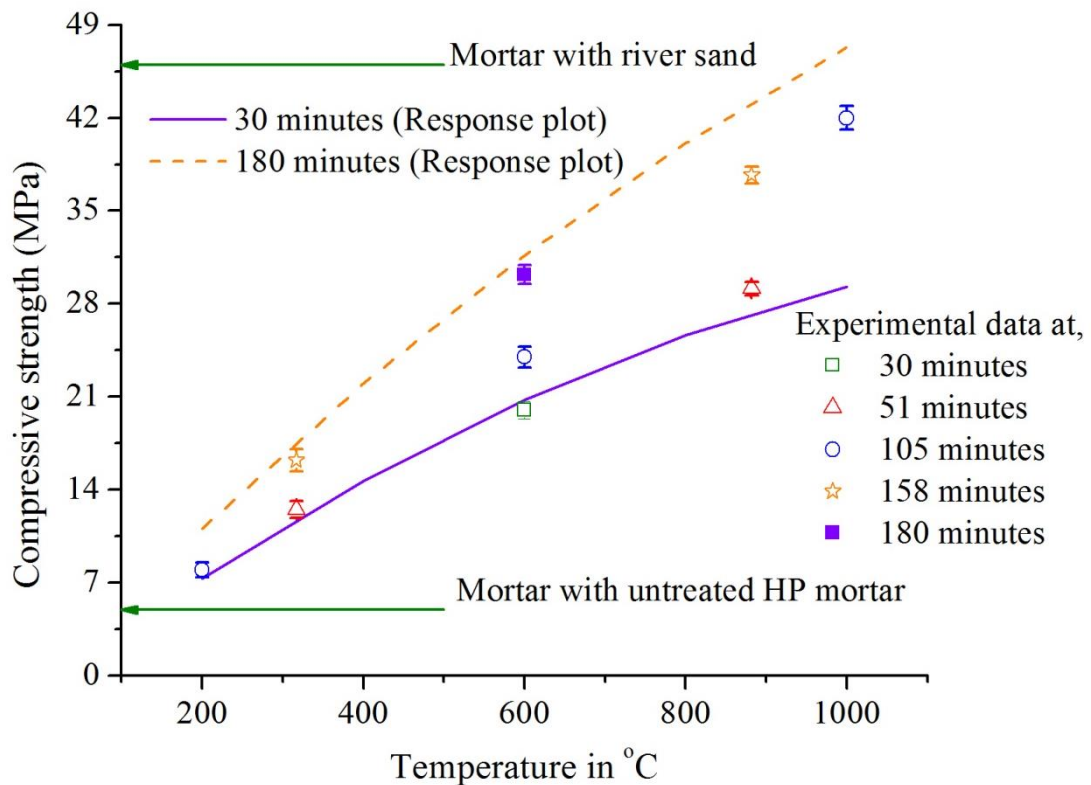


Figure 7.6 Dry density of cement mortar with thermally treated soils



(a) Medium plastic soil



(b) High plastic soil

Figure 7.7 Compressive strength of cement mortar with thermally treated soils

For any duration of treatment, compressive strength of thermally treated medium plastic soil increases with treatment temperature (Figure 7.7a). However, it could attain only 76% of compressive strength of mortar with river sand at an exposure temperature of 1000°C for a duration of 180 minutes. It may be because of the combination and proportion of illitic clay present in the medium plastic soil which does not respond to the thermal treatment below 1000°C as shown in XRD curves in Figure 7.2a. This response of illitic clay to thermal treatment is also supported by Bhatnagar and Goel 2002.

For soil with high expansive clay content, compressive strength of mortar with thermally treated soil increases with treatment duration and temperature (Figure 7.7b). This increase in strength of mortar with HP soil is linear with increasing temperature from 200 to 1000°C. This can be related to the high percentage of montmorillonite type of clay mineral present in HP soil which may need high temperature treatment for the complete disintegration of the clay structure. Additionally, pore refinement with montmorillonite type of expansive clay helps in improving the strength better than the non-expansive clay in higher temperatures (Joshi et al. 1994). This could be the reason for the drastic strength improvement in mortar with HP soil which even exceeded the strength of mortar with river sand at a treatment temperature of 1000°C.

7.4.3.1 Strength to density ratio

Strength to density ratio of cement mortar with thermally treated MP and HP soil are compared with that of mortar with river sand in Figure 7.8. Though mortar with medium plastic soil shows an increase in strength to density ratio from 0.4 to 1.4, it could not reach a value as significant as strength of mortar with HP soil. This can be attributed to the temperature requirement to activate illite type of clay mineral which is why there is a marked difference in the effect of duration only at a treatment temperature of 1000°C. Strength to density ratio of mortar with HP soil is widespread and exceeds mortar with river sand at 1000°C. Mortar with HP soil could give comparable strength of river sand at a lower density range which makes it a potential light weight material for structural applications.

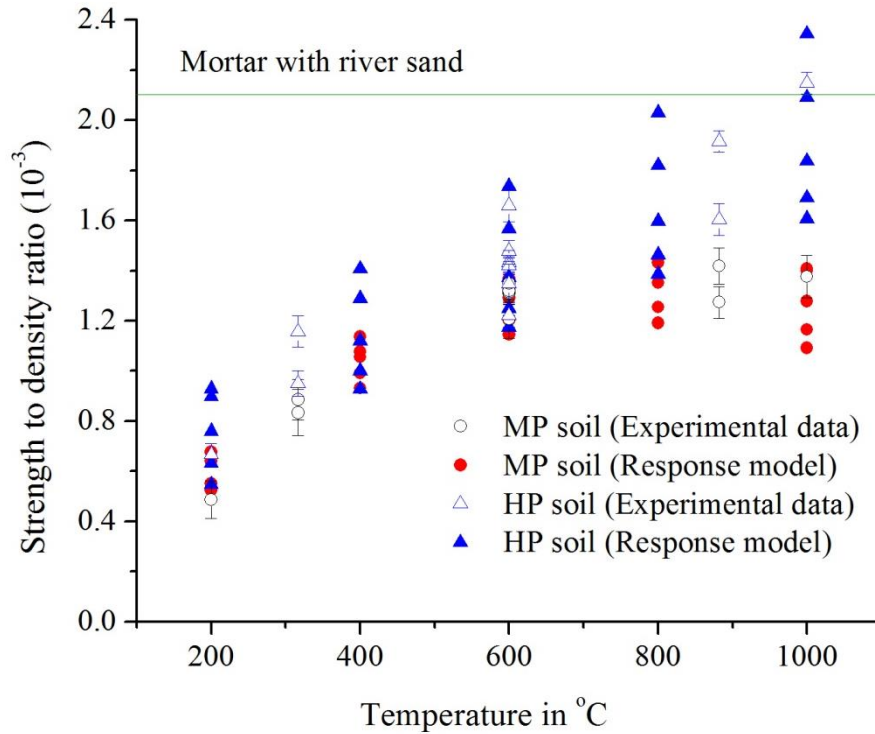
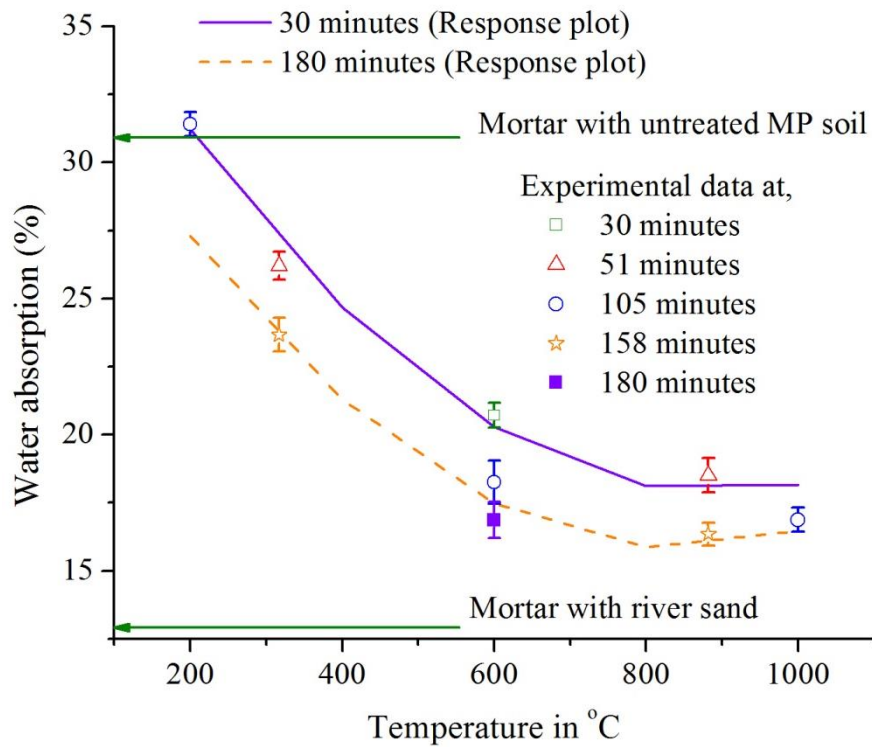


Figure 7.8 Strength to density ratio of cement mortar with thermally treated soils

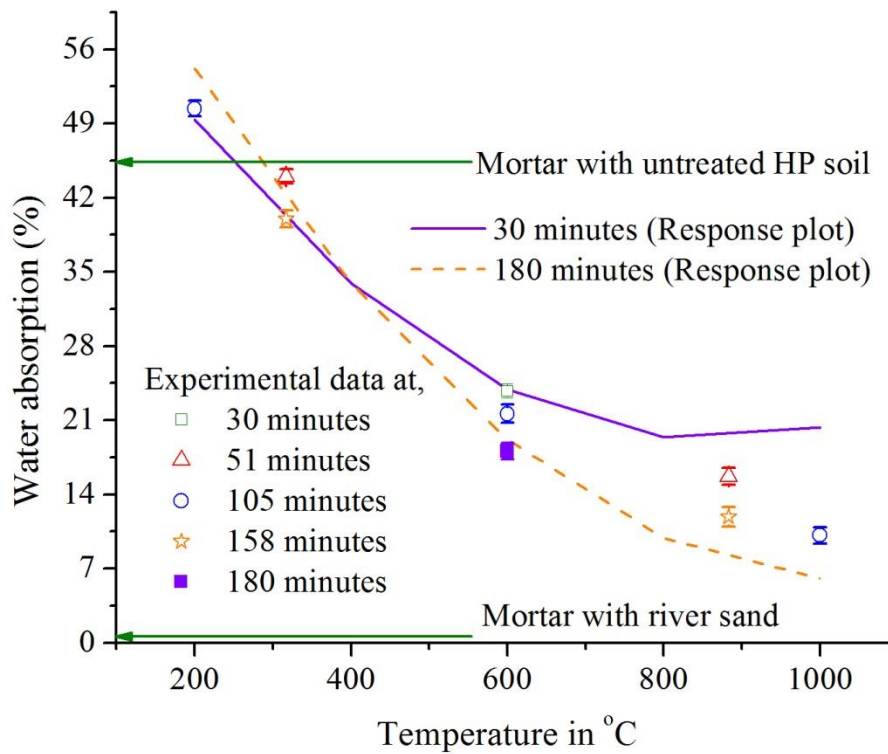
7.4.4 Water Absorption

Influence of treatment temperature and duration on water absorption of mortar with thermally treated MP and HP soil are shown in Figure 7.9. Considering the influential parameters, both the temperature and duration of treatment plays major role in bringing down the water absorption which is presented in ANOVA data in Table 7.4 and Table 7.5.

With MP soil, water absorption of mortar reduces with increasing duration of treatment. As MP soil needs much higher temperature for actual change in crystal structure, there is a need for longer treatment time at lower temperatures $< 1000^{\circ}\text{C}$ (Figure 7.9a). Mortar with HP soil shows similar behavior as mortar with MP soil with an exception at 180 minutes. This may be due to the breakdown of clay structure at lower temperatures ($< 600^{\circ}\text{C}$) and sintering effect at higher temperatures ($> 600^{\circ}\text{C}$) by sustaining the treatment duration beyond 120 minutes which influences the particle size and pore refinement (Figure 7.3b).



(a) Medium plastic soil



(b) High plastic soil

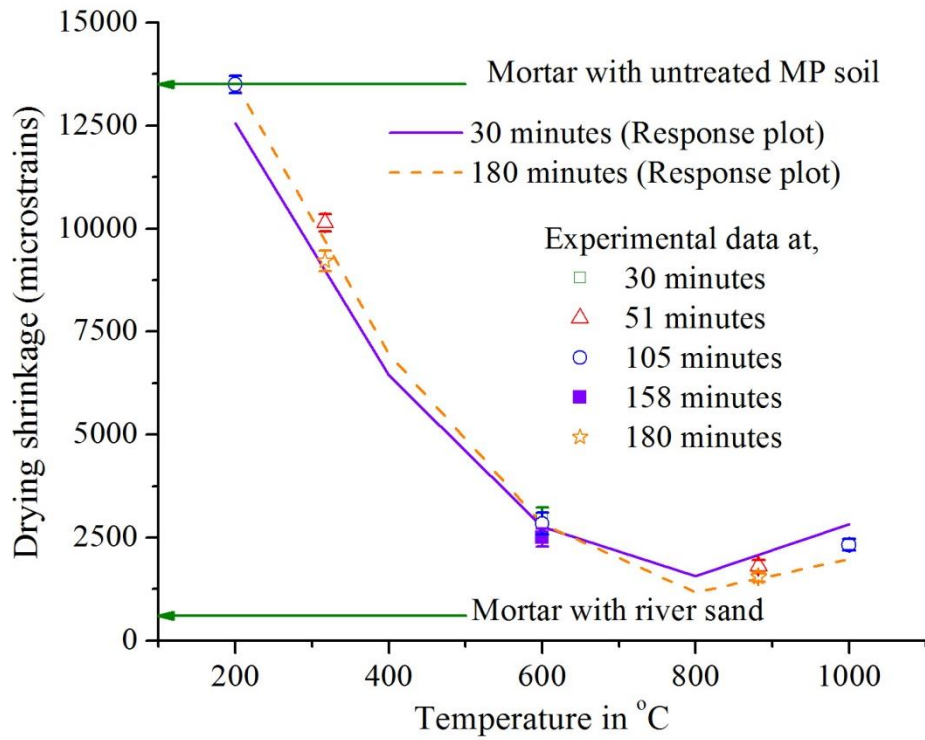
Figure 7.9 Water absorption of cement mortar with thermally treated soils

For a given treatment duration and type of fine aggregate, the water absorption of mortar with thermally treated soil reduces with increasing treatment temperature (Figure 7.9). Mortars with medium plastic soil shows 50% reduction of water absorption when temperature is raised from 200°C to 800°C whereas, mortar with HP soil shows 80% reduction. Highly expansive clay mineral in HP soil responds drastically to the raise in temperature and it can be noted that 800°C could be an optimum treatment temperature to bring down the water absorption value below 10% (Figure 7.9b).

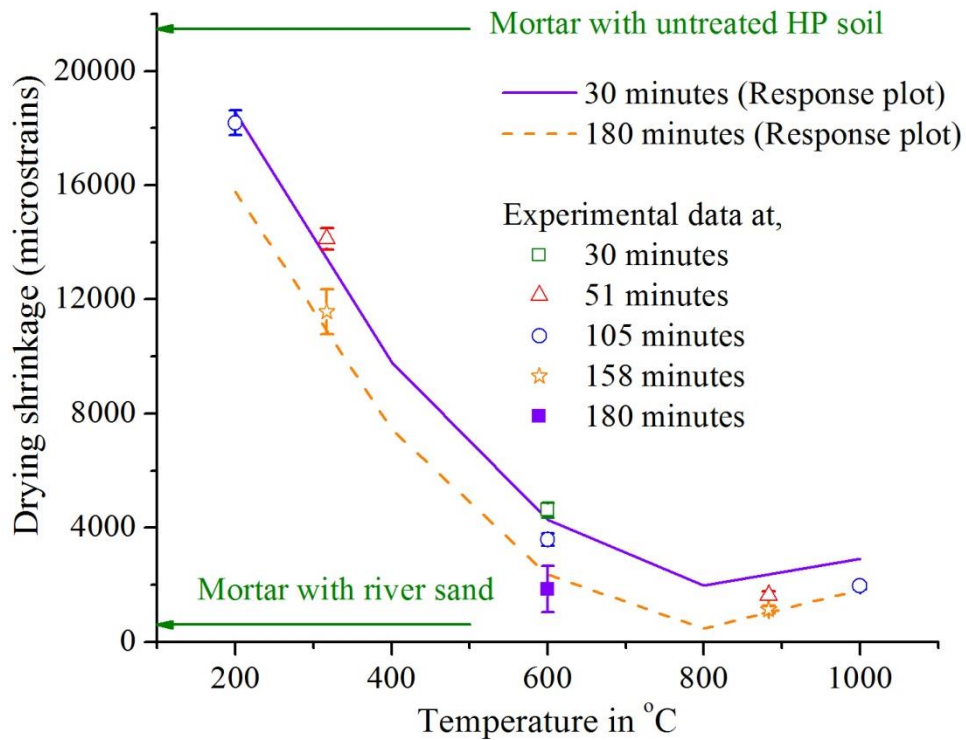
7.4.5 Drying Shrinkage

Drying shrinkage strains of mortar with thermally treated MP and HP soil are compared with that of mortar with untreated corresponding soils and control mortar with river sand in Figure 7.10. From ANOVA data presented in Table 7.4 and Table 7.5, it can be considered that temperature of treatment is the only significant parameter for drying shrinkage of mortar with MP and HP soils.

Drying shrinkage strain of mortar with MP and HP soil reduces by 85 and 95% respectively when the treatment temperature is raised from 200 to 800°C. This can be related to the reduction in clay size particles with increasing treatment temperature in both the soils (Figure 7.3). Egan et al., (2017) mentioned that increase in aggregate volume is a possible parameter to alter the shrinkage strain and here, with increasing temperature i.e., sintering of clay particles and growth in particle size helps in shrinkage reduction of mortars with expansive clay. However, there is an average 10% increase in drying shrinkage strain at 1000°C for mortars with MP and HP soil (Figure 7.10b) which can be explained with critical pore present in hardened mortar.



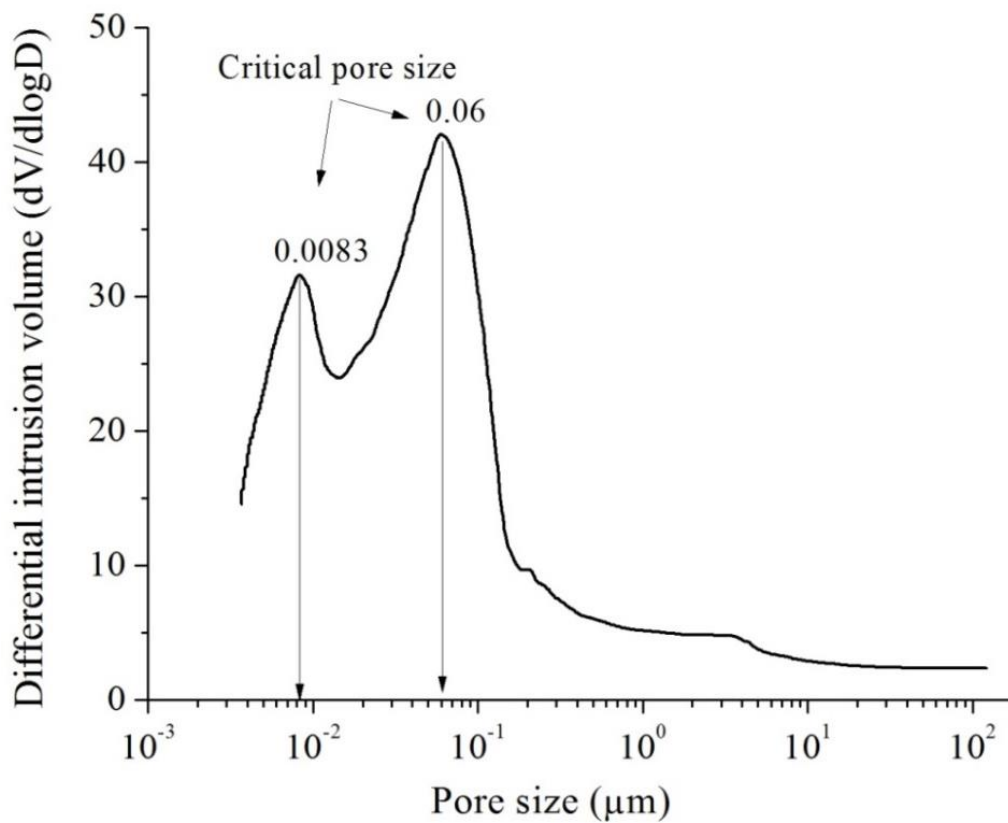
(a) Medium plastic soil



(b) High plastic soil

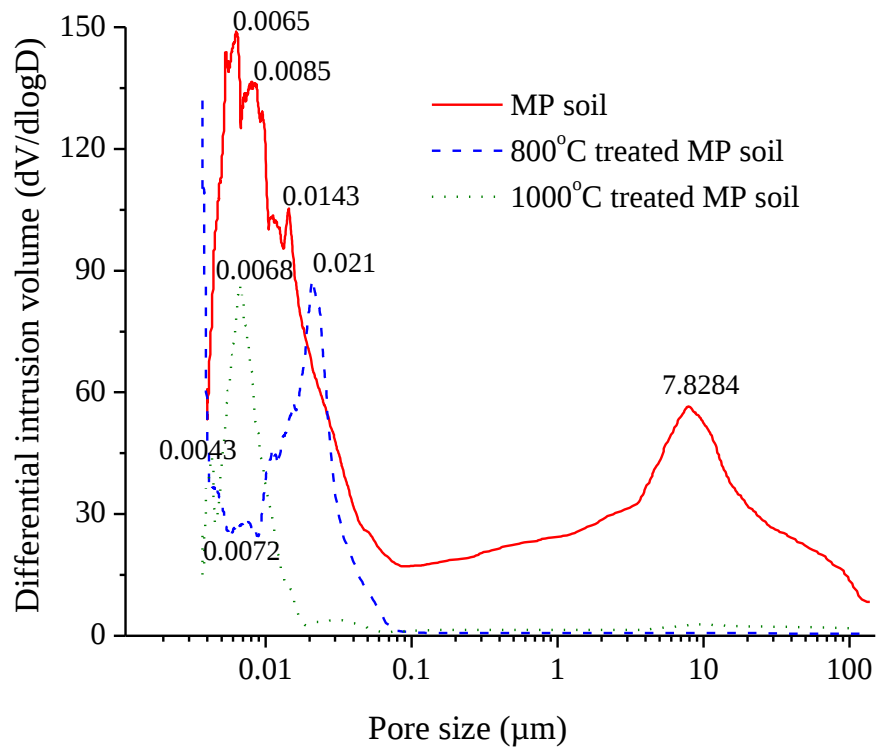
Figure 7.10 Drying shrinkage of cement mortar with thermally treated soils

Critical pore size distribution was calculated from Mercury Intrusion Porosimetry data of mortar specimens following the procedure followed in Chapter 6 (Section 6.2). With MIP curves of hardened mortar with MP and HP soil, mortar with untreated soils show 3 to 4 critical pore diameters without any definite shape which looks more like a MIP curve of a plain soil specimen. MIP curves of mortar with thermally treated MP and HP soils show bimodal curves that are typical of cement mortar specimens (Figure 7.11a). There is a reduction in pore size with thermal treatment that helps in bringing down the shrinkage strains. However, the formation of nano-pores in mortar with thermally treated MP (Figure 7.11b) and HP soil (Figure 7.11c) at 1000°C causes slight increase in shrinkage strain.

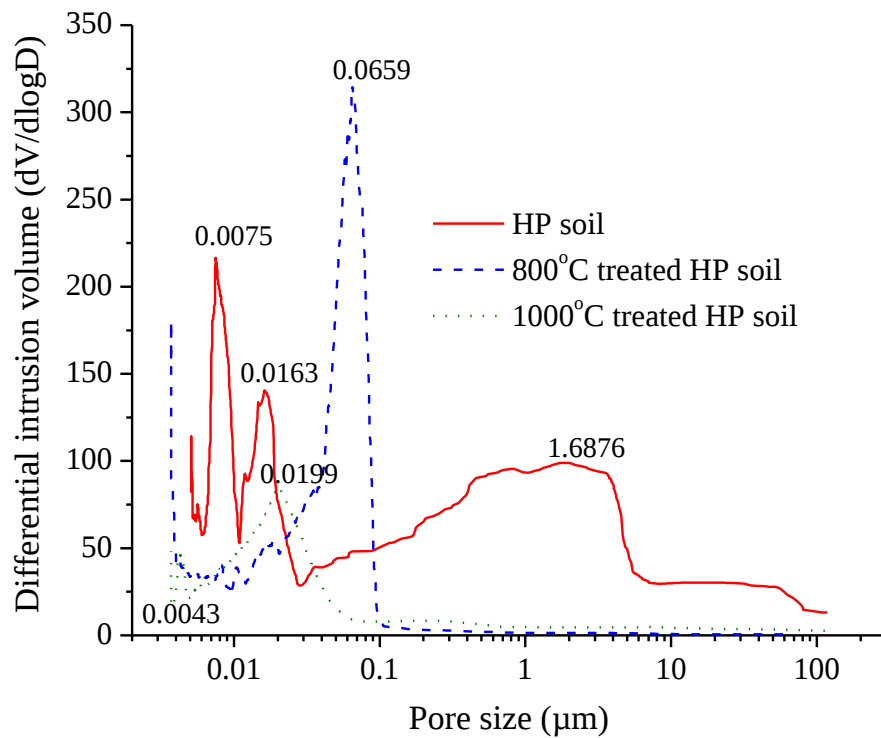


(a) River sand

Figure 7.11 continued...



(b) Medium plastic soil



(c) High plastic soil

Figure 7.11 Pore size distribution curves of cement mortar

7.4.6 Permeable Pore Volume

The permeable pore volume and the differential intrusion volume from MIP curve as shown in Figure 7.12 gives the total permeable porosity. Permeable porosity of mortar/concrete is considered to be the main factor governing the durability of the material by which water/aggressive chemicals enters the hydration products. ANOVA table indicates that permeable pore volume is affected by treatment temperature in case of mortar with MP soil (Table 7.4) and is affected by temperature and duration for mortar with HP soil (Table 7.5).

Influence of treatment temperature and duration on permeable pore volume present in the hardened cement mortar with thermally treated MP and HP soil are shown in Figure 7.13. The trend in reduction of permeable porosity gives a clear idea for the improvement in mortar property of thermally treated soil. For any soil type, total porosity of mortar reduces with increasing treatment temperature. This can be related to the growth in particle size with increasing temperature that alters the particle size distribution; thereby, the porosity (voids content) of mortar reduces.

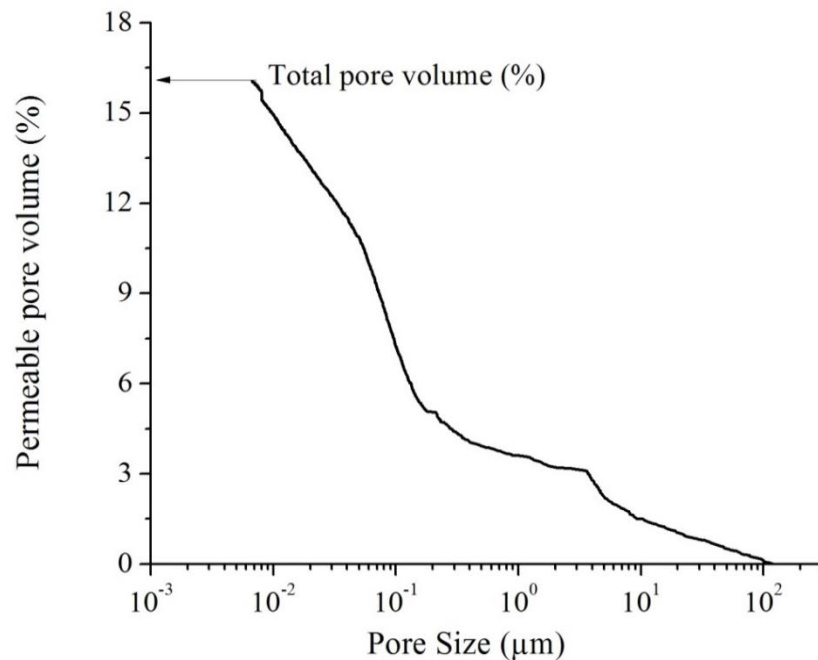
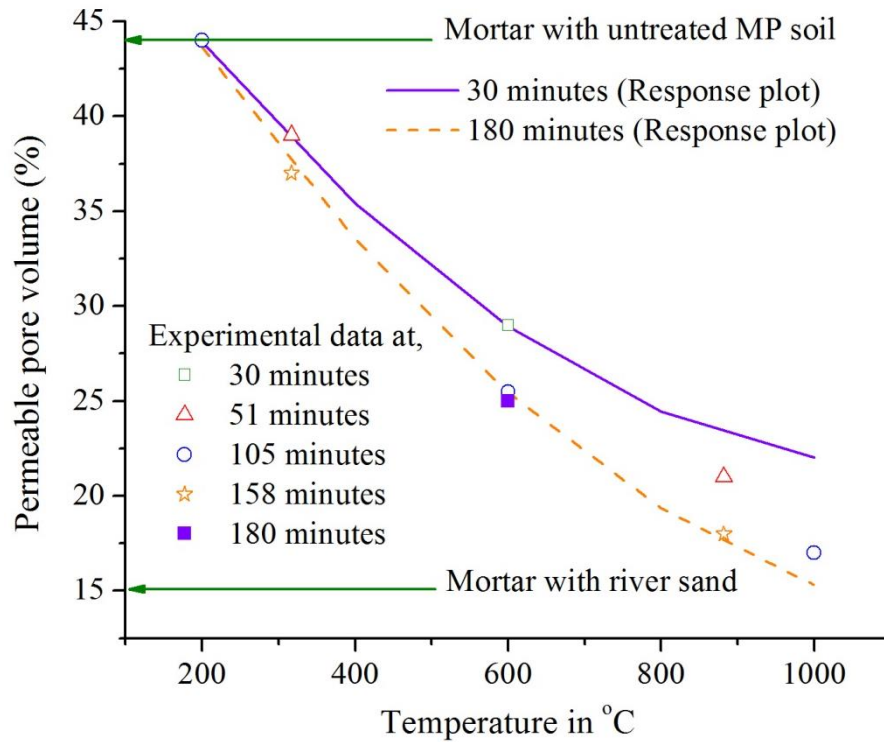
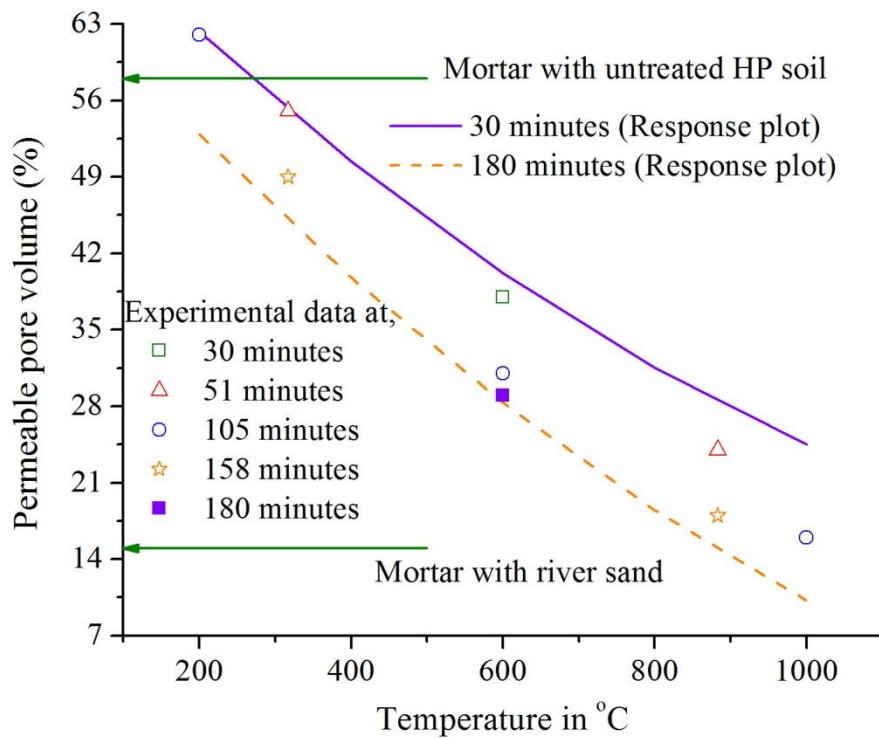


Figure 7.12 Permeable pore volume of mortar with river sand



(b) Medium plastic soil



(c) High plastic soil

Figure 7.13 Permeable pore volume in cement mortar with thermally treated soils

For a treatment temperature of 1000°C and duration of 120 minutes, mortar with medium plastic soil produces a microstructure with least permeable pore volume (Figure 7.13a). Considering HP soil, porosity of mortar is affected by both temperature and duration of treatment. There is a marked reduction in permeable pore volume with increasing duration at any given temperature. HP soil with almost 50% of clay content responds well for the heat treatment and the crystal structure modification occurs gradually with increasing duration of treatment (Figure 7.13b). Mortar with HP soil can achieve porosity even below that of control mortar when treated at a temperature > 800°C for duration in the range of 120 – 180 minutes.

7.5 EMBODIED ENERGY ANALYSIS

As this treatment method involves high temperature, it becomes inevitable to do an embodied energy analysis. In order to illustrate the methodology, a jobsite is assumed to be located at the Chennai city, Tamilnadu (India). The nearest availability of river sand for a construction site in Chennai city is from Chengalpattu (62 km). For extraction of loose material like river sand, mechanical shovel operated on diesel (energy - 43.1 MJ/kg) is used (da Rocha et al. 2016; Demiral, 2012). A potential replacement for river sand which is already accepted in construction industry is crushed stones. Production of crushed stone involves various process such as mining, crushing, sieving, washing and proportioning. Energy involved in these operations is calculated from available data in literatures and equipment manuals (Landfield and Karra 2000). Mining of rock involves use of percussion drill, hydraulic shovel, dump truck and bulldozer (EEP, 2013). Nearest location of crushed stone aggregate quarry to Chennai city is Sriperumbudur (40 km). It could be identified that natural sand/crushed stone consumes 1.75 MJ/m³ of energy for every 1 km of transport distance (Reddy and Jagadish 2003).

For the embodied analysis of a construction material, there are several approaches for comparisons of different compositions involved in the manufacturing process. In this study drying shrinkage strain is considered to be the most important parameter as fine aggregate plays an influential role in shrinkage property of soil-based mortar. Cement mortar which could produce a shrinkage strain equivalent to that of mortar with river sand is made as reference to fix the manufacturing parameters of alternative fine aggregates (excavation soil). Based on the experimental data, this is fixed as 800°C for 120 minutes for MP and HP soil. Additionally, 90 minutes of treatment duration was considered for both the types of soil which gives a shrinkage strain less than 2000 micro strains. An industrial grade electrical

furnace of capacity 500 kg/hr with a power consumption of 27 MJ/hr was considered for the energy and cost calculations of thermal treatment of different soil. Thermal conductivity of soil found from calorimetric data of the soil samples increases with its fineness and clay content and found to be in the range of 0.45 – 0.6 kJ/kg.°C for medium to high plastic soil.

Transportation cost of excavated soil involves both from construction site to treatment plant and from plant to the site. Considering a treatment plant in the fringes of the City, maximum transportation distance for excavation soil before and after treatment could be 40 km. The embodied energy comparison is made for gate to gate which would serve as a better comparison factor in this case and presented in Table 7.6. Also, the processing of crushed stone involves non-renewable fuel usage such as diesel/electricity which cannot be replaced by alternative energy.

Table 7.6 Comparison of embodied energy of conventional and alternative fine aggregates

Type of fine aggregate	Processing					Transportation		Total embodied energy (MJ/tonne)
	category	Type of fuel	Fuel consumption	Production (tonne/hr)	Energy (MJ/tonne)	Distance (km)	Energy (MJ/tonne)	
River sand	Extraction	Diesel	0.15 ltr/h	143	54.00	62	108.5	162.5
Crushed stone	Extraction	Diesel	15 ltr/h	For granite	182.67	40	70	255.47
	Crushing	Electric			1.60			
	Screening	Electric	27 MJ/hr	100	0.20			
	Washing	Electric	54 MJ/hr	50	1.00			
Excavated soil	(0.45 – 0.6) kJ/kg.°C × (800°C for 90 to 180 minutes)	Electric	27 MJ/hr	0.41 - 0.65	81 - 162	40 – 60	70 – 122.5	151 – 284.5

7.6 SUMMARY

Thermal treatment of soil with expansive clay significantly improved the material properties by sintering of clay particles and particle size growth. This helped in improving the cement mortar properties with thermally treated soils. Comparatively, soil with high clay content (>50%) with desirable clay mineralogy responded well to thermal treatment. Based on the analysis, it is clear that for producing mortar of comparable properties with thermally treated excavation soil with high clay content, demands high energy. With low plastic soil, alternative treatment methods with low energy usage such as washing and sieving can be adopted. However, high plastic soil with more than 50% clay content does not work with other treatment methods such as granulometry or stabilization. More than that, other alternatives such as crushed stone is a product from mineral rocks that are non-renewable source of construction material which may go extinct similar to river sand. Whereas, excavation soil are waste generated by construction industries which can be reused by this treatment method. This paves way for a circular economy in construction industry.

CHAPTER 8

CONCLUSIONS AND SCOPE FOR FUTURE WORK

8.1 CONCLUSIONS

The salient conclusions arising out of this research work are summarised in this section. The studies on excavation soil as fine aggregate in mortar comes under four stages viz.; (i) performance of unprocessed excavation soil in geopolymer mortar; (ii) performance of stabilized excavation soil in cement mortar; (iii) performance of wet sieved excavation soil in cement mortar; and (iv) performance of thermally treated excavation soil in cement mortar. These conclusions are applicable to the characteristics of the material used, range of parameters investigated and the methodology adopted in this study.

8.1.1 Performance of Unprocessed Excavation Soil in Geopolymer Mortar

In fly ash based geopolymer mortar, all three types of soil (low, medium and high plastic) were used as fine aggregate and the properties were compared with control mortar made of river sand. Molarity of NaOH, fly ash to fine aggregate ratio and curing temperature were the factors considered in this study.

- i) Molarity of NaOH is a significant factor that affects all the properties of the geopolymer mortar with different fine aggregates. Workability got reduced with increasing NaOH concentration whereas dry density and compressive strength are improved. The percentage increase in compressive strength with molarity of NaOH ranges from 78% to 128% for geopolymer mortar with plastic soils. Water absorption and shrinkage are positively affected by increasing molarity of NaOH.
- ii) Increasing curing temperature increases the dry density and compressive strength and reduces the shrinkage strains of the geopolymer mortar mixes. Higher curing temperature helps in geopolymerisation process and makes the matrix dense and stiff, resulting in better mortar properties. With increase in curing temperature from 60 °C to 90°C, shrinkage strains reduced in the range of 21% to 48%.
- iii) Increasing the fly ash to fine aggregate ratio, increases the fly ash content which improves the workability of geopolymer mortar with plastic soils. Further, fly ash

participates actively in geopolymerisation thereby increasing the compressive strength up to 88% and reduces the shrinkage strains up to 72%.

- iv) Considering the dry density, mixes with clayey fine aggregates helps to achieve enhanced properties at a lower dry density range. By suitably designing the parameters, geopolymer mortar with properties comparable to control mix can be achieved using plastic soils as fine aggregates.

8.1.2 Performance of Stabilized Excavation Soil in Cement Mortar

Low, medium and high plastic soils were dry sieved and used as fine aggregate in cement mortar. Raw soil and dry sieved soil were then stabilized using lime and ground granulated blast furnace slag (GGBS). Properties of mortar with stabilized soils were compared with that of mortar with river sand.

- i) Dry sieving of soil using 600 μm sieve partially removes some of the fines and clay particles, thereby improves soil-based mortar properties. However, this can be an effective method to improve cement mortar properties with non-plastic or low plastic soils only.
- ii) Water demand of mortar increased with increasing soil plasticity which could be reduced by stabilization to a maximum of 35, 64 and 135% for low, medium and high plastic soils, respectively. Combination of sieving and stabilization of soil helps in controlling the water demand to maintain a constant workability.
- iii) Dry density and compressive strength improved better with slag stabilized plastic soil mortars compared to lime stabilized soil mortar. The reactive silica and alumina with Ca(OH)_2 in slag helped in improving the hydration process and results in the formation of dense microstructure.
- iv) Low plastic soil mortar reached a strength value equivalent to river sand by dry sieving and stabilizing with slag. Medium and high plastic soil mortar though could not reach an equivalent strength, there was a remarkable improvement of 145 and 171%, respectively, compared to mortar with unprocessed soils.
- v) Water absorption and shrinkage are influenced by factors such as clay mineralogy, stabilizer type and dosage. Soil with non-reactive clay minerals, stabilized with slag

gives lowest water absorption and shrinkage strains. Whereas, lime stabilization gives enhanced mortar properties in soil with high plasticity.

8.1.3 Performance of Wet Sieved Excavation Soil in Cement Mortar

Low and medium plastic soils were wet sieved through 75 μm size sieve, resulting in three products such as, sand particles, wash water and residual clay.

- i) Wet sieving technique is very effective in removing the clay and fines smaller than 75 μm . The difference in properties of excavated soils in mortar is mainly due to the particle size distribution and pore formation inside the mortar samples.
- ii) Excessive water demand to wet the large specific surface area of fines and clay in excavated soil was reduced by 80–100% in wet sieved sand.
- iii) Wet sieving improved the compressive strength up to 160% and reduced the water absorption by 50% when compared to the raw soil mortar.
- iv) Compared to raw soil mortars, the removal of fines and clay content by wet sieving helped in reducing the shrinkage strain by 86%. Though fineness and pore inter-connectivity played a vital role in shrinkage property of mortar specimens, shrinkage strain of cement mortar with wet sieved sand was comparable to that of mortar with river sand.
- v) Cement hydration is not affected by the use of wash water and mortar properties are comparable to those with tap water. This concludes that the wash water used for wet sieving can be wisely reused in the mortar or concrete production, making this treatment method more eco-friendly and sustainable.
- vi) The pozzolanic performance of residual clay with a major portion of kaolinite is superior compared to residual clay with montmorillonite. However, both the residual clays satisfy the requirement to be used as pozzolan in cementitious systems.

8.1.4 Performance of Thermally Treated Excavation Soil in Cement Mortar

Medium and high plastic soils were thermally treated at a temperature range of 200 to 1000°C for the treatment duration between 30 to 180 minutes.

- i) Dehydration occurs at 100°C irrespective of type of soil, followed by dehydroxylation of kolinite at 400–600°C and montmorillonite at 600–800°C and illite at temperature greater than 800°C.
- ii) Heat treatment resulted in grain growth due to sintering effect on clay particles in both medium and high plastic soils. Maximum effect was noted in high plastic soil with 40% of expansive clay.
- iii) Treatment temperature is the major influential parameter that affects all the properties of mortar with thermally treated soils. Mortar with MP soil shows comparatively less property enhancement due to the presence of illite/stilbite clay that needs treatment temperature more than 1000°C. Whereas mortar with HP soil behaves well with increasing temperature and reaches its maximum at 1000°C.
- iv) In cement mortar with thermally treated excavation soil with high clay content (>50%) at 800°C for 90 minutes, percentage reduction in water demand was 68%, due to the growth of clay size particles by sintering. Shrinkage strain of mortar with high plastic soil got reduced by 96% and strength improved to 85% compared to mortar with raw soil.
- v) The behavior of cement mortar with thermally treated soils can be related to the pore refinement in the hardened structure which results in less permeable pore volume with reduced pore size.

8.2 SCOPE FOR FUTURE WORK

The present study gives a platform for carrying out detailed long term investigations on excavation soil as alternative material for fine aggregate. This work needs extensive research in various aspects like:

- i) Investigations on effect of super plasticizers on enhancing the mortar/concrete properties with excavation soil.
- ii) Studies on plastic shrinkage, long-term durability studies including alkali-silica reaction.
- iii) Methods of reducing shrinkage by incorporation of fibers, shrinkage reducing admixtures.
- iv) Treatment methods addressing other deleterious materials such as organic content in soil.

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PUBLICATIONS BASED ON THE THESIS

Journal Publications

- **Priyadharshini, P.**, Ramamurthy, K. and Robinson, R.G. (2017). “Excavated soil waste as fine aggregate in fly ash based geopolymer mortar.” *Applied Clay Science*. 146, 81–91.
- **Priyadharshini, P.**, Ramamurthy, K. and Robinson, R.G. (2018). “Sustainable Reuse of Excavation Soil in Cementitious Composites.” *Journal of Cleaner Production*. 176, 999–1011.

Conference Proceedings

- **Priyadharshini, P.**, Ramamurthy, K. and Robinson, R.G. (2017). “Effect of raw earth as fine aggregate in mortar properties.” In Proceedings of 32nd International Conference on Solid Waste Technology and Management (ICSW-2017). 79-88.
- **Priyadharshini, P.**, Ramamurthy, K. and Robinson, R.G. (2017). “Use of lime stabilized lake sediments as fine aggregates in cement mortar.” In Proceedings of International Conference on Advances in Construction Materials and Systems (ICACMS-2017), vol.3, 183-192.

DOCTORAL COMMITTEE

CHAIR PERSON

Dr. Manu Santhanam
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Department of Civil Engineering

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Department of Civil Engineering

Dr. Manu Santhanam
Professor
Department of Civil Engineering

Dr. Abhijit P. Deshpande
Professor
Department of Chemical Engineering

CURRICULUM VITAE

Name : P. Priyadharshini

Date of birth : 10 - 05 - 1986

Educational qualification

2007 : Bachelor of Engineering (B.E.)

Institution : Thiagarajar College of Engineering,
Madurai, Tamil Nadu

University : Anna University

Specialization : Civil Engineering

2013 : Master of Science (M.S., By Research)

Institution : Vellore Institute of Technology, Vellore, India

University : VIT University

Specialization : Structural and Geotechnical Engineering

2018 : Doctor of Philosophy (Ph. D.)

Institution : Indian Institute of Technology, Madras, Chennai,
India

Specialization : Building Technology and Construction Management
(Construction Materials and Concrete Technology)

Registration date : 02/01/2014