SUSTAINABLE PRODUCTION OF FLY ASH BASED COARSE AGGREGATES AND ITS PERFORMANCE IN CONCRETE

Thesis

Submitted in partial fulfilment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

by

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DEPARTMENT OF CIVIL ENGINEERING
NATIONAL INSTITUTE OF TECHNOLOGY KARNATAKA,
SURATHKAL, MANGALORE - 575025
JANUARY 2020

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DECLARATION

I hereby declare that the Research Thesis entitled "**Sustainable Production of Fly Ash Based Coarse Aggregates and its Performance in Concrete"** which is being submitted to the **National Institute of Technology Karnataka, Surathkal** in partial fulfilment of the requirements for the award of the Degree of **Doctor of Philosophy** in **Civil Engineering** is a bonafide report of the research work carried out by me. The material contained in this Research Thesis has not been submitted to any University or Institution for the award of any degree.

Place: NITK, SURATHKAL
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CERTIFICATE

This is to certify that the Research Thesis entitled "**Sustainable Production of Fly Ash Based Coarse Aggregates and its Performance in Concrete"** submitted by **SHIVAPRASAD K N** (Registration number: 155088CV15F10) as the record of research work carried out by him, is accepted as Research Thesis submission in partial fulfilment of the requirements for the award of degree of **Doctor of Philosophy**.

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Dedicated to

My Beloved Parents

and

Teachers

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Shivaprasad K N

ABSTRACT

An optimization study was carried out for the sustainable production of fly ash based coarse aggregates from the industrial by-products such as fly ash, considering the combined effect of geopolymerisation and pelletization. The trial mixes during the process of producing the fly ash based coarse aggregates were designed through Taguchi's experimental design method. The combined effect of geopolymerisation and pelletization factors in the production process and engineering properties of the produced fly ash based coarse aggregates were evaluated using response indices at different curing ages. Further, the influence of each individual factor of geopolymerisation and pelletization on the engineering properties was determined through Grey relational analysis to identify the most influencing factors in the production of fly ash based coarse aggregates. The results obtained from Grey relational analysis indicate that the properties of produced fly ash aggregates are governed mostly by geopolymerisation. It is also observed that water content of 20 % by mass of fly ash found to be essential for the suitable production of fly ash based coarse aggregates. Artificially produced fly ash aggregate were used in the production of concrete with partial replacement of natural aggregates and it is found that for the production of M40 grade durable concrete, up to 30% by its volume can be replaced effectively.

CONTENTS

LIST OF FIGURES

LIST OF TABLES

NOMENCLATURE

Abbreviations

AH : Alkali Hydroxide

AS : Alkali Silicate

GRC : Grey Relational Coefficient

GRG : Grey Relational Grade

OPC : Ordinary Portland Cement

RCPT : Rapid Chloride ion Penetration Test

SEM : Scanning Electron Microscope

XRF : X-Ray Fluorescence test

IS : Indian Standard

ASTM : American Society for Testing and Materials

BS : British Standards

F/A : Fly ash to Alkaline solution ratio

A/F : Alkaline solution to Fly ash ratio

w/b : water to binder ratio

w/c : water to cement ratio

RPM : Rotations Per Minute

TM : Taguchi Method preliminary study

TMG : Taguchi Method Geopolymerisation factors study

TMP : Taguchi Method Pelletization factors study

TMC : Taguchi Method Combined factor study

CHAPTER – 1

INTRODUCTION

1.1 GENERAL

Concrete is the second most consumed commodity by volume after water. The statistics depict global concrete production is approximately 5.3 billion cubic meters per year (Hasanbeigi et al 2012). Typically, ordinary concrete contains about 70-80% aggregate, 12% cement, and 8% mixing water by mass (Mehta 2002). To produce one cubic meter of concrete, approximately two tons of aggregates are required. A recent study from Freedonia Group (2013), a USA-based industry market research company reported that the global demand for construction aggregate is about 51.7 billion metric tons in 2018 and is expected to grow 5.2% annually. In India, the consumption of aggregate was about 2.2 billion metric tons in 2010 and further the demand is going to be more than 5 billion metric tons by 2020. At the present growth rate of the developing countries of the world, the consumption rate of natural aggregates has reached an alarming level and as of now the aggregate component in concrete is quarried from the mines. Mining of aggregate leads to serious environmental impacts.

On the other hand, due to the industrialization, it is becoming very complicated to fulfil the primary energy requirements. So coal based energy production methods have gained attention in mass energy producing units. During the energy extraction, pulverized coal is used in power plants which produce fly ash. In 2017, the global fly ash market is accounted for 2.80 billion USD and is expected to reach 5.55 billion USD by 2026 growing at a compound annual growth rate of 7.9% during the forecast period (Absolute 2018). The quality of produced fly ash depends on the coal quality used; for instance, Indian coal has high ash content of 35 – 45 % and is of poorer quality. In India, fly ash production is about 196.44 MT in the year 2017-18 (Central Electricity Authority 2018) and it is reported that by the year 2025 fly ash production is expected to increase by around 300 – 400 MT per year (Karnamprabhakara et al 2019). The major portion of fly ash produced is disposed of in ash pond and landfill. A small fraction of it is being utilised in the cement and concrete additives, ceramics, agriculture application, building materials, water treatment, etc. Due to its toxic contents and high salinity, disposal in landfill leads to soil pollution.

For last two decades fly ash is being used as a pozzolanic material for manufacturing of Portland Pozzalona Cements or blended cements in the production of building materials such as fly ash bricks and precast units, as a suitable material in ready mix concrete, in agriculture, construction of roads, land filling of mines and low lying areas and several other applications. Recently, it is found from the literature that fly ash can be replaced with cement even up to 100% by introducing suitable alkaline activators are known as geopolymer.

Enough studies were reported on the production of artificial aggregates using fly ash and natural materials through agglomeration process (Baykal and Doven 2000, Ramamurthy and Harikrishnan 2006, Gesoglu et al 2007, Clarke 2014, Gunasekara et al 2018). In the production of fly ash aggregates, it is noted that researchers have adopted different techniques such as agglomeration, expansion (bloating), pressing and crushing, etc., (Baykal and Doven 2000, Clarke 2014, Gunasekara et al 2018). The pelletization theory or agglomeration of fines was developed in 1940's and it is found to be well developed technique for producing fly ash aggregates (Baykal and Doven 2000). Fly ash aggregates produced through the process of pelletization is still not being adopted widely by the construction industry and hence the production cost of fly ash aggregates is found to be high as compared to the cost of natural aggregates.

The formation of pellets or aggregates in the production process i.e., the particle size distribution of aggregates and their properties can be better controlled with a disc type pelletizer as compared to other types such as a cone or drum type of pelletizer (Baykal and Doven 2000, Ramamurthy and Harikrishnan 2006). Some effort has been made on utilizing the fly ash in the production of artificial aggregates using different additives such as cement, clay and glass powder (Ramamurthy and Harikrishnan 2006, Priyadharshini et al. 2011, Gomathi and Sivakumar 2014). Many researchers reported that the factors affecting the production process in producing the aggregates are mainly depending on the type of raw materials, type of binder, size of the particles, specific surface area, the wettability of particles, water content and production process involved

(Ramamurthy and Harikrishnan 2006, Priyadharshini et al. 2011, Gomathi and Sivakumar 2014, Gesoglu et al. 2007). It is also reported that the hardening methods adopted for producing pellets are sintering, normal water curing, autoclaving and steam curing (Manikandan and Ramamurthy 2008, Priyadharshini et al. 2011, Gomathi and Sivakumar 2014). The engineering performance of produced pellets is predominantly depending on the binder used in the process (Ramamurthy and Harikrishnan 2006, Priyadharshini et al. 2011, Gomathi and Sivakumar 2014, Gesoglu et al. 2007).

It is also reported that aggregates can be produced with fly ash as the main constituent material through the process of pelletization. Whereas, the artificially produced aggregates through the process of pelletization do not meet the basic requirements of engineering properties to be adopted as a suitable construction material by the construction industry. Hence, utilization of fly ash can be used in the production of artificial fly ash based coarse aggregates by introducing a suitable binder such as an alkaline solution (geopolymer).

It is reported that geopolymers have many applications including replacement of cement in building materials and as binder for concretes (Davidovits 1999). In geopolymerisation, alkali activator or chemical activator solution plays a vital role in the initiation of surface hydrolysis of the particles present in the alumino-silicate materials as the raw material (Rangan et al. 2005). The raw aluminosilicate materials used in geopolymerisation can be broadly classified into two groups: (1) calcined materials such as metakaolin, fly ash, slag and pozzolanic wastes; (2) non-calcined materials such as clays, feldspar, kaolinite, and mine tailings. The effect of the process is greatly affected by a number of factors such as type of alkali activator, concentration of the alkali solution, binder to alkali ratio and curing regime (Davidovits 1989, Hardjito et al. 2004, Rangan et al. 2005, Khale and Rubina 2007, Hardjito et al. 2008, Rattanasak and Prinya 2009, Komljenovic et al. 2010, Mustafa et al. 2012, Gorhan and Kurklu 2014, Patankar et al. 2014, Rahim et al. 2014, Rahmiati et al. 2014, Phoo-ngernkham et al. 2015).

However, use of alkaline solution as a binding media in the production of fly ash aggregate is a complex and challenging task. The complexity involves identifying the important factors of geopolymerisation and pelletization. To study these complexities of relative influence of geopolymerisation and pelletization on the properties of aggregates and to identify the optimum value of factors, the experimental design plays an important role. There are several experimental designs that can be used to prepare or reformulate an optimal process. Several experimental designs such as factorial designs (Soudki et al. 2001, Cavazzuti 2013, Krishnan and Purushothaman 2017), Taguchi method (Olivia and Nikraz 2012, Cavazzuti 2013) response surface methodology (Hanrahan and Lu 2006, Cavazzuti 2013, Vasugi and Ramamurthy 2014) and other techniques are available which can be formulated and used to optimize the production process. One of the designs is an orthogonal array or Taguchi method defined by Taguchi, which is a powerful optimization technique and unique method that allows to study the influence of selected factors with a small number of trials (Cavazzuti 2013). It also provides a clear understanding between the factors considered with the individual parameter. However, for getting clear understanding among the response indices which one is the most influential in the experimental design program, can be determined using Grey relational analysis (Sahoo et al. 2016, Srinivasan et al 2018).

Whereas, sufficient information is available in the literature in the production of pelletized fly ash aggregate with alkaline solution (geopolymer) as the binding media will lead to a new level of mitigating the requirement of such a large amount of aggregate in concrete, which is otherwise is being quarried from the mines. Sustainable solutions for the utilization of such a large volume of industrial by-products in developing a value added product also pose a great challenge to the research fraternity.

1.2 NEED FOR THE PRESENT WORK

The amount of utilization of aggregates is increasing because of the rapid change in the infrastructure development of the country. However, the availability of natural aggregates is getting scarce and mining leads to serious environmental impact. Hence, it is necessary to find alternate material which can replace the natural mined aggregate for construction works. Along with alternate material, technology plays a vital role in the manufacturing of artificial aggregates with the local available resources. In this scenario, considering the industrial by products as one of the important raw material in producing artificial aggregates poses a great challenge to the researchers. A substantial utilization of this industrial waste in producing coarse aggregates can be one of the good solutions to waste management, disposal problems and also to alleviate potential health hazards.

1.3 THESIS STRUCTURE

The thesis is divided into seven chapters. Chapter one, a brief description of general introduction and need of the study has been given. Chapter two presents a comprehensive review of relevant literature and critical review towards the research gap. Third chapter contains the materials used in the present study, basic tests conducted on the materials characterisation. Chapter four contains the experimental methodology of the study, i.e., the influence of combined factors of pelletization and geopolymerisation on sustainable production of artificial fly ash aggregates planned through Taguchi’s experimental design is discussed. Further, concrete mix proportions along with relevant tests on aggregates and concrete are also discussed in detail. The test results and discussion on the influence of factors affecting the production of aggregates are presented in chapter five. The results of mechanical properties along with the results of durability studies on the concrete produced with artificially produced fly ash aggregates as a partial replacement to natural coarse aggregates are presented in chapter six. Conclusions along with the major findings from the present work and the scope for future research are presented in chapter seven.

CHAPTER – 2

LITERATURE REVIEW

2.1 GENERAL

This chapter provides a comprehensive review of relevant literature on aggregate production through pelletization process with different types of binder used in the production. Further, geopolymer binder is reviewed for the suitability in the fly ash aggregate production. Finally, the use of produced aggregates in concrete production and its performance.

2.2 PELLETIZATION THEORY

The pelletization theory was developed in 1940’s. Pelletization of fine powders has grown greatly in importance as an industrial process and particularly in the metallurgical industry (Harikrishnan and Ramamurthy 2006). Even though it is a known technique in the production of artificial aggregates, it has not been extensively used in construction sector. The reason behind it was the abundant availability of natural resources and production costs relatively high for the artificial aggregate production when compared to natural aggregate (Baykal and Doven 2000).

2.2.1 Pelletization process

Pelletization process is defined as the process of forming larger spherical bodies by rolling moisturized fine particles on a smooth surface without application of external pressures. The required grain size distribution of artificial aggregates may be obtained either by crushing of large size aggregates or by the agglomeration of fines that has cementitious property either by themselves and/or by blending them with binders. In other words, the agglomeration of moisturized fines in a rotating drum or disc is referred as pelletization process. The product at the end of the pelletization process is referred as ‘fresh pellet’. The fresh pellet’s crushing strength must be enough for hauling and stockpiling purposes. The pellets may be sintered for specified interval of time after production to improve its engineering performance depending on the properties of the fines used in pelletization process and the intend purpose of use (Baykal and Doven 2000).

2.2.2 Factors affecting the pelletization process

Many researchers reported that the factors affecting the pelletization process in producing the pellet from finer particles such as raw materials, moisture content, binder type, dosage and duration of pelletization (Geetha and Ramamurthy 2011) and some of the parameters should be considered for the efficiency of the production of pellet i.e., mainly the type of pelletizer for the pelletization process. Further, it is also reported that the hardening methods adopted for producing pellets are sintering, normal water curing, autoclaving and steam curing (Manikandan and Ramamurthy 2008). However, the energy required for the procedures to obtain the agglomerated material is high (i.e., curing regime and sintering) and the engineering performance of produced pellets depends mostly on the binder used in the process (Baykal and Doven 2000, Ramamurthy and Harikrishnan 2006).

2.2.2.1 Type of pelletizer and its factors

The different types of pelletizer machine such as disc or pan type, drum type, cone type and mixer type were used to make the pellet (Sivakumar and Gomathi 2012; Sastry et al 2003). Among these pelletizers, in disc type pelletizer, the pellet size distribution is easier to control than drum type pelletizer. With disc type pelletizer, the small grains are formed initially and are subsequently increased in particle size (Sivakumar and Gomathi 2012). Disc type pelletizer is most commonly used in production of artificial aggregate with good efficiency (Harikrishnan and Ramamurthy 2006; Gomathi and Sivakumar 2014).

Typical laboratory disc type pelletizer is of 0.40-0.60 m in diameter and 0.15-0.25 m depth (Harikrishnan and Ramamurthy 2006; Priyadharshini et al 2011; Sivakumar and Gomathi 2012) and it is fixed in an adjustable frame where angle of the disc can be varied from 0° to 70°. In these types, pelletizer is provided with motors and gear arrangement where speed of the pelletizing disc can control with different speeds varying from 0 to 55 rpm. A sketch of components of pelletization disc and dimensional details of pelletizer is presented in Figures 2.1 – 2.2.

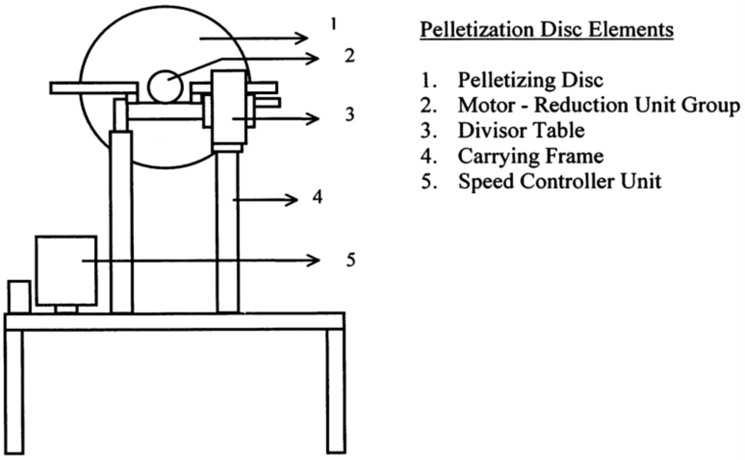


Figure 2.1: Details of pelletization disc - back view (Source: Baykal and Doven 2000)

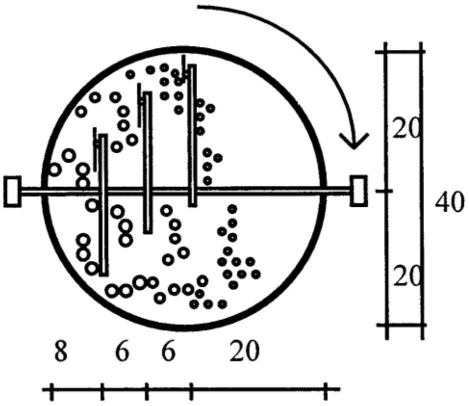


Figure 2.2: Plan of scraping blades positioning (Source: Baykal and Doven 2000)

Pelletization process mainly depends on the (a) speed of pelletizer disc (b) angle of pelletizer disc (c) moisture content and (d) pelletization duration. Along with raw materials particle size and their distribution, the wettability of particles and moisture content, along with the other process related parameters also affects the pelletization process (Harikrishnan and Ramamurthy 2006; Gomathi and Sivakumar 2012).

2.2.2.2 Properties of raw materials and binder factors

The study on engineering performance of moist cured fly ash pellets with mineral additives such as cement and lime for production of artificial light weight fly ash aggregates were investigated by Baykal and Doven (2000). The optimum moisture content for pelletization process for the artificial aggregates ranges between 29 and 33%. The crushing value of artificial pellets with mineral additives increased up to

319% for 28 days curing period. The specific gravity of fly ash group aggregates ranges from 2.00 (for 20+ mm sieve size) to 2.17 (for 2–4 mm sieve size). The specific gravity values for fly ash lime group aggregates were 2.10 and 2.28 and for fly ash cement group aggregates were 2.17 and 2.35 for the same sieve sizes intervals. The bulk specific gravity and water absorption values for fly ash group aggregates vary between 1.19-1.29 and 33.9-31.4%, for fly ash lime group aggregates are 1.26-1.33 and 31.6-31.4% and finally for fly ash cement group aggregates are 1.33-1.40 and 29.3-28.8% for the same sieve size intervals, respectively. All three groups of aggregates succeed in the soundness test since the loss of weights is well below 12%.

Ramamurthy and Harikrishnan (2006) investigated the fly ash aggregates properties with different binders such as cement, lime and bentonite. Among the binders, aggregate with bentonite additive has shown lower specific gravity when compared to lime and cement. The 24 hrs water absorption of sintered fly ash aggregate without binders is in the range of 21–22%. Addition of lime reduces the water absorption marginally. However, cement as binder in the production of the fly ash aggregates showed relatively better than lime as a binder in reducing water absorption and it is reported that addition of 20% sodium bentonite resulted in optimal strength and minimum water absorption characteristics.

Influence of fineness of fly ash on the aggregate pelletization process is studied by Manikandan and Ramamurthy (2007) and reported that finer fly ash (fineness of 414 m^2/kg) does not require any binder for achieving maximum pelletization efficiency. However, the use of fly ash with a lower fineness (257 m^2/kg) exhibit lower pelletization efficiency of only 12%. Addition of clay binders (bentonite or kaolinite) improves the pelletization efficiency up to 98%.

Gesoglu et al. (2007) studied effects of fly ash properties on characteristics of cold-bonded fly ash lightweight aggregates and aggregates surface treated with water glass for 30 min. Surface treated fly ash aggregates showed reduction in water absorption as high as 85%. The water glass treatment provided a marked improvement in the aggregate strength.

Priyadharshini et al. (2011) studied the production of light weight aggregates using pelletizer and curing has been done in cold bonding technique. Light weight aggregates are produced using class C fly ash with cement as binder for pelletization. Light weight fly ash aggregate showed 31.8% lower crushing value than the natural aggregate and 26.4% higher impact value and reported that the water absorption of fly ash aggregate is 9 times higher than that of natural gravel.

Gomathi and Sivakumar (2014) investigated the efficiency of the production of the pelletized fly ash aggregates with different binders such as cement, lime and alkali solution and showed that 100% efficiency can be achieved by the use of cement in the pelletization process of fly ash in short duration. The specific gravity of fly ash aggregates with cement binder is 2.12 which is higher than other types of aggregates. The lowest specific gravity is 1.68 for fly ash aggregates without binder and for lime binder it is 1.83. The water absorption of fly ash aggregate ranges from 12% to 77%. The strength of artificial pelletized fly ash aggregates are higher (13.72 MPa) for the mean size of aggregates is 13.53 mm and lower (0.8 MPa) for the mean size of aggregates is maximum 26.68 mm.

Efficiency of pelletization was investigated by Gomathi and Sivakumar (2014a) on the fly ash based aggregates incorporating clay binders such as bentonite and metakaolin with alkali activator (NaOH solution). Authors have reported that the pelletization efficiency and strength of fly ash aggregates increases with the addition of binders. Addition of alkali activator during pelletization of aggregates provided a stable formation pellets as well as the increase in the early strength properties. The water absorption of the fly ash aggregates produced with bentonite binder cured at 100°C oven curing observed lower water absorption (16.39%) as compared to other types.

Researchers even investigated the utilization pyrite ash wastes, low-calcium bottom ash, mining residues, heavy metal sludge, volcano ash etc., with suitable binder such as bentonite, calcium hydroxide, sodium hydroxide and sodium silicate (Turgul et al, 2006; Huang et al, 2007; Geetha and Ramamurthy, 2010; Rafiza et al, 2013) under different curing conditions. It is observed from the reported results that increasing the amount of clay binder increases the strength properties of produced pellets (Tugul et al, 2006; Geetha and Ramamurthy, 2010). Addition of $Ca(OH)_2$ reduces the duration of pelletization process from an average of 7−14 min and reduced the binder dosage for a given pelletization efficiency (Geetha and Ramamurthy, 2010). However, the produced aggregates which is sintered or treated at high temperature will increase the strength properties of aggregates (Huang et al, 2007; Rafiza et al, 2013).

2.2.2.3 Curing regime

Type of curing methods for pellets mainly depends on the raw materials and binders used for the production. Ramamurthy and Harikrishnan (2006) studied on the sintering of fly ash aggregates with different binders such cement, lime and bentonite and reported that the sintered fly ash aggregates observed that the properties of aggregates depend on the type of binder and its dosage. Sintering of fly ash aggregate significantly improves strength and reduction in water absorption when bentonite is added with fly ash. The binders used did not alter the chemical composition, while it influence the microstructure of the aggregate, which results in enhancement in the properties of aggregates.

Kockal and Ozturan (2011) studied on the characterization of sintered light weight aggregates produced by fly ash and different binders such bentonite and glass powder and reported that specific gravity of fly ash aggregates continuously increased with sintering temperature. The use of binder led to a considerable reduction in specific gravity of aggregates sintered at 1200 °C due to expansion and bloating. Water absorption of all aggregates decreased with the increase in temperature. Strength of aggregates with bentonite and glass powder binders at 1100 °C and 1150 °C were much higher than fly ash aggregate and increases with binder content. The highest strength for fly ash aggregates was obtained at 1200 °C whereas the strength of aggregates with binder at 1200 °C was reduced due to bloating effect resulting in large closed pores.

Rafiza et al. (2014) studied the pelletized artificial geopolymer aggregate made from volcano ash and sintered at 500 °C to 800°C and reported that specific gravity of artificial geopolymer aggregate was in the range of 1.7 - 2.0 g/cm^3. The range of water absorption for artificial geopolymer aggregate with volcano ash was 9-16 %. Further, authors reported that the water absorption can be modulated by controlling the expansion sintering temperature.

The characterization of artificial fly ash aggregates produced from a pelletization process with clay binders such as bentonite and metakaolin shows enhanced stability and strength properties. Artificial fly ash aggregates are investigated for different environmental conditions, controlled humidity curing at 40% RH and 35°C, hot air oven curing at two different temperatures 50°C and 100°C and reported that a maximum crushing strength of 17.97 MPa was obtained when aggregate subjected to hot air oven curing at 100°C (Gomathi and Sivakumar 2014).

The detailed investigation carried by the researchers on the effect of curing methods on pelletized aggregates of class C fly ash and reported that the normal water curing greatly improved the aggregate properties with increase in curing duration. However, accelerated curing methods and autoclaving exhibit only a negligible improvement in the properties of aggregates (Gesoglu et al. 2007; Manikandan and Ramamurthy 2008; Priyadharshini et al. 2011).

It is observed from literature that the various factors affecting the pelletization efficiency and properties of produced aggregates are moisture content, process duration, type of binder, amount of binder added and curing regime. Many researchers reported that the moisture content is the major factor that influences size growth of pellets followed by the angle of the pelletizer (Harikrishnan and Ramamurthy, 2006; Baykal and Doven, 2000). However, from the literature it can be observed that the artificial aggregates satisfying the few standards such as strength properties of aggregates and grain size distribution. However, the water absorption of artificial aggregate is very high and predominantly influenced by the raw materials, type and content of binder, pelletization factors and moisture content (Harikrishnan and Ramamurthy, 2006; Geetha and Ramamurthy 2010; Gomathi and Sivakumar 2012).

A review of different raw material used in the pelletization process with suitable binders and along with the hardening method adopted is presented in Table 2.1. It can be noted that in the pelletization process such as speed of pelletizing disc, the angle of pelletizing disc, water content and duration of the pelletization are the responsible factors along with the binder type and curing regime adopted that affect the production process and engineering properties of artificially produced fly ash aggregates.

Table 2.1: Review of literature on the factors in the production of aggregates

Author(s) name (Year)	Raw materials	Binder type	Angle of disc (°)	Speed of disc (RPM)	Duration (minutes)	Moisture content	Hardening method
Meyer (1980)	Iron ore	Bentonite	45 - 48	10 - 14	Continuous	8 - 12%	Sintering
Suzuki et al (1987)	Iron ore	Bentonite	47	10 - 26	20	10 - 30 %	Sintering
Baykal and Doven (2000)	Fly ash	Lime and cement	20 - 50	35 - 55	20	29 - 33 %	Cold bonding
Veverka and Hinkle (2001)	Lime stone	Lignosulfonate, Brewex, Molasses	40 - 60	12 - 37	Continuous	8%	Sintering
Su et al (2004)	Sludge and dust	Cement	45	18	20	12.5 - 13.5%	Cold bonding
Ahmed and Mohamed (2005)	Iron ore	Bentonite, calcium hydroxide	55	17	10	10 - 30 %	Sintering
Harikrishnan and Ramamurthy (2006)	Fly ash	Clay binders	40 & 70	20 & 40	10 and 20	15% & 35%	Sintering
Tugrul et al (2006)	Iron ore	Bentonite, calcium hydroxide and calcium chloride	60	20 - 30	30 - 60	10 - 30 %	Sintering

Author(s) name (Year)	**Raw materials**	**Binder type**	**Angle of disc (°)**	**Speed of disc (RPM)**	**Duration (minutes)**	**Moisture content**	**Hardening method**
Manikandan and Ramamurthy (2007)	Fly ash	Bentonite and kaolinite	35 & 55	35 & 55	8 - 16	23 - 35 %	Sintering
Gesoglu et al. (2007)	Fly ash	Class C Fly ash	43	35	20	23 - 35 %	Cold bonding
Manikandan and Ramamurthy (2008)	Fly ash	Bentonite and kaolinite	55	55	8 - 16	31	Cold bonding
Geetha and Ramamurthy (2010)	Fly ash and Bottom ash	Kaolinite, metakaolin and cement	55	50	8 - 16	25 - 33 %	Cold bonding and Sintering
Kockal and Ozturan (2011)	Fly ash	Bentonite and glass powder	43	45	20	22 - 25 %	Sintering
Gomathi and Sivakumar (2012)	Fly ash	Lime, cement and alkali solution	35 - 50	55	10 - 20	25%	Cold bonding
Colangelo and Cioffi (2013)	Marble Sludge	Cement Kiln Dust, Blast Furnace Slag	50	35 - 55	Continuous	10 - 20 %	Cold bonding
Gul et al 2015	Iron ore	Bentonite	45	40	8 - 16	8 - 12%	Sintering

2.3 ARTIFICIAL AGGREGATES IN THE CONCRETE

Baykal and Doven (2000) investigated on the use of artificial aggregates in the concrete production and reported that the compressive strengths of concrete specimens prepared by artificial aggregates cured 7 and 28 days are almost the same. The compressive strength of those specimens prepared by 7 days cured artificial fly ash aggregates ranges from 17.2MPa to 22.1 MPa and those prepared with 28 days cured aggregates ranges from 17.6MPa to 23.0 MPa. However, authors reported that lime blended fly ash artificial aggregates give better performance in production of lightweight concrete.

Gesoglu et al (2007) studied the properties of fly ash aggregate in lightweight concrete production and reported that the compressive strength of the concretes was increased by increasing crushing strength of the artificial fly ash lightweight aggregates used. The surface treatment with water glass provided a significant increase in the aggregate strength and a reduction in the water absorption. The lightweight concretes made with such light weight aggregates had a compressive strength of as high as 60 MPa.

Priyadharshini et al (2011) investigated the use of fly ash aggregates in the concrete production and reported that the rounded shape of artificial fly ash aggregate gives better workability compared to the angular natural gravel and compressive strength of artificial fly ash aggregate concrete is 48% lesser compared to normal concrete mix.

2.4 DESIGN OF EXPERIMENTS

In experimental investigation, to identify the effects of various factors, which are affecting the results of experiment, requires a series of experiments. Experimental design techniques become extremely important in studies to develop new products and processes in an effective method. In design of experiments, there are different experimental design methods such as full factorial, fractional factorial design, response surface method and central composites are available (Cavazzuti 2013).

Harikrishnan and Ramamurthy (2006) investigated the influence of pelletization process on the properties of the produced fly ash aggregates with statistically designed experiments. The study was carried out with fractional factorial experiments using the concept of Taguchi’s orthogonal array. Authors reported that the moisture content is major parameter whereas the angle is second parameter that influences the size of the pellets and maximum influence on strength of pellet depends on the speed and angle of the pelletizer. The water absorption of the sintered aggregates is significantly governed by speed of the pelletizer followed by the moisture content. Among the interaction of parameters, the angle and moisture content interaction has considerable influence on the size growth and strength of the pellets.

Manikandan and Ramamurthy (2007) investigated the influence of fineness of fly ash in the production of aggregate through pelletization process. The influence of fineness of fly ash was studied by collecting distinctive samples of fly ash from two thermal power plants. The significant influencing factors in the production of fly ash aggregates through pelletization process were statistically determined using fractional factorial design. Fractional factorial design of 2^4 with eight run and 2^5 with sixteen run for fly ash with fineness of 414 m^2/kg and 257 m^2/kg, respectively were used. Authors reported that introducing the clay binders like bentonite and kaolinite in the pelletization process, enhances the pelletization efficiency of coarser fly ash and also the amount of binder content and moisture content varies with the type of binder used. Statistically designed experiments with fractional factorial design revealed that only angle and speed significantly influence the pelletization of fly ash with fineness of 414 m^2/kg. However, all the factors significantly influenced the pelletization process for fly ash with fineness of 257 m^2/kg.

Geetha and Ramamurthy (2011) studied the properties of the sintered low calcium bottom ash aggregates with clay binders using central composite design of response surface methodology with five factors at five levels. The factors involved in the process are moisture content, binder, $Ca(OH)_2$ dosage, sintering temperature and duration. Authors reported that the engineering properties of produced aggregate mainly dependent on sintering temperature, binding ability of binders and the internal pore structure of the material. The properties of aggregates depended on the type of binder used and it was observed that an increase in binder dosage and sintering temperature resulted in aggregates with higher 10% fines value and low water absorption.

Vasugi and Ramamurthy (2014) investigated the design parameters influencing the manufacturing and properties of cold bonded pond ash aggregates. Central composite design of response surface methodology a statistical technique was adopted to design the experimental runs with different parameters such moisture content, binder dosage, pelletization and strength enhancer dosage with varied range. The engineering properties of produced aggregate such as bulk density, water absorption and the strength property of aggregate characterised through 10% fines value have been determined for the influence of parameters. Authors reported that the dosage of binder, strength and pelletization enhancing admixture improved the engineering properties of aggregate.

2.5 GEOPOLYMERISATION THEORY

In 1950s, Glukhovsky was the first to discover the material alkali activated aluminosilicate which enables this as binding material to be cement like construction material. However, in 1979, Davidovits introduced the term “geopolymer” for his binder, which consisted of alkalis mixed with a burnt mixture of lime stone, kaolinite and dolomite did the research on the alkali activated aluminosilicate materials. According to Davidovits, geopolymer is a new type of binder which is distinct from alkali activated aluminosilicate, most of the researchers preferred the name “geopolymer” to name all the alkali activated aluminosilicate binders.

2.5.1 Geopolymerisation process

Geopolymerisation is a geo-synthesis – process involves a substantially fast chemical reaction with integration of minerals. The exposure of aluminosilicate materials such as fly ash, blast furnace slag, metakaolin, etc., to high-alkaline environment gives rise to geopolymerisation and leads to the formation of a geopolymers. These types of polymers are inorganic polymers, where chain structures formed on a backbone of Al and Si ions. The chemical composition of this geopolymer material is similar to natural zeolitic materials, but they have amorphous microstructure instead of crystalline. The polymerisation process involves a substantially fast chemical reaction under highly alkaline condition on Si-Al minerals that result in a three dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds, as follows (Davidovits 1999; Nikolic et al. 2015):

$$M_n\,[-(SiO_2)_z\text{-}AlO_2]_n\,.\,wH_2O \qquad (\textit{Eq}\ 2.1)$$

where; M is the alkaline element (cation such as potassium, sodium or calcium); z is 1, 2, or 3 and n is the degree of polycondensation or polymerisation.

Duxson et al (2007) proposed conceptual model for geopolymerisation mechanism in five stages as follows: (i) dissolution, (ii) speciation equilibrium, (iii) gelation, (iv) reorganization, and (v) polymerization and hardening (Figure 2.3).

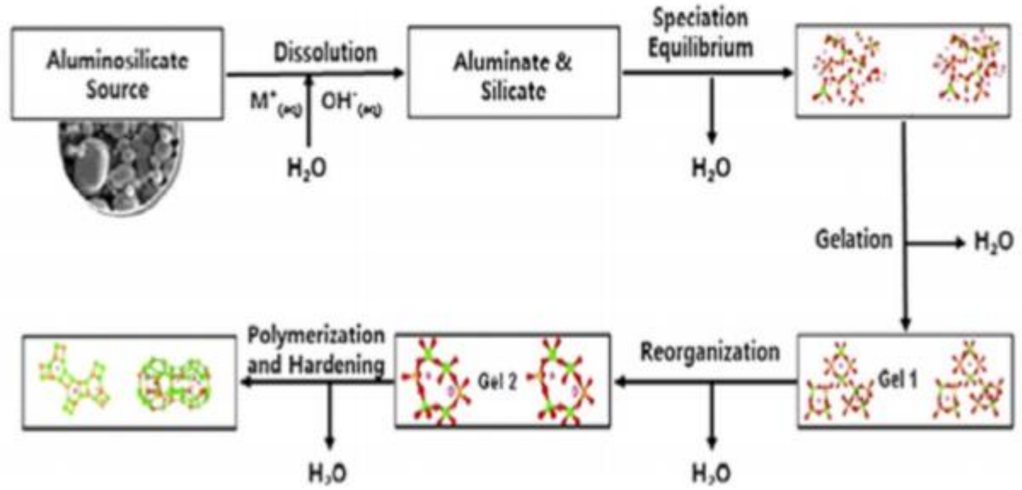
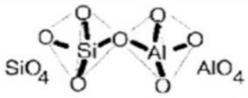
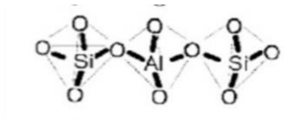
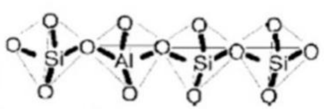
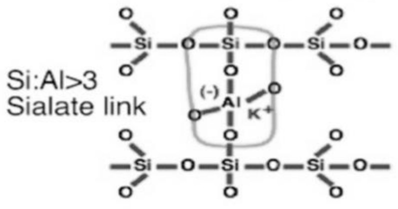


Figure 2.3: Conceptual model for geopolymerisation (Source: Duxson et al. 2007)

A geopolymer can take one of the three basic forms as presented in the Table 2.2. Davidovits (1999) proposed the possible applications of the geopolymer materials depending on the molar ratio of Si to Al, as given in Figure 2.4.

Table 2.2: The geopolymer terminology (Source: Davidovits., 1999)

Monomers	Polymers	Si:Al ratio	Formation
Sialate (silicon-oxo-aluminate)	Poly (sialate)	1	
Sialate-siloxo (silicon-oxo-aluminate; silicon-oxo)	Poly (sialate-siloxo)	2	
Sialate-disiloxo (silicon-oxo-aluminate; silicon-oxo; silicon-oxo)	Poly (sialate-disiloxo)	3	
Sialate (silicon-oxo-aluminate)	Sialate link	>3	

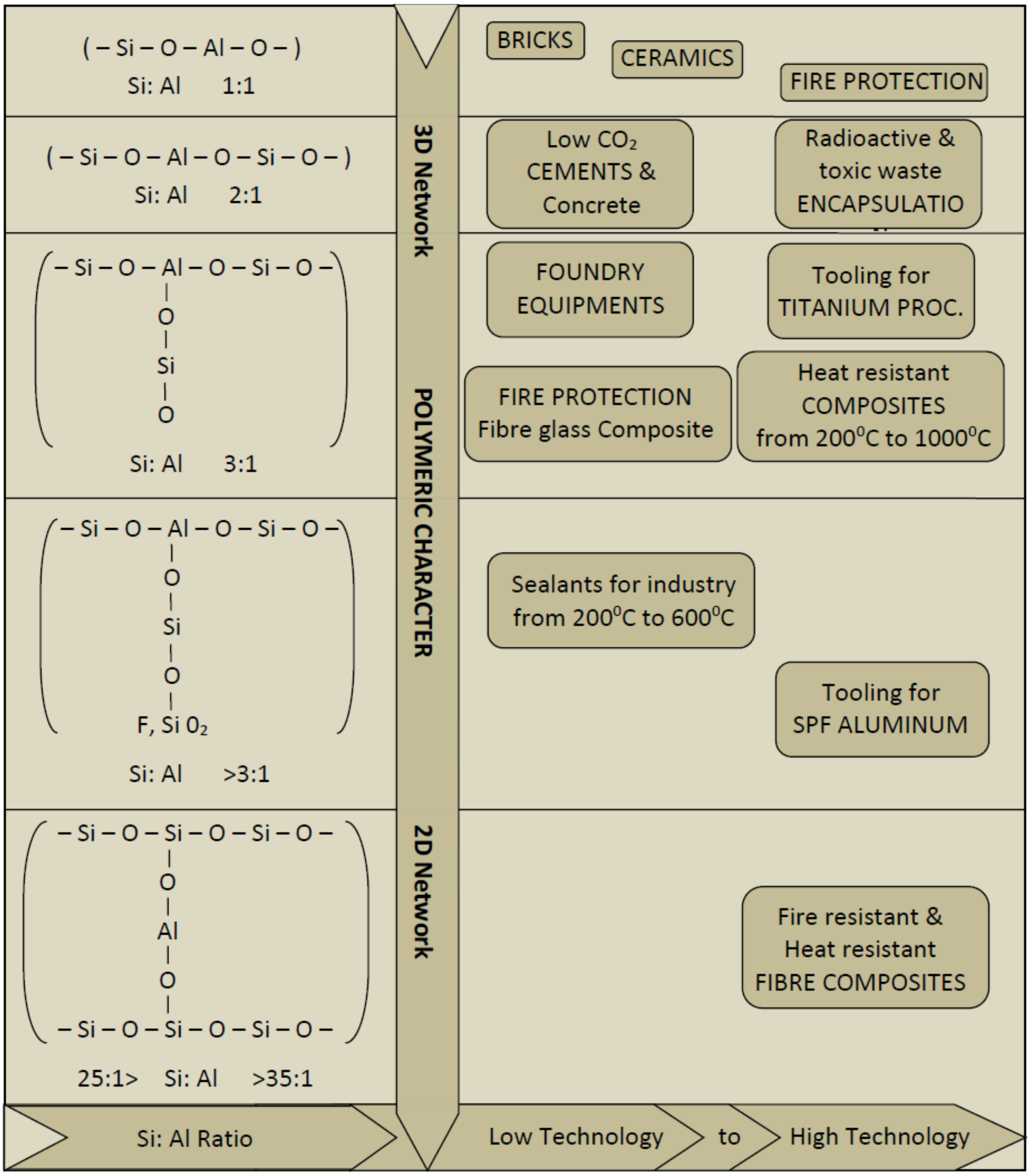


Figure 2.4: Applications of Geopolymer Material (Source: Davidovits 1994)

2.5.2 Factors affecting the properties of fly ash based geopolymers

In geopolymerisation, alkali activator or chemical activator solution plays a vital role in the initiation of surface hydrolysis of the particles present in the aluminosilicate materials as the raw material. Generally, a strong alkaline medium is essential to increase the surface hydrolysis of the fly ash. The effect of the process is greatly affected by the number of factors such as type of alkali activator, concentration of the alkali solution, binder to alkali ratio, curing regime, etc (Rangan et al. 2005 and Ridtirud et al. 2011).

2.5.2.1 Type of chemical activator

In geopolymerisation mechanism, strong alkalis are required to activate the silica and aluminium present in the fly ash such as NaOH, Na_2SO_4, Na_2SiO_3, Na_2CO_3, K_2CO_3, KOH, K_2SO_4, $Ca(OH)_2$ or a little amount of cement clinker or combination of the alkali solution can be used to in the process of geopolymerisation (Leong et al 2016; de Vargas et al 2014; Komljenovic et al 2010; Khale and Rubina 2007). According to researchers the type and concentration of alkali activator is the most important parameter in geopolymerisation process.

Whereas, Phoo-ngernkham et al. (2015) used sodium hydroxide and sodium silicate solutions for fly ash based geopolymer and observed that the combine use of solutions results in the good mechanical properties due the formation of crystalline calcium silicate hydrate which co-existed with amorphous gel. However, Leong et al investigated the combination of alkali hydroxides (i.e. NaOH, KOH or $Ca(OH)_2$) and sodium silicate in their research. Experimental results show that the compressive strength and workability of $Ca(OH)_2$-based geopolymer were the weakest in comparison to NaOH and KOH based geopolymers. The lower activation potential of KOH compared to NaOH was due to the difference in ionic diameter difference between potassium and sodium.

2.5.2.2 Concentration of alkali activators

Many researchers reported that the dissolution of Si and Al species during the synthesis of geopolymer is very much dependent on the concentration of alkali solution (Singh et al 2005; Khale and Rubina 2007; Rattanasak and Prinya 2009). A study by Khale and Rubina (2007) on the influence of NaOH concentration in geopolymerisation mechanism on the release of silicate and aluminate monomers from aluminosilicate source and reported that as the concentration increases the release of silicate and aluminate monomers from aluminosilicate source also increases. Rattanasak and Prinya (2009) studied the effect of leaching of fly ash mixed with NaOH solution. Leaching of SiO_2 and Al_2O_3 were investigated by mixing fly ash with different concentration of

NaOH solution for different time intervals and reported that when fly ash comes in contact with NaOH solution, leaching of Si, Al and other minor ions started. The optimum leaching time depends on the concentration of NaOH solution. The leaching time of 5 to 10 minutes is reported to be sufficient as further increase the leaching time of 20 to 30 min did not increase in the concentration of Si^{4+} and Al^{3+} ions.

Al-Bakri et al (2011) investigated the effect of the NaOH concentration on the compressive strength of fly ash based geopolymer paste. The different concentrations of NaOH were used and while other fly ash to alkali activator and sodium silicate to sodium hydroxide ratios maintained constant. Based on the compressive strength which is cured at 60°C for 24 hrs and tested for 7 days resulted that the optimum NaOH concentration of 12M is contributed high compressive strength of 94.59MPa. Whereas, Somna et al. (2011) investigated the influence of NaOH concentration on the ground fly ash geopolymer cured at ambient temperature. NaOH concentrations of 4.5 – 16.5 M (Molar) were used as an alkali activator and reported that increase in NaOH concentration from 4.5 to 14.0 M increased the strength of fly ash based geopolymer pastes. Microstructure studies showed that at higher concentrations of NaOH (12.0–14.0 M), new crystalline products of sodium aluminosilicate were created. The compressive strengths of geopolymer paste at 28 days of 20.0–23.0 MPa are obtained with the NaOH concentrations of 9.5–14.0 M. however, increasing the NaOH concentration beyond this point resulted in a decrease in the strength of the paste due to early precipitation of aluminosilicate products.

Further researchers, investigated the effect of the NaOH concentration in the fly ash based geopolymer mortar or concrete (Hardjito et al 2008; Mishra et al 2008; Ridtirud et al 2011; Patankar et al 2014; Gorhan and Kurklu 2014; Atis et al 2015). Whereas, compressive strength of geopolymer mortar or concrete significantly affect with increase in concentration of NaOH solution from 3M to 16M. From Figure 2.5 and Table 2.3 it is clear that the concentration of alkali activator increases the mechanical properties of the geopolymers up to certain limit, further increase in the concentration will decrease in the mechanical properties. However, the workability of the geopolymers shows that the flow increases with increase in concentration of sodium hydroxide solution and that the density of geopolymers does not depend on concentration of sodium hydroxide solution (Patankar et al 2014).

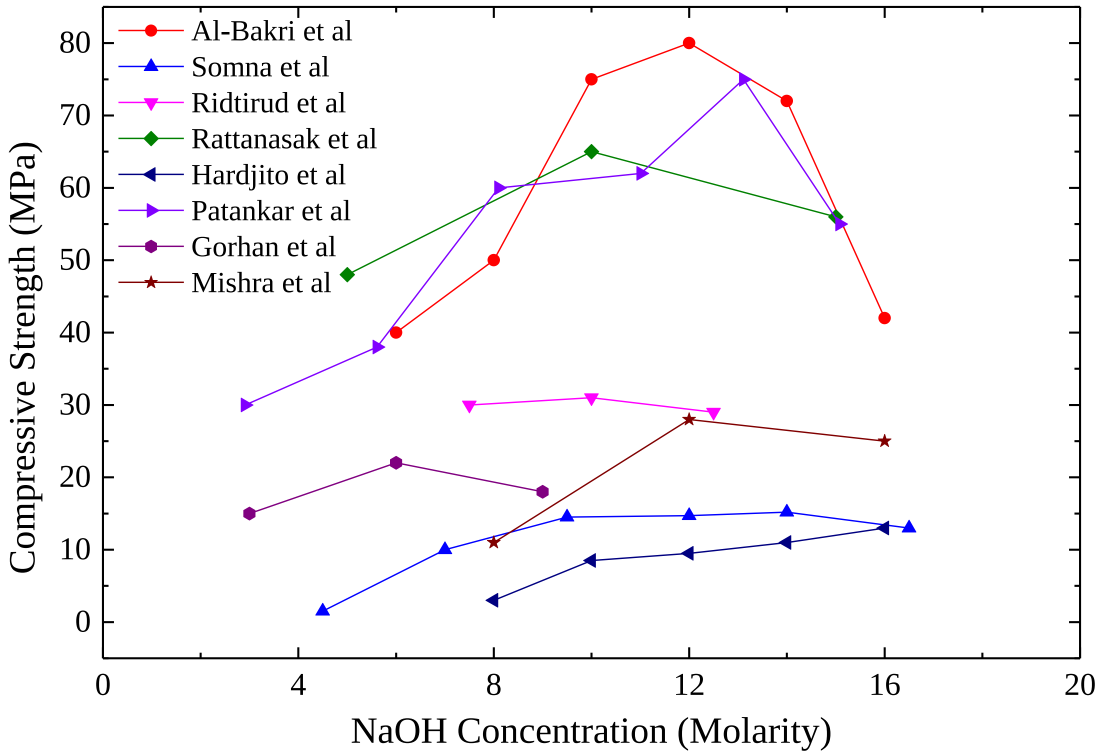


Figure 2.5: Effect of alkali concentration on the compressive strength of the fly ash based geopolymers

Table 2.3: Effect of alkali solution concentration on the properties of fly ash based geopolymers

Sl. No.	Author(s) name (Year)	Type of geopolymer (Paste/ mortar/ concrete)	Binder/Solution or F/A ratio or A/F ratio or w/b ratio	NaOH / KOH / $Ca(OH)_2$ (M)	AS/AH Ratio	Curing Regime	Remarks
1	Somna et al. (2011)	Paste	A/F: 0.3	4.5 - 16.5	-	Room Temperature	Optimal NaOH concentration is 14M
2	Al-Bakri et al. (2011)	Paste	F/A : 2.5	6 - 16	2.5	70 °C for 24 hrs	Optimal NaOH concentration is 12M
3	Hardjito et al. (2008)	Mortar	A/F: 0.35	8 - 16	2.5	65 °C for 24 hrs	Concentration of the NaOH solution proportionate to the compressive strength
4	Rattanasak et al. (2009)	Mortar	F/A : 1.5	5 - 15	1.5	65 °C for 48 hrs	Optimal NaOH concentration is 10M
5	Ridtirud et al. (2011)	Mortar	A/F: 0.6	7.5 - 12.5	0.67	40 °C for 24 hrs	Optimal NaOH concentration is 10M.
6	Gorhan and Kurklu (2014)	Mortar	A/F: 1	3 - 9	2	85 °C for 24 hrs	Optimal NaOH concentration is 6M
7	Patankar et al. (2014)	Mortar	A/F: 0.45	2.91 - 15.08	1	90 °C for 24 hrs	Optimal NaOH concentration is 11.01M
8	Mishra et al. (2008)	Concrete	A/F: 0.5	8 - 16	0.6	60 °C for 48 hrs	Optimal NaOH concentration is 12M

2.5.2.3 Alkali silicate to alkali hydroxide ratio

It is reported that the use of alkali silicate (AS) or alkali hydroxide (AH) separately resulted in the lower activation of the aluminosilicate materials. According to Phoongernkham et al (2015) use of both sodium hydroxide and sodium silicate solutions in combination for the production fly ash based geopolymer revelled that the solutions resulted in the formation of crystalline structures which co-existed with amorphous gel. However, the use of sodium silicate solution alone resulted mainly in the amorphous products. The use of sodium hydroxide solution or sodium silicate solution alone gave low strengths when cured at ambient temperature. It is recommended that for better strength development the use of combination of sodium hydroxide and sodium silicate solutions need to be chosen.

Mustafa et al (2012) investigated the influence of Na_2SiO_3 to NaOH ratios on the compressive strength of fly ash based geopolymer paste with different ratios (0.5, 1, 1.5, 2, 2.5 and 3) and NaOH concentration (10M) and fly ash to alkali activator ratio (2.5) maintained constant throughout the experimental work. Based on the compressive strength which is cured at 60°C for 24 hrs and tested on 7 days resulted that the optimum sodium silicate to sodium hydroxide ratio of 2.5 contributed high compressive strength of 57MPa. The compressive strength of geopolymer paste for a sodium silicate to sodium hydroxide ratio of 3.0 is reported to be low, due to excess hydroxyl ion concentration in the mixture.

However, detailed investigation by many researchers was carried out on the fly ash based geopolymers with varying alkali silicate to alkali hydroxide ratio on compressive strength and reported that the alkali silicate to alkali hydroxide ratio has significant effect on fly ash based geopolymers. The increase in the alkali silicate to alkali hydroxide ratio resulted in the increase of M^+ (alkalie ions) content in the mixture, which is important to the formation of geopolymers as it acts as charge balancing ions (Hardjito et al 2008; Ridtirud et al 2011; Morsy et al 2014; Risdanareni and Ekaputri 2015; Leong et al 2016). Further, authors reported that fly ash based geopolymer with a high alkali silicate to alkali hydroxide of 3.0 gives low drying shrinkage as compared to other ratios with the alkali silicate to alkali hydroxide of 0.3 - 1.5. This can be attributed to high silicate content the reaction or condensation is fairly quick (Ridtirud et al 2011).

Research reported that compressive strength of the fly ash geopolymer mortars or concrete increases with the increase alkali silicate to alkali hydroxide ratio from 0.5 to 2.5. From Figure 2.6 and Table 2.4 it is clear that the alkali silicate to alkali hydroxide ratio of alkali activator increases the mechanical properties of the geopolymers up to certain limit, further increase in the concentration will decrease in the mechanical properties. Whereas, Leong et al (2016) investigated the fly ash based geopolymer mortar and observed that the workability decreases with the increase in Na_2SiO_3/NaOH ratio and commercial grade Na_2SiO_3 shows considerable increase in the workability of fly ash based geopolymers when compared to industrial grade Na_2SiO_3. The compressive strength of geopolymer using commercial grade Na_2SiO_3 is significantly higher than those using industrial grade Na_2SiO_3 and it increases with the increase of Na_2SiO_3/NaOH ratio.

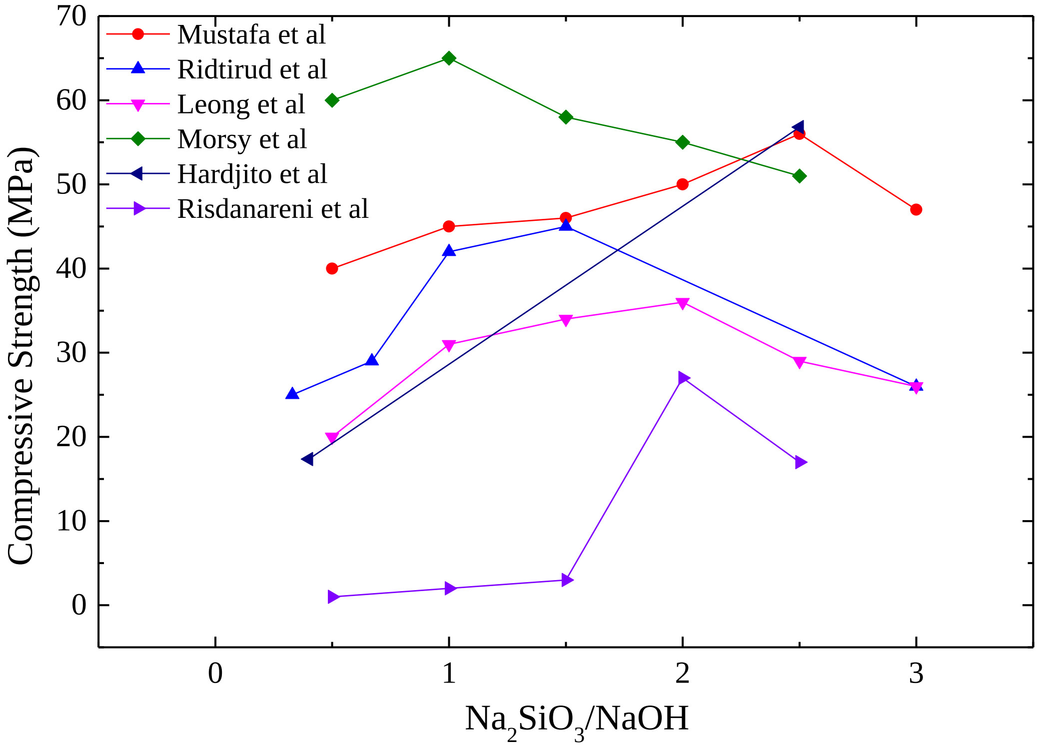


Figure 2.6: Effect of alkali silicate to alkali hydroxide ratio on the compressive strength of fly ash based geopolymers

Table 2.4: Effect of the alkali silicate to alkali hydroxide ratio on properties of geopolymers

Sl. No.	Author(s) name (Year)	Type of geopolymer (Paste/ mortar/ concrete)	Binder/Solution or F/A ratio or A/F ratio or w/b ratio	NaOH / KOH / $Ca(OH)_2$ (M)	AS/AH Ratio	Curing Regime	Remarks
1	Mustafa et al. (2012)	Paste	F/A : 2.5	10M	0.5 - 2.5	70 °C for 24 hrs	Optimal ratio is 2.5
2	Ridtirud et al. (2011)	Mortar	A/F: 0.6	10M	0.33 - 3.0	40 °C for 24 hrs	Optimal ratio is 1.5
3	Morsy et al. (2014)	Mortar	F/A : 2.5	10 M	0.5 - 2.5	80 °C for 24 hrs	Optimal ratio is 1
4	Leong et al. (2016)	Mortar	A/F: 0.4	8M	0.5 - 3.0	60 °C for 24 hrs	Optimal ratio is 2. Workability decreases with the increase in Na_2SiO_3/NaOH ratio
5	Hardjito et al. (2004)	Concrete	A/F: 0.35	8M	0.4 - 2.5	60 °C for 24 hrs	Ratio is proportionate to Compressive strength
6	Risdanareni et al. (2015)	Concrete	A/F: 0.33	8M & 10M	0.5 - 2.5	Room temperature	Optimal ratio is 2 for 8M and proportionate for 10M

Although concentration of activator (in terms of NaOH molarity) in fly ash based geopolymer binder is an important parameter as reported by many researchers (Hardjito and Rangan 2005, Fernandez-Jimenez and Palomo 2005, Al-Bakri et al 2011), the effect of Na^+ ion concentration in the mixes on the engineering properties of alkali activated binder especially when the activator contains both sodium silicate and sodium hydroxide cannot be recognized. Therefore, the dosage of activator in terms of the mass ratio of total Na_2O in the activator solution to fly ash was adopted by many researchers in their experimental investigations as the main indicator of the Na concentration in the mixes.

The dosage of activator in terms of Na_2O percentage is defined as the ratio of the Na_2O content of the alkaline activator to the mass of the binder. From literature, it is found that Na_2O have significant influence on the strength of alkali activated geopolymer paste/mortar/concrete. Study by Hardjito and Rangan (2005), on the fly ash based geopolymer concrete having the Na_2O dosage varies from 4.5 to 6.5 % showed increase in the compressive strength of the concrete with increase in the Na_2O dosage and high strength of 89 MPa was achieved with heat curing of samples at 90 °C for 24h. However, Fernandez-Jimenez and Palomo (2005) investigated on the alkali activated fly ashes using sodium hydroxide in terms Na_2O dosage varies from 6.5 to 13.67% in the production of mortar using sodium hydroxide showed the highest compressive strength of 70.4 MPa was achieved with heat curing of samples at 85 °C for 20 h.

2.5.2.4 Alkali activator to binder ratio

The properties of fresh mixtures, mainly their flowability or workability and setting rate are very significant for the practical application of cementitious materials. The alkali activator to binder ratio is the important factor to understand the behaviour of the geopolymerisation process, fresh and harden properties of the geopolymers. So, fresh properties of the geopolymer mixtures such as workability and setting, which is strongly depends on the quantity of water added and alkali content in the solution. This quantity of water added or water/binder ratio significantly influences the workability of the mixtures, their setting and also the engineering properties of the hardened products. Water to binder ratio is important in geopolymer mixtures which act as the water to cement ratio in the Portland cement mixtures.

According to the researchers, binder to alkali activator ratio appeared to be the most critical parameter in development of the geopolymers (Kong et al 2007; Patankar et al 2014; Leong et al 2016). From literature, it can be observed from experimental investigation by different researchers showed that fly ash based geopolymers having different binder to alkali activator or solids to liquids ratio with other parameters has constant and reported that workability increases with the increase of alkali to binder ratio. Leong et al (2016) investigated the influence of alkaline activator to fly ash ratio on the workability of geopolymer mortar by varying from 0.3 to 0.6 and reported that workability of the geopolymer mortar increases with increase of alkaline activator to fly ash ratio. It is reported that at alkali activator to binder ratio of 0.3 the workability cannot be measured due to collapse of mixture instead of flowing when it is subjected to flow table test. The highest workability (i.e., approx. 250 mm) is observed at alkali activator to fly ash ratio of 0.6. It is observed that, as the alkaline activator to fly ash ratio increases the water content in mix increases and shows good workability. But the rate of gain of flow is not very significant as solution to binder ratio increases. The fly ash based geopolymers achieved significantly higher solids to liquid ratios due to mainly attributed to the spherical shapes and fineness of fly ash particles, which greatly increases workability.

Researchers reported that fly ash based geopolymers generally take a long time (>24 hours) to set due to its slow rate of chemical reaction in its molecular level (Deb and Sarker 2016; Saha and Rajasekaran 2017). It is concluded that, the increase of solid to liquid ratio results in reduction of setting time. This can be associated to less water available as the solid to liquid ratio increased and result in faster reaction of gel phase and more rapidly bonding of the geopolymeric framework.

The ratio of binder to alkali activator affects several critical properties of the geopolymers. However, overall strength is significantly affected by this variable. Many researchers studied the effect of the binder to alkali activator ratio, which varied from 1 to 5 and test results on the compressive strength of geopolymer paste revealed that optimum range from 2 to 4 depending upon the properties of raw materials, chemical composition of alkali activator and water content in the alkali activator has used in their study (Abdullah et al 2011; Rahmiati et al 2014; Rahim et al 2014). Effect of binder to alkali activator solution ratio on compressive strength of geopolymer paste is compiled and presented in the Figure 2.7. The details are given in the Table 2.5.

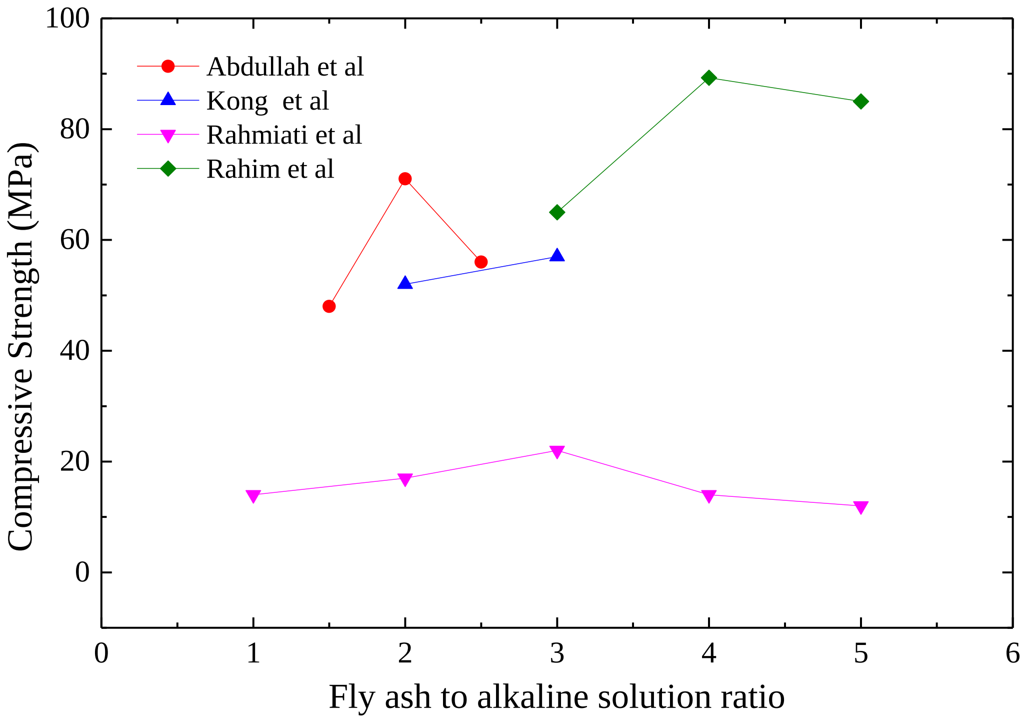


Figure 2.7: Effect of binder to alkaline activator solution ratio on compressive strength of geopolymer paste

Further, many researchers investigated the effect of alkaline activator to binder ratio on geopolymer mortar and concrete (Hardjito et al 2008; Patankar et al 2011; Ridtirud et al 2011; Yellaiah et al 2014; Shinde 2015; Leong et al 2016). Based on the compressive strength of geopolymer mortars, strength increases with the increase in liquid to binder ratios. The high liquid to fly ash ratio contributes to the high porosity of the hardened geopolymer and causes a decline in strength. However, it is observed from the experimental test results in literature that optimum range of alkali activator to binder ratio ranges from 0.3 to 0.5. The complied details on the effect of alkali activator to binder ratio on compressive strength of geopolymers are given in Table 2.5 and also is presented in the Figure 2.8.

Table 2.5: Effect of binder to alkali solution ratio on the compressive strength of fly ash based geopolymers

Sl. No.	Author Name	Type of geopolymer (Paste/ mortar/ concrete)	Binder/Solution or F/A ratio or A/F ratio or w/b ratio	NaOH / KOH / $Ca(OH)_2$ (M)	AS/AH Ratio	Curing Regime	Remarks
1	Kong et al. (2007)	Paste	F/A : 2.0 - 3.0	7	2	80°for 24 hrs	Optimal Ratio is 3
2	Abdullah et al. (2011)	Paste	F/A : 1.5 - 2.5	12	2.5	70 °C for 24 hrs	Optimal Ratio is 2
4	Rahmiati et al. (2014)	Paste	F/A : 1.0 - 5.0	4.5	-	Room temp and 60°C	Optimum solid to liquid ratio of 3 using KOH only.
3	Rahim et al. (2014)	Paste	F/A : 3.0 - 5.0	10	-	60 °C for 24 hrs	Optimum at solid/liquid ratio is 4 using NaOH only.
5	Hardjito et al. (2008)	Mortar	A/F: 0.35-0.45	10	2.5	65 °C for 24 hrs	Optimal Ratio is 0.40
6	Ridtirud et al. (2011)	Mortar	A/F: 0.4-0.7	10	0.67	40 °C for 24 hrs	The strength decreases with the increase in liquid-to-fly ash ratios from 0.4 to 0.7.
7	Patankar et al. (2014)	Mortar	A/F: 0.35-0.45	11.01	1	30 - 120 °C for 24 hrs	The compressive strength increases with the increase in solution-to-fly ash ratio.
8	Yellaiah et al. (2014)	Mortar	A/F: 0.3-0.4	14	2	60 °C for 24 hrs	The compressive strength increases with the increase alkaline activator to fly ash ratio from 0.3 to 0.4.

Sl. No.	Author Name	Type of geopolymer (Paste/ mortar/ concrete)	Binder/Solution or F/A ratio or A/F ratio or w/b ratio	NaOH / KOH / $Ca(OH)_2$ (M)	AS/AH Ratio	Curing Regime	Remarks
9	Shinde (2015)	Mortar	A/F: 0.2-0.8	14	1.5	80 °C for 24 hrs	The ultimate compressive strength (i.e., 38.65 MPa) is reported at the solution to fly ash ratio of 0.5 with rest period 4 days.
10	Leong et al. (2016)	Mortar	A/F: 0.35-0.45	8	2.5	Room temp and 60°C	Optimal Ratio is 0.40, regardless of the type of alkali activators and curing conditions
11	Thakur and Somnath (2009)	Paste	w/b: 0.157-0.366	-	-	85 °C for 48 hrs	The concentration of solution expressed in terms of alkali and silica content. Based on the compressive strength, the optimum w/b is 0.3.
12	Ghosh and Partha (2012)	Paste	w/b: 0.225-0.35	-	-	85 °C for 48 hrs	The concentration of solution expressed in terms of alkali and silica content. Based on the compressive strength, the optimum w/b is 0.3.
13	Leong et al. (2016)	Mortar	w/b: 0.4-0.7	8	0.5	60 °C for 24 hrs	Optimum w/b ratio is 0.4.
14	Rangan et al. (2005)	Concrete	w/b: 0.16-0.24	8	2.5	30 - 90 °C for 48 hrs	The compressive strength decreases as the w/b ratio by mass increases.

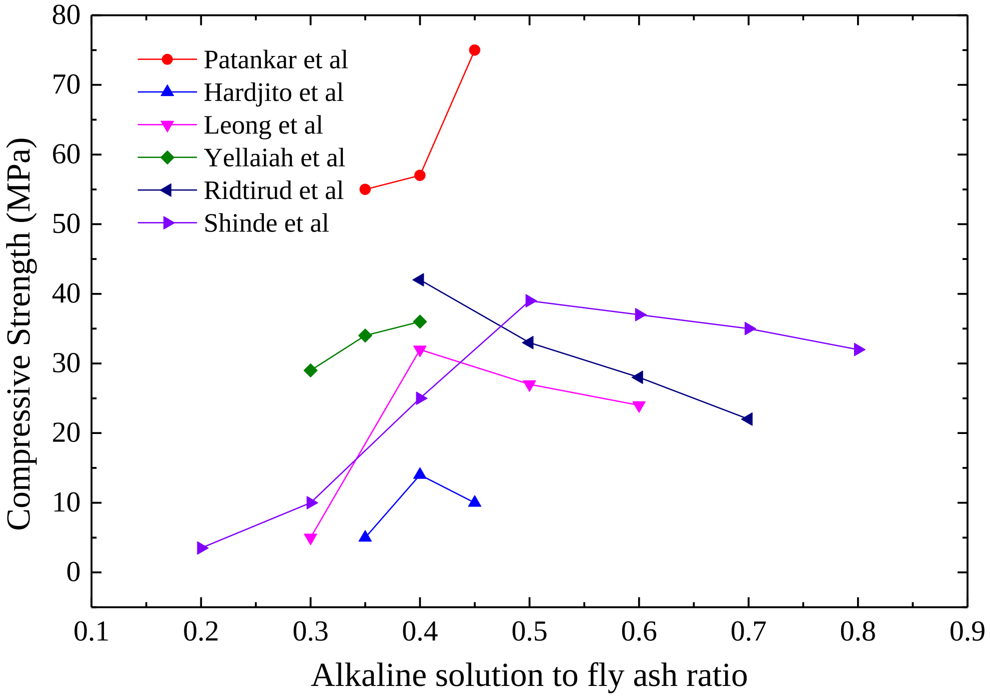


Figure 2.8: Effect of binder to alkaline activator solution ratio on compressive strength of geopolymers

It is clear from literature that water content in the mix plays a vital role in synthesis of geopolymeric material and also it acts as the medium for polymerisation of Al and Si precursors. The effect of water content expressed as water to geopolymer binder (W/B) (geopolymer binder is sum total of mass of fly ash + mass of solids in activating solution). This effect is investigated by many researchers in their study and it is reported that compressive strength of geopolymer paste more or less increased as the water to binder ratio was increased to certain level. Further increase in the water to binder ratio beyond the point it prevents the geopolymerisation process from reaching super saturation, thus the dissolution of precursors is likely to be prolonged resulting in slow gel formation and reduction in strength (Rangan et al 2005; Thakur and Somanth 2009; Ghosh and Partha 2012). The compiled results on the effect of water to binder ratio on compressive strength of geopolymer with details are given in the Table 2.5 and also presented in the Figure 2.9. This test trend is similar to the well-known effect of water to cement ratio on the compressive strength of ordinary Portland cement concrete.

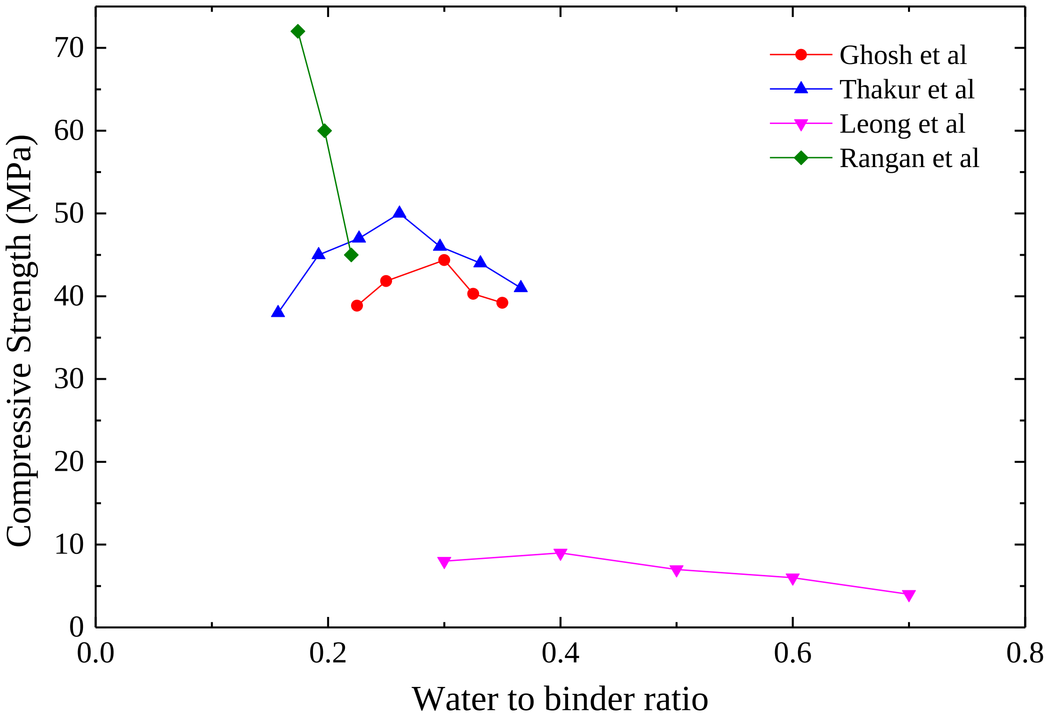


Figure 2.9: Effect of water to binder ratio on compressive strength of geopolymers

Whereas other properties of the fly ash based geopolymers show excellent resistance such as shrinkage, which suffers very little dry shrinkage (Rangan et al 2005), the most important factor affecting compressive strength in geopolymers is pH, with a range of 13–14 being the most suitable for development of good mechanical strength (Thokchom et al 2010; Khale and Rubina 2007; Patankar et al 2014). An increase of alkaline activator concentration directly raises the pH and consequently enhances the degree of reaction. Further the investigation by Patankar et al on alkalinity of geopolymer mortar reported that maximum pH value of geopolymer mortar is 10.92 which is less than that of conventional cement mortar (pH = 11.3–11.6). That means there is less chance of alkali-aggregate reaction even though highly alkaline solution is used for the preparation of geopolymer mortar.

2.5.2.5 Curing regime

It is reported in the literature that heating is necessary for the fly ash based geopolymers (Rangan et al 2005; Thakur and Somanth 2009; Ghosh and Partha 2012; Yellaiah et al 2014; Shinde 2015; Leong et al 2016). Many researchers are reported that the optimum temperature of 60 °C to 90 °C for 24 hrs. The data was compiled and the effect of the heat curing with respect to alkaline solution to fly ash ratio or water to binder ratio on the geopolymer is presented in Figure 2.10.

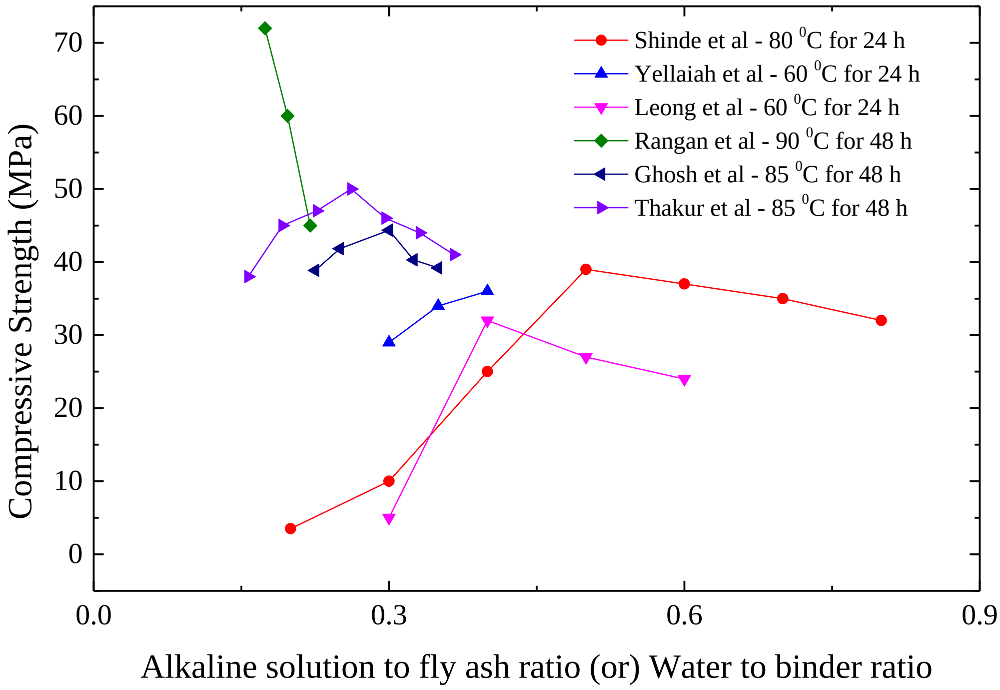


Figure 2.10: Effect of alkali solution to binder (or) water to binder ratio on compressive strength of fly ash based geopolymers

2.6 CRITICAL REVIEW

Effective utilization of fly ash is very much essential to reduce the environmental problems. After having an extensive literature survey, it is observed that these huge quantities of generated waste can be accommodated in the construction sector if planned systematically. The need of the hour for the concrete and cement technologists is relatively a lower carbon foot prints. This can only be achieved through the process of geopolymerisation. It is understood from the literature that the process of geopolymerisation can completely eliminate the usage of cement in the concrete.

Considering the requirements of the concrete manufacturing industry, it is observed that mining of aggregate, which is an important ingredient to concrete and occupies around 70% by its volume, pose a great challenge to the depletion of natural resources. Most of the regulatory authorities have levied additional taxes for the mining of the aggregates or in some places it is completely banned.

Production of artificial aggregates with fly ash, bottom ash, volcano ash etc., is found in literature. The produced artificial aggregates should satisfy minimum acceptance criteria as per the Bureau of Indian Standards (BIS). Some of the aggregates are meeting the strength criteria successfully; however, the water absorption is not satisfied. On the other hand, geopolymers produced from such waste achieves strength parameters. However, geopolymer binder in utilizing for the production of fly ash aggregates is not tried so far. If it is produced successfully, it will be a boon for construction industry, large scale fly ash would be utilized and this will become a major contribution factor for reducing pollution.

The outcome of this research investigation will give enough information about utilization of fly ash admixed with suitable amount of alkaline solution in production of fly ash based coarse aggregate. These artificial aggregates which will be utilized partial or full replacement to natural aggregates in the production of concrete. All the ingredients used in this investigation are the wastes or the by-products generated from different industries, which ultimately helps the disposal problems and environmental hazards.

2.7 OBJECTIVES

The objectives of this research work are:

- To investigate the effects and suitability of different dosages of alkaline solution and type of curing regime on the engineering properties of fly ash based coarse aggregates.
- To produce fly ash based coarse aggregates satisfying the engineering properties of natural coarse aggregates.
- To evaluate the comparative performance of concrete prepared with different proportions of fly ash based coarse aggregate to that of concrete mixed with natural coarse aggregates.

CHAPTER – 3

MATERIALS AND METHODS

3.1 GENERAL

In this chapter, the details of the various materials used in this study are discussed and the tests conducted on these materials along with the results are presented. Further, the methodology adopted for testing the effect of the parameters on the production of fly ash based coarse aggregates and standard methods of characterization of the aggregates are discussed in this chapter. Similarly, methodology adopted for concrete production are discussed along with the tests conducted on the concrete to check its performance.

3.2 RAW MATERIALS FOR AGGREGATE PRODUCTION

3.2.1 Fly ash

Fly ash was collected from Udupi thermal power plant, Karnataka, India. The physical properties of the fly ash were analysed and it is presented in Table 3.1. Further, chemical composition was determined through X-Ray Fluorescence (XRF) test at IIT Bombay and details are presented in Table 3.1. It is classified as class F type as per IS 3812 (part 1) – 2013 classification. The fineness of the fly ash was assessed using Blaine's air permeability apparatus as per the guidelines prescribed in IS 1727 – 1967. The SEM photomicrographs of the fly ash are provided in the Figure 3.1. The particle size distribution of the fly ash was analysed through standard IS sieves from 300 µm to 10 µm and same is presented in the Figure 3.2.

Table 3.1: Physical properties and chemical composition of fly ash

Parameters	Fly ash
Specific gravity	2.2
Blaine's fineness (m^2/kg)	260.3
Chemical composition (%)	
SiO_2	60.65
Al_2O_3	28.62

Fe_2O_3	3.95
CaO	1.70
MgO	1.84
SO_3	1.26
Na_2O	1.11
K_2O	0.11

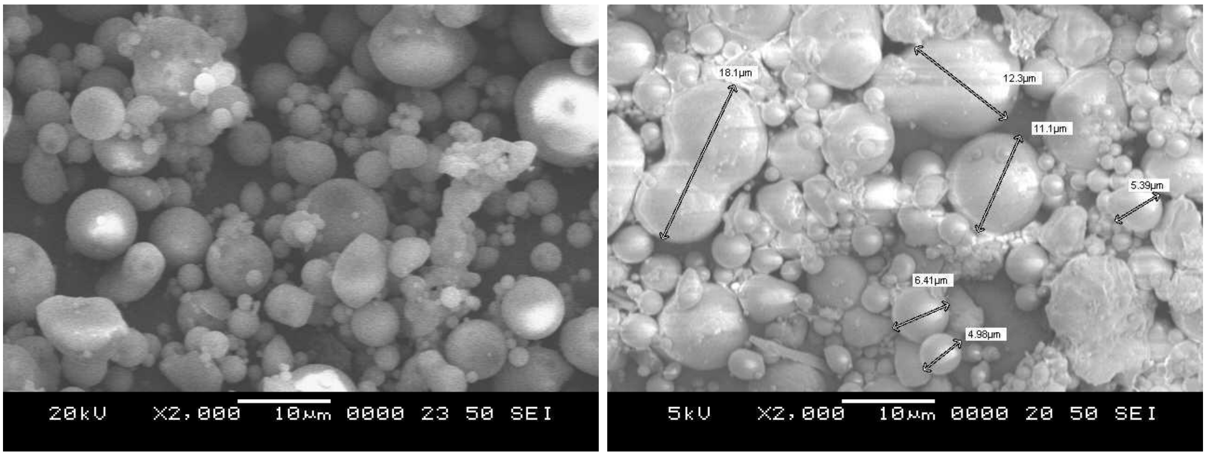


Figure 3.1: SEM photomicrographs of fly ash

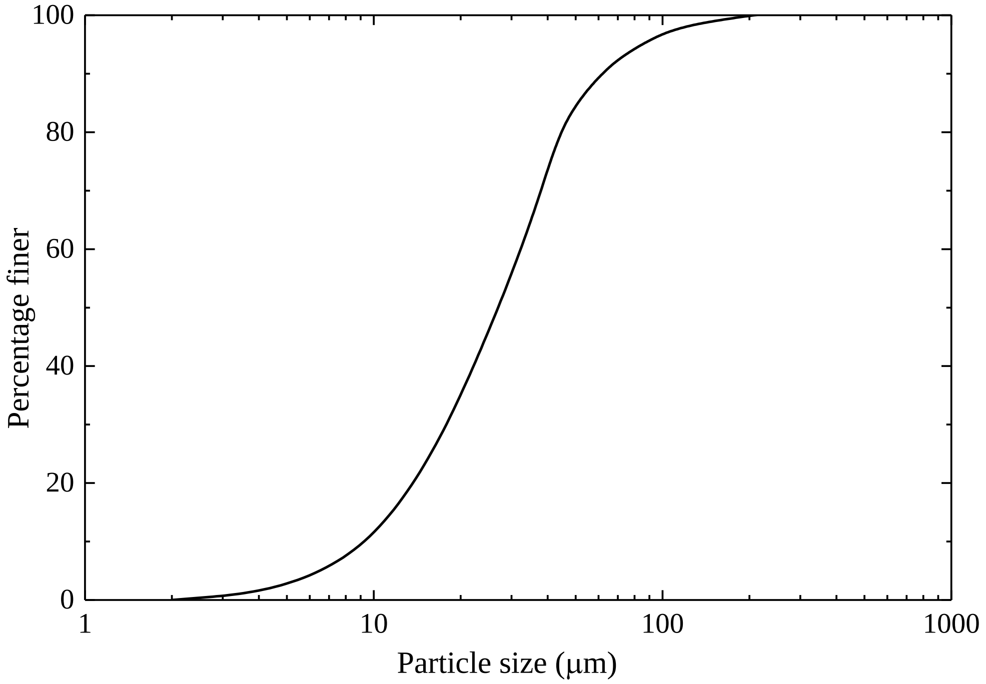


Figure 3.2: Particle size distribution of fly ash

3.2.2 Alkaline solution

Laboratory grade sodium silicate solution (Na_2SiO_3) with silica modulus (SiO_2/Na_2O) of 3.3 (8.0% Na_2O, 26.5% SiO_2, 65.5% H_2O by mass) and sodium hydroxide (NaOH) flakes of 98% purity were used to prepare the alkaline solution. Sodium hydroxide in flakes form was dissolved in deionised water to produce a sodium hydroxide solution. The alkaline solution prepared by mixing both sodium silicate and sodium hydroxide in the required proportions for different mixes and transferred to an air tight container with cap and allowed to cool for 24 h, before using the same in the mix.

3.3 PRODUCTION OF FLY ASH BASED COARSE AGGREGATES

Present study was taken with different set of trial mixes and the pelletization of fly ash was carried out in a laboratory disc pelletizer. A fabricated disc pelletizer as shown in Figure 3.3 was used in this study which has a disc diameter of 500 mm and depth 125 mm. Pelletization was carried out in the following steps.

i) The required proportion of alkaline solution was prepared.

ii) The necessary adjustments were made for the pelletizer.

iii) Fly ash which is free from lumps was transferred to the pelletizing disc.

iv) Alkali solution was sprayed within three minutes to the fly ash during the process of pelletization.

Pelletization in disc pelletizer

Fly ash aggregates

Raw Materials

Figure 3.3: Production of fly ash based coarse aggregates using disc pelletizer.

3.4 CURING OF AGGREGATES

In the present investigation produced aggregates were subjected to three different curing regimes. They are as follows

Ambient curing: The artificially produced fly ash based coarse aggregates through the pelletization process was kept at a temperature of 27 ± 2 ºC and relative humidity of 80% and were characterised for aggregates properties.

Heat curing: The artificially produced fly ash based coarse aggregates through the pelletization process was allowed in ambient temperature as rest period for 24 hours. After that the fly ash aggregates are subjected to 80°C for 24 hours. Further, aggregates removed after heat curing were kept in ambient temperature conditions until it is tested for their engineering properties.

Solution curing: The artificially produced fly ash based coarse aggregates through the pelletization process was allowed in ambient temperature as rest for a period of 24 hours. After that produced aggregates are subjected to solution curing in laboratory grade sodium silicate solution for 30 minutes. Further, this fly ash based coarse aggregates are removed and kept in an ambient temperature conditions until it is tested for their engineering properties.

3.5 TESTS ON PRODUCED AGGREGATES

3.5.1 Efficiency and particle size distribution

The efficiency of pelletization is expressed in percentage weight of aggregates of size greater than 4.75mm (amount of aggregates retained on IS sieve no 480) produced against the total weight materials used in the production and it is calculated using the Eq 3.1 (Gomathi and Sivakumar 2014).

$$Efficiency\ of\ pelletization = \frac{Weight\ retained\ on\ the\ IS\ sieve\ no\ 480}{Total\ weight\ of\ materials\ used} * 100 \qquad (Eq\ 3.1)$$

The distribution of particle sizes present was determined using standard set of sieves in accordance with the Bureau of Indian Standards – IS 383: 2016 (ASTM C136/136M – 14).

3.5.2 Specific gravity, water absorption, aggregate crushing value and aggregate impact value

Specific gravity, water absorption, aggregate crushing value and aggregate impact value were carried out in accordance with the Bureau of Indian Standards - IS 2386:1963 part 3 (ASTM C127 – 15) and IS 2386:1963 part 4 (BS 812 :1990), respectively.

3.5.3 Crushing strength of individual aggregates

The crushing strength of individual fly ash based coarse aggregate was determined using a crushing testing machine as shown in Figure 3.4 and crushing strength (σ) of aggregates was obtained from the equation 3.2 (Gomathi and Sivakumar 2012) and the average of the batch of aggregates is reported as the crushing strength of the aggregates or aggregates crushing strength.

$$\sigma = \frac{2.8 * P}{\pi * x^2} \qquad (Eq\ 3.2)$$

Where, P represents the failure load for the sample and x is the size of aggregates or distance between the two plates. The crushing strength of aggregates was tested in a batch which has a sample size of 20 numbers and the diameter of the aggregates in the range of 6 to 20mm.

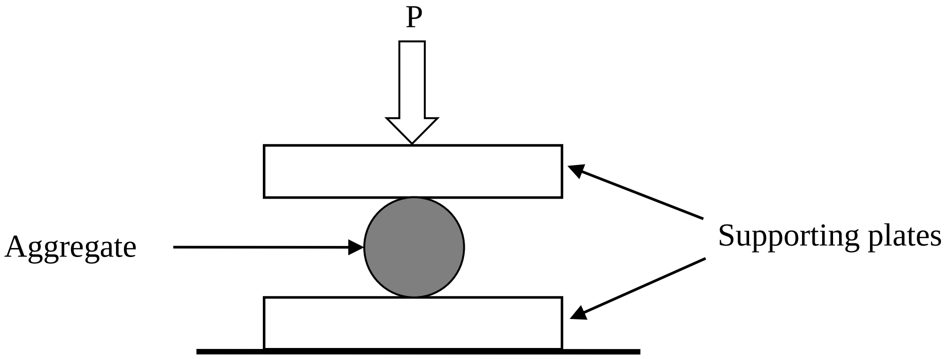


Figure 3.4: Crushing strength testing arrangement

3.5.4 Leaching test on the fly ash based coarse aggregates

Produced fly ash aggregates are examined for leaching of NaOH using titration method by using hydrochloric acid along with methyl orange indicator. Leaching test was conducted on produced aggregates to estimate the amount of NaOH leaching and it was carried out on produced aggregates after 28 days curing. The procedure adopted for leaching tests is carried out in two stages namely preparation of leachate solution and standard titration. The detailed procedure are as follows

Preparation of leachate

Produced fly ash aggregate of known weight is mixed with known weight of distilled water to prepare the leachate solution. The aggregates were left for 24 hours in same water with regular intervals of stirring (3 to 4 times in 24 hours). The above solution is filtrated using filter paper under vacuum. This filtered solution is referred as leachate solution which is used to estimate the leaching of NaOH.

Titration and estimation of NaOH leached from produced aggregates

The leachate sample is titrated against the hydrochloric acid of known concentration with methyl orange as indicator. Titration value is noted down when the solution turns red and the amount of NaOH present in leachate solution is estimated. Average of three titration values is considered as the average leached out NaOH content from produced fly ash based coarse aggregates.

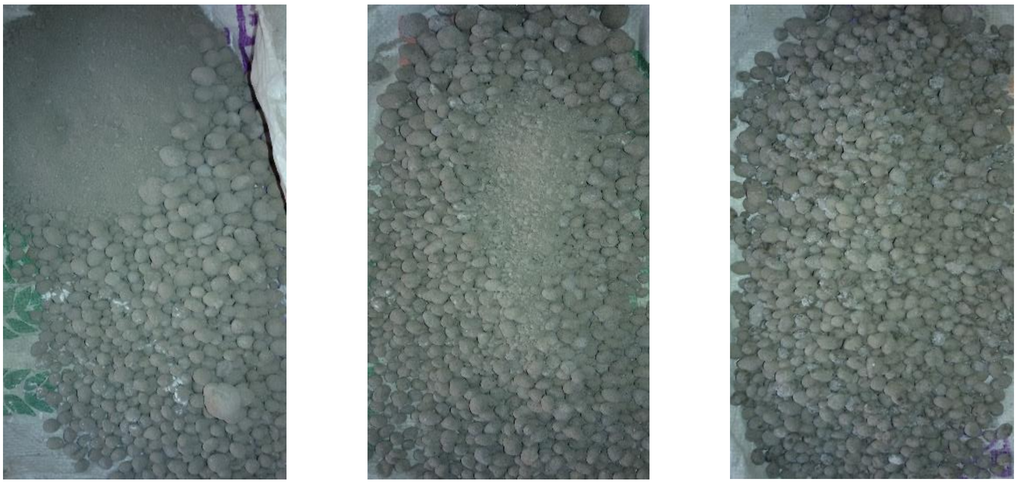
3.5.5 Fixation of water content for pelletization process

An appropriate water content for the pelletization process is the most important factor. To achieve this, few trials with different content were carried out and the same is presented in Table 3.2. Further, produced fly ash aggregates were kept in ambient curing conditions for one day before the efficiency of pelletization was determined as per Eq 3.1.

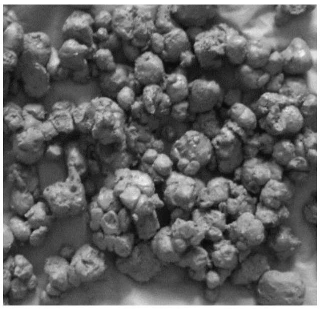
The value of efficiency of the pelletization for different contents is presented in Table 3.2. Further, photographs of the produced aggregates are presented in Figure 3.5. It can be observed from the table that the efficiency is directly proportional to the dosage of water content (percentage by mass of fly ash). From Figure 3.5, it can be observed that with increase in water content (up to 21.58%) the pellets were formed with perfect spherical shape. Further increase in water content formation of the pellets became irregular in shape (muddy balls) by agglomerating with the other pellets.

Table 3.2: Fixation of water content for pelletization process

Proportion No	Mix Code	Fly ash content (%)	Water content (%)	Efficiency (%)
1	F100S1	100	18.26	82.14
2	F100S2		19.92	91.29
3	F100S3		21.58	99.00
4	F100S4		22.57	-



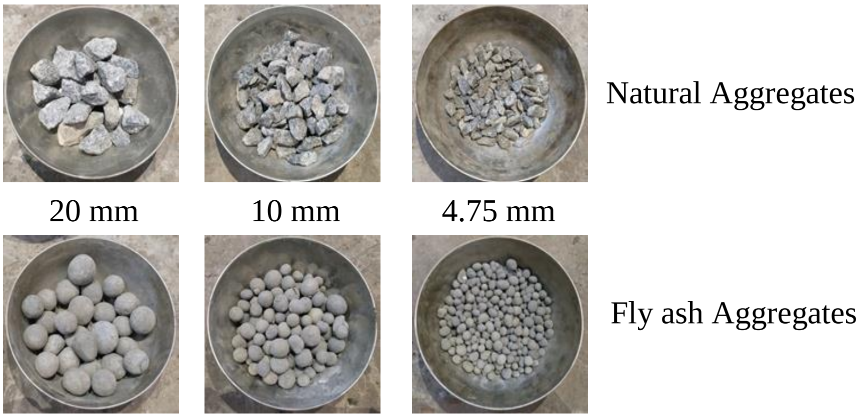
F100S1 F100S2 F100S3



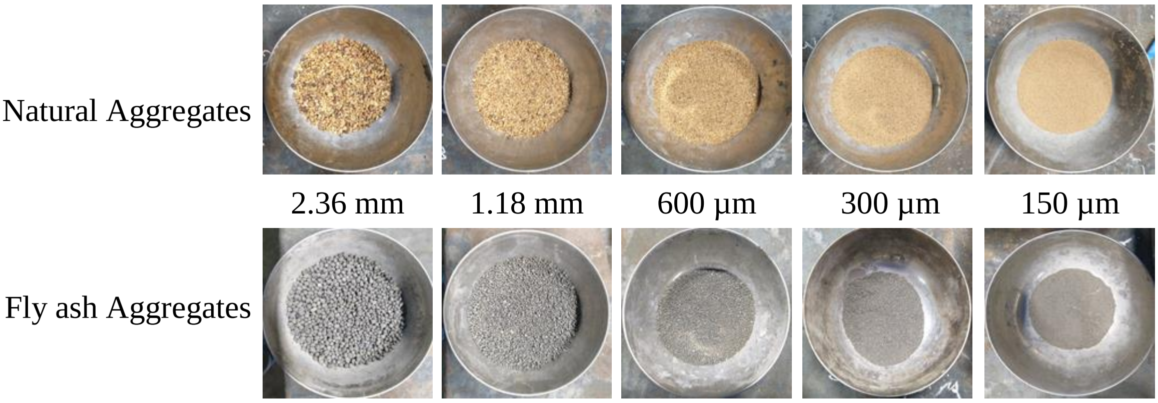
F100S4

Figure 3.5: Aggregates production with different dosage of water content

The produced aggregates are analysed for the particle sizes as per IS 383-2016 and it is observed that produced fly ash aggregates are having complete range of aggregates from 150 µm to 20 mm as shown in Figure 3.6. From Table 3.2, it is noted that water content of minimum of 19 to 21 % is required to produce fly ash based coarsse aggregates efficiently.



i) Coarse aggregates



ii) Fine aggregates

Figure 3.6: Different size of aggregates produced during pelletization process

3.6 MIX PROPORTION AND CONCRETE PRODUCTION

All the concrete mixes were designed for a minimum strength of M40, their mix proportioning were based on the guidelines of IS 10262-2009. The details of the materials used in the concrete production and mix proportion are given in following section.

3.6.1 Materials

3.6.1.1 Cement

Locally available ordinary Portland cement of 53 grade was used for all the mixes in this investigation. Physical tests were carried out as per IS 4031-1988 and the results are presented in Table 3.3. It is observed that all pertinent parameters have values which are within the limits specified by the Indian standards. It is observed that cement used satisfies all the requirements for 53 grade OPC cement as per IS 269-2015.

Table 3.3: Physical test results on ordinary Portland cement

Sl. No.	Test conducted	Results obtained	Requirement as IS code
1	Specific gravity	3.12	--
2	Normal consistency	34 %	--
3	Setting times (minutes) – Initial – Final	150 290	≥ 30 ≤ 600
4	Fineness (m^2 /kg)	330	≥ 300
5	Soundness – Le Chatelier test (mm)	2.5	Not more than 10
6	Average Compressive strength, MPa – 7 days – 14 days – 28 days	 31 41 54	 ≥ 53

3.6.1.2 Fine Aggregate

For the present investigation, the locally available natural river sand was used. The sand was characterised as per IS 2386 - 1963 for their properties and found to be conforming to zone II as per IS 383 - 2016. The physical properties of natural river sand are evaluated and same is presented in Table 3.4. The particle size distribution is presented in the Figure 3.7.

Table 3.4: Physical properties of river sand and coarse aggregates

Properties	River sand	Coarse aggregates
Specific Gravity	2.63	2.72
Bulk density, (kg/m^3) - Loose - Compacted	1440 1700	1410 1620
Water absorption (%)	1.00	0.5

3.6.1.3 Natural coarse (crushed stone) aggregates

Locally available natural (crushed stone) coarse aggregates of 20 mm downsize were used. The various properties of aggregates are determined as per IS 2386-1963 and the specifications are checked as per IS 383-2016 requirements. The particle size distribution is presented in the Figure 3.7.

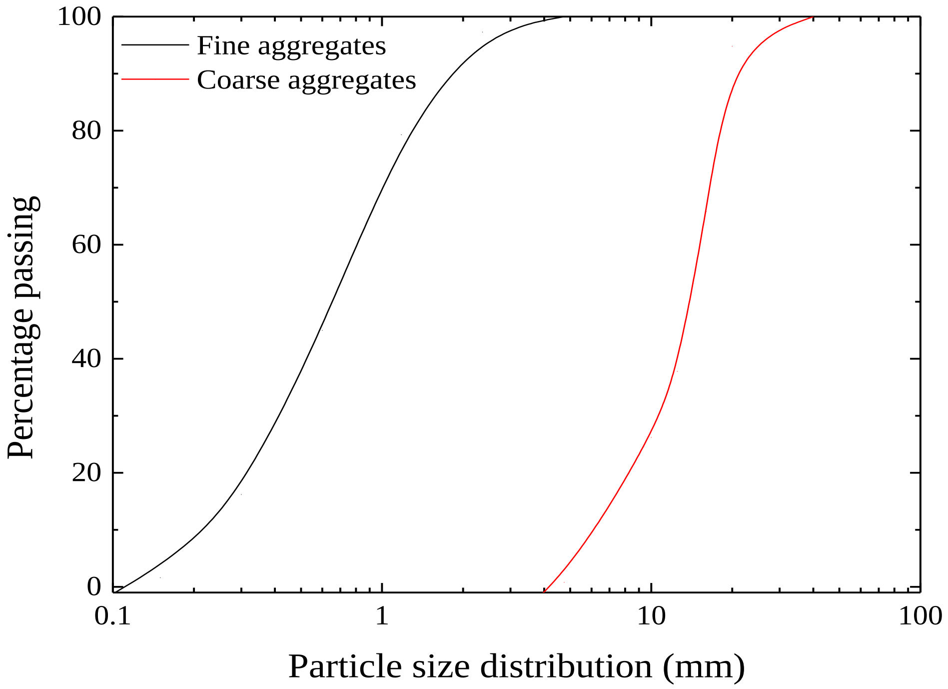


Figure 3.7: Particle size distribution of aggregates

3.6.1.4 Water

Potable tap water was used in production of concrete and curing of the produced concrete.

3.6.2 Formulation of concrete mixes and concrete production

The concrete mix proprortion was carried out according to IS: 10262-2009 to have desired compressive strength of 40 MPa. The addition of any plasticizers is avoided in the concrete mix design, to investigate only the effect of produced fly ash based coarse aggregates on the strength development of concrete. The mix proportions are presented in the Table 3.5 with varying replacement of crushed stone aggregates from 10 to 50 % with produced fly ash based coarse aggregates. The concrete is produced with the help of a ribbon type drum mixer after necessary corrections for aggregate water absorption.

Table 3.5: Mix proportion for concrete production with mix designation

Mix designation	Quantities (kg/m^3)				
	Cement	Fine aggregate	Coarse aggregate	Fly ash based coarse aggregate	Water
M0	380	648	1154	0	186
M1	380	648	1039	85	186
M2	380	648	923	169	186
M3	380	648	808	254	186
M4	380	648	692	338	186
M5	380	648	577	423	186

3.7 TESTS ON CONCRETE

3.7.1 Mechanical properties

3.7.1.1 Compressive strength

Compression testing of the cube specimens was carried out in a compression testing machine of capacity 3000 kN, as per IS 516-1959. Compression testing of cube specimens of size 100 mm × 100 mm × 100 mm was used. A set of three cubes were tested for their compressive strengths at 7, 14 and 28 days of curing.

3.7.1.2 Split tensile strength

Splitting tension tests are carried out, on cylindrical specimens for all the concrete mixes, as per IS 5816:1999. Cylinder of size 100 mm diameter and 200 mm length are used for split tensile strength tests.

3.7.1.3 Flexure strength

Flexure tests are carried out on prisms of size (100 mm ×100 mm ×500 mm) as per IS 516-1959 in a standard flexural testing machine. The load is applied at a rate of 1.8 kN/min, till the specimens fail.

3.7.1.4 Bond strength

To assess the bond strength, cubical specimens of the size 100 mm x 100 mm x 100 mm were prepared and single ribbed reinforcement bar (Fe 500) of diameter 10 mm was placed at the centre of the cubical specimen, as presented in Figure 3.8. The casting direction was vertical with respect to reinforcement bar position and the ultimate bond strength was measured by a pull-out test according to the specifications given in IS 2770 part 1.

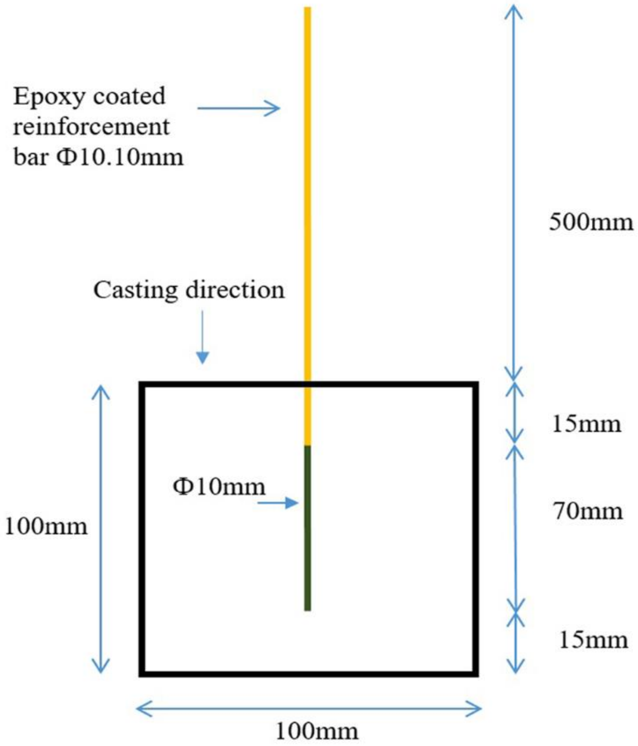


Figure 3.8: Schematic representation of the sample used for bond strength measurement

3.7.2 Water absorption and rate of water absorption

Produced concrete was checked for water absorption and rate of water absorption of concrete by preparing the separate samples prepared for the both the tests and prepared samples was tested as per ASTM C642 and ASTM C1585, respectively.

3.7.3 Durability tests

The durability tests such rapid chloride penetration test (RCPT) was carried out on the produced concrete at 28 and 56 days as per the ASTM C1202. Further, durability study on concrete was performed by considering the strength loss and weight loss when it is exposed chemicals like sulfuric acid and sodium sulphate solution. The specimens are cured in normal water for a period of 28 days, the samples were taken out of the curing tank and allowed to dry naturally. Specimen were immersed in 1% of sulfuric acid solution (H_2SO_4) and 5% sodium sulphate solution (Na_2SO_4), separately in chemical curing containers for an exposure period of 30 and 60 days. All these tests were carried out at room temperature in a laboratory environment for desired period.

3.8 SCANNING ELECTRON MICROSCOPIC STUDIES

The artificially produced fly ash based coarse aggregates and its concrete were analysed for microstructure through scanning electron microscopy (JEOL JSM – 6380LA). The fractured surface/portion of the aggregates was exposed to electron beam and characterized for micrographs and surface morphology. Further, the scanning electron microscopy was also used to identify the microstructure and the interfacial zone (between aggregate and paste) in the concrete. Before taking the SEM images, the samples were gold sputtered in a vacuum container. Two samples are considered for microscopic study for each experimental trial. All the images were characterized at a magnification scale of x1000.

3.9 METHODOLOGY FLOW CHART

The methodology applied in this experimental investigation through a flow chart (Figure 3.9) represents the method of production and testing of fly ash based coarse aggregates and its performance in concrete

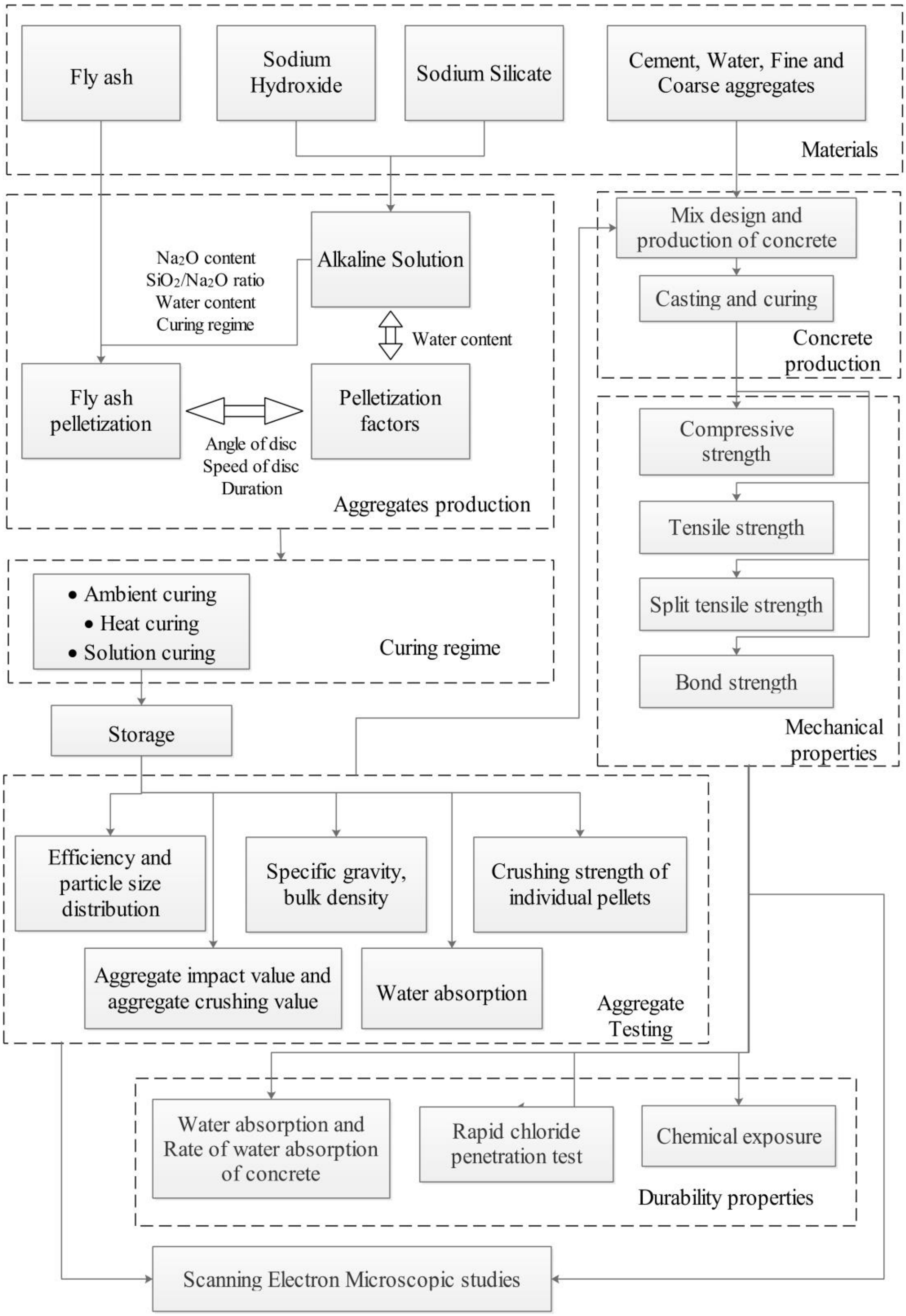


Figure 3.9: Flow chart showing the methodology adopted in the present investigation

CHAPTER – 4

TAGUCHI'S EXPERIMENTAL DESIGN FOR THE PRODUCTION OF FLY ASH BASED COARSE AGGREGATES

4.1 GENERAL

In this chapter factors affecting the production process and engineering properties of produced fly ash based coarse aggregates are discussed. The influence of each factor in the production and properties has been investigated through design of experiments to understand its influence on the overall performance of the produced aggregates.

4.2 FACTORS AFFECTING THE PRODUCTION OF FLY ASH BASED COARSE AGGREGATES

In the present study, the factors which are responsible for the production of fly ash based coarse aggregates are planned to be investigated in detail with help of a matrix presented in the Table 4.1. To optimize the mixtures of fly ash and alkali solution in the production of fly ash based coarse aggregates and to improve the production efficiency and properties of aggregates the design of experiments was used. This study is carried out to analyse the effects of different factors on the production and engineering properties of produced fly ash coarse aggregates.

The most influencing factors related to binder is geopolymerisation and the factors related to geopolymerisation were identified as Na_2O content, SiO_2/Na_2O ratio, water content and curing methods. With an increase of Na_2O content and SiO_2/Na_2O ratio in alkaline solution will lead to higher level of binding property in the geopolymer (Fernandez-Jimenez and Palomo 2005; Dimas et al. 2009; de-Vargas et al. 2014). The different composition in alkaline solution with of Na_2O content from 3% to 7% (percentage of the mass of fly ash) and SiO_2/Na_2O ratio from 0.1 to 2.0. With increase in Na_2O content and SiO_2/Na_2O ratio with constant water content the alkaline solution was found to be highly viscous. This can be attributed to high content of solids in the solution (Leong et al. 2016). Hence, in the present study alkaline solution composition is restricted to less than 7 % of Na_2O content along with SiO_2/Na_2O less than 0.5.

Further, factors related to pelletization process such as speed of pelletizing disc, the angle of pelletizing disc and duration of the pelletization (Baykal and Doven 2000; Harikrishnan and Ramamurthy 2006; Gomathi and Sivakumar 2012) were identified. From literature it is noted that the speed and slope of pelletizing disc were varied from 10 to 70 RPM and 20° to 60° respectively. It is also observed from the literature, minimum of 10 – 15 minutes is needed for the formation of pellets (Baykal and Doven 2000; Gomathi and Sivakumar 2012).

Water content is a factor where both geopolymerisation and pelletization are getting affected, it is observed from literature that water content (percentage is with respect to mass of fly ash) is varied from 15 to 25 %. The water added in the preparation of sodium hydroxide solution and water present in the sodium silicate solution together referred as the water content in the alkaline solution.

Table 4.1: Factors responsible in aggregates production process

Factors		**Factors affecting the pelletization**						
		Raw material	**Type of Pelletizer**	**Speed of Pelletizer**	**Angle of Pelletizer**	**Duration of Pelletization**	**Dosage of Binder**	**Type of Binder**
Factors affecting geopolymerisation	**Raw materials**	-	Key Factor					
	Dosage (Concentration)	Key Factor	Yes	Yes	Yes	Yes	Key Factor	Key Factor
	Operating Temperature		-	Yes	Yes	Yes		
	Curing Regime		-	-	-	-		

4.3 EXPERIMENTAL DESIGN

This section provides information on the experimental methodology followed for the research work. The project's main purpose is concerned with the investigation on effect of geopolymerisation factors and pelletization factors in the production of coarse aggregates using fly ash with alkali solution as a binder. To optimize the mixtures of fly ash and alkali solution in the production of fly ash aggregates in order to improve the efficiency and properties of aggregates the design of experiments was used. The study is carried out using Taguchi's method in different stages to analyse the effects of the factors on the production and engineering properties of artificially produced fly ash coarse aggregates.

4.3.1 Preliminary study on the aggregate production

The production process involves several factors which affect the process and properties of the fly ash aggregates. In this stage, the effect of different factors related to strength and efficiency were identified such as Na_2O content, water content, and curing methods. Alkaline solution with Na_2O content within the range of 3.5 – 4.5 % of the mass of fly ash (Fernandez-Jimenez and Palomo 2005, Hardjito and Rangan 2005) and water content within the range of 19 – 21 % by mass of total solids were used in the mixture are investigated. Three curing methods were used such as curing at ambient temperature (28 ± 2 ºC), heat curing at 80°C for 24 h (Hardjito et al. 2004, Kong et al. 2007, Mustafa et al. 2012, Gorhan and Kurklu 2014, Patankar et al. 2014) and solution curing for 30 min (Gesoglu et al. 2007).

In order to evaluate the interaction with other parameters in the production of fly ash aggregates, Taguchi orthogonal array was used to assess the influence of selected factors in the production of fly ash based coarse aggregates. The different levels of the selected factors were presented in Table 4.2.

Table 4.2: Different selected factors and their different levels tested in preliminary study

Factors	Level 1	Level 2	Level 3
A: Na_2O Content (%)	4.5	4.0	3.5
B: Water Content (%)	19	20	21
C: Curing Regime	Ambient Temperature	Heat Cured	Solution Cured

Orthogonal array, i.e., L9 (3^3) developed by Taguchi to represent a full factorial experiment were used in the present study (Table 4.3). The component variables for each of the mixes (TM 1 – TM 9) are presented in Table 4.4. Three curing ages of 14, 28 and 56 days are selected in this study. The response index for each factor was assessed using Taguchi method.

Table 4.3: Taguchi L9 (3^3) orthogonal array used for preliminary study

Experimental trial runs	Factor A	Factor B	Factor C
TM 1	1	1	1
TM 2	1	2	2
TM 3	1	3	3
TM 4	2	1	2
TM 5	2	2	3
TM 6	2	3	1
TM 7	3	1	3
TM 8	3	2	1
TM 9	3	3	2

Table 4.4: Preliminary study factors and their values used in production of fly ash based coarse aggregates

Trial runs	Factors		
	Na_2O content (%)	Water content (%)	Curing regime
TM 1	4.5	19	Ambient temperature
TM 2	4.5	20	Heat cured
TM 3	4.5	21	Solution cured
TM 4	4.0	19	Heat cured
TM 5	4.0	20	Solution cured
TM 6	4.0	21	Ambient temperature
TM 7	3.5	19	Solution cured
TM 8	3.5	20	Ambient temperature
TM 9	3.5	21	Heat cured

In this stage, geopolymerisation factors are varied which is presented in Table 4.4 in the preparation of alkaline solution with constant SiO_2/Na_2O ratio of 0.3 in each trial mixes. However, the angle of disc maintained at 45° and 15 min duration of pelletization is used in the process of pelletization. For this present investigation rotational speed of disc is maintained at 10 rotations per minute for all mixes.

4.3.2 Detailed investigation on the aggregate production

To evaluate the production performance and engineering properties of the fly ash based coarse aggregates with respect to all the factors, the detailed study was carried out in stages. The relative influence of geopolymerisation factors such as Na_2O content, Si_2O/Na_2O ratio, water content and curing method along with the pelletization factors such as pelletizing disc, angle of pelletizing disc, water content and duration of the pelletization were investigated simultaneously in the production of fly ash based coarse aggregates.

Stage 1: Geopolymerisation factors

In this stage, the effect of geopolymerisation factors related to strength and efficiency were identified such as Na_2O content, water content, and curing method are investigated, same as preliminary study. However, the SiO_2/Na_2O ratio is also an important factor in the geopolymerisation process along with the above factors which is included in this stage (Ridtirud et al 2011, Mustafa et al 2012, Morsy et al 2014). For this stage, an alkaline solution with Na_2O content within the range of 4 – 6 % are selected along with the varying SiO_2/Na_2O ratio from 0.3 to 0.5. Whereas water content within the range of 19 – 21 % of total solids were maintained. Three curing methods were used such as curing at ambient temperature (28 ± 2 ºC), at 80°C for 24 hours, and at solution curing for 30 minutes. The different levels of the all selected geopolymerisation factors were finalised and presented in Table 4.5.

Table 4.5: Different geopolymerisation factors and tested levels

Factors	**Level 1**	**Level 2**	**Level 3**
Na_2O Content (%)	4.0	5.0	6.0
SiO_2/Na_2O	0.3	0.4	0.5
Water Content (%)	19	20	21
Curing Regime	Ambient Temperature	Heat Cured	Solution Cured

Orthogonal array, i.e., L9 (3^4) developed by Taguchi to represent a full factorial experiment were used in the present study (Table 4.6). The component variables for each trial mixes were designated from TMG 1 – TMG 9 and they are presented in Table 4.7. Three curing ages of 14, 28 and 56 days are selected in this study. The response index for each factor was assessed using Taguchi method.

Table 4.6: Taguchi L9 (3^4) orthogonal array for geopolymerisation factors

Experimental trial runs	Factor A	Factor B	Factor C	Factor D
TMG 1	1	1	1	1
TMG 2	1	2	2	2
TMG 3	1	3	3	3
TMG 4	2	1	2	3
TMG 5	2	2	3	1
TMG 6	2	3	1	2
TMG 7	3	1	3	2
TMG 8	3	2	1	3
TMG 9	3	3	2	1

Table 4.7: Geopolymerisation factors and their values used in production of fly ash based coarse aggregates for different trial runs

Trial runs	Factors			
	Na_2O content (%)	SiO_2/Na_2O	Water content (%)	Curing regime
TMG 1	4.0	0.3	19	Ambient temperature
TMG 2	4.0	0.4	20	Heat cured
TMG 3	4.0	0.5	21	Solution cured
TMG 4	5.0	0.3	20	Solution cured
TMG 5	5.0	0.4	21	Ambient temperature
TMG 6	5.0	0.5	19	Heat cured
TMG 7	6.0	0.3	21	Heat cured
TMG 8	6.0	0.4	19	Solution cured
TMG 9	6.0	0.5	20	Ambient temperature

In this stage, geopolymerisation factors are varied and is presented in Table 4.7 for preparation of alkaline solution in each trial mixes. However, the angle of disc maintained at 45° and 15 min duration of pelletization is used in the process of pelletization. For this stage of investigation rotational speed of disc is maintained at 40 rotations per minute for all trial runs.

Stage 2: Pelletization factors

The relative influence of pelletization factors in the production of fly ash based coarse aggregates is very much supportive. In this stage, four factors related to production were identified such as speed of pelletizing disc, angle of pelletizing disc, water content and duration of the pelletization (Baykal and Doven 2000, Harikrishnan and Ramamurthy 2006, Gomathi and Sivakumar 2012). The speed and slope of pelletizing disc varied from 30 to 50 RPM with every 10 RPM interval and 35° to 55° with every 10° intervals, respectively. Water content is varied from 19% to 21% (by weight of fly ash) with every 1% interval. It is also observed from the literature, a minimum of 10 – 15 minutes is needed for the formation of aggregates (Baykal and Doven 2000, Gomathi and Sivakumar 2012) and in the present study 12 and 18 minutes were selected with an interval variation of 3 minutes. The different pelletization factors and their levels were identified and pelletization factors having significant influence in the production process are presented in Table 4.8.

Table 4.8: Different pelletization factors and tested levels

Factors	Level 1	Level 2	Level 3
Speed of pelletizing disc (RPM)	30	40	50
Angle of pelletizing disc (°)	35	45	55
Water content (%)	19	20	21
Duration of pelletization (minutes)	12	15	18

In this stage, the Taguchi method was used in which L9 (3^4) orthogonal array is selected and presented in Table 4.9. The constituent variables for each trial run are designated from TMP 1 to TMP 9 and they are presented in Table 4.10.

Table 4.9: Taguchi L9 (3^4) orthogonal array for pelletization factors

Experimental trial runs	Factor A	Factor B	Factor C	Factor D
TMP 1	1	1	1	1
TMP 2	1	2	2	2
TMP 3	1	3	3	3
TMP 4	2	1	2	3
TMP 5	2	2	3	1
TMP 6	2	3	1	2
TMP 7	3	1	3	2
TMP 8	3	2	1	3
TMP 9	3	3	2	1

Table 4.10: Pelletization factors and their values used in the production of fly ash based coarse aggregates for different trial runs

Trial runs	Factors			
	Speed of pelletizing disc (RPM)	Angle of pelletizing disc (°)	Water Content (%)	Duration of pelletization (minutes)
TMP 1	30	35	19	12
TMP 2	30	45	20	15
TMP 3	30	55	21	18
TMP 4	40	35	20	18
TMP 5	40	45	21	12
TMP 6	40	55	19	15
TMP 7	50	35	21	15
TMP 8	50	45	19	18
TMP 9	50	55	20	12

In this stage, pelletization factors are varied and is presented in Table 4.10 for each trial mixes. However, the alkaline solution was prepared by mixing both sodium hydroxide solution and sodium silicate solution such that Na_2O content of 5% and Si_2O/Na_2O ratio of 0.3 is maintained constant. Water content was also varied with respect to proportions for different mixes (Table 4.10) by adding extra water to alkaline solution.

Stage 3: Combined (Geopolymerisation and pelletization) factors

In this stage, the factors studied in the geopolymerisation stage and pelletization stage are combined to evaluate the production performance and engineering properties of fly ash based coarse aggregates. To study the relative influence of geopolymerisation factors such as Na_2O content, Si_2O/Na_2O ratio, water content and curing method along with the pelletization factors such as pelletizing disc, angle of pelletizing disc, water content and duration of the pelletization were examined simultaneously in the production of pelletized fly ash aggregates. The different factors and their levels were identified in the process having significant influences in the production process are presented in Table 4.11.

Table 4.11: Different combined factors and tested levels

Factors	Level 1	Level 2	Level 3
Na_2O Content	4	5	6
SiO_2/Na_2O	0.3	0.4	0.5
Water content	19	20	21
Speed of pelletizing disc	30	40	50
Angle of pelletizing disc	35	45	55
Duration of pelletization	12	15	18
Curing Regime	Ambient Temperature	Heat cured	Solution cured

In this stage, the Taguchi method was used in which L27 (3^7) orthogonal array is selected and presented in Table 4.12. The constituent variables for each trial run are designated from TMC 1 to TMC 27 and they are presented in Table 4.13.

Table 4.12: Taguchi L27 (3^7) orthogonal array for combined factors of geopolymerisation and pelletization

Trial runs	Factor A	Factor B	Factor C	Factor D	Factor E	Factor F	Factor G
TMC 1	1	1	1	1	1	1	1
TMC 2	1	1	1	1	2	2	2
TMC 3	1	1	1	1	3	3	3
TMC 4	1	2	2	2	1	1	1

Trial runs	Factor A	Factor B	Factor C	Factor D	Factor E	Factor F	Factor G
TMC 5	1	2	2	2	2	2	2
TMC 6	1	2	2	2	3	3	3
TMC 7	1	3	3	3	1	1	1
TMC 8	1	3	3	3	2	2	2
TMC 9	1	3	3	3	3	3	3
TMC 10	2	1	2	3	1	2	3
TMC 11	2	1	2	3	2	3	1
TMC 12	2	1	2	3	3	1	2
TMC 13	2	2	3	1	1	2	3
TMC 14	2	2	3	1	2	3	1
TMC 15	2	2	3	1	3	1	2
TMC 16	2	3	1	2	1	2	3
TMC 17	2	3	1	2	2	3	1
TMC 18	2	3	1	2	3	1	2
TMC 19	3	1	3	2	1	3	2
TMC 20	3	1	3	2	2	1	3
TMC 21	3	1	3	2	3	2	1
TMC 22	3	2	1	3	1	3	2
TMC 23	3	2	1	3	2	1	3
TMC 24	3	2	1	3	3	2	1
TMC 25	3	3	2	1	1	3	2
TMC 26	3	3	2	1	2	1	3
TMC 27	3	3	2	1	3	2	1

Table 4.13: Combined factors and their values used in the production of fly ash based coarse aggregates for different trial runs

Trial runs	Factors						
	Na_2O content	SiO_2/ Na_2O ratio	Water content	Speed of pelletizing disc	Angle of pelletizing disc	Duration of pelletization	Curing regime
TMC 1	4	0.3	19	30	35	12	Ambient Temperature
TMC 2	4	0.3	19	30	45	15	Heat cured
TMC 3	4	0.3	19	30	55	18	Solution cured
TMC 4	4	0.4	20	40	35	12	Ambient Temperature
TMC 5	4	0.4	20	40	45	15	Heat cured
TMC 6	4	0.4	20	40	55	18	Solution cured
TMC 7	4	0.5	21	50	35	12	Ambient Temperature
TMC 8	4	0.5	21	50	45	15	Heat cured
TMC 9	4	0.5	21	50	55	18	Solution cured
TMC 10	5	0.3	20	50	35	15	Solution cured
TMC 11	5	0.3	20	50	45	18	Ambient Temperature
TMC 12	5	0.3	20	50	55	12	Heat cured
TMC 13	5	0.4	21	30	35	15	Solution cured
TMC 14	5	0.4	21	30	45	18	Ambient Temperature
TMC 15	5	0.4	21	30	55	12	Heat cured
TMC 16	5	0.5	19	40	35	15	Solution cured
TMC 17	5	0.5	19	40	45	18	Ambient Temperature
TMC 18	5	0.5	19	40	55	12	Heat cured
TMC 19	6	0.3	21	40	35	18	Heat cured
TMC 20	6	0.3	21	40	45	12	Solution cured
TMC 21	6	0.3	21	40	55	15	Ambient Temperature
TMC 22	6	0.4	19	50	35	18	Heat cured
TMC 23	6	0.4	19	50	45	12	Solution cured
TMC 24	6	0.4	19	50	55	15	Ambient Temperature
TMC 25	6	0.5	20	30	35	18	Heat cured

Trial runs	Factors						
	Na_2O content	SiO_2/ Na_2O ratio	Water content	Speed of pelletizing disc	Angle of pelletizing disc	Duration of pelletization	Curing regime
TMC 26	6	0.5	20	30	45	12	Solution cured
TMC 27	6	0.5	20	30	55	15	Ambient Temperature

In this stage, both geopolymerisation factors and pelletization factors are varied and is presented in Table 4.13 for each trial mixes. The necessary adjustments in the production stage for both pelletizer adjustments and alkaline solution adjustments were taken care in each trial run.

4.4 DETERMINATION OF RESPONSE INDEX

For understanding the effect of selected factors on the properties of produced fly ash based coarse aggregates, it is very much necessary to calculate the value of the response index. For this step, Minitab software was used and calculation was done as follows. The response index for each factor was assessed by taking the average values of the test results from trial mixes, in which factor is involved in that trial. For example, factor A1 was tested in trial mixes TM 1 – TM 3 (Table 4.4). The response index for factor A1 will be the average of the value for trials TM 1 – TM 3. Similarly, the response index for all factors were calculated.

4.5 GREY RELATIONAL ANALYSIS

To understand the level of influence of the various factors in the production and engineering properties of fly ash based coarse aggregates, the Grey relation analysis was carried out. In the present study, the aggregate impact value, aggregate crushing value, crushing strength of aggregates and water absorption have been determined experimentally and considered as responses. For grey relation analysis, the present investigation carried out with the response yielded through Taguchi’s orthogonal array trial sets in each stage which is explained in section 4.3.

4.5.1 Assessment of Grey relational coefficients

Taguchi’s orthogonal array design has been adopted to optimize the multiple performance characteristics of aggregates concurrently with the different curing durations or ages. Grey relational generation was carried out on a scale of 0 – 1 with the experimental data. The normalization of responses is done using the principle that lower the obtained value, it is better for aggregate impact value, aggregate crushing value and water absorption. However, the higher value obtained is better for crushing strength of individual aggregates. Grey relational generation was obtained using the following equation.

$$x_i = \frac{max\, y_i(k) - y_i(k)}{max\, y_i(k) - min y_i(k)} \qquad (Eq\ 4.1)$$

Where, $x_i(k)$ = value after the Grey relational generation; min $y_i(k)$ = smallest value of $y_i(k)$ for the k^{th} response; and max $y_i(k)$ = largest value of $y_i(k)$ for the k^{th} response.

Grey relational coefficients (GRC) provide information on the correlation between the desired and actual experimental data. The Grey relational coefficient was computed by using the following equation.

$$\xi i\ (k) = \frac{\Delta_{min} + \psi\, \Delta_{max}}{\Delta_{0i}(k) + \psi\, \Delta_{max}} \qquad (Eq\ 4.2)$$

Where, $\Delta_{0i} = \| x_0\ (k) - x_i(k)\ \|$ is the difference of the absolute value between $x_0\ (k)$ and $x_i(k)$ and Δ_{min} and Δ_{max} = minimum and maximum values of the absolute differences of all comparing sequences, respectively. ψ is a distinguishing coefficient ($0 \leq \psi \leq 1$) and in the present study, $\psi = 0.5$ is taken (Sahoo et al. 2016, Liu et al. 2017).

4.5.2 Grey relational grade

The Grey relational grade (GRG) is calculated by summing up the weighted Grey relational coefficients corresponding to the responses. The Grey relational grade γ_i is computed using the following equation.

$$\gamma_i = \frac{1}{n}\sum_{k=1}^{n} \xi i(k) \qquad (Eq\ 4.3)$$

Where, n = number of process responses.

Higher Grey relational grade indicates a stronger relational degree between the ideal sequence and the given sequence. Further, Grey relation response table is generated using the Grey relational grade for various curing ages. The higher the difference between maximum and minimum values of Grey relational grade levels indicates the high level significance of the factor. It is ranked according to the higher differences in the values of Grey relational grade.

CHAPTER – 5

CHARACTERISATION OF FLY ASH BASED COARSE AGGREGATES

5.1 GENERAL

In this chapter, results of the various tests conducted on the factors affecting the production of fly ash based coarse aggregates are presented. The engineering properties of the produced aggregates are characterised as per the specifications prescribed in Bureau of Indian standards and necessary discussion are presented.

5.2 TEST RESULTS OF PRELIMINARY STUDY ON AGGREGATES PRODUCTION

Effect of selected factors on the production and properties of fly ash aggregates were investigated as defined in the section 4.3.1. The produced aggregates were evaluated for pelletization efficiency, particle size distribution and their properties such as impact value, water absorption, crushing strength of individual aggregates as explained in section 3.5.

5.2.1 Pelletization efficiency and particle size distribution

The pelletization efficiency for the produced aggregates was determined using Eq 3.1 is plotted with respect to the water content of the trial mixes and presented in Figure 5.1. It can be noted from the figure that the highest degree of efficiency is found to be more than 99% with a water content of 21%. However, efficiency was only 89.72% at 19% water content.

The results of the particle size distribution of the produced pelletized aggregates were presented in Figure 5.2. To check the suitability of the produced aggregates size distribution, the lower limit and higher limit of the specified grading for 20 mm size of IS 383 – 2016 is also plotted in Figure 5.2. It can be observed from the figure that the selected water content levels are found to be appropriate in the production of graded aggregates of 20 mm maximum size. Hence, the produced pelletized aggregates satisfy the grading requirements.

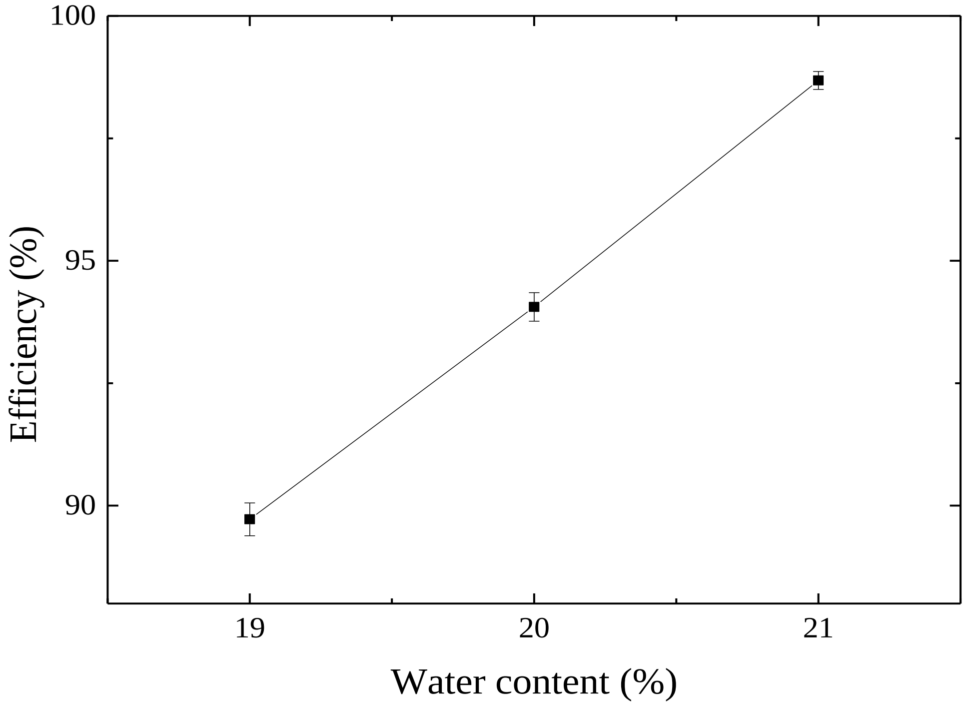


Figure 5.1: Efficiency of pelletization with respect to the water content in preliminary study trial runs

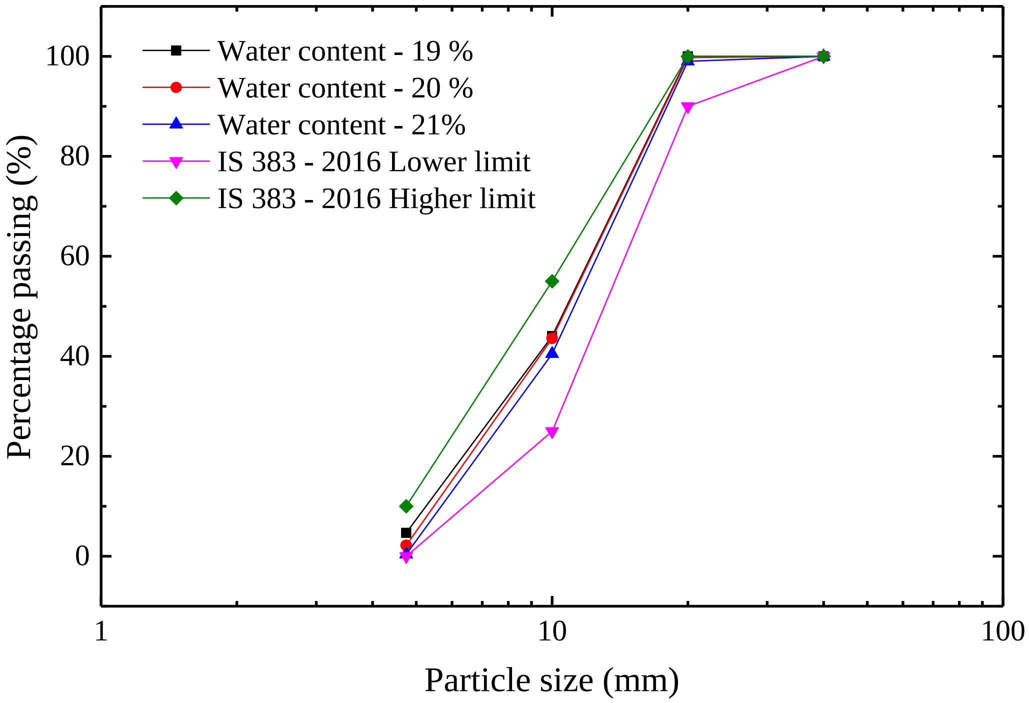


Figure 5.2: Grading of produced fly ash aggregates in the preliminary study with Bureau of Indian Standards grading requirements

5.2.2 Effect of selected factors in preliminary study on aggregates impact value

Test results of aggregates impact value of fly ash aggregates are presented in Table 5.1. It can be observed from the data presented in table that mix TM 9 showed better impact value as compared to all other trial mixes. The better impact value at 28 days (19.23 %) for mix TM 9 corresponds to the high water content and the associated heat curing. Whereas, the lowest (30.73 %) impact value at 28 days was measured for mix TM 8, which is cured in ambient temperature conditions.

The data of the calculated response indices of geopolymerisation factors for the impact value, which is available in Table 5.2, is plotted in Figure 5.3. It can be observed from the figure that Na_2O and water content have the least effect on the impact values of the pelletized aggregates. However, the curing regime governs a pivotal role on the aggregates impact value and it can be noted that heat cured aggregates showed better aggregates impact value than the ambient and solution cured aggregates at all ages. Aggregates impact values of all trial mix increased over the period of 56 days and it can be observed that the solution cured aggregates showed significant improvement.

Table 5.1: Test results of aggregate properties on preliminary study trial runs

Trial runs	Combination	Aggregate impact value (%)			Crushing strength of individual aggregates (MPa)			Water absorption (%)		
		14 days	28 days	56 days	14 days	28 days	56 days	14 days	28 days	56 days
TM 1	A1B1C1	30.48	30.19	29.58	2.10	2.48	2.76	10.54	9.00	11.74
TM 2	A1B2C2	26.85	25.24	25.12	3.89	3.91	3.77	10.61	10.60	9.01
TM 3	A1B3C3	29.65	27.68	22.85	2.99	3.46	4.11	6.44	5.96	4.31
TM 4	A2B1C2	25.25	24.75	24.27	2.38	2.41	2.39	17.79	17.22	17.48
TM 5	A2B2C3	28.38	27.27	24.45	2.41	2.86	3.04	6.00	5.59	5.68
TM 6	A2B3C1	30.58	29.73	27.78	2.60	2.86	3.04	10.83	7.57	8.33
TM 7	A3B1C3	29.73	26.79	27.03	1.77	2.35	3.02	6.74	4.97	5.40
TM 8	A3B2C1	31.07	30.73	29.77	2.53	2.89	2.89	13.71	12.91	10.70
TM 9	A3B3C2	19.80	19.23	20.19	3.01	3.47	3.94	12.15	11.92	11.54

Table 5.2: Response indices of aggregate properties on the preliminary study trial runs

Factors	Aggregate impact value (%)			Crushing strength of individual aggregates (MPa)			Water absorption (%)		
	14 days	28 days	56 days	14 days	28 days	56 days	14 days	28 days	56 days
A1	28.991	27.700	25.847	2.994	3.284	3.547	9.199	8.519	8.351
A2	28.071	27.252	25.501	2.463	2.808	2.965	11.540	10.125	10.495
A3	26.867	25.583	25.662	2.438	2.901	3.286	10.866	9.934	9.214
B1	28.486	27.242	26.959	2.085	2.414	2.726	11.692	10.395	11.539
B2	28.766	27.747	26.447	2.943	3.316	3.375	10.106	9.701	8.460
B3	26.677	25.546	23.605	2.867	3.262	3.696	9.806	8.482	8.061
C1	30.709	30.217	29.041	2.408	2.744	2.900	11.693	9.827	10.256
C2	23.969	23.072	23.194	3.092	3.264	3.369	13.517	13.245	12.673
C3	29.251	27.246	24.775	2.394	2.984	3.529	6.394	5.506	5.131

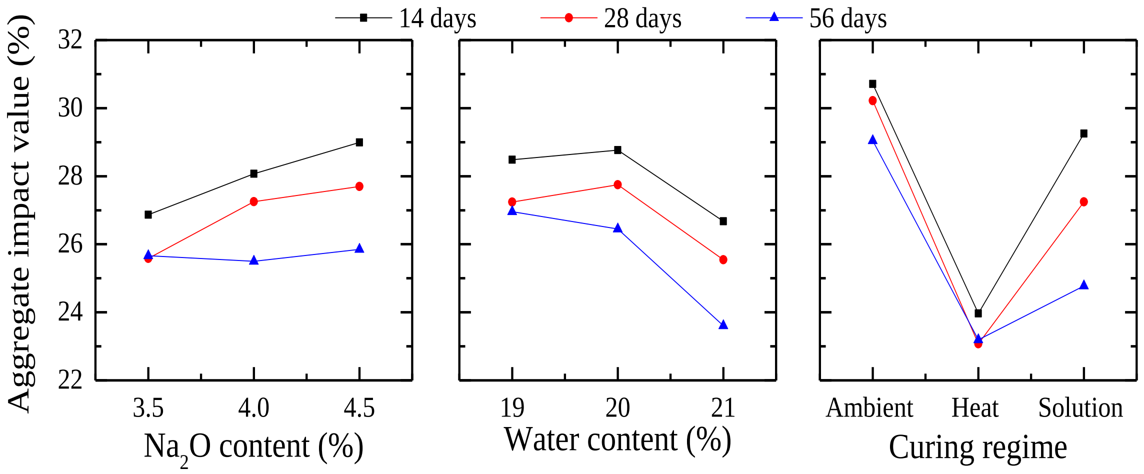


Figure 5.3: Response index relationship between aggregate impact value and selected factors in preliminary study

5.2.3 Effect of selected factors in preliminary study on crushing strength of individual aggregates

Test results of crushing strength of the fly ash aggregates are presented in Table 5.1. It can be understood that the high aggregate crushing strength of 4.11 MPa obtained for Mix TM 3 corresponds to high Na_2O content and solution based curing. Whereas, the trial mix TM 7 resulted in the lowest crushing strength of 1.77 MPa at the age of 14 days which can be attributed to low Na_2O content. However, with an increase in curing ages, the crushing strength increased from 1.77 to 3.02 MPa which can be attributed to the solution curing of produced aggregates.

The data of the calculated response indices of geopolymerisation factors for the crushing strength, which is available in Table 5.2, is plotted in Figure 5.4. It can be observed from the figure that as Na_2O and water content increases, the crushing strength of the individual fly ash aggregates increases. It is also to be noted that the curing regime has considerably influenced the crushing strength of the individual aggregates. However, the rate of gain in crushing strength of the individual aggregates with respect to their age is directly being influenced more in the case of solution based curing as compared to heat curing and ambient curing. This can be attributed to continuous geopolymerisation process, since the aggregates has absorbed sodium silicate solution during the curing period and that contributes to strength properties at later ages (Gesoglu et al. 2007).

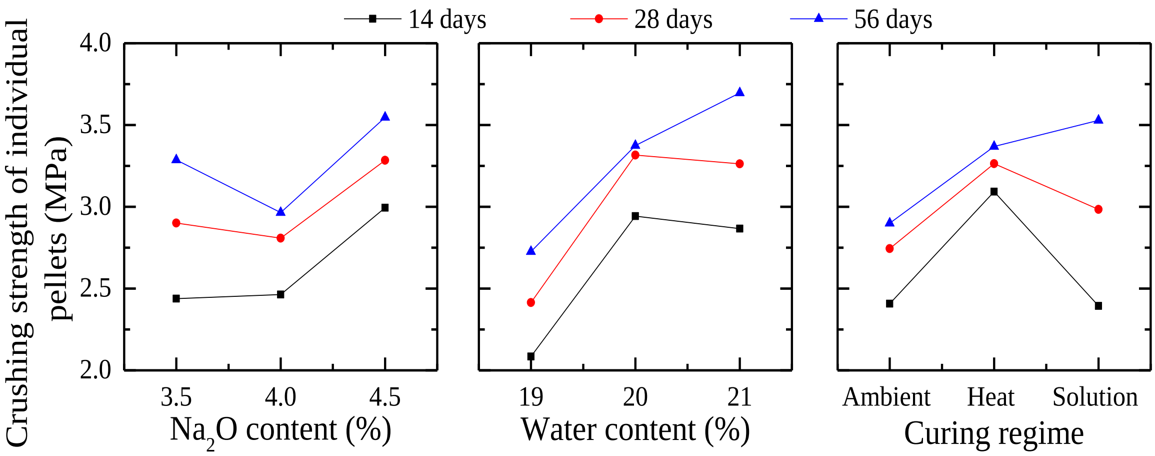


Figure 5.4: Response index relationship between crushing strength of individual aggregates and selected factors in preliminary study

5.2.4 Effect of selected factors in preliminary study on water absorption

Test results of water absorption value of the pelletized fly ash aggregates are presented in Table 5.1. It can be observed that mix TM 3 showed the least water absorption of 4.31 %. The water absorption values for mix TM 5 and mix TM 7 found to be 5.68 % and 5.40 %, respectively is relatively low as compared to other trial mixes. The solution curing which was employed for all three mixes (TM 3, TM 5 and TM 7) can be one of the reasons of having low water absorption values (Gesoglu et al. 2007). The aggregates produced with Mix TM 4 absorbed the highest percentage of water (17.48 %) which is cured with the heat curing.

The data of the calculated response indices of geopolymerisation factors for the water absorption, which is available in Table 5.2, is plotted in Figure 5.5. It can be observed from the figure that the water absorption value of the pelletized aggregates is directly being influenced by the type of curing regimes. High water absorption value is measured in case of heat curing, whereas, the lowest in case of solution curing. It can be also observed in Figure 5.5 that percentage of Na_2O and water content has improved the water absorption value of fly ash aggregates marginally as compared to curing regime.

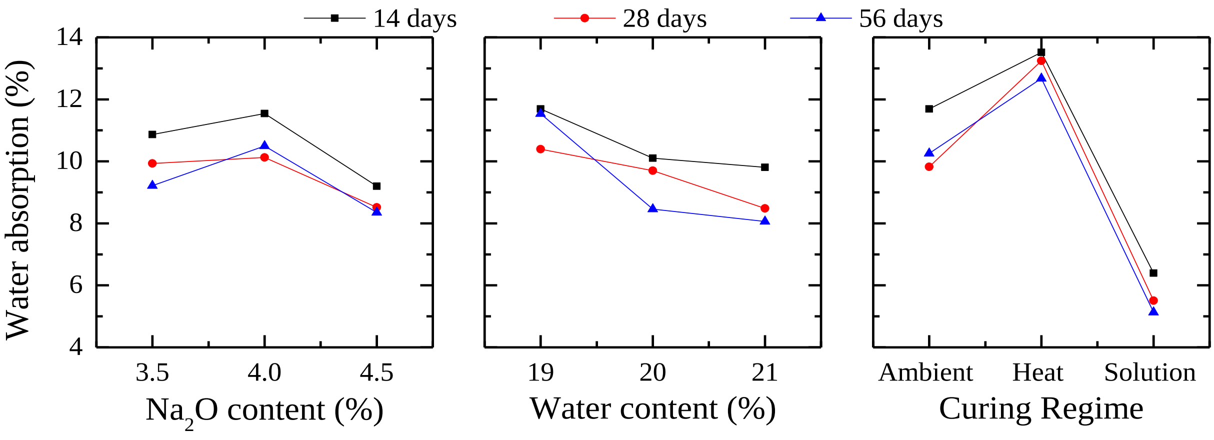


Figure 5.5: Response index relationship between water absorption and selected factors in preliminary study

5.2.5 Grey relational analysis on selected factors in preliminary study

The Grey relational generation and Grey relational coefficient were computed by using the Eq 4.1 and Eq 4.2 respectively. The computed Grey relational generations and Δ_{0i} values are presented in Table 5.3, Table 5.4 and Table 5.5 for 14 days, 28 days and 56 days respectively, while the Grey relational coefficients (GRC) are furnished in Table 5.6. Further, the Grey relational grades were computed using Eq 4.3 are presented in Table 5.7 for various curing ages. Higher Grey relational grade indicates a stronger relational degree between the ideal sequence and the given sequence. The Grey relational grades were computed for all the selected factors in the preliminary study with respect to various ages and the response table is presented in Table 5.8.

Table 5.3: Grey relational generation for 14 days with respect to selected factors in preliminary study

Trial runs	Response			Grey relational generations			Δ_{0i}		
	Aggregate impact value (%)	Aggregates Crushing strength (MPa)	Water absorption (%)						
TM 1	30.48	2.10	10.54	0.053	0.153	0.615	0.947	0.847	0.385
TM 2	26.85	3.89	10.61	0.374	1.000	0.609	0.626	0.000	0.391
TM 3	29.65	2.99	6.44	0.126	0.579	0.963	0.874	0.421	0.037
TM 4	25.25	2.38	17.79	0.516	0.288	0.000	0.484	0.712	1.000
TM 5	28.38	2.41	6.00	0.239	0.302	1.000	0.761	0.698	0.000
TM 6	30.58	2.60	10.83	0.043	0.389	0.591	0.957	0.611	0.409
TM 7	29.73	1.77	6.74	0.119	0.000	0.937	0.881	1.000	0.063
TM 8	31.07	2.53	13.71	0.000	0.359	0.346	1.000	0.641	0.654
TM 9	19.80	3.01	12.15	1.000	0.584	0.478	0.000	0.416	0.522

Table 5.4: Grey relational generation for 28 days with respect to selected factors in preliminary study

Trial runs	Response			Grey relational generations			Δ_{0i}		
	Aggregate impact value (%)	Aggregates Crushing strength (MPa)	Water absorption (%)						
TM 1	30.19	2.48	9.00	0.047	0.086	0.671	0.953	0.914	0.329
TM 2	25.23	3.91	10.60	0.478	1.000	0.541	0.522	0.000	0.459
TM 3	27.68	3.46	5.96	0.266	0.708	0.919	0.734	0.292	0.081
TM 4	24.75	2.41	17.22	0.520	0.041	0.000	0.480	0.959	1.000
TM 5	27.27	2.86	5.59	0.301	0.329	0.949	0.699	0.671	0.051
TM 6	29.73	2.86	7.57	0.087	0.329	0.788	0.913	0.671	0.212
TM 7	26.79	2.35	4.98	0.343	0.000	1.000	0.657	1.000	0.000
TM 8	30.73	2.89	12.91	0.000	0.345	0.351	1.000	0.655	0.649
TM 9	19.23	3.47	11.92	1.000	0.715	0.432	0.000	0.285	0.568

Table 5.5: Grey relational generation for 56 days with respect to selected factors in preliminary study

Trial runs	Response			Grey relational generations			Δ_{0i}		
	Aggregate impact value (%)	Aggregates Crushing strength (MPa)	Water absorption (%)						
TM 1	29.58	2.76	11.74	0.020	0.217	0.436	0.980	0.783	0.564
TM 2	25.12	3.77	9.01	0.486	0.807	0.643	0.514	0.193	0.357
TM 3	22.85	4.11	4.31	0.723	1.000	1.000	0.277	0.000	0.000
TM 4	24.27	2.39	17.48	0.574	0.000	0.000	0.426	1.000	1.000
TM 5	24.45	3.04	5.68	0.555	0.380	0.896	0.445	0.620	0.104
TM 6	27.78	3.04	8.33	0.208	0.380	0.694	0.792	0.620	0.306
TM 7	27.03	3.02	5.40	0.286	0.369	0.917	0.714	0.631	0.083
TM 8	29.77	2.89	10.70	0.000	0.292	0.515	1.000	0.708	0.485
TM 9	20.19	3.94	11.54	1.000	0.904	0.451	0.000	0.096	0.549

Table 5.6: Grey relation coefficients for various curing ages with respect to selected factors in preliminary study

Trial runs	Curing age								
	14 days			28 days			56 days		
	Aggregate impact value	Aggregates Crushing strength	Water absorption	Aggregate impact value	Aggregates Crushing strength	Water absorption	Aggregate impact value	Aggregates Crushing strength	Water absorption
TM 1	0.345	0.371	0.565	0.344	0.353	0.603	0.338	0.390	0.470
TM 2	0.444	1.000	0.561	0.489	1.000	0.521	0.493	0.721	0.584
TM 3	0.364	0.543	0.930	0.405	0.632	0.860	0.643	1.000	1.000
TM 4	0.508	0.413	0.333	0.510	0.343	0.333	0.540	0.333	0.333
TM 5	0.396	0.417	1.000	0.417	0.427	0.907	0.529	0.446	0.828
TM 6	0.343	0.450	0.550	0.354	0.427	0.702	0.387	0.446	0.621
TM 7	0.362	0.333	0.888	0.432	0.333	1.000	0.412	0.442	0.857
TM 8	0.333	0.438	0.433	0.333	0.433	0.435	0.333	0.414	0.508
TM 9	1.000	0.546	0.489	1.000	0.637	0.468	1.000	0.839	0.477

Table 5.7: Grey relational grades for various curing ages with respect to selected factors in preliminary study

Trial runs	Grey relational grade		
	14 days	28 days	56 days
TM 1	0.427	0.434	0.399
TM 2	0.668	0.670	0.599
TM 3	0.612	0.632	0.881
TM 4	0.418	0.395	0.402
TM 5	0.605	0.584	0.601
TM 6	0.448	0.494	0.485
TM 7	0.528	0.589	0.570
TM 8	0.402	0.400	0.418
TM 9	0.678	0.702	0.772

Table 5.8: Response table for Grey relational grade for various ages with respect to selected factors in preliminary study

Factor	Curing ages	Mean Grey relational grade			Maximum value – minimum value	Rank
		Level 1	Level 2	Level 3		
Na_2O Content (%)	**14 days**	0.569	0.490	0.536	0.079	3
Water Content (%)		0.458	0.558	0.580	0.122	2
Curing Regime		0.425	0.588	0.582	0.163	1
Na_2O Content (%)	**28 days**	0.579	0.491	0.564	0.088	3
Water Content (%)		0.472	0.551	0.609	0.137	2
Curing Regime		0.443	0.589	0.602	0.159	1
Na_2O Content (%)	**56 days**	0.627	0.496	0.587	0.130	3
Water Content (%)		0.457	0.540	0.713	0.255	1
Curing Regime		0.434	0.591	0.684	0.250	2

It is evident from the data presented in Table 5.8 that the Grey relational grade is highest at Level 1 for Na_2O content, Level 3 for water content and Level 2 for curing regime at 14 days. However, at 28 and 56 days, the Grey relational grade remains the same for

Na_2O and water content, whereas the curing regime is changed from Level 2 to Level 3. Thus, it can be inferred that simultaneous optimization of aggregates properties such as aggregate impact value, crushing strength of individual aggregates and water absorption can be carried out in aggregates made using high Na_2O and water content with heat curing at 14 days. However, at the curing age of 28 and 56 days, pelletized aggregate properties can be optimized using high Na_2O and water content with solution curing.

The higher the difference between maximum and minimum values of Grey relational grade levels indicates the high level significance of the factor. It is ranked according to the higher differences in the values of Grey relational grade and is presented in Table 5.8. It can be observed from the table that at curing ages of 14 and 28 days pelletized fly ash aggregates is getting influenced in the order, first by curing regime (Rank 1), water content (Rank 2) and followed by Na_2O content (Rank 3).

However, at the curing age of 56 days, fly ash aggregates are highly influenced in the order, first by water content (Rank 1), curing regime (Rank 2) and followed by Na_2O content (Rank 3). This can be attributed to the fact that the properties of produced fly ash aggregates are very much sensitive to the type of adopted curing regime until the curing age of 28 days. However, at a later age (56 days), the amount of water content added in the trial mix plays the vital role in the properties of the pelletized aggregates.

5.3 DETAILED INVESTIGATION TEST RESULTS ON AGGREGATE PRODUCTION

Trial runs were carried out as described in the section 4.3.2 and artificially produced fly ash aggregates are evaluated for their efficiency of production, particle size distribution, aggregate impact value, aggregate crushing value, crushing strength of individual aggregates and water absorption of aggregates as explained in section 3.5.

5.3.1 Pelletization efficiency and particle size distribution

The pelletization efficiency for the produced aggregates was determined using Eq 3.1. It can be observed that the efficiency is increased from 77.96 % to 96.41% as water content increased from 19 to 21 %. The distribution of aggregates size in the production with respect to different water content used is presented in Figure 5.6. From the figure, it can be observed that the particle size distribution is uniformly graded in all cases of water content.

The percentage passing for a graded aggregate of the nominal size of 20 mm as per IS 383 – 2016 were compared with artificially produced fly ash based coarse aggregates and presented in Figure 5.6. It can be noted from the figure that particle size distribution of aggregates produced through pelletization process as the significantly affected due to increases in the water content from 19 % to 21 % and with content of 21 % is particle size distribution found to be the almost near to the lower limit of IS 383 – 2016 which consists of coarser fraction. However, the artificially produced fly ash aggregates particle size distribution is within the IS 383 – 2016 limits.

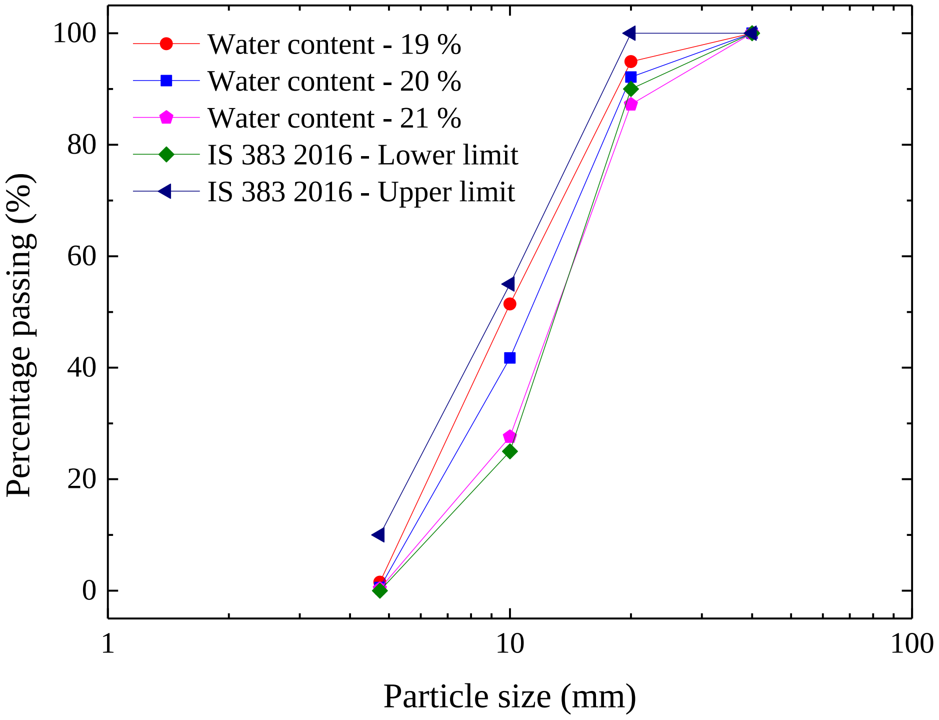


Figure 5.6: Particle size distribution of aggregates with respect to water content and IS 383 – 2016 limits

It can be understood from the test results that the selected factors considered in this stage have a significant influence on production of aggregates. However, water content plays a crucial role in the agglomeration process in production of pellets and also acts as a medium for the binder. The selected water content from 19 % to 21 % showed an enhanced efficiency of production and also the engineering properties. On the other hand, the effect of speed and angle of pelletizing disc in the production of aggregates where the movement of materials at initial and final stage of pelletization process can be clearly observed in the Figure 5.7 and Figure 5.8, respectively.

Figure 5.7: Movement of materials at the initial stage of pelletization process

At lower speed and low angle, the materials movement in the pelletizing disc is very slow because the effects of the gravitational and centrifugal force on the raw materials are at its minimum (Meyer 1980, Baykal and Doven 2000). It can be clearly observed in Figure 5.7 (TMP 7, TMP 8, TMP 9) that as the speed increases the movement of materials increase which in turn help in the movement of materials only with respect to the centrifugal force. Where as, with increase in angle of the pelletizing disc, materials movement is mainly observed with respect to the influence of gravitational force (Meyer 1980, Rao 1994, Baykal and Doven 2000). However, when the pelletizing disc is at an angle of 45^{0} with the minimal varying speed, the materials movement found to be more when compared to other angles and can be visible in Figure 5.8 (TMP 2, TMP 5, TMP 8). This is because of the combined influence of centrifugal and gravitational forces in the production process of fly ash aggregates (Sastry and Fuerstenau 1973; Allen 1987).

Figure 5.8: Movement of materials at the final stage of pelletization process

5.3.2 Specific gravity

The artificially produced fly ash based coarse aggregates which are produced with alkaline solution as a binder were assessed for specific gravity as explained in section 3.5. The average specific gravity of ambient temperature, heat cured and solution cured pelletized fly ash were found to be 1.95, 1.97 and 1.97, respectively. It is to be noted from the reported literature that the specific gravity of pelletized aggregates varies from 2.00 to 2.35 for aggregates produced with cement as a binder (Baykal and Doven 2000, Priyadharshini et al. 2011, Gomathi and Sivakumar 2012), 1.83 for fly ash aggregates produced with lime as a binder (Gomathi and Sivakumar 2012) and 1.80 to 1.99 for aggregates produced with clay minerals as binder (Gomathi and Sivakumar 2014).

5.3.3 Effect of geopolymerisation, pelletization and combined factors on aggregates impact value

The effect of various selected factors on aggregate impact value as defined in the section 4.3.2 is calculated through response indices and presented in Figure 5.9. From Figure 5.9 (i), response index relationship between aggregate impact value and geopolymerisation factors can be observed that as the Na_2O content increases from 4% to 6 % and heat curing has improved the aggregate impact value. Whereas the aggregate impact value was improved when Si_2O/Na_2O ratio increased up to 0.4. Further, increase in the Si_2O/Na_2O ratio as the negligible effect on the aggregates impact value. However, increase in the water content as shown has negligible effect on the aggregate impact value. The response index relationship between aggregate impact value and pelletization factors, which is presented in Figure 5.9 (ii), it can be understood that speed of pelletizing disc, water content and duration of pelletization plays a crucial role on the aggregate impact value of the produced fly ash aggregates. However, with increase in angle of pelletizing disc, the aggregate impact value found to be increasing. This can be attributed to the fact that with increase in angle of pelletizing disc, the gravitational force to each and individual pellets is getting higher as compared to the centrifugal force (Harikrishnan and Ramamurthy 2006).

From the Figure 5.9 (iii) it can be observed that with increase in Na_2O content from 4% to 6%, aggregate impact value was improved from 27.88% to 22.78% at 56 days. Similarly, increase in SiO_2/Na_2O ratio from 0.3 to 0.4 has improved the aggregate impact value from 26.71% to 25.47%, further increase in SiO_2/Na_2O ratio from 0.4 to 0.5 had marginal affect. Significant improvement of aggregate impact value of 23.69% was observed for heat cured fly ash aggregates when compared to aggregate impact values of 28.74% and 25.58% for ambient cured and solution cured aggregates at 56 days, respectively. The aggregate impact value gets marginally improved from 25.83% to 24.98% at 56 days with an increase in water content from 19% to 20%, further increase in the water content from 20% to 21% found to have an adversarial effect where the aggregates impact value has decreased from 24.98% to 27.21% at 56 days. It is also noted from figure that the aggregates impact value has marginally improved from 25.93% to 25.59% due to the increase in the speed of pelletizing disc from 30 RPM to 50 RPM. Further increase in the angle of pelletizing disc from 35° to 55° and duration of pelletization 12 to 18 minutes has showed the negligible improvement in the aggregate impact values from 26.04% to 25.96% and 26.08% to 26.25% at 56 days, respectively.

From Figure 5.9, it is to be understood that the geopolymerisation factors such as higher Na_2O content, SiO_2/Na_2O ratio of 0.4, and heat curing significantly improved the aggregate impact value. It is also observed that increase in water content has a negative effect on the aggregate impact value and solution curing of produced aggregates shown the improved aggregate impact value at later ages (56 days). Further, the pelletization factors in this stage are introduced along with the geopolymerisation factors and the influence of combined factors on the aggregate impact value is presented in Figure 5.9 (iii). On introducing the pelletization factors in the production process, the influence of Na_2O content, SiO_2/Na_2O ratio and curing regime on aggregates impact value has reduced up to 8.9 %, 11.0 %, and 8.6 %, respectively. Whereas, the aggregate impact value gets improved when water content increased up to 20%. Further, an increase in the water content found to have a negative effect on the aggregates impact value. However, the influence of the pelletization factors on the aggregates impact value in the water content response in the study is reduced by 11.3 %. It is also noted that from the pelletization factors response indices, Figure 5.9(iii), indicates that the aggregates impact value has improved due to the increase in the speed of pelletizing disc. However, increase in the angle of pelletizing disc and duration of pelletization has shown the negligible effect on the aggregate impact value.

It is to be noted that local available natural aggregates have the impact value of less than 19.2%. However, the impact value of fly ash aggregates produced with cement and clay minerals as a binder is reported to be 25.4 and 38.0, respectively (Priyadharshini et al. 2011, Gomathi and Sivakumar 2014).

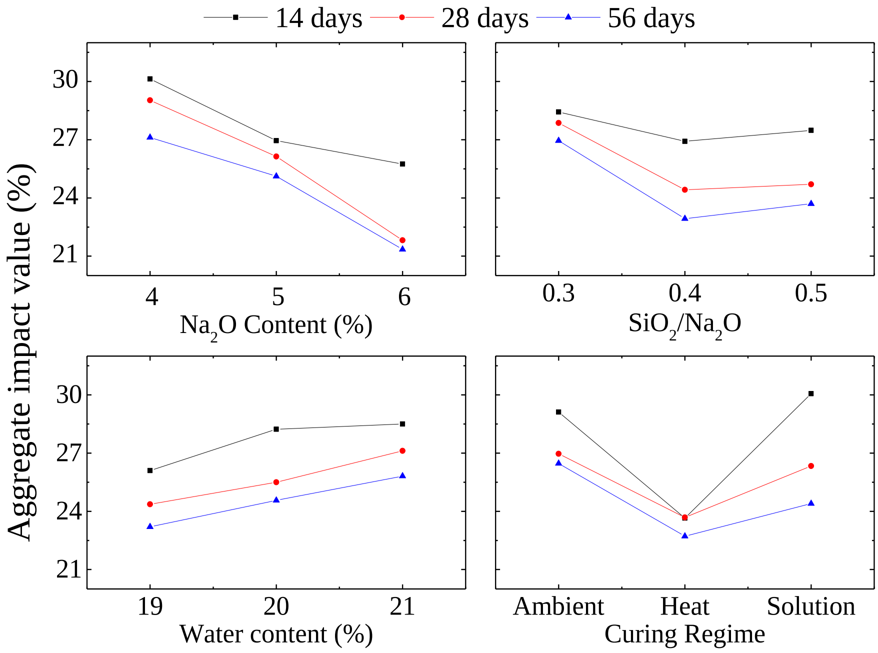


Figure 5.9 (i) Geopolymerisation factors

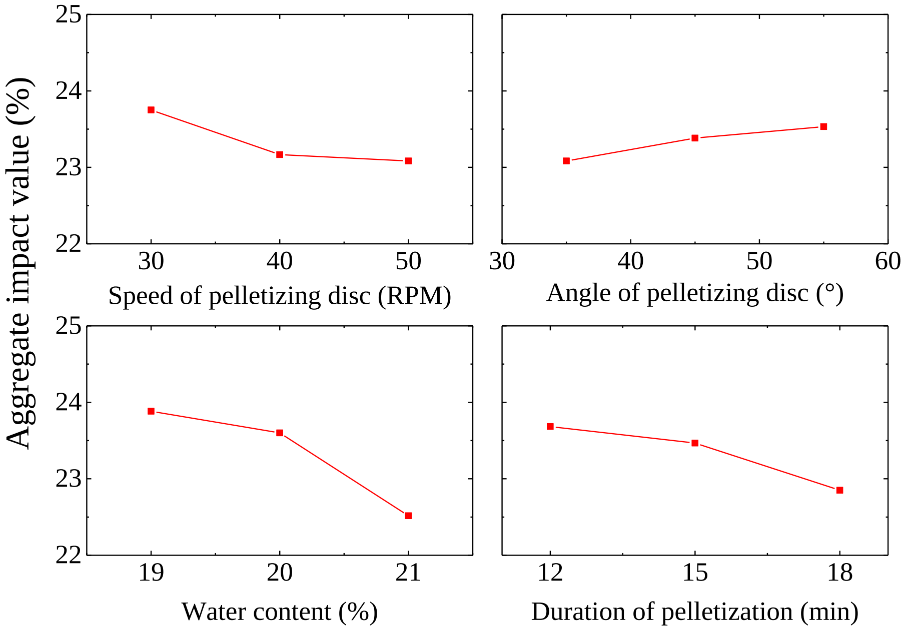


Figure 5.9 (ii) Pelletization factors

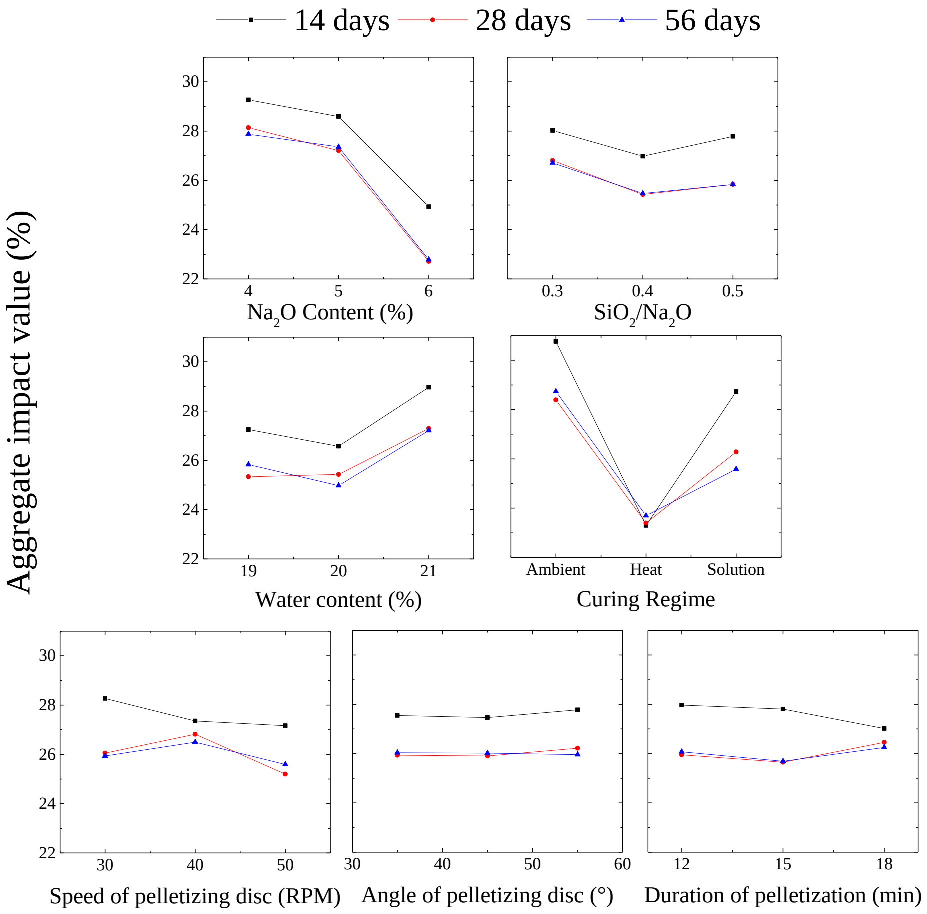


Figure 5.9 (iii) combined factors

Figure 5.9: Response index relationship between aggregate impact value and i) geopolymerisation factors ii) pelletization factors and iii) combined factors

5.3.4 Effect of geopolymerisation, pelletization and combined factors on aggregate crushing value

The response indices are calculated from the experimental test results to know the effect of various selected factors on aggregate crushing value as defined in the section 4.3.2 and is presented in Figure 5.10. It can be observed from response index relationship between aggregate crushing value and geopolymerisation factors that as the increase in Na_2O content, Si_2O/Na_2O ratio and curing regime (heat cured and solution cured) has improved the aggregate crushing value. However, the increase in the water content from 19 % to 20 % has shown the negative effect and from 20 % to 21% negligible effect on the aggregate crushing value. The effect of factors of pelletization on aggregate crushing value which is presented in Figure 5.10 (ii), it is observed that angle of pelletizing disc, duration of pelletization and water content governs a crucial role on the aggregate crushing value of the artificially produced fly ash aggregates. In case of angle of pelletizing disc, it can be noted that 45º found to be the optimum suitable value. However, speed of pelletizing disc is having least influence on the aggregate crushing value.

The response index relationship for combined factors with aggregate crushing value presented in Figure 5. 10 (iii). It can be observed from the figure that the geopolymerisation factors such as Na_2O content increase from 4% to 6%, aggregate crushing value was improved from 27.21% to 23.92% at 56 days. Similarly, increase in SiO_2/Na_2O ratio from 0.3 to 0.5 has marginally improved the aggregate crushing value from 26.72% to 25.33% at 56 days. It was also observed that heat curing and solution curing of aggregates has significantly improved the aggregate crushing value of 23.37% and 25.34%, respectively at 56 days when compared to the aggregates crushing value 28.72% at 56 days of ambient cured fly ash aggregates. Aggregates crushing value has declined from 25.13% to 26.35% at 56 days due to the increased water content in trial mixes. However, response indices from Figure 5.10 (ii) for pelletization factors indicate that the duration of pelletization of 12 to 18 minutes has improved the aggregates crushing value from 26.62% to 24.17% at 56 days. Further, increase in speed of pelletizing disc from 30 RPM to 50 RPM and angle of pelletizing disc from 35° to 55° has negligible improvement on aggregate crushing value from 26.01% to 25.93% and 25.93% to 25.98%, respectively at 56 days.

From Figure 5.10, it can be noted that the geopolymerisation factors such as higher Na_2O content, higher SiO_2/Na_2O ratio, and curing regime (heat curing and solution curing) has significantly improved the aggregate crushing value. Further, the pelletization factors are introduced in the process and the effect of combined factors on the aggregates crushing value is presented in the Figure 5.10 (iii). It is observed that because of the introduction of the pelletization factors in the process the influence of Na_2O content, SiO_2/Na_2O ratio and curing regime on aggregates crushing value has reduced by 30.8 %, 33.9 %, and 24.5 %, respectively. Further, an increase in the water content found to have a negative effect on the aggregates crushing value and its response in the study significantly reduced by 26.5 %. However, the pelletization factors response indices indicates that the speed of pelletizing disc and duration of pelletization has improved the aggregates crushing value and angle of pelletizing disc has negligible effect on the aggregate crushing value.

It is to be noted that local available natural aggregates have the aggregate crushing value of less than 24.5%. However, the aggregate crushing value of fly ash aggregates produced with cement as a binder is reported to be 22.7 (Priyadharshini et al. 2011).

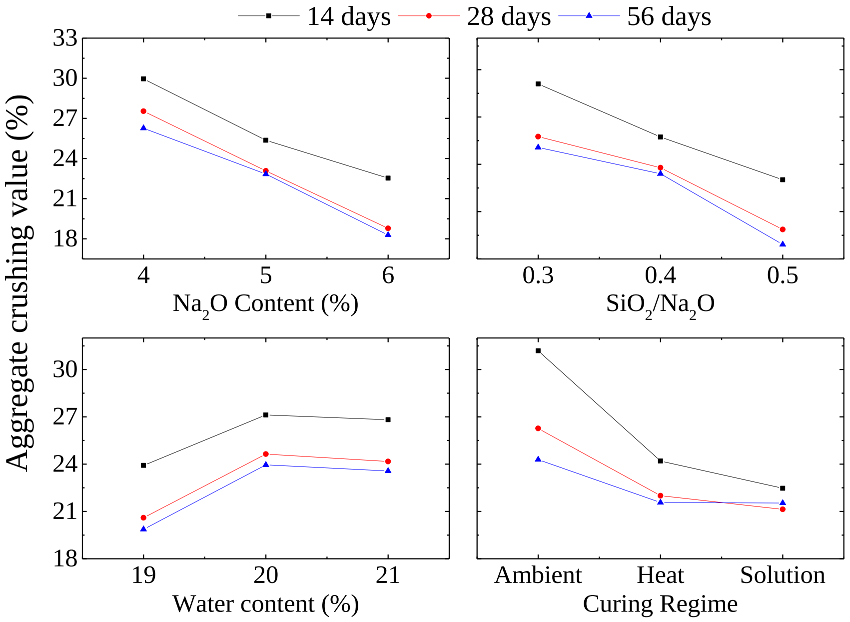


Figure 5.10 (i) Geopolymerisation factors

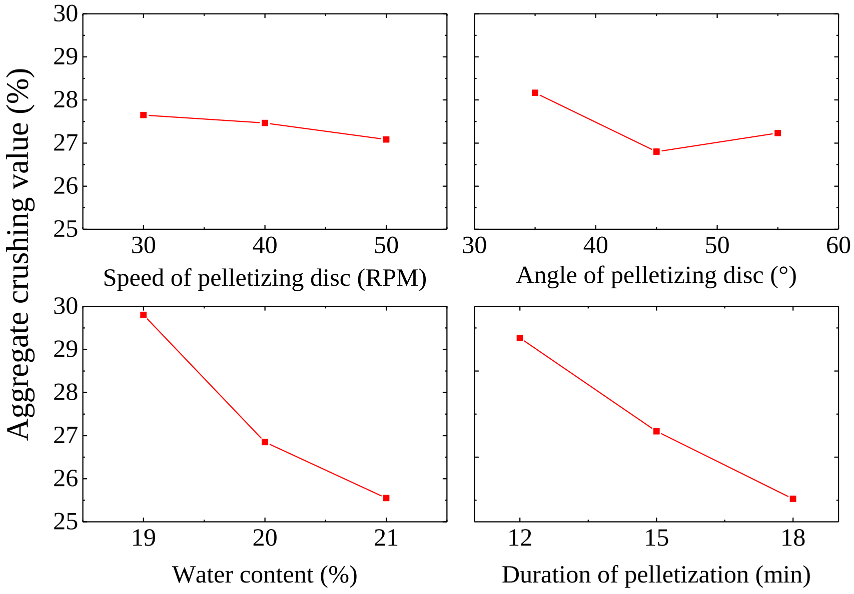


Figure 5.10 (ii) Pelletization factors

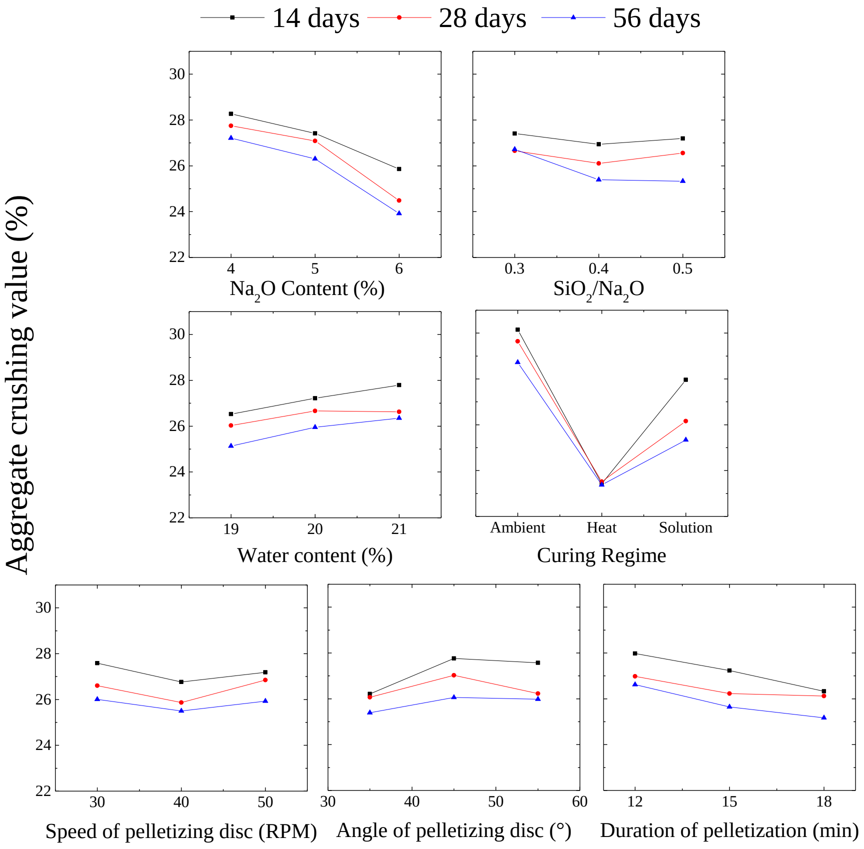


Figure 5.10 (iii) Combined factors

Figure 5.10: Response index relationship between aggregate crushing value and i) geopolymerisation factors ii) pelletization factors and iii) combined factors

5.3.5 Effect of geopolymerisation, pelletization and combined factors on crushing strength of individual aggregates

The effect of various selected factors on aggregate impact value as defined in the section 4.3.2 is calculated through response indices and presented in Figure 5.11. From response index relationship between crushing strength of individual aggregates and geopolymerisation factors, it is clear that as the increase in Na_2O content, Si_2O/Na_2O ratio and heat curing has improved the crushing strength of individual aggregates. However, increase in the water content from 19 % to 20 % as shown the negative effect and increase from 20 % to 21% crushing strength of individual aggregates increased at 56 days of curing. The response index relationship between crushing strength of individual aggregates and pelletization factors, which is presented in Figure 5.11 (ii), it is observed that all factors of pelletization have a decisive influence on the crushing strength of individual aggregates. However, it is noted that angle of pelletizing disc is 45° found to be the suitable value.

From the Figure 5.11 (iii), response index relationship for combined factors with crushing strength of individual aggregates. it can be observed that the geopolymerisation factors such as the Na_2O content increase from 4% to 6%, improved the crushing strength of individual aggregates from 3.04 MPa to 3.79 MPa, respectively at 56 days. Similarly, increase in SiO_2/Na_2O ratio from 0.3 to 0.5 has improved the crushing strength of individual aggregates from 3.24 MPa to 3.57 MPa at 56 days. Significant improvement in crushing strength of individual aggregates was observed for heat cured aggregates (3.81 MPa at 56 days) as compared to that of ambient cured and solution cured aggregates values of 3.08 MPa and 3.28 MPa, respectively at 56 days. Aggregate crushing strength of individual aggregates declined from 3.50 MPa to 3.21 MPa at 56 days with the increase in water content from 19% to 21%. It can also be understood from figure that pelletization factors such as response indices indicates a trivial improvement in aggregates crushing strength from 3.36 MPa to 3.49 MPa due to the increase in the duration of pelletization from 12 to 18 minutes. However, increase in the angle of pelletizing disc from 35° to 55° and speed of pelletizing disc from 30 RPM to 50 RPM has shown the negligible improvement on the individual aggregates crushing strength from 3.38 MPa to 3.39 MPa and 3.39 MPa to 3.41 MPa, respectively at 56 days.

From Figure 5.11, it is to be understood that the geopolymerisation factors, with higher Na_2O content, high SiO_2/Na_2O ratio, and heat curing has significantly improved the aggregates crushing strength. It can be also observed that the increase in the water content has a negative effect on the aggregate crushing strength of individual aggregates. However, high water content and solution curing of produced aggregates shown the improved aggregates crushing strength at later ages (56 days). The pelletization factors introduced later in the production process and the influence of combined factors on the crushing strength of aggregates is presented in Figure 5.11 (iii). On introducing the pelletization factors in the process, the influence of Na_2O content, SiO_2/Na_2O ratio, curing regime and water content on aggregates crushing strength has reduced up to 25.2 %, 27.1 %, 26.2 % and 24.2 %, respectively. It can be also understood that from Figure that pelletization factors response indices indicates an improvement in aggregates crushing strength due the increase in the duration of pelletization. However, increase in the angle of pelletizing disc and speed of pelletizing disc has shown the negligible effect on the aggregates crushing strength.

It is to be noted from the research reported that fly ash aggregates produced with cement and lime as a binder have the aggregates crushing strength of 13.72 MPa and 1.65 MPa, respectively (Gomathi and Sivakumar 2012) and 2.92 to 9.98 MPa for fly ash aggregates produced with clay minerals as a binder (Gomathi and Sivakumar 2014).

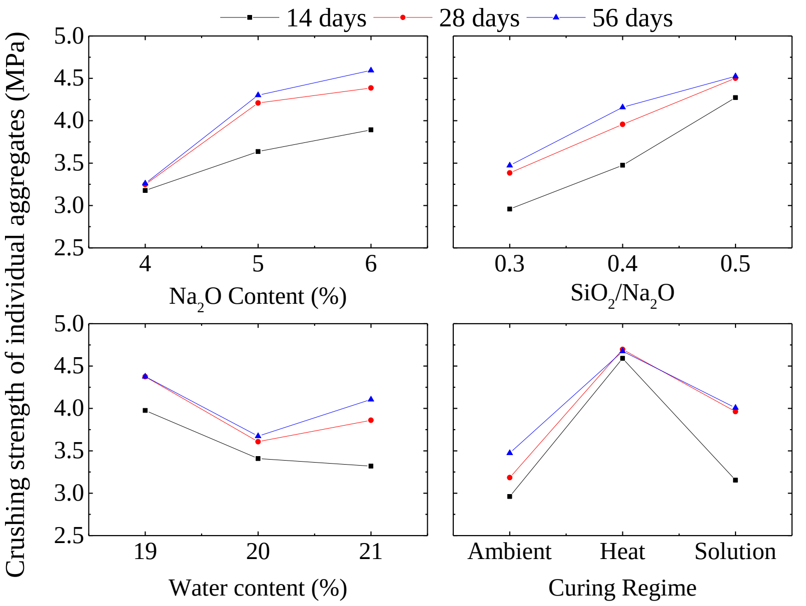


Figure 5.11 (i) Geopolymerisation factors

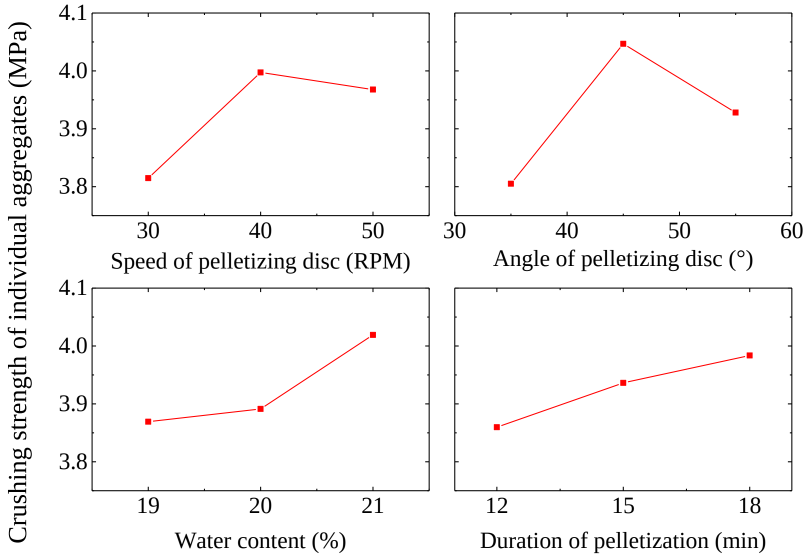


Figure 5.11 (ii) Pelletization factors

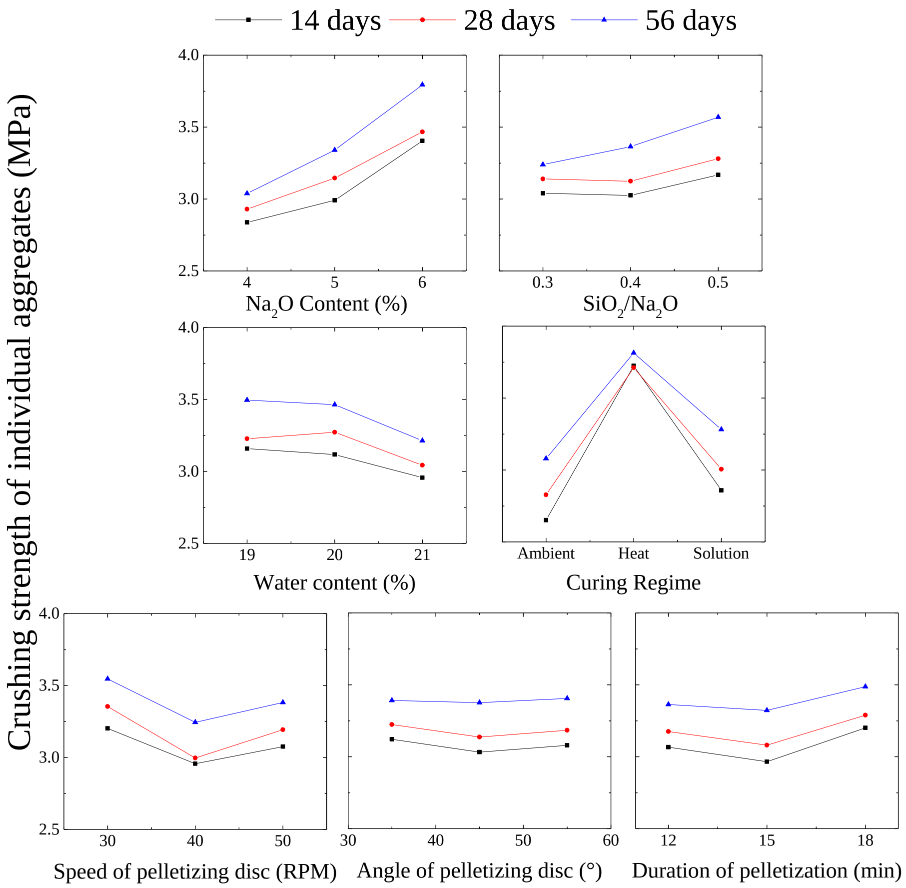


Figure 5.11 (iii) Combined factors

Figure 5.11: Response index relationship between the crushing strength of individual aggregates and i) geopolymerisation factors ii) pelletization factors and iii) combined factors

5.3.6 Effect of geopolymerisation, pelletization and combined factors on water absorption

The response indices are calculated from the experimental test results to know the effect of various selected factors on water absorption as defined in the section 4.3.2 and is presented in Figure 5.12. From response index relationship between water absorption and geopolymerisation factors, it is clear that as the increase in Na_2O content, Si_2O/Na_2O ratio and solution curing has improved the water absorption of aggregates. Whereas, increase in the water content has shown the negative effect and negligible effect on the water absorption of aggregates. However, the heat curing of aggregates has increased the water absorption of the aggregates. The effect of pelletization factors on water absorption which is presented in Figure 5.12 (ii), it is clear that speed of pelletizing disc, angle of pelletizing disc and water content governs a crucial role on the water absorption of produced aggregates. However, pelletization duration is found to be having least effect on the water absorption of artificially produced fly ash aggregates.

The response index relationship for combined factors with water absorption presented in Figure 5.12 (iii), it can be observed that geopolymerisation factors such as Na_2O content from 4% to 6%, improved water absorption value from 11.37% to 9.04% and with increase in SiO_2/Na_2O ratio from 0.3 to 0.5 also reduced the water absorption from 10.91% to 9.92% at 56 days. It was also observed that solution curing of artificially produced aggregates has significantly reduced the water absorption value to 8.42% when compared to water absorption values of 10.58% for ambient cured and 12.44% for heat cured aggregates at 56 days. However, increase in speed of pelletizing disc from 30 RPM to 50 RPM and change in the angle of pelletizing disc from 35° to 55° has marginally improved the water absorption of aggregates from 10.47% to 9.82% and 10.76% to 10.26%, respectively at 56 days. It was also observed that increase in duration of pelletization from 12 to 18 minutes showed the negligible effect on the water absorption values of 10.42% to 10.68%.

From Figure 5.12, it is found that geopolymerisation factors such as higher Na_2O content, higher SiO_2/Na_2O ratio, and solution curing has significantly reduced the water absorption. Further the pelletization factors are introduced in the process, the effect of combined factors on the aggregates crushing value is presented in the Figure 5.12 (iii). It is observed that after introduction of the pelletization factors in the process, the influence of Na_2O content, SiO_2/Na_2O ratio and curing regime on the water absorption of aggregates has reduced by 7.3 %, 10.3 %, and 8.8 %, respectively. Further, an increase in the water content found to have a negative effect on the water absorption of aggregates and its response in the study significantly reduced by 7.6 % due to pelletization factors in the process. However, the pelletization factors response indices indicates the speed of pelletizing disc and duration of pelletization has improved the water absorption of aggregates and it is observed that angle of pelletizing disc on the water absorption of aggregates has negligible effect in the production process.

It is to be noted from the research reported that the water absorption varies from 18.33 % to 46.79 % for aggregates produced with cement as a binder (Ramamurthy and Harikrishnan 2006, Priyadharshini et al. 2011, Gomathi and Sivakumar 2012), 21.82 % to 48.75 % for fly ash aggregates produced with lime as binder (Ramamurthy and Harikrishnan 2006, Gomathi and Sivakumar 2012) and 17.86 % to 22.22 % for aggregates produced with clay minerals as binder (Gomathi and Sivakumar 2014).

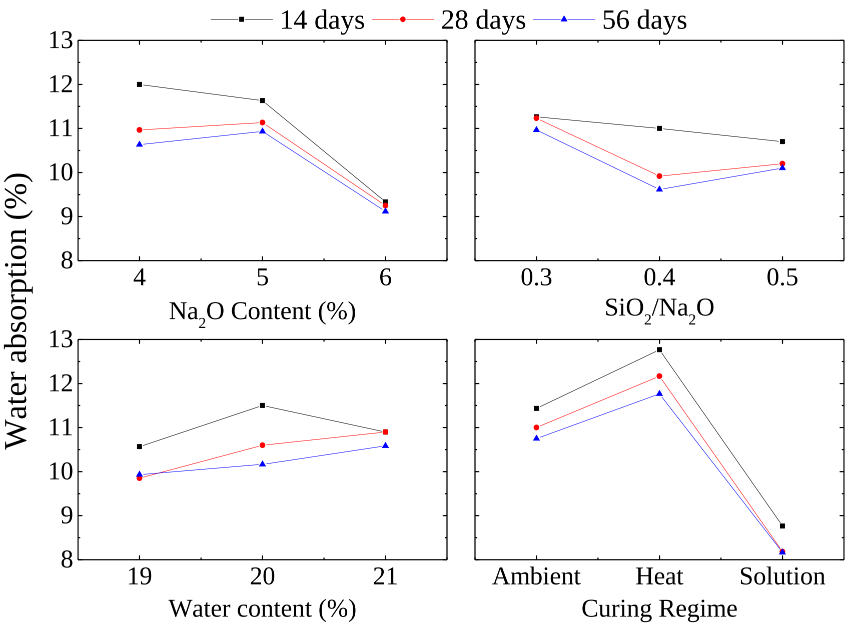


Figure 5.12 (i) Geopolymerisation factors

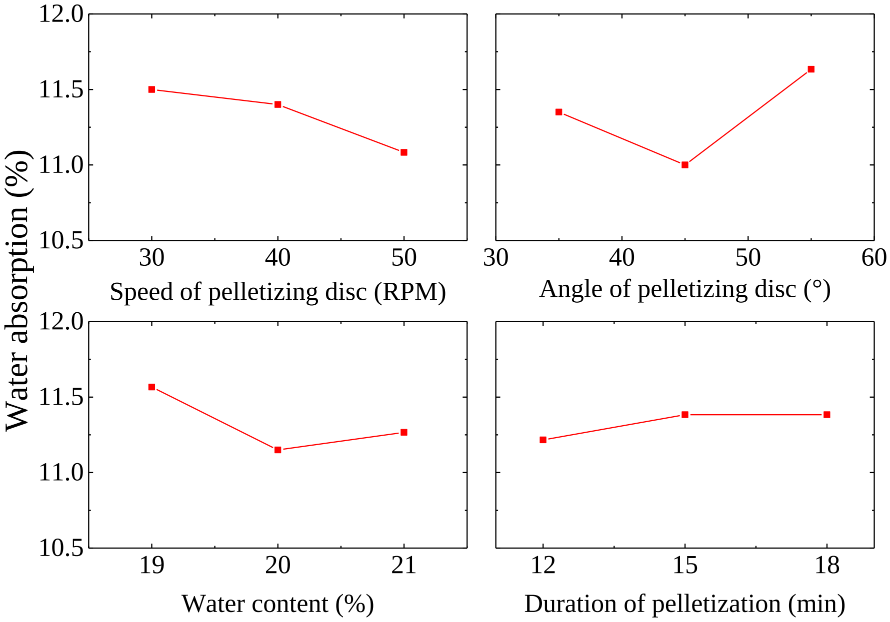


Figure 5.12 (ii) Pelletization factors

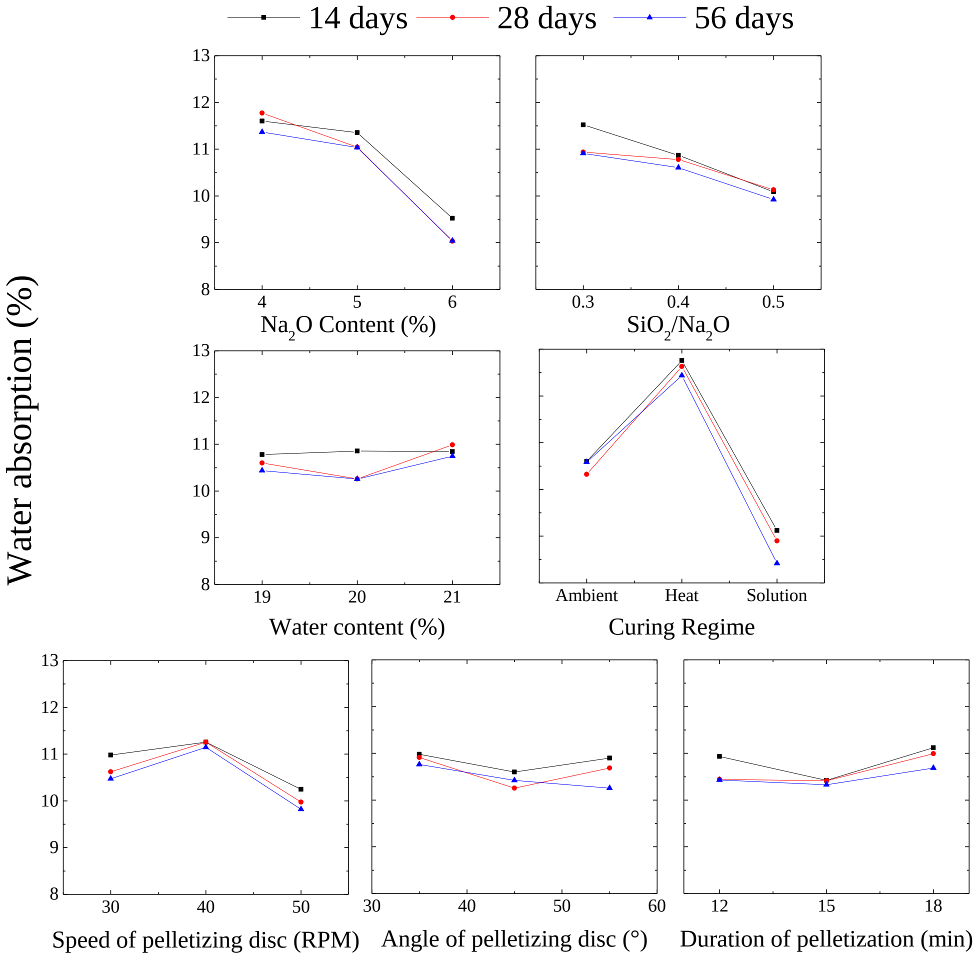


Figure 5.12 (iii) Combined factors

Figure 5.12: Response index relationship between water absorption and i) geopolymerisation factors ii) pelletization factors and iii) combined factors

5.3.7 Grey relational analysis on geopolymerisation, pelletization and combined factors in detailed study

The Grey relational generation was computed by using the Eq 4.1. Grey relational generations for the geopolymerisation, pelletization and combined factors are presented in Table 5.9 – 5.11, Table 5.12 and Table 5.13 – 5.15, respectively at selected different ages.

Table 5.9: Grey relational generation with respect to geopolymerisation factors for 14 days

Trial runs	Responses				Grey relational generations			
	Aggregate impact value	Water absorption	Aggregates crushing strength	Aggregate crushing value	Aggregate impact value	Water absorption	Aggregates crushing strength	Aggregate crushing value
TMG 1	30.95	12.30	2.37	36.30	0.200	0.263	0.000	0.000
TMG 2	26.10	14.30	3.95	29.15	0.604	0.000	0.464	0.367
TMG 3	33.35	9.40	3.22	24.40	0.000	0.645	0.250	0.610
TMG 4	30.85	10.20	2.45	26.20	0.208	0.539	0.025	0.518
TMG 5	28.65	12.00	2.69	31.25	0.392	0.303	0.094	0.259
TMG 6	21.35	12.70	5.77	18.65	1.000	0.211	1.000	0.905
TMG 7	23.50	11.30	4.06	24.80	0.821	0.395	0.496	0.590
TMG 8	26.00	6.70	3.79	16.80	0.613	1.000	0.419	1.000
TMG 9	27.75	10.00	3.83	26.00	0.467	0.566	0.430	0.528

Table 5.10: Grey relational generation with respect to geopolymerisation factors for 28 days

Trial runs	Responses				Grey relational generations			
	Aggregate impact value	Water absorption	Aggregates crushing strength	Aggregate crushing value	Aggregate impact value	Water absorption	Aggregates crushing strength	Aggregate crushing value
TMG 1	31.25	11.70	2.35	30.75	0.000	0.093	0.000	0.000
TMG 2	25.65	12.30	3.67	28.55	0.496	0.000	0.367	0.139
TMG 3	30.20	8.90	3.73	23.30	0.093	0.527	0.385	0.470
TMG 4	28.85	9.80	3.32	25.20	0.212	0.388	0.271	0.350
TMG 5	27.65	11.60	3.37	27.90	0.319	0.109	0.285	0.180
TMG 6	21.90	12.00	5.94	16.15	0.827	0.047	1.000	0.921
TMG 7	23.50	12.20	4.49	21.30	0.686	0.016	0.595	0.596
TMG 8	19.95	5.85	4.84	14.90	1.000	1.000	0.694	1.000
TMG 9	22.00	9.70	3.84	20.15	0.819	0.403	0.415	0.669

Table 5.11: Grey relational generation with respect to geopolymerisation factors for 56 days

Trial runs	Responses				Grey relational generations			
	Aggregate impact value	Water absorption	Aggregates crushing strength	Aggregate crushing value	Aggregate impact value	Water absorption	Aggregates crushing strength	Aggregate crushing value
TMG 1	30.15	11.60	2.43	28.10	0.000	0.076	0.000	0.000
TMG 2	23.75	11.50	3.61	27.80	0.540	0.093	0.360	0.024
TMG 3	27.45	8.80	3.74	22.90	0.228	0.551	0.400	0.419
TMG 4	27.45	9.55	3.30	26.00	0.228	0.424	0.265	0.169
TMG 5	26.75	11.20	3.88	26.70	0.287	0.144	0.443	0.113
TMG 6	21.15	12.05	5.72	15.80	0.759	0.000	1.000	0.992
TMG 7	23.25	11.75	4.69	21.10	0.582	0.051	0.688	0.565
TMG 8	18.30	6.15	4.98	15.70	1.000	1.000	0.775	1.000
TMG 9	22.50	9.45	4.11	18.05	0.646	0.441	0.511	0.810

Table 5.12: Grey relational generation with respect to pelletization factors

Trial runs	Responses				Grey relational generations			
	Aggregate impact value	Aggregate crushing value	Aggregates crushing strength	Water absorption	Aggregate impact value	Aggregate crushing value	Aggregates crushing strength	Water absorption
TMP 1	24.40	31.80	3.57	11.65	0.000	0.000	0.000	0.318
TMP 2	24.20	26.40	3.91	11.05	0.089	0.766	0.592	0.864
TMP 3	22.65	24.75	3.97	11.80	0.778	1.000	0.691	0.182
TMP 4	22.70	26.80	3.90	11.30	0.756	0.709	0.572	0.636
TMP 5	22.75	26.00	4.14	10.90	0.733	0.823	1.000	1.000
TMP 6	24.05	29.60	3.95	12.00	0.156	0.312	0.665	0.000
TMP 7	22.15	25.90	3.95	11.10	1.000	0.837	0.661	0.818
TMP 8	23.20	28.00	4.09	11.05	0.533	0.539	0.903	0.864
TMP 9	23.90	27.35	3.87	11.10	0.222	0.631	0.519	0.818

Table 5.13: Grey relational generation with respect to combined factors for 14 days

Trial runs	Responses				Grey relational generations			
	Aggregate impact value	Aggregate crushing value	Aggregates crushing strength	Water absorption	Aggregate impact value	Aggregate crushing value	Aggregates crushing strength	Water absorption
TMC 1	34.00	30.90	2.57	13.50	0.000	0.036	0.100	0.129
TMC 2	25.80	24.95	3.63	12.60	0.516	0.514	0.508	0.235
TMC 3	30.25	28.90	2.82	11.10	0.236	0.197	0.198	0.412
TMC 4	31.40	30.95	2.31	11.80	0.164	0.032	0.000	0.329
TMC 5	22.95	24.65	3.14	13.20	0.695	0.538	0.321	0.165
TMC 6	27.80	27.35	2.66	11.30	0.390	0.321	0.136	0.388
TMC 7	32.85	31.35	2.46	9.60	0.072	0.000	0.060	0.588
TMC 8	26.40	25.85	3.21	12.90	0.478	0.442	0.345	0.200
TMC 9	31.95	29.50	2.75	8.40	0.129	0.149	0.169	0.729
TMC 10	28.50	28.30	2.51	9.60	0.346	0.245	0.079	0.588
TMC 11	31.15	30.70	2.56	11.60	0.179	0.052	0.097	0.353
TMC 12	23.05	24.05	3.90	13.30	0.689	0.586	0.612	0.153
TMC 13	32.50	28.70	2.76	9.50	0.094	0.213	0.174	0.600
TMC 14	31.80	30.90	2.64	10.60	0.138	0.036	0.129	0.471
TMC 15	25.75	25.00	3.42	14.60	0.519	0.510	0.429	0.000
TMC 16	28.65	24.85	2.84	9.10	0.336	0.522	0.204	0.647
TMC 17	30.20	28.75	2.72	11.40	0.239	0.209	0.160	0.376
TMC 18	25.75	25.50	3.56	12.50	0.519	0.470	0.483	0.247
TMC 19	22.60	21.45	3.86	11.80	0.717	0.795	0.597	0.329
TMC 20	27.70	29.30	2.77	9.40	0.396	0.165	0.177	0.612
TMC 21	29.15	28.10	2.74	10.80	0.305	0.261	0.166	0.447
TMC 22	18.10	18.90	3.88	11.30	1.000	1.000	0.605	0.388
TMC 23	24.55	27.20	3.60	7.60	0.594	0.333	0.496	0.824
TMC 24	27.95	28.80	2.81	7.90	0.381	0.205	0.194	0.788
TMC 25	19.30	20.50	4.91	12.60	0.925	0.871	1.000	0.235
TMC 26	26.65	27.55	3.02	6.10	0.462	0.305	0.273	1.000
TMC 27	28.35	30.90	3.05	8.20	0.355	0.036	0.285	0.753

Table 5.14: Grey relational generation with respect to combined factors for 28 days

Trial runs	Responses				Grey relational generations			
	Aggregate impact value	**Aggregate crushing value**	**Aggregates crushing strength**	**Water absorption**	**Aggregate impact value**	**Aggregate crushing value**	**Aggregates crushing strength**	**Water absorption**
TMC 1	30.40	31.25	2.68	12.40	0.132	0.058	0.092	0.167
TMC 2	26.00	24.70	3.60	12.80	0.431	0.602	0.475	0.117
TMC 3	28.40	27.20	3.04	11.05	0.268	0.394	0.239	0.333
TMC 4	32.35	30.15	2.46	11.95	0.000	0.149	0.000	0.222
TMC 5	22.95	24.80	3.20	13.75	0.637	0.593	0.309	0.000
TMC 6	27.95	26.25	2.67	10.95	0.298	0.473	0.088	0.346
TMC 7	28.30	31.95	2.69	10.45	0.275	0.000	0.095	0.407
TMC 8	26.15	26.10	3.14	13.10	0.420	0.485	0.283	0.080
TMC 9	30.75	27.35	2.88	9.50	0.108	0.382	0.176	0.525
TMC 10	26.40	27.85	2.74	8.65	0.403	0.340	0.114	0.630
TMC 11	30.90	31.45	3.10	9.95	0.098	0.041	0.266	0.469
TMC 12	22.45	24.50	3.79	12.50	0.671	0.618	0.554	0.154
TMC 13	29.65	26.10	3.08	10.10	0.183	0.485	0.258	0.451
TMC 14	28.65	30.75	2.54	10.95	0.251	0.100	0.033	0.346
TMC 15	25.45	24.45	3.75	13.70	0.468	0.622	0.540	0.006
TMC 16	25.00	24.70	2.92	9.50	0.498	0.602	0.190	0.525
TMC 17	30.15	29.45	2.93	11.40	0.149	0.207	0.193	0.290
TMC 18	26.25	24.50	3.47	12.65	0.414	0.618	0.423	0.136
TMC 19	23.95	22.15	3.66	12.45	0.569	0.813	0.499	0.160
TMC 20	26.10	25.75	2.74	8.40	0.424	0.515	0.115	0.660
TMC 21	26.65	25.05	2.91	10.25	0.386	0.573	0.188	0.432
TMC 22	17.60	20.55	3.93	10.85	1.000	0.946	0.613	0.358
TMC 23	20.75	23.50	3.65	6.30	0.786	0.701	0.495	0.920
TMC 24	23.50	28.40	2.83	8.45	0.600	0.295	0.153	0.654
TMC 25	19.80	19.90	4.86	11.85	0.851	1.000	1.000	0.235
TMC 26	21.50	26.75	3.33	5.65	0.736	0.432	0.364	1.000
TMC 27	24.60	28.35	3.30	7.10	0.525	0.299	0.351	0.821

Table 5.15: Grey relational generation with respect to combined factors for 56 days

Trial runs	Responses				Grey relational generations			
	Aggregate impact value	Aggregate crushing value	Aggregates crushing strength	Water absorption	Aggregate impact value	Aggregate crushing value	Aggregates crushing strength	Water absorption
TMC 1	31.00	30.45	2.62	12.40	0.106	0.032	0.000	0.163
TMC 2	26.65	25.30	3.84	13.10	0.413	0.498	0.511	0.068
TMC 3	27.35	27.15	2.99	9.75	0.364	0.330	0.155	0.524
TMC 4	31.10	30.30	2.75	12.55	0.099	0.045	0.055	0.143
TMC 5	23.30	24.45	3.10	13.60	0.650	0.575	0.202	0.000
TMC 6	26.00	25.10	2.97	9.65	0.459	0.516	0.147	0.537
TMC 7	30.05	30.80	2.73	10.15	0.173	0.000	0.048	0.469
TMC 8	25.85	24.80	3.19	12.65	0.470	0.543	0.242	0.129
TMC 9	29.60	26.55	3.17	8.45	0.205	0.385	0.229	0.701
TMC 10	26.30	27.80	2.87	8.20	0.438	0.271	0.106	0.735
TMC 11	30.35	29.60	3.02	10.90	0.152	0.109	0.167	0.367
TMC 12	23.20	25.00	3.87	12.65	0.657	0.525	0.526	0.129
TMC 13	29.80	25.85	3.25	9.75	0.191	0.448	0.266	0.524
TMC 14	28.20	29.45	2.98	11.30	0.304	0.122	0.150	0.313
TMC 15	25.85	24.55	3.64	13.20	0.470	0.566	0.430	0.054
TMC 16	23.85	22.10	3.36	9.35	0.611	0.787	0.310	0.578
TMC 17	32.50	27.40	3.34	11.65	0.000	0.308	0.302	0.265
TMC 18	26.15	25.00	3.73	12.30	0.449	0.525	0.467	0.177
TMC 19	23.85	21.50	3.76	12.90	0.611	0.842	0.479	0.095
TMC 20	24.55	26.70	3.14	7.65	0.562	0.371	0.220	0.810
TMC 21	27.15	26.95	3.05	10.65	0.378	0.348	0.183	0.401
TMC 22	18.35	19.75	4.18	10.30	1.000	1.000	0.653	0.449
TMC 23	22.25	22.75	4.06	6.70	0.724	0.729	0.606	0.939
TMC 24	24.35	26.30	3.35	8.40	0.576	0.407	0.307	0.707
TMC 25	20.05	20.00	5.00	11.25	0.880	0.977	1.000	0.320
TMC 26	20.55	24.05	3.72	6.25	0.845	0.611	0.462	1.000
TMC 27	23.95	27.25	3.88	7.25	0.604	0.321	0.529	0.864

The Grey relation coefficients (GRC) and Grey relational grades (GRG) are computed using Eq 4.2 and Eq 4.3, respectively for geopolymerisation factors and the same are presented in Table 5.16, Table 5.17 and Table 5.18 for 14, 28 and 56 days of curing, respectively. The response table for the Grey relational grade for various geopolymerisation factors at various curing ages is presented in Table 5.19. Similarly, for pelletisation factors Grey relation coefficients and Grey relational grades are calculated and presented in Table 5.20. The response table for the Grey relational grade for various pelletization is presented in Table 5.21. Further, The Grey relation coefficients and Grey relational grades are computed for combined factors are presented in Table 5.22, Table 5.23 and Table 5.24 for 14, 28 and 56 days of curing respectively. The response table for the Grey relational grade for various geopolymerisation factors at various curing ages is presented in Table 5.25. The detailed discussion on the grades with respect to factors are discussed as follows.

Table 5.16: GRC and GRG with respect to geopolymerisation factors for 14 days

Trial runs	**Δ_{0i}**				**Grey relation coefficients**				**GRG**
	Aggregate impact value	**Water absorption**	**Aggregates crushing strength**	**Aggregate crushing value**	**Aggregate impact value**	**Water absorption**	**Aggregates crushing strength**	**Aggregate crushing value**	**γ_i**
TMG 1	0.800	0.737	1.000	1.000	0.385	0.404	0.333	0.333	0.364
TMG 2	0.396	1.000	0.536	0.633	0.558	0.333	0.483	0.441	0.454
TMG 3	1.000	0.355	0.750	0.390	0.333	0.585	0.400	0.562	0.470
TMG 4	0.792	0.461	0.975	0.482	0.387	0.521	0.339	0.509	0.439
TMG 5	0.608	0.697	0.906	0.741	0.451	0.418	0.356	0.403	0.407
TMG 6	0.000	0.789	0.000	0.095	1.000	0.388	1.000	0.841	0.807
TMG 7	0.179	0.605	0.504	0.410	0.736	0.452	0.498	0.549	0.559
TMG 8	0.388	0.000	0.581	0.000	0.563	1.000	0.463	1.000	0.756
TMG 9	0.533	0.434	0.570	0.472	0.484	0.535	0.467	0.515	0.500

Table 5.17: GRC and GRG with respect to geopolymerisation factors for 28 days

Trial runs	Δ_{0i}				Grey relation coefficients				GRG
	Aggregate impact value	Water absorption	Aggregates crushing strength	Aggregate crushing value	Aggregate impact value	Water absorption	Aggregates crushing strength	Aggregate crushing value	γ_i
TMG 1	1.000	0.907	1.000	1.000	0.333	0.355	0.333	0.333	0.339
TMG 2	0.504	1.000	0.633	0.861	0.498	0.333	0.441	0.367	0.410
TMG 3	0.907	0.473	0.615	0.530	0.355	0.514	0.449	0.485	0.451
TMG 4	0.788	0.612	0.729	0.650	0.388	0.449	0.407	0.435	0.420
TMG 5	0.681	0.891	0.715	0.820	0.423	0.359	0.411	0.379	0.393
TMG 6	0.173	0.953	0.000	0.079	0.743	0.344	1.000	0.864	0.738
TMG 7	0.314	0.984	0.405	0.404	0.614	0.337	0.553	0.553	0.514
TMG 8	0.000	0.000	0.306	0.000	1.000	1.000	0.620	1.000	0.905
TMG 9	0.181	0.597	0.585	0.331	0.734	0.456	0.461	0.602	0.563

Table 5.18: GRC and GRG with respect to geopolymerisation factors for 56 days

Trial runs	Δ_{0i}				Grey relation coefficients				GRG
	Aggregate impact value	Water absorption	Aggregates crushing strength	Aggregate crushing value	Aggregate impact value	Water absorption	Aggregates crushing strength	Aggregate crushing value	γ_i
TMG 1	1.000	0.924	1.000	1.000	0.333	0.351	0.333	0.333	0.338
TMG 2	0.460	0.907	0.640	0.976	0.521	0.355	0.439	0.339	0.413
TMG 3	0.772	0.449	0.600	0.581	0.393	0.527	0.454	0.463	0.459
TMG 4	0.772	0.576	0.735	0.831	0.393	0.465	0.405	0.376	0.410
TMG 5	0.713	0.856	0.557	0.887	0.412	0.369	0.473	0.360	0.404
TMG 6	0.241	1.000	0.000	0.008	0.675	0.333	1.000	0.984	0.748
TMG 7	0.418	0.949	0.312	0.435	0.545	0.345	0.615	0.534	0.510
TMG 8	0.000	0.000	0.225	0.000	1.000	1.000	0.690	1.000	0.923
TMG 9	0.354	0.559	0.489	0.190	0.585	0.472	0.505	0.725	0.572

Table 5.19: Response table for Grey relational grade for various ages with respect to geopolymerisation factors in detailed study

Factors	**Curing ages**	**Grey relational grade**			**Maximum value – minimum value**	**Rank**
		Level 1	**Level 2**	**Level 3**		
Na_2O Content (%)	**14 days**	0.429	0.551	0.605	0.176	3
SiO_2/Na_2O		0.454	0.539	0.592	0.138	4
Water Content (%)		0.642	0.464	0.479	0.178	2
Curing Regime		0.424	0.607	0.555	0.183	1
Na_2O Content (%)	**28 days**	0.400	0.517	0.661	0.261	1
SiO_2/Na_2O		0.424	0.569	0.584	0.160	3
Water Content (%)		0.661	0.464	0.453	0.208	2
Curing Regime		0.432	0.554	0.592	0.160	3
Na_2O Content (%)	**56 days**	0.403	0.520	0.668	0.265	1
SiO_2/Na_2O		0.419	0.580	0.593	0.174	3
Water Content (%)		0.669	0.465	0.458	0.212	2
Curing Regime		0.438	0.557	0.597	0.159	4

It is clear from the data presented in the Table 5.19 that grey relation grade is highest at level 3 for Na_2O content, level 3 for SiO_2/Na_2O, level 1 for water content and level 2 for curing regime at 14 days. However, at 28 days and 56 days, the Grey relation grade remains the same for Na_2O content, SiO_2/Na_2O and water content. Whereas the curing regime is changed from level 2 to level 3. Thus, is it understood that the overall engineering properties of the aggregates produced with high Na_2O content, high SiO_2/Na_2O ratio, low water content and heat cured showed better performance at 14 days. However, aggregates produced with similar proportion and solution cured showed better engineering properties at 28 days and 56 days.

The higher the difference between maximum and minimum values of Grey relational grade levels indicates the high level significance of the factor. It is ranked according to the higher differences in the values of Grey relational grade and is presented in the Table 5.19. It can be observed from the Table that at 14 days of curing age pelletized fly ash aggregates is getting influenced in the order, first by curing regime (Rank 1), water content (Rank 2), Na_2O content (Rank 3) and followed by the SiO_2/Na_2O ratio (Rank 4). However, at the curing age of 28 and 56 days, fly ash aggregates are highly influenced in the order, first by Na_2O content (Rank 1), water content (Rank 2), SiO_2/Na_2O ratio (Rank 3) and followed by curing regime (Rank 4).

This can be attributed to the fact that the properties of fly ash aggregates produced with alkaline binder are very much sensitive to the factors of the geopolymerisation. It is to be noted that from the above investigation Na_2O content and curing regime plays a prominent role in the production, in which Na_2O content and heat curing of aggregates will improve the engineering properties at an early age, because of the faster rate of the degree of polymerisation subjected to a higher temperature. However, the solution curing of pelletized fly ash aggregates has improved overall engineering properties of aggregates at 56 days.

Table 5.20: GRC and GRG with respect to pelletization factors

Trial runs	**Δ_{0i}**				**Grey relational coefficients**				**GRG**
	Aggregate impact value	**Aggregate crushing value**	**Aggregate crushing strength**	**Water absorption**	**Aggregate impact value**	**Aggregate crushing value**	**Aggregate crushing strength**	**Water absorption**	**y_i**
TMP 1	1.000	1.000	1.000	0.682	0.333	0.333	0.333	0.423	0.356
TMP 2	0.911	0.234	0.408	0.136	0.354	0.681	0.551	0.786	0.593
TMP 3	0.222	0.000	0.309	0.818	0.692	1.000	0.618	0.379	0.672
TMP 4	0.244	0.291	0.428	0.364	0.672	0.632	0.539	0.579	0.605
TMP 5	0.267	0.177	0.000	0.000	0.652	0.738	1.000	1.000	0.848
TMP 6	0.844	0.688	0.335	1.000	0.372	0.421	0.599	0.333	0.431
TMP 7	0.000	0.163	0.339	0.182	1.000	0.754	0.596	0.733	0.771
TMP 8	0.467	0.461	0.097	0.136	0.517	0.520	0.837	0.786	0.665
TMP 9	0.778	0.369	0.481	0.182	0.391	0.576	0.510	0.733	0.552

Table 5.21: Response table for Grey relational grade with respect to pelletization factors in detailed study

Factors	Mean Grey relational grade			Maximum value – minimum value	Rank
	Level 1	Level 2	Level 3		
Speed of pelletizing disc	0.540	0.628	0.663	0.122	3
Angle of pelletizing disc	0.577	0.702	0.552	0.150	2
Water content	0.484	0.584	0.764	0.280	1
Duration of pelletization	0.585	0.598	0.648	0.062	4

It can be noted from Table 5.21 that speed of pelletizing disc, water content and duration of pelletization scores the highest GRG at Level 3, whereas at Level 2 angle of pelletizing disc scores the best. This gives the understanding that optimized engineering properties of produced fly ash aggregates can be obtained by having high speed, an angle of 45 °, water content of 21% and with longer duration of pelletization.

Table 5.21 also represents values of GRG levels obtained from the difference of maximum and minimum values of the factors of pelletization. This helps in determining the order of influence for the factors responsible for pelletization. It can be observed from the table that water content found to be the most influencing (Rank 1) followed by angle of pelletizing disc (Rank 2), speed of pelletizing disc (Rank 3) and duration of pelletization (Rank 4).

Table 5.22: GRC and GRG with respect to combined factors for 14 days

Trial runs	Δ_{0i}				Grey relation coefficients				GRG
	Aggregate impact value	Aggregate crushing value	Aggregates crushing strength	Water absorption	Aggregate impact value	Aggregate crushing value	Aggregates crushing strength	Water absorption	γi
TMC 1	1.000	0.964	0.900	0.871	0.333	0.342	0.357	0.365	0.349
TMC 2	0.484	0.486	0.492	0.765	0.508	0.507	0.504	0.395	0.479
TMC 3	0.764	0.803	0.802	0.588	0.396	0.384	0.384	0.459	0.406
TMC 4	0.836	0.968	1.000	0.671	0.374	0.341	0.333	0.427	0.369
TMC 5	0.305	0.462	0.679	0.835	0.621	0.520	0.424	0.374	0.485
TMC 6	0.610	0.679	0.864	0.612	0.450	0.424	0.367	0.450	0.423

Trial runs	Δ_{0i}				Grey relation coefficients				GRG
	Aggregate impact value	Aggregate crushing value	Aggregates crushing strength	Water absorption	Aggregate impact value	Aggregate crushing value	Aggregates crushing strength	Water absorption	γi
TMC 7	0.928	1.000	0.940	0.412	0.350	0.333	0.347	0.548	0.395
TMC 8	0.522	0.558	0.655	0.800	0.489	0.472	0.433	0.385	0.445
TMC 9	0.871	0.851	0.831	0.271	0.365	0.370	0.376	0.649	0.440
TMC 10	0.654	0.755	0.921	0.412	0.433	0.398	0.352	0.548	0.433
TMC 11	0.821	0.948	0.903	0.647	0.379	0.345	0.356	0.436	0.379
TMC 12	0.311	0.414	0.388	0.847	0.616	0.547	0.563	0.371	0.524
TMC 13	0.906	0.787	0.826	0.400	0.356	0.388	0.377	0.556	0.419
TMC 14	0.862	0.964	0.871	0.529	0.367	0.342	0.365	0.486	0.390
TMC 15	0.481	0.490	0.571	1.000	0.510	0.505	0.467	0.333	0.454
TMC 16	0.664	0.478	0.796	0.353	0.430	0.511	0.386	0.586	0.478
TMC 17	0.761	0.791	0.840	0.624	0.397	0.387	0.373	0.445	0.401
TMC 18	0.481	0.530	0.517	0.753	0.510	0.485	0.491	0.399	0.471
TMC 19	0.283	0.205	0.403	0.671	0.639	0.709	0.554	0.427	0.582
TMC 20	0.604	0.835	0.823	0.388	0.453	0.374	0.378	0.563	0.442
TMC 21	0.695	0.739	0.834	0.553	0.418	0.404	0.375	0.475	0.418
TMC 22	0.000	0.000	0.395	0.612	1.000	1.000	0.559	0.450	0.752
TMC 23	0.406	0.667	0.504	0.176	0.552	0.429	0.498	0.739	0.554
TMC 24	0.619	0.795	0.806	0.212	0.447	0.386	0.383	0.702	0.479
TMC 25	0.075	0.129	0.000	0.765	0.869	0.796	1.000	0.395	0.765
TMC 26	0.538	0.695	0.727	0.000	0.482	0.418	0.407	1.000	0.577
TMC 27	0.645	0.964	0.715	0.247	0.437	0.342	0.412	0.669	0.465

Table 5.23: GRC and GRG with respect to combined factors for 28 days

Trial runs	Δ_{0i}				Grey relation coefficients				GRG
	Aggregate impact value	Aggregate crushing value	Aggregates crushing strength	Water absorption	Aggregate impact value	Aggregate crushing value	Aggregates crushing strength	Water absorption	γi
TMC 1	0.868	0.942	0.908	0.833	0.366	0.347	0.355	0.375	0.361
TMC 2	0.569	0.398	0.525	0.883	0.468	0.557	0.488	0.362	0.468
TMC 3	0.732	0.606	0.761	0.667	0.406	0.452	0.397	0.429	0.421
TMC 4	1.000	0.851	1.000	0.778	0.333	0.370	0.333	0.391	0.357
TMC 5	0.363	0.407	0.691	1.000	0.580	0.551	0.420	0.333	0.471
TMC 6	0.702	0.527	0.912	0.654	0.416	0.487	0.354	0.433	0.423
TMC 7	0.725	1.000	0.905	0.593	0.408	0.333	0.356	0.458	0.389
TMC 8	0.580	0.515	0.717	0.920	0.463	0.493	0.411	0.352	0.430
TMC 9	0.892	0.618	0.824	0.475	0.359	0.447	0.378	0.513	0.424
TMC 10	0.597	0.660	0.886	0.370	0.456	0.431	0.361	0.574	0.456
TMC 11	0.902	0.959	0.734	0.531	0.357	0.343	0.405	0.485	0.397
TMC 12	0.329	0.382	0.446	0.846	0.603	0.567	0.528	0.372	0.518
TMC 13	0.817	0.515	0.742	0.549	0.380	0.493	0.402	0.476	0.438
TMC 14	0.749	0.900	0.967	0.654	0.400	0.357	0.341	0.433	0.383
TMC 15	0.532	0.378	0.460	0.994	0.484	0.570	0.521	0.335	0.477
TMC 16	0.502	0.398	0.810	0.475	0.499	0.557	0.382	0.513	0.488
TMC 17	0.851	0.793	0.807	0.710	0.370	0.387	0.383	0.413	0.388
TMC 18	0.586	0.382	0.577	0.864	0.460	0.567	0.464	0.367	0.464
TMC 19	0.431	0.187	0.501	0.840	0.537	0.728	0.499	0.373	0.535
TMC 20	0.576	0.485	0.885	0.340	0.465	0.507	0.361	0.596	0.482
TMC 21	0.614	0.427	0.812	0.568	0.449	0.539	0.381	0.468	0.459
TMC 22	0.000	0.054	0.387	0.642	1.000	0.903	0.564	0.438	0.726
TMC 23	0.214	0.299	0.505	0.080	0.701	0.626	0.497	0.862	0.671
TMC 24	0.400	0.705	0.847	0.346	0.556	0.415	0.371	0.591	0.483
TMC 25	0.149	0.000	0.000	0.765	0.770	1.000	1.000	0.395	0.791
TMC 26	0.264	0.568	0.636	0.000	0.654	0.468	0.440	1.000	0.641
TMC 27	0.475	0.701	0.649	0.179	0.513	0.416	0.435	0.736	0.525

Table 5.24: GRC and GRG with respect to combined factors for 56 days

Trial runs	Δ_{0i}				Grey relation coefficients				GRG
	Aggregate impact value	Aggregate crushing value	Aggregate crushing strength	Water absorption	Aggregate impact value	Aggregate crushing value	Aggregate crushing strength	Water absorption	γ_i
TMC 1	0.894	0.968	1.000	0.837	0.359	0.341	0.333	0.374	0.352
TMC 2	0.587	0.502	0.489	0.932	0.460	0.499	0.506	0.349	0.453
TMC 3	0.636	0.670	0.845	0.476	0.440	0.427	0.372	0.512	0.438
TMC 4	0.901	0.955	0.945	0.857	0.357	0.344	0.346	0.368	0.354
TMC 5	0.350	0.425	0.798	1.000	0.588	0.540	0.385	0.333	0.462
TMC 6	0.541	0.484	0.853	0.463	0.480	0.508	0.370	0.519	0.469
TMC 7	0.827	1.000	0.952	0.531	0.377	0.333	0.344	0.485	0.385
TMC 8	0.530	0.457	0.758	0.871	0.485	0.522	0.397	0.365	0.442
TMC 9	0.795	0.615	0.771	0.299	0.386	0.448	0.394	0.626	0.463
TMC 10	0.562	0.729	0.894	0.265	0.471	0.407	0.359	0.653	0.472
TMC 11	0.848	0.891	0.833	0.633	0.371	0.359	0.375	0.441	0.387
TMC 12	0.343	0.475	0.474	0.871	0.593	0.513	0.513	0.365	0.496
TMC 13	0.809	0.552	0.734	0.476	0.382	0.475	0.405	0.512	0.444
TMC 14	0.696	0.878	0.850	0.687	0.418	0.363	0.370	0.421	0.393
TMC 15	0.530	0.434	0.570	0.946	0.485	0.535	0.467	0.346	0.458
TMC 16	0.389	0.213	0.690	0.422	0.563	0.702	0.420	0.542	0.557
TMC 17	1.000	0.692	0.698	0.735	0.333	0.419	0.417	0.405	0.394
TMC 18	0.551	0.475	0.533	0.823	0.476	0.513	0.484	0.378	0.463
TMC 19	0.389	0.158	0.521	0.905	0.563	0.759	0.490	0.356	0.542
TMC 20	0.438	0.629	0.780	0.190	0.533	0.443	0.390	0.724	0.523
TMC 21	0.622	0.652	0.817	0.599	0.446	0.434	0.380	0.455	0.429
TMC 22	0.000	0.000	0.347	0.551	1.000	1.000	0.590	0.476	0.766
TMC 23	0.276	0.271	0.394	0.061	0.645	0.648	0.559	0.891	0.686
TMC 24	0.424	0.593	0.693	0.293	0.541	0.458	0.419	0.631	0.512
TMC 25	0.120	0.023	0.000	0.680	0.806	0.957	1.000	0.424	0.797
TMC 26	0.155	0.389	0.538	0.000	0.763	0.562	0.482	1.000	0.702
TMC 27	0.396	0.679	0.471	0.136	0.558	0.424	0.515	0.786	0.571

Table 5.25: Response table for Grey relational grade for various ages with respect to combined factors in detailed study

Factors	Curing ages	Mean Grey relational grade			Maximum value – minimum value	Rank
		Level 1	Level 2	Level 3		
Na_2O Content (%)	14 days	0.421	0.439	0.559	0.138	2
SiO_2/Na_2O		0.446	0.481	0.493	0.047	6
Water Content (%)		0.486	0.491	0.443	0.048	4
Speed of pelletizing disc (RPM)		0.478	0.452	0.489	0.037	7
Angle of pelletizing disc (°)		0.505	0.461	0.453	0.051	3
Duration of pelletization (min)		0.460	0.456	0.504	0.048	4
Curing Regime		0.405	0.551	0.464	0.146	1
Na_2O Content (%)	28 days	0.416	0.445	0.590	0.175	1
SiO_2/Na_2O		0.455	0.492	0.504	0.049	4
Water Content (%)		0.497	0.509	0.446	0.062	3
Speed of pelletizing disc (RPM)		0.501	0.452	0.499	0.049	4
Angle of pelletizing disc (°)		0.504	0.481	0.466	0.038	6
Duration of pelletization (min)		0.484	0.469	0.499	0.030	7
Curing Regime		0.416	0.542	0.494	0.126	2
Na_2O Content (%)	56 days	0.424	0.451	0.614	0.190	1
SiO_2/Na_2O		0.455	0.505	0.530	0.076	3
Water Content (%)		0.513	0.523	0.453	0.070	4
Speed of pelletizing disc (RPM)		0.512	0.466	0.512	0.047	5
Angle of pelletizing disc (°)		0.519	0.493	0.478	0.041	6
Duration of pelletization (min)		0.491	0.482	0.517	0.034	7
Curing Regime		0.420	0.542	0.528	0.123	2

The GRG was computed for individual factors with respect to combined factors and the values of GRG are presented in Table 5.25. From table, it is observed that highest GRG among the geopolymerisation factors is 6% of Na_2O content (level 3) followed by curing regime – heat curing (level 2), 0.5 of SiO_2/Na_2O ratio (level 3) and among the pelletization factors highest GRG is observed for angle of pelletization disc (level 1) and followed by 18 minutes of duration of pelletization (level 3) and speed of pelletization disc at 50 RPM (level 3). Whereas water content is the important factors which plays important role in geopolymerisation and pelletization process, it is observed that higher GRG for 20% of water content (level 2).

The level of significance of each individual factors is estimated through difference of maximum and minimum values. It is ranked according to the higher differences in the values of Grey relational grade and is presented in the Table 5.25. From table, based on the rank given in the Grey relational analysis for the geopolymerisation factors, it clearly indicates that the curing regime and higher Na_2O content has major role in the aggregates production at early ages. However, the higher Na_2O content plays significant role in improving the engineering properties of the produced aggregates. Further, it is observed that curing regime rank is changed since solution curing of produced aggregates has improved overall engineering properties of aggregates at later ages. It is clear that in the fly ash aggregates production with alkaline solution as a binder, where Na_2O content, curing regime and SiO_2/Na_2O ratio play major role in the rate of geopolymerisation of fly ash. Further, the GRG for factors of pelletization such as speed of pelletizing disc, angle of pelletizing disc and duration of pelletization has relatively less influence as compared to the factors of geopolymerisation such as Na_2O content, SiO_2/Na_2O ratio, water content and curing regime in the production of artificial fly ash aggregates. However, water content plays a prominent and dual role in geopolymerisation process and pelletization which acts as a medium in both the process.

5.4 SCANNING ELECTRON MICROSCOPIC STUDIES ON THE AGGREGATES

The micrographs taken in secondary electron mode is presented in Figure 5.13 and Figure 5.14 for surface morphology and microstructure, respectively. It can be observed from the figure that samples studied through SEM have obtained relatively different surface morphology and microstructure. This can be attributed to different types of curing regimes. The surface SEM images (TM 1, TM 6 and TM 9) morphology shows the ambient cured samples which have a large amount of unreacted fly ash particle when compared to other micrograph images. On the other hand, the dense matrices were observed on the surface of the produced fly ash based coarse aggregates of the heat cured samples (TM 2, TM 4 and TM 8) and solution cured samples (TM 3, TM 5 and TM 7) (Al-Bakri 2011).

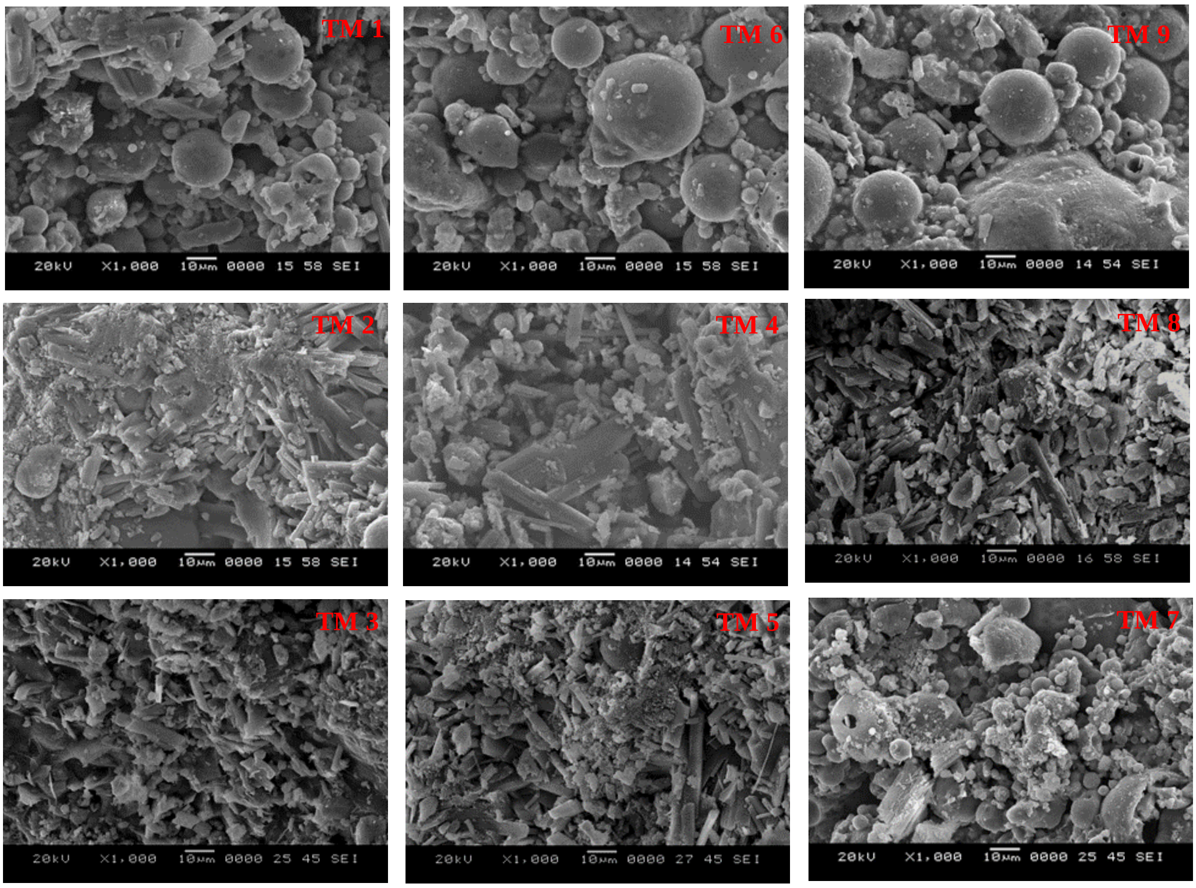


Figure 5.13: SEM images showing surface morphology of the produced fly ash aggregates

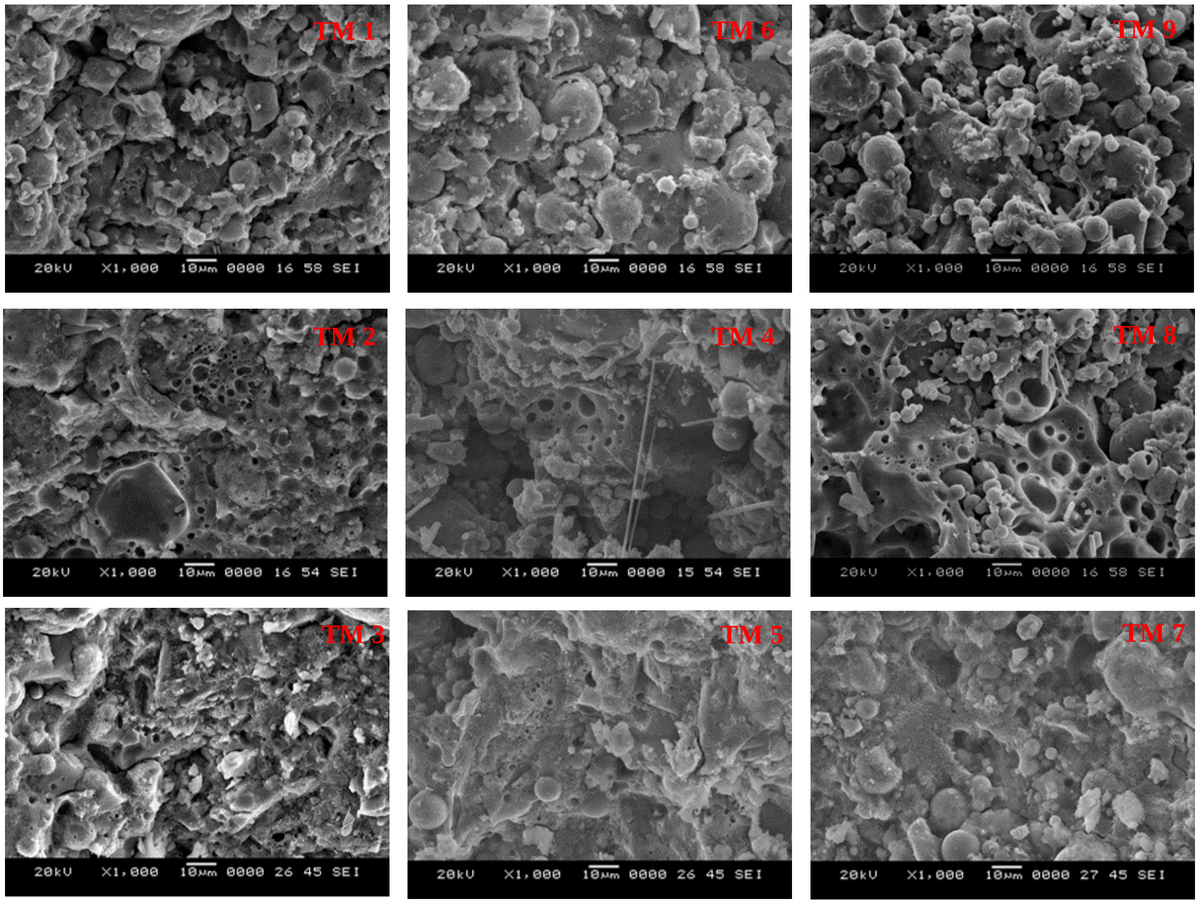


Figure 5.14: SEM images showing microstructure of the produced fly ash aggregates

5.5 LEACHING TEST ON THE AGGREGATES

Leaching test was conducted on produced fly ash aggregates to estimate the amount of NaOH leaching. The leachate sample is titrated against the prepared hydrochloric acid with methyl orange as indicator. Titration value is noted down when the solution turns red and the amount of NaOH present in leachate solution is estimated by equating number of moles. Experimental test results for the NaOH leaching test on produced fly ash aggregates are presented in Figure 5.15 with respect to Na_2O concentration. It was found that leaching is minimum at 5% Na_2O concentration irrespective of curing type. Further addition of Na_2O in the production phase would lead to an increase in the NaOH leaching.

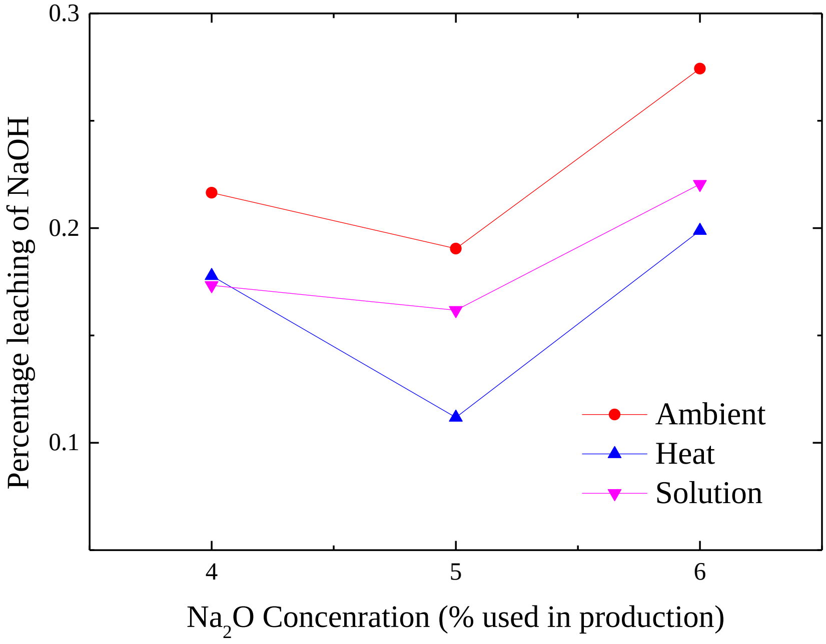


Figure 5.15: Percentage leaching of NaOH from produced fly ash aggregates

5.6 LARGE SCALE PRODUCTION OF AGGREGATES

From the present investigation, the factors influencing the production of fly ash based coarse aggregates were analysed and the optimized factors which are suitable for the large scale production are presented in Table 5.26. The production process was studied well in the laboratory, which in turn helped to study the effect of the optimised factors of pelletization in the production of fly ash based coarse aggregates in an industrial scale pelletizer. Industrial scale laboratory study was carried out in the Kudremukh Iron Ore Company Ltd., (KIOCL), Mangalore, India. An industrial scale disc pelletizer of size 800 mm disc diameter and 180 mm depth was employed for this study.

Table 5.26: Factors selected for large scale production of aggregates

Factors	**Lab scale study**	**Industrial scale study**
Na_2O content	5 %	5 %
Si_2O/ Na_2O ratio	0.3	0.3
Curing regime	Heat curing	Heat curing
Water content	20 %	20 %
Angle of pelletization	45 °	45 °

Factors	Lab scale study	Industrial scale study
Speed of pelletization	40 RPM	13 - 15 RPM
Duration of pelletization	15 Minutes	15 Minutes
Curing period	28 days	28 days

The properties of natural coarse aggregates and artificially produced fly ash based coarse aggregate at laboratory and industry were characterised as per IS standards as explained in the section 3.5 and the same is presented in Table 5.27.

Table 5.27: Properties of natural coarse aggregates, fly ash based coarse aggregates produced at laboratory and industrial scale

Properties	Natural coarse aggregates	Laboratory Scale Produced Aggregate	Industrial Scale Produced Aggregate
Specific gravity	2.72	1.95	1.95
Aggregate impact value (%)	19	23.15	23.60
Aggregate crushing value (%)	24.7	26.75	27.30
Crushing strength of individual aggregates (MPa)	-	4.20	3.80
Water absorption (%)	0.5	10.10	9.80

Figure 5.16: Laboratory scale and industrial scale produced fly ash based coarse aggregates

CHAPTER 6

PERFORMANCE OF FLY ASH BASED COARSE AGGREGATES IN CONCRETE

6.1 GENERAL

This chapter is focused on the study of different concrete mixes which are produced with conventional ingredients and artificial produced fly ash based coarse aggregates which are used in this study. The concrete mixes need careful selection of materials as the type, quality and the mix proportions have a critical influence on the properties of resulting concrete mixes. The effect of alternative materials used in this work in the different trial mixes are discussed in this chapter along with the mechanical and durability test results.

6.2 MECHANICAL PROPERTIES

6.2.1 Compressive strength

The compressive strength of the concrete specimens, after various water curing ages were tested and results are presented in the Figure 6.1. It can be observed that the concrete produced with complete natural aggregates has achieved the compressive strength of 48.33 MPa (M0) at 28 days. Whereas, the compressive strength of concrete produced with partial replacement of natural coarse aggregate with fly ash based coarse aggregate has reduced with the increase in the replacement level and it is observed from the figure that compressive strength results for the mixes from M1 to M5, has reduction in compressive strength up to 28.3% with respect to control mix M0 at 28 days. However, it is observed that increase up to 30% of artificial fly ash based coarse aggregate in concrete has compressive strength more than 40 MPa. Further, increase in the fly ash based coarse aggregates in concrete, the compressive strength is reduced to less than 40 MPa, which is less than the design strength.

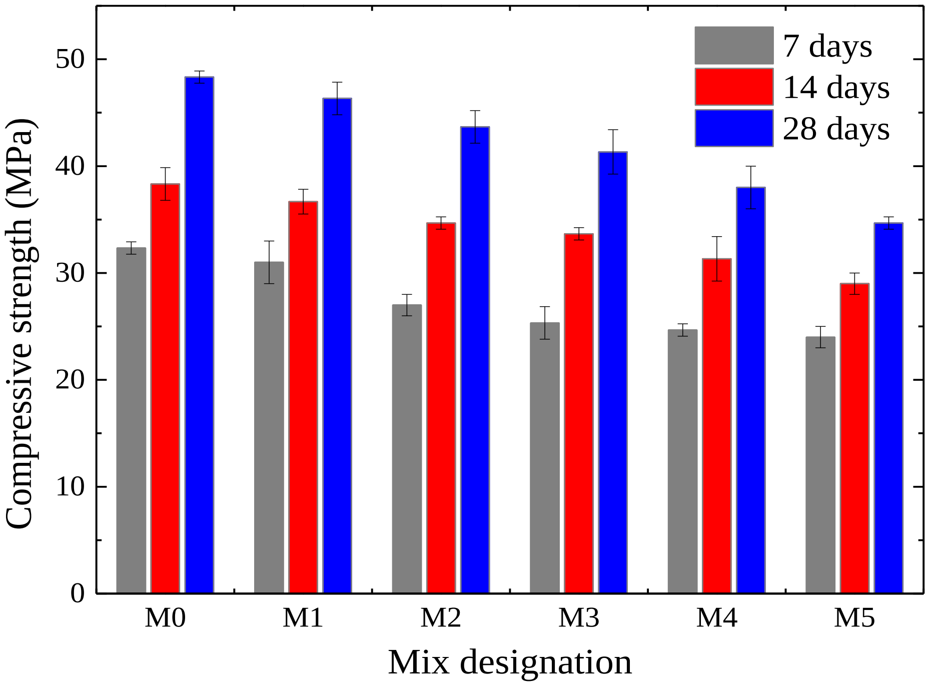


Figure 6.1: Compressive strength of different concrete mixes

6.2.2 Flexural strength

The flexural strength for all the concrete mixes were evaluated at 28 days and test results are presented in Figure 6.2. It is observed that, mix M0 has the flexural strength of 5.33 MPa. Whereas, the flexural strength of different mixes with increase in the replacement of fly ash based coarse aggregate with natural aggregates from M1 to M5, has reduction in strength from 2.4 % to 9.9 % with respect to control mix M0. However, as per IS: 456-2000, flexural strength of M40 grade concrete should obtained as 4.43 MPa from standard equation ($f_{cr} = 0.7\sqrt{f_{ck}}$). The flexural strength of all produced concrete mixes were more than the IS specifications.

6.2.3 Split tensile strength

For all the concrete mixes split tensile strength was carried out and test results are presented in Figure 6.3. It is observed that, mix M0 has the flexural strength of 3.40 MPa. The values for mix M1 is found to be marginally higher as compared to the control one and also among other mixes. Whereas, the flexural strength of different mixes with increase in the replacement of fly ash based coarse aggregate with natural aggregates from M2 to M5, has reduction in strength from 3.2 % to 15.6 % with respect to control mix M0.

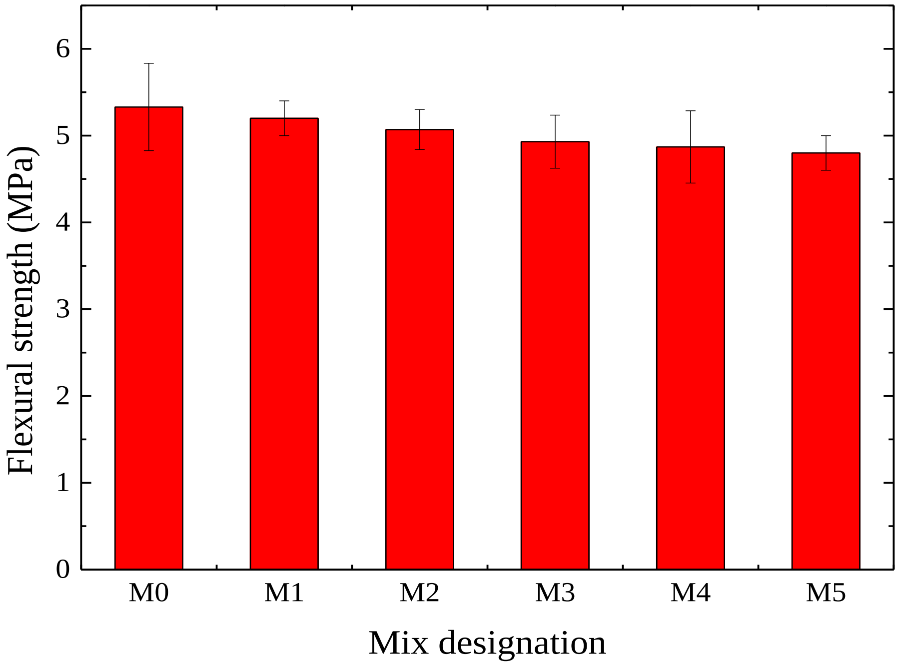


Figure 6.2: Flexural strength of different concrete mixes

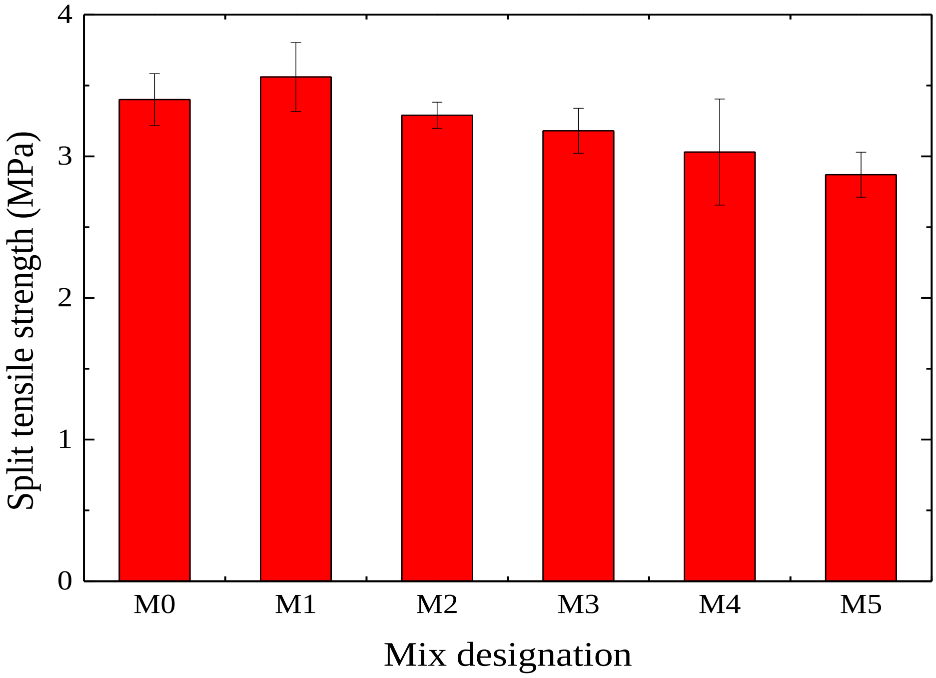


Figure 6.3: Split tensile strength of different concrete mixes

6.2.4 Bond strength

Bond strength of all the concrete mixes were evaluated at 28 days and test results are presented in Figure 6.4. It is observed that, bond strength of the concrete is increased by 3.62 % with 10 % of replacement artificially produced fly ash based coarse aggregates with natural aggregates. Further increase in the fly ash based coarse aggregate more than 10 %, bond strength of concrete has reduced significantly up to 28.59 %.

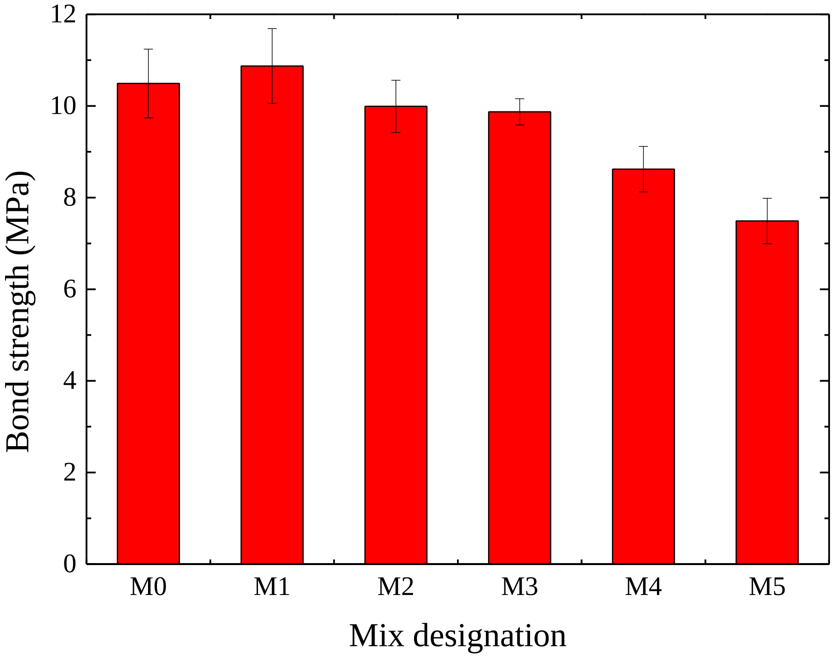


Figure 6.4: Bond strength of different concrete mixes

These strength variations in mechanical properties of the concrete may be attributed to type of aggregates used in production of concrete. In this study artificially produced fly ash based coarse aggregates are used, which are having spherical in shape and has a smooth surface.

6.3 WATER ABSORPTION AND RATE OF WATER ABSORPTION

The water absorption of the concrete produced with produced aggregates was found to be similar in the range of natrual coarse aggregate concrete, where the water absorption values were less then 4% for all the concrete mixes and same is presented in Figure 6.5. Further, the rate of water absorption of concrete mixes produced with different replacement level by artificially produced fly ash based coarse aggregate were evaluated and same is presented in Figure 6.6. The initial and secondary rate of water absorption are evaluated as per ASTM C1585 and these values are presented along with r square values in Table 5.1, it is observed from the table, that as the increase in the level of replacement of aggregates in the mixes the rate of water absorption also increases.

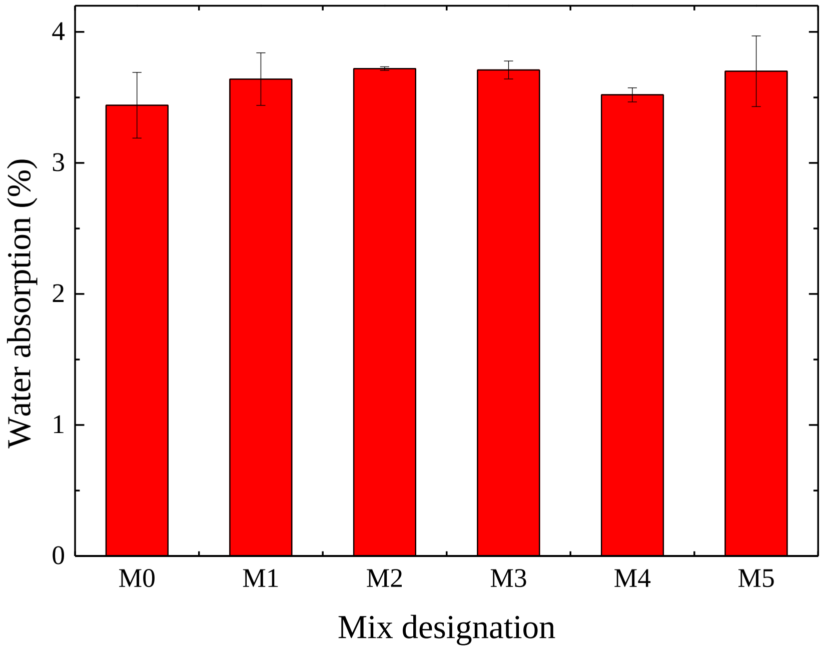


Figure 6.5: Water absorption of different concrete mixes

Table 6.1: Initial and secondary rate of water absorption for different concrete mixes

Mix designation	Rate of absorption		R square value	
	Initial	Secondary	Initial	Secondary
M0	4.38E-04	1.56E-04	0.98	0.99
M1	3.73E-04	1.24E-04	0.91	1.00
M2	6.69E-04	1.54E-04	0.98	0.99
M3	5.71E-04	1.74E-04	0.97	0.99
M4	5.69E-04	1.98E-04	0.96	0.99
M5	6.57E-04	1.82E-04	0.99	0.99

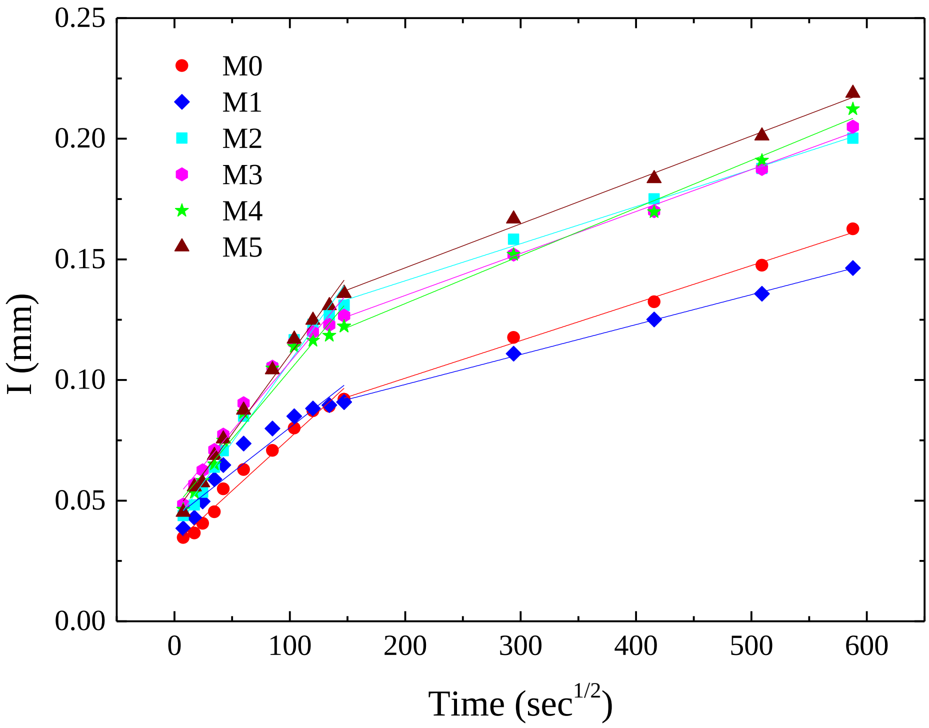


Figure 6.6: Rate of water absorption of different concrete mixes

6.4 DURABILITY TESTS

6.4.1 Rapid chloride ion penetration test

The different concrete mixes produced with the use of artificially produced fly ash coarse aggregate was evaluated for the durability test such as rapid chloride ion penetration test (RCPT) and test was conducted at 28 and 56 days. Experimental test results are presented in Figure 6.7. It is observed from the figure that the charge passed for all the concrete specimens are found to be in the range 380 - 450 Coulombs and 250 to 280 Coulombs at 28 and 56 days respectively. Further, it is noted that these values are less than 1000 Coulombs which is considered to be low category as per ASTM standards.

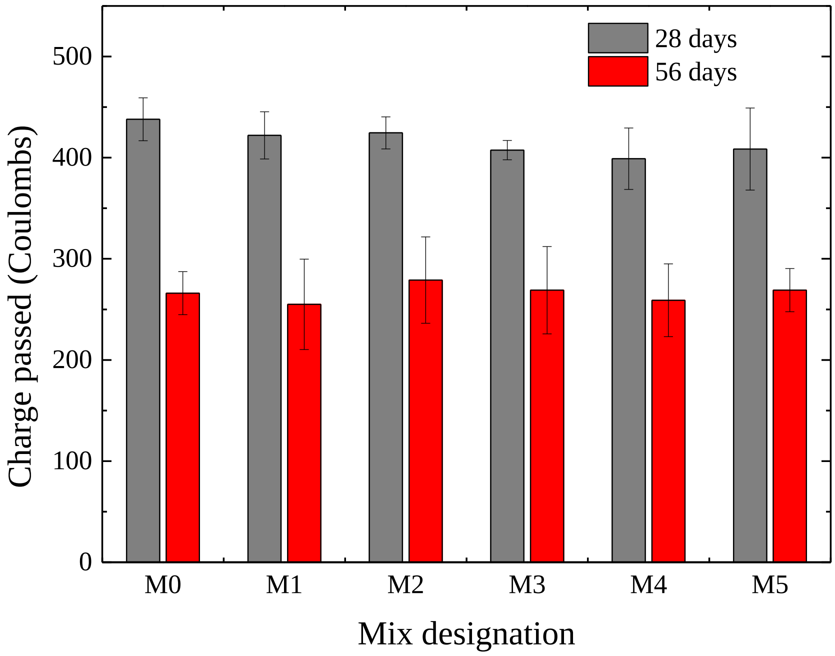


Figure 6.7: Rapid chloride ion penetration test values on different concrete mixes

6.4.2 Acid and sulphate exposure

Durability test like acid exposure (1% of sulphuric acid (H_2SO_4) solution) and sulphate exposure (5% sodium sulphate (Na_2SO_4) solution) were carried out on the 28 days cured concrete specimens for exposure period of 30 and 60 days. Tests were carried out at room temperature in a laboratory environment for the desired periods. Experimental test results with respect to percentage weight reduction and percentage strength reduction was evaluated at 30 days and 60 days for acid and sulphate chemical exposure conditions are presented in Figure 6.8 and Figure 6.9, respectively. It can be noted that the percentage weight change is less than 2 percent in both the exposure conditions. However, the percentage of compressive strength reduction of concrete mixes is found to be less than 6% and 12% at 30 and 60 days, respectively for sulphuric acid chemical exposure. For sulphate solution chemical exposure, the percentage of compressive strength reduction of concrete mixes is found to be less than 4% and 6% at 30 and 60 days, respectively.

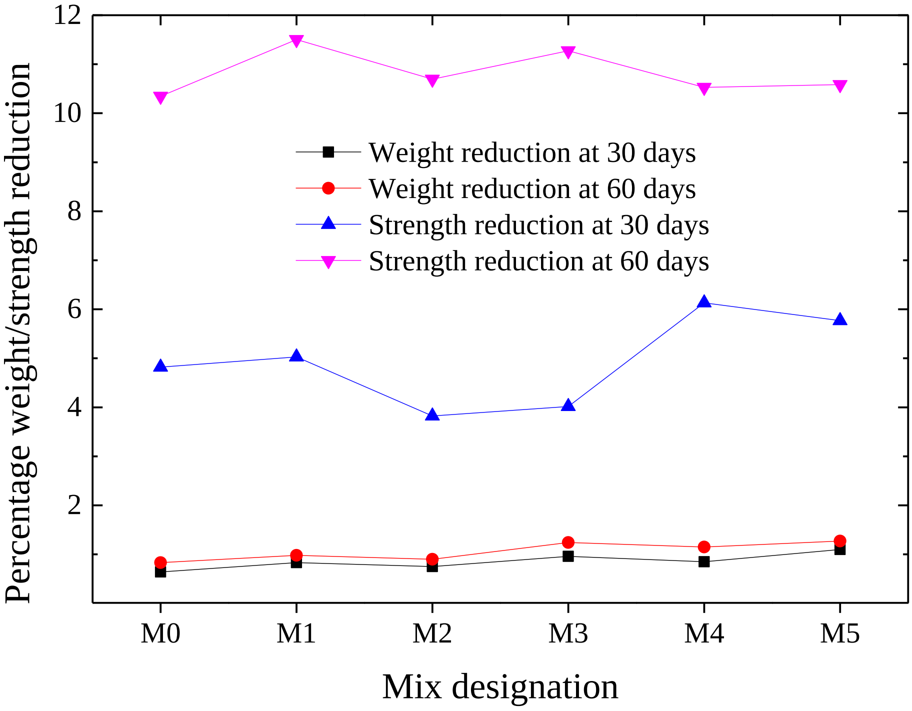


Figure 6.8: Durability test on concrete under acid exposure

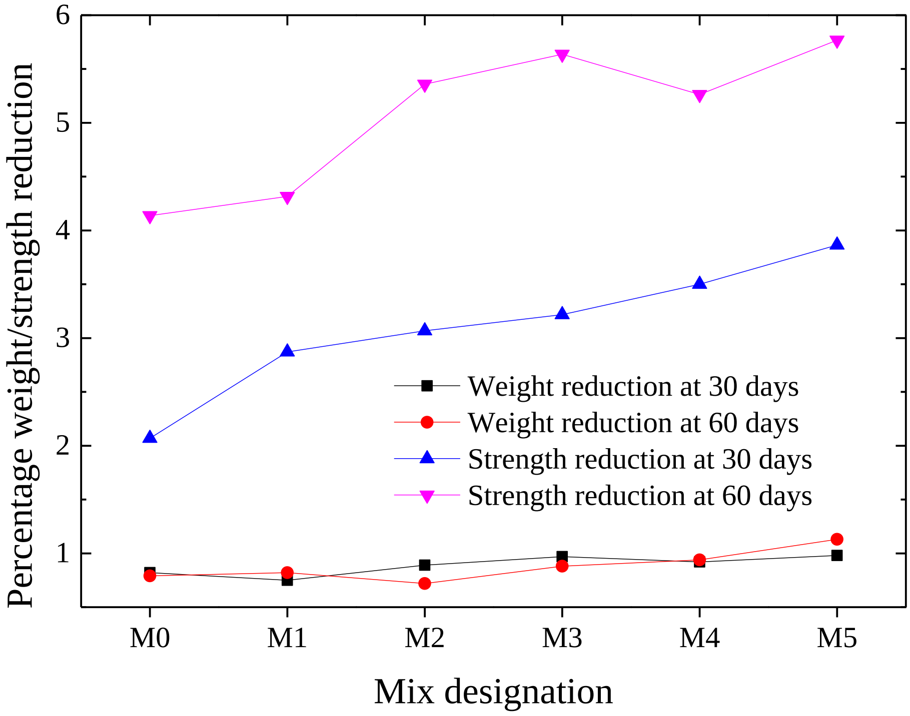


Figure 6.9: Durability test on concrete under sulphate exposure

6.5 VISUAL INSPECTION OF TESTED CONCRETE CUBES

After compression testing of concrete specimens, their failure patterns and surfaces were visually inspected. The distribution of aggregates across the fractured surfaces can be observed visually in Figure 6.10 that the aggregates distribution is uniform through the failed surface. The specimens generally failed in a standard pyramidal fracture shape and the same can be observed in the tested concrete which is presented in Figure 6.10. Also, it can be observed in Figure 6.10 (ii) presents failure pattern of concrete produced with artificially produced fly ash based coarse aggregates has failed through the aggregates and most of the aggregates are intact, which designates that aggregates are having good engineering properties. However, the concrete failed through the surface of aggregates may be due to the smooth surface and round shape of the produced fly ash aggregates (Figure 6.10 (ii)).

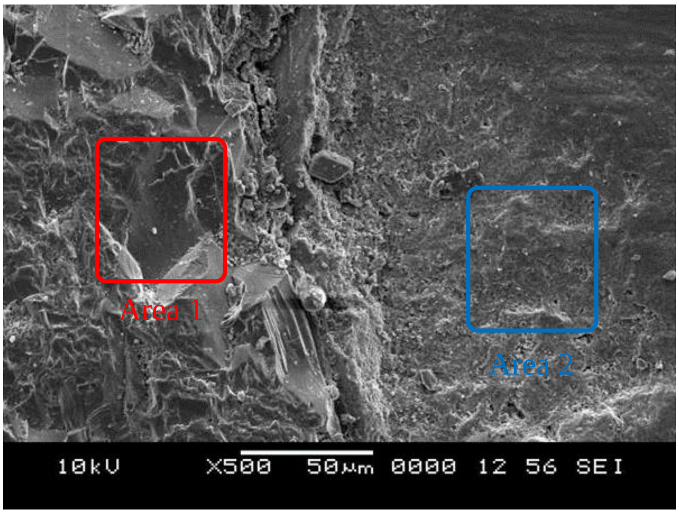
(i) Concrete - Natural aggregate

(ii) Concrete – Fly ash based coarse aggregate

Figure 6.10: Failure pattern of the concrete produced with a) Natural aggregates and b) Fly ash based coarse aggregates.

6.6 SCANNING ELECTRON MICROSCOPIC STUDIES ON CONCRETE

Concrete produced with partial replacement of fly ash aggregate with natural aggregates are studied with scanning electron microscopic (SEM) and energy dispersive spectrum (EDS) analysis on the interfacial zone between aggregate and paste. The SEM images and EDS results of interfacial zone between aggregates and cement paste for different concrete are presented in Figure 6.11 and Figure 6.12. From the Figure 6.11, it is observed that the EDS of the area 1 clearly shows the presence of predominant Si, Al and composition table shows the percentage atomic weight of Si, Al is significantly higher, which indicates the presence of a natural coarse aggregate. Similarly, in Figure 6.12, it can be noted that from area 1, Si and Al presence is predominant due the alumina-silicaous (fly ash) aggregate and same can be observed in the composition tables. However, in area 2 of Figure 6.11 and Figure 6.12, it is noted that presence Ca, Si, Al, Fe, Na which occur commonly in cement hydration products and same can be observed in the composition tables of area 2 shows that percentage atomic weight of Ca/Si ratio is 2.6 which indicates the presence of calcium hydroxide – cement hydration products (Escalante-Garcia et al 1999, Goudar et al 2019).



Element	Atomic %	
	Area 1	Area 2
Ca K	2.14	18.66
Si K	20.73	7.20
Al K	9.60	2.78
Na K	7.82	0.93
O K	53.59	56.81
Fe L	0.39	0.62
Mg K	0.44	0.95
K K	0.31	0.66
C K	4.98	11.40

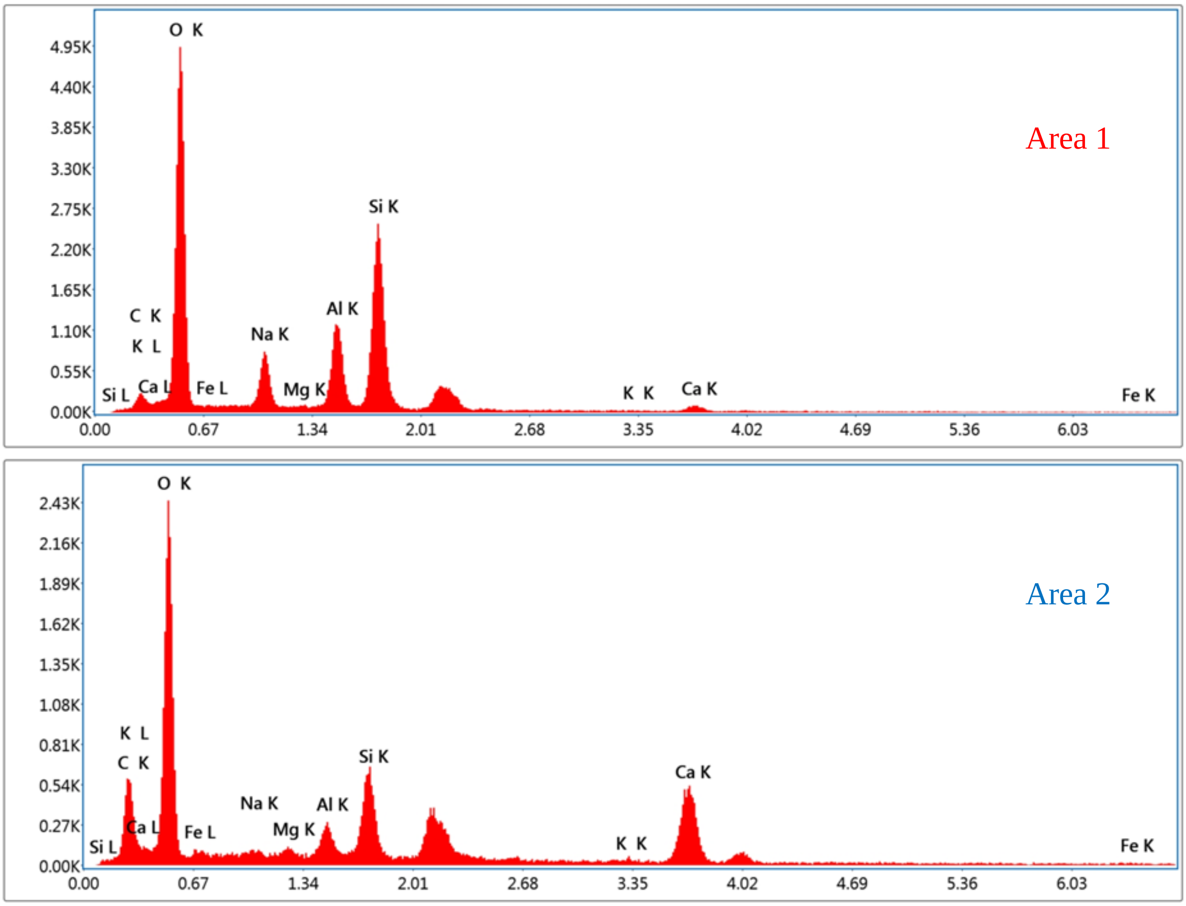
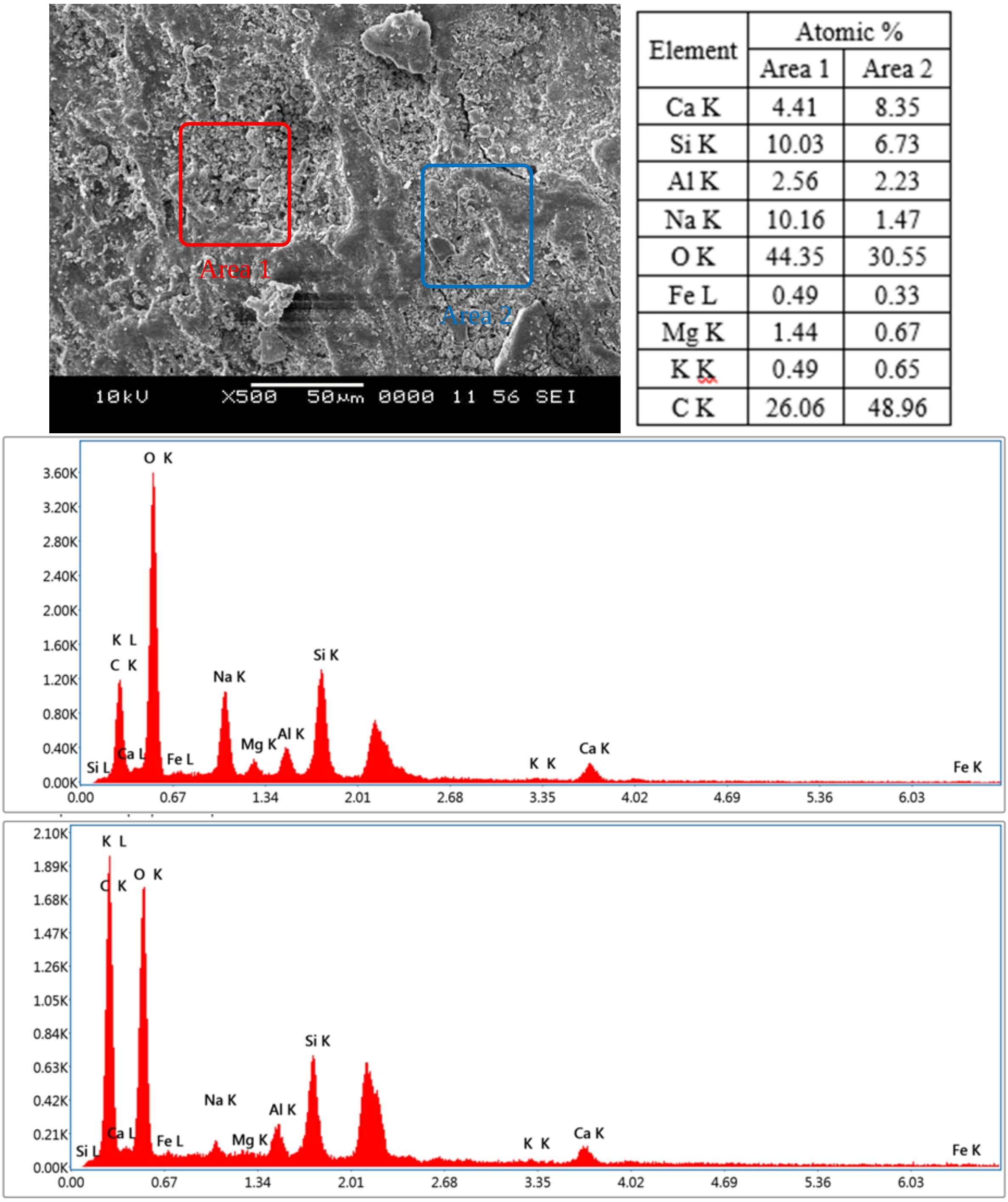


Figure 6.11: SEM and EDS of natural aggregate concrete



Element	Atomic %	
	Area 1	Area 2
Ca K	4.41	8.35
Si K	10.03	6.73
Al K	2.56	2.23
Na K	10.16	1.47
O K	44.35	30.55
Fe L	0.49	0.33
Mg K	1.44	0.67
K K	0.49	0.65
C K	26.06	48.96

Figure 6.12: SEM and EDS of fly ash based coarse aggregate concrete

CHAPTER 7

CONCLUSIONS AND SCOPE FOR FUTURE WORK

7.1 GENERAL

In this chapter major findings and conclusion are presented with respect to the study of different factors affecting the production of fly ash based coarse aggregates with alkaline solution as a binder. The produced aggregates were used in the production of concrete with appropriate mix design and trial mixes are carried out. Test results showed that the production of fly ash aggregates with alkaline solution and these aggregates in cement concrete can be produced. Further, scope of future work is also presented in this chapter.

7.2 CONCLUSIONS

The following conclusions are made based on the experimental investigations on the effect of different factors in the production and engineering properties of produced fly ash aggregates. It is observed that all the selected factors have shown significant effect either in the production stage or on the engineering properties and also its performance in the cement concrete with partial replacement of these produced aggregates with natural aggregates. The following section presents the conclusions from each of the phases of this research work.

Aggregate production and engineering properties of produced aggregates

The fly ash based coarse aggregates can effectively be produced with alkali solution as a binder.

The water content in the alkaline solution is an important factor that influences the efficiency of the pelletization process and particle size distribution of produced aggregates. Based on the production efficiency and particle size distribution of aggregates, the minimum water content required for the production of fly ash aggregates is found to be 20 % of weight of fly ash.

The average specific gravity of pelletized fly ash is found to be in the range of 1.95 to 2.01, which can be considered as lightweight aggregates irrespective type curing regime adopted.

The increase in the Na_2O content and SiO_2/Na_2O ratio has the significant influence on the engineering properties of pelletized fly ash aggregates. water content has shown a slightly negative and negligible effect on the engineering properties of pelletized fly ash aggregates.

Heat cuing of pelletized fly ash aggregates has improved the mechanical properties of aggregates at early ages and increase of the water absorption of aggregates. Whereas, the solution curing showed improved mechanical properties at later ages and it has significantly reduced the water absorption of the aggregates also.

The strength properties produced fly ash aggregates are mainly influenced by the water content followed by speed of pelletizing disc and angle of pelletizing disc. It can be concluded from the study that good quality of fly ash aggregates are produced with an optimum angle of 45°.

The engineering properties of the artificially produced fly ash based coarse aggregates are significantly influenced by the factors of geopolymerisation and pelletization. Statistically designed experiments showed that geopolymerisation factors significantly influenced the production and engineering properties of the pelletized fly ash aggregates as compared to pelletization factors.

Fly ash based coarse aggregate - cement concrete

Artificially produced fly ash based coarse aggregate were used in the production of concrete with partial replacement of natural aggregates and it is found that for the production of M40 grade concrete, up to 30% by its volume can be replaced effectively to produce durable concrete.

SEM and EDS analysis indicate that interfacial zone between aggregates and cement paste is uniform and with fewer pores. However, the strength of the fly ash based geopolymer aggregate concrete is slightly lower than control concrete, which can be attributed to smooth surface of aggregates and same can be observed in the visual inspection of the fractured surface of concrete.

7.3 SCOPE FOR FUTURE WORK

In present investigation, fly ash aggregates properties were attained at 28 to 56 days. However, the scope is there to attain the required properties of produced fly ash aggregates within 3 to 7 days.

Detailed investigation on the production of concrete is necessary to overcome the strength loss due to spherical shape and smooth surface of aggregates.

The use of these produced aggregates in the fly ash based geopolymer concrete and ground granulated blast furnace slag based alkali activated concrete can be taken up by the future generation researchers.

The economic analysis for the production of fly ash based coarse aggregates and the concrete production using these produced aggregates is very much needed.

Further, produced fly ash aggregates can be crushed into required sizes and graded for the specific applications which can be utilized as a sustainable alternative material in the construction industry. Research in this direction can be carried out.

The produced aggregates are having a smooth surface, further investigation can be carried out in the production of aggregates having rough surface.

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K N Shivaprasad and B B Das, “Effect of Duration of Heat Curing on the Artificially Produced Fly Ash Aggregates” Materials Science and Engineering, IOP publications vol. 431, no. 9, October 2018, 092010 (1-8).

K N Shivaprasad and B B Das “Determination of optimized geopolymerisation factors on the properties of pelletized fly ash aggregates” Construction and Building Materials, Elsevier, Vol. 163, February 2018, 428-437.

K N Shivaprasad and B B Das, “Influence of Alkali Binder Dosage on the Efficiency of Pelletization of Aggregates from Iron Ore Tailing and Fly ash.” International Journal of Engineering Research in Mechanical and Civil Engineering, ISSN (Online) 2456-1290, 2017, Vol 2, No 3, 388-392.

International Journals - In review

K N Shivaprasad and B B Das, “Influence of combined effect of pelletization and geopolymerisation on sustainable production of artificial fly ash aggregates”, (Communicated to international peer review journal).

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M. Roshan, **K N Shivaprasad**, B B Das, “Study on Mechanical Properties and Leaching of Heavy Metals in the Artificially Produced Fly Ash Aggregates”, Select Proceedings of ICSCBM 2018, Springer Nature Singapore Pte Ltd. 2019, A Book Chapter, Pages: 201-212.

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K N Shivaprasad, B B Das and S Krishnadas, “Effect of curing methods on the artificial production of fly ash aggregates” International Conference "Trending Moments & Steer Forces" - Civil Engineering Today, Don Bosco College of Engineering, Goa, November 2019.

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K N Shivaprasad and B B Das, “Effect of liquid to solid ratio on the fly ash based geopolymer: A review.” International Conference on Advances in Concrete Technology, Materials & Construction Practices, UKIERI project, June 2016, 331 – 338.

Achievements/Awards

Best presenter award to paper “Effect of Duration of Heat Curing on the Artificially Produced Fly Ash Aggregates” under the theme of Geopolymer” in the 14th International Conference on Concrete Engineering & Technology 2018 (CONCET 2018), University of Malaya, Kuala Lumpur, Malaysia, August 8 - 9, 2018.

Best Paper Award to “Effect of Liquid to Solid Ratio of Alkaline Activator to Cement Based Materials on the Efficiency of Geo-Polymerization, UKIERI International Conference on Advances in Concrete Technology, Materials & Construction Practices (CTMC2016), 22-24, June 2016.

OTHER PUBLICATIONS

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Sharath B P, **K N Shivaprasad**, Athikkal M M and B B Das, “Some Studies on Sustainable Utilization of Iron Ore Tailing (IOT) as Fine Aggregates in Fly Ash Based Geopolymer Mortar”, Materials Science and Engineering, IOP publications, vol. 431, no. 9, October 2018, 092013(1-8).

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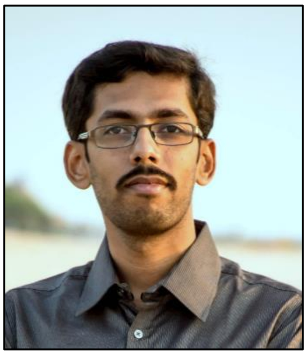

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WORK EXPERIENCE:

Organization	Post held	Duration
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Nagarjuna College of Engineering and Technology, Bangalore	Assistant Professor	Aug 2014 to Jul 2015
National Institute of Technology Karnataka, Surathkal, Mangalore	Research Scholar	Jul 2015 to Jul 2019
National Institute of Technology Karnataka, Surathkal, Mangalore	Assistant Lecture	Jul 2019 to Sept 2019
JSS science and technology university (formerly SJCE), Mysuru.	Assistant Professor	Sept 2019 to till date

RESEARCH PUBLICATIONS:

Type of Publication	No of Publications (Under Review)
International Journal Papers	8 (1)
International Conference	4
Book Chapters	4