A STUDY ON STRENGTH AND DURABILITY OF CEMENT MORTARS CONTAINING GRANITE POWDER

Submitted in

fulfilment of the requirements for the degree of

Doctor of Philosophy

by

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JAIPUR - 302017

January 2020

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This thesis is dedicated to

My Niece

VAISHALI,

PARENTS

and

"GOVIND DEVJI"

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This is to certify that the thesis entitled 'A Study on Strength and Durability of Cement Mortars Containing Granite Powder' being submitted by Lalit Kumar Gupta (2015RCE9035) is a bonafide research work carried out under my supervision and guidance in fulfilment of the requirement for the award of the degree of Doctor of Philosophy in the Department of Civil Engineering, Malaviya National Institute of Technology, Jaipur, India. The matter embodied in this thesis is original and has not been submitted to any other University or Institute for the award of any other degree.

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ACKNOWLEDGEMENT

I express my sincere gratitude to my supervisor Dr. Ashok Kumar Vyas, Professor of Department of Civil Engineering, MNIT for his unflinching support, valuable guidance, motivation and consistent encouragement throughout my research work. He will always remain a source of inspiration for me.

I take this opportunity to offer my thanks to Dr. A. B. Gupta, Professor, Department of Civil Engineering, MNIT for painstakingly identifying the areas where I can grow and achieve my goals with greater ease.

I also offer my thanks to Dr. Mahesh Kumar Jat, Professor and Head of the Department of Civil Engineering, MNIT; along with Prof. Gunwant Sharma, former Head of the Department of Civil Engineering, MNIT, for maintaining a conducive environment for all the research scholars and students so that we can pursue our academic and research goals without any hindrances.

I am grateful to my esteemed DREC members Dr. Rajesh Gupta, Dr. Pawan Kalla and Dr. Sandeep Shrivastava and the DPGC convenor Prof. Sanjay Mathur for providing me with valuable feedback at every opportunity presented to them. I also offer my thanks to Dr. Neha Shrivastava, Dr. Sanyam Dangayach, Dr, Manviri and Dr. Uma Shankar for their motivation.

I would acknowledge the support of Mr. Sita Ram Jat, Mr. Om Prakash Gared, Mr. Sadiq Ansari, Mr. Nitesh Kumar Sunaria, Mr. Anil Kumar Sharma, Mr. Rajesh Saxena and Mr. R. S. Mandolia for their technical assistance. I am also thankful to the staff of Material Research Centre for their backing in conducting the microstructural studies required for completion of this thesis.

I am also obligated towards my material suppliers, Mr. Abhishek Jain (Savita Scientific and Plastic Products, Jaipur), Ankur Speciality Gases and Technologies (Jaipur), and Rohit Traders (Jaipur); and experimental setup manufacturer, HEICO (New Delhi) for providing me with their best efforts to complete my experimental programme.

It is an honour to acknowledge the blessings of my mother Mrs. Santosh Devi and father Mr. Mahesh Kumar Gupta for their continuous support and motivation for the completion of my thesis. I am also humbled by the love shown by my elder brother Mr. Rakesh Kumar Gupta and sister - in - law Mrs. Asha Gupta towards me. I am also lucky to have countless prayers of my younger brother Mr. Manish Kumar Gupta and cousins Mr. Govind Gupta, Ms. Jagriti Gupta, Ms. Shruti Gupta and Mr. Mukund Gupta for the successful completion of my research work.

The contribution made by my teachers Prof. P.R. Sindhi, Sh. S.D. Thanvi, and Dr. D.K. Sharma to my life is also profound, who inspired me to take up this doctoral degree. So just thanking him will be an understatement. I also recognize how lucky I was to have an humble but

short company of Mr. Harshwardhan Singh Chouhan, Dr. Kunal Bisht, Mr. Ajay, Mr. Vishal Singhal, Mr. Sumit Mukund Choudhary, Mr. Kulviabhav Sharma, Mr. Rakesh Choudhary, Mr. Abhishek Jain, Mr. Rohit Yadav, Mr. Santosh, Mrs. Kusum Shekhwat, Mrs. Prathitha B. Gupta, Ms. Kanika Saxsena and Mr. Sandeep Singh Shekhawat who have been the best of friends whose influence in my life is more than just sublime during the difficult times.

I am thankful to my fellow research scholars, Dr. Sudarshan D. Kore, Dr. Aditya Choudhary, Dr. Pradeep Kumar Gautam, Dr. Aneesh Mathew, Dr. Shushendra Kumar Gupta, Mr. Priyamitra, Dr. Nishant Roy, Dr. Jitendra Kumar Yadav, Dr. Arnav Anuj Kasar, Dr. Salma Banu along with; post graduate students Mr. Rajendra Kumar Khyaliya, Mr. Vikash Kumar, Ms. Anjali Chawla, Mr. Vikas Babu, Mr. Manish Kumar Meena, Mr Mohit Vyas and Ms Apporva Mudhgal; and undergraduate students Mr. Rakesh Meena, Mr. Arnubh, Mr. Vivek, Ms. Pooja and Mr. Mudit.

I am more than just fortunate to have Mr. Phukraj Leelawat to assist me in my experimental ventures, without whom this journey would not have been as easy it seemed to be.

At the last I just want to express my thanks to my boss and very near to my heart Dr. K.I. Syed Ahmed Kabeer without whom this journey would never achieve this goal. I always keep in mind for entire life for his support during my work.

(Lalit Kumar Gupta)

ABSTRACT

Solid waste management is a big challenge in all around the world. Granite slurry generated from granite stone processing industry is a prime source of solid waste produced in Rajasthan. The generated slurry is indiscriminately dumped on vacant lands, river banks or forest areas. These slurry particles being fine enough are capable of filling pores of the soil, preventing water percolation and making the land futile. Construction industry requires a huge quantity of natural sand for the execution of all type of construction works. Since extraction of this material is associated with environmental degradation, governing authorities have imposed restriction on their usage. Such restrictions demand assessing alternative materials that can be used to replace conventional ones. Therefore this study aims to find an optimum utilizing ratio of granite waste generated from granite processing industries in production of cement mortars. This granite waste was intended to be used as a replacement of fine aggregate.

In this experimental study, granite powder (dried granite slurry) was used for the production of 1:4 and 1:6 cement mortar mixes. In order to improve performance parameters of mortars containing granite powder (GP), it was planned to use coarse sand (CS) of zone-II to negate the effect of excessive fines. Ratio of 60:40 and 70:30 of CS:GP satisfied gradation recommended by standards for plaster and masonry mortars. For 1:4 mortar proportion, the adhesive strength increased by 21% and 23% for CS30 and CS40 mixes, respectively as compared to control mortar (FS). Drying shrinkage of cementsand-granite mortar found to be similar with that of control mortar in 1:4 mix proportion but drying shrinkage in 1:6 proportion exceeded in the range of 20 to 60%. To understand the effect on microstructure of mortar with GP, microstructure study was also conducted by SEM, XRD, FTIR and TGA techniques. It can be concluded from SEM images that both CS30 and CS40 mortar mixes are more compact and denser than control mixes. On the basis of such outcomes of mechanical properties, same mortars were exposed to the adverse environmental condition and compared with outcome of conventional mortar. When the mortar specimens were subjected to 15 cycles of salt crystallization it was seen that the residual strength of all mixes of both series increase marginally due to absorbed salt (crystallized sodium sulphate). Filling of voids by GP also reduced the penetration of carbon di-oxide in mortars. On exposure of mortars to a 5% sulphuric acid medium, portlandite and katoite are converted into gypsum and ettringite, respectively which reduced adhesion between particles of mortars and results in 22% decline in compressive strength at 28 days exposure period. No significant variation in performance across all the mixes was observed when mortar mixes were subjected to 20 cycles of alternate wetting and drying. Hence, granite powder as partial substitute (30-40%) of sand in cement mortar mixes has no adverse effect on mechanical as well as durability properties of cement mortar.

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ASR Alkali silica reaction ASTM American Society for Testing and Materials BIS Bureau of Indian standard BS **British Standard** CaCl₂ Calcium chloride CaCO₃ Calcium carbonate / calcite CAH Calcium aluminate hydrate / katoite CaO Lime Ca(OH)₂ or Calcium hydroxide / Portlandite CH CASH Calcium aluminate silicate hydrate / gismondine CaSO₄.0.5H₂O Calcium sulphate hemihydrate / bassanite CaSO₄.2H₂O Calcium sulphate dihydrate / gypsum Carbon dioxide CO_2 CS Coarse river sand CSH Calcium silicate hydrate / tobermorite **CS30** Mortar with 30% granite powder and 70% coarse sand CS40 Mortar with 40% granite powder and 60% coarse sand C_3S Tri-calcium silicate 4CS30 1:4 mortar with 30% granite powder and 70% coarse sand 4CS40 1:4 mortar with 40% granite powder and 60% coarse sand 6CS30 1:6 mortar with 30% granite powder and 70% coarse sand 6CS40 1:6 mortar with 30% granite powder and 60% coarse sand C&D Construction & demolition FESEM / SEM Field emission scanning electron microscope FS Fine river sand FTIR Fourier transform infrared 4FS 1:4 mortar prepared with fine river sand only 6FS 1:6 mortar prepared with fine river sand only GCW Granite cutting waste Ground granulated blast furnace slag GGBFS GP Granite Powder H₂CO₃ Carbonic acid H_2SO_4 Sulphuric acid IS Indian Standard ISF **Imperial Smelting Furnace** LOI Loss on ignition $Mg(OH)_2$ Magnesium hydroxide / brucite

Abbreviations and Symbols

MGR	Marble granite waste
MSME	Ministry of Micro, Small and Medium Enterprises
NaCl	Sodium chloride
NaSO ₄	Anhydrous sodium sulphate / thenardite
NaSO ₄ .10H ₂ O	Hydrous sodium sulphate / mirabilite / Glauber's salt
$Na_2Ca(SO_4)_2$	Sodium calcium sulphate / glauberite
NaOH	Sodium hydroxide
OPC	Ordinary Portland cement
PET	Polyethylene terephthalate
PPC	Portland pozzolana cement
QD	Quarry dust
RGD	Red granite dust
RHA	Rice husk ash
SCC	Self-consolidating concrete
SCM	Supplementary cementitious material
SEM	Scanning electron microscope
TGA	Thermogravimetric analysis
UPV	Ultrasonic pulse velocity
XRD	X- ray diffraction
XRF	X- ray fluorescence

CHAPTER 1

INTRODUCTION

1.1 General

Development of society demands the urbanisation. Urbanisation requires a good infrastructure to achieve economic growth. In order to achieve the economic growth a rapid industrialization is required in society. The industrialization promotes the consumption of natural resources and generates waste as by-product. Such wastes are dumped in open lands which create a lot of pollution for surroundings. The waste also has disturbed the ability of nature to self-regulation of temperature and atmosphere. For the betterment of upcoming generation, an adoption of sustainable development of society should take place which would balance both living and non-living being on earth.

1.2 Waste Management

Total population on this planet in 20th century was six billion about seven years ago (Hammed et al., 2012). Now this population has reached at 7.2 billion (Dong et al., 2017). This increase in population demands increase in infrastructure. About 30 billion tonnes of raw materials are required for construction of necessary infrastructure each year worldwide (Behera et al., 2014).

Creating infrastructure and industrialization generates a lot of waste during processing. Total quantity of solid waste generated in India is 69 million tonnes. Out of this only 12 million tonnes waste is treated and remaining waste is dumped on open lands. This untreated waste creates a lot of problems to surroundings (Mohan et al., 2018).

Approximately 25% need of nutrients for crop can be achieved from recycled agriculture waste. Most of wastes generated from agriculture are bio-degradable which can be used as composts and for production of bio-fuel. One of agriculture wastes is rice husk ash which has potential to be used in manufacturing of bricks (Hwang and Huynh, 2015) and concrete (M. Jamil et al., 2016). Self-compacting concrete prepared with 10% metakaolin and 10% rice husk ash improves its compressive strength by 27% (Gill and Siddique, 2018).

Developing countries have recycled 80% of waste generated from electronics industry (Ohajinwa et al., 2018). European regulations has made it mandatory to use 85% components of automobile of recyclable products (Ortego et al., 2018). Cathode ray tube glass waste was

used as fine aggregate in high density concrete after treatment by nitric acid (Xiao et al., 2018).

Construction and demolition (C&D) waste also has potential for utilization in new construction. But, it requires a lot of energy to process it for the use of construction as an ingredient. Coarse aggregate obtained from C&D waste was used by 50% of total coarse aggregate with 10% glass powder in place of cement for the production of concrete without compromising its performance (Akhtar and Sarmah, 2018). In upcoming paragraph, requirement of construction industry which also has potential to use waste is discussed.

1.3 Industrial waste used as ingredient for cement composites

A good number of researches are available which have proved that the industrial wastes have potential to be used as cement and aggregates (both coarse and fine aggregate) in concrete as well as in mortar. Some of recommendations are given in standards. IS 455:1989 recommended that 65% ordinary Portland cement (OPC) can be replaced by ground granulated blast furnace slag (GGBFS). Recommendations for 25% replacement of OPC by fly ash has been given by IS 1489:1991. Use of recycled aggregate, steel slag, iron slag and bottom ash as fine and coarse aggregate in both plain and reinforced concrete to some extent has been allowed by IS 383:2016.

Chawla et al. (2018) and Kore and Vyas (2016) have used the marble waste as conventional coarse aggregate in concrete mixes. Kumar et al. (2017) have used sandstone waste as coarse aggregate with higher dose of admixture in concrete. Singh et al. (2015) prepared concrete with 25% granite cutting waste as fine aggregate which have shown better performance against adverse condition. Rana et al. (2016) have successfully used Kota stone slurry (dimensional lime stone) as fine aggregate in concrete.

Siddique et al. (2018) prepared concrete with ceramic waste as fine aggregate without compromising its durability. Tripathi et al. (2013) used a toxic waste i.e. ISF (Imperial Smelting Furnace) slag as fine aggregate for the production of concrete. Bisht and Ramana (2018) produced concrete with 21% discarded beverage glass as fine aggregate which have improved it`s mechanical properties. Thomas and Gupta (2016) produced high strength concrete of strength more than 60MPa by using 12.5% crumb rubber as fine aggregate. Natural fine aggregate in concrete also replaced successfully by 20% sanitary-ware waste as reported by farinha et al. (2012).

Singh et al. (2017) prepared cement mortar with 50% ISF slag and 50% Marble powder as fine aggregate and found that compressive strength was 63% higher than that of conventional mortar. Khayaliya et al. (2017) reported that performance of lean cement mortar with 25% marble waste as fine aggregate against aggressive environment was improved. Kabeer and Vyas (2018) obtained dense microstructure with 20% substitution of river sand by marble powder in cement mortar. Strength parameter was improved with 40% replacement of natural fine aggregate by dimensional lime stone slurry in cement mortar as documented by H.S. Chouhan et al. (2018). H.K. Kim et al. (2012) obtained 10-20% increment of flow of mortar with fine bottom ash aggregate.

1.4 Granite waste in cement composite

Granite is one of the most desired dimension stones available in India for construction projects. It is a type of igneous rock which is formed from slow crystallization of magma present below the earth surface. The major minerals present in granite are quartz and feldspar. India's part in granite production in 2014 was roughly about 45-55 million square meter (WONASA: World Natural Stone Association).

Approximate 250-400 tons of granite waste is generated every year from the cutting and finishing of granite block. It is estimated that 30% of the above quantity is lost as dust (Rajgor and Pitroda, 2013). The quantity of waste generated during processing also depends on technology. Table 1.1 presents the data for waste generated during sawing and finishing obtained from MSME Development Institute, 2009. This fine granite powder is accumulated in the form of slurry at the cutting and finishing industries engaged in processing of granite blocks. With time, water present in the slurry gets evaporated. The remaining residue of fine granite powder is a type of non-biodegradable industrial waste. The disposal of this waste is a major problem as it creates pollution of air as well as unwanted deposit on land. Air pollution causes eye infections and asthmatic problems while land pollution includes filling pores of soil which prevent the recharge of ground water and also reduces the soil fertility.

Apart from concrete, mortar also consumes a large amount of fine aggregate for masonry and plaster purposes. A report prepared by German company detailed that 1.4 billion tonnes of sand will be required by the year 2020. Out of this huge requirements of 248 million tonnes of sand shall be required only for mortar work (Material Consumption Patterns in India, 2016). This has led to an uncontrolled exploitation of river sand, which in turn leads to scarcity of sand. The Honourable Supreme Court of India has also banned the mining of

fine aggregate from river beds in Rajasthan in November 2017. This ban on fine aggregate has promoted its illegal mining which has raised its prices. As a consequence, there is an immediate need to find an economic alternative to natural sand due to the huge demand in the construction industries.

 Table 1.1 Percentage waste generated depending upon the mining technology used during processing (Source: MSME Development Institute, 2009)

Stage	Mechanised mines with gang saw cutting machines	Mechanised mines using blasting	Semi – mechanised mines using blasting	Weighted average	
Cutting	10%	15%	18%	15%	
Grinding and polishing	5%	5%	5%	5%	

There are a large number of research works available, where attempts have been made to reduce the granite waste by utilizing it in concrete. When granite powder was used as fine aggregate in concrete, the slump of concrete decreased due to rough and angular texture of granite powder (Vijyalakshmi et al., 2013). When the granite powder was used to replace cement in concrete up to 7.5%, mechanical properties i.e. compressive strength, flexural strength and split tensile strength were increased. For 10% substitution, above properties were comparable to the control concrete. Therefore strength required in concrete can be achieved with the 10% substitution of cement by granite powder (Chiranjeevi et al., 2015). With 5% addition or replacement of cement by granite powder in concrete, the corrosion cracking time of concrete was increased (Elmoaty, 2013). Plastic and drying shrinkage strain in concrete with granite powder were more than that of control concrete (Flexikala and Partheeban, 2010). From microstructural aspects, 30% replacement of sand by granite cutting waste in concrete showed maximum calcium silicate hydrate (C-S-H) gel (observed from XRD analysis) and densest matrix of concrete mixture (observed from SEM images) (Singh et al., 2016). Concrete mixes prepared with 30% red granite dust showed superior fresh and mechanical properties and excellent surface finish (Abukersh and Fairfield, 2011). With regard to the durability, concrete with 25% granite cutting waste in place of river sand showed better resistance to carbonation and corrosion for 0.30 w/c (Singh et al., 2016).

Potential of granite waste for the production of mortar as cement and fine aggregate substitute has also been discussed by some researchers. Marmol et al. (2010) used the granite

sludge as pigment in plaster mortar. They prepared granite sludge as pigment by heating it at 700-900°C for 4-24 hours in presence of KNO₃ mineralizer and using it for preparing colour plaster mortar in place of CaCO₃ filler. Due to the crystallizing of Fe_2O_3 present in granite at heat of 700-900°C, the plaster mortar prepared with granite sludge waste has shown middle intensity yellow orange colour with acceptable compressive strength. Ramos et al. (2013) studied the effect of 5-10% granite powder (PG) and superfine granite powder (PGS) as replacement of cement in mortar. The chloride-ion penetration was reduced by 70% in mortar with 10% PGS due to development of chloroaluminates. Li et al. (2018) investigated the durability and dimensional stability of mortar with substitution of cement by 5%, 10% and 15% replacement of cement by granite dust. The drying shrinkage found to be reduced up to 38% with the presence of 15% granite dust in place of cement. Mashaly et al. (2018) prepared mortar with substitution of cement in mortar by granite sludge from 0 to 40% by weight. The maximum reduction in compressive strength and flexural strength was 16% and 31% for mortar prepared with 40% granite sludge. With 10% granite sludge has improved the water absorption and apparent density of cement paste. Bonavetti and Irassar (1993) replaced by granite stone dust by 5, 10, 15 and 20% by weight in 1:3 cement mortar. Increment of granite stone dust in mortar has increased its drying shrinkage due to higher water demand. Jeyaprabha et al. (2016) prepared three mortar mixes i.e. mortar with 100% natural river sand (called RSM), mortar with 85% natural river sand and 15% granite powder (called G15M) and mortar with 100% manufactured sand (called MSM). The mortar mix of 1:3 with 0.5 as w/c ratio was exposed to direct fire (200°, 500°, 700° and 900°C) in this experimental survey. No change in chemistry of mortars prepared with granite powder (G15M) and manufactured sand (MSM) after direct exposure to fire were observed through micro-structure analysis.

In the light of results obtained by various researchers it can be observed that granite waste can be used as a constituent in mortars and concrete as a partial substitute to natural river sand. Granite powder is available in a huge quantity because of 80% granite production of India comes from Rajasthan and its chemical composition is similar to conventional fine aggregate. The proportion can be decided based on application of material. Hence detailed study is needed to find out feasibility of using this waste.

1.5 Objectives of study

There is a need for comprehensive evaluation of performance of mortar mixes with a wide range of mix proportions with granite powder in place of river sand. Therefore the objectives of the study are:

- 1. To characterize the waste granite powder obtained from granite processing industries.
- 2. To evaluate physical and mechanical properties of mortars made with waste granite powder as a partial substitute for fine aggregate.
- 3. To assess the durability characteristics of mortars made with granite powder when exposed to adverse environment.

1.6 Structure of the thesis

The thesis comprises of five chapters. **First chapter** describes the expansion of infrastructure works and the details of problems faced by construction industry due to unavailability of fine aggregate. Possibility of alternate materials as fine aggregate in construction activities has been discussed. Specific objectives of the study have also been briefly outlined. **Second chapter** describes the extensive review of literature on granite fines as substitute of cement and fine aggregate in concrete and mortar. Research gap in literature has also been highlighted in this chapter. **Third chapter** of thesis describes the procedures with relevant standards followed to evaluate the physical, mechanical and durability properties of cement mortars. The methodology followed in the present study is also discussed at length. **Fourth chapter** incorporates the results and discussions of experimental investigations for mechanical and durability properties with graphs and figures. Detailed microstructural investigations are also discussed in this chapter. **Fifth chapter** concludes the outcome of the study and recommendations for future study are highlighted.

CHAPTER 2

LITERATURE REVIEW

2.1 General

High demand of fine aggregate with limited sources has raised the question for alternatives in the construction industry. There are a number of sites where wastes like marble, granite, Kota stone etc. generated from stone processing industries are dumped. These dumping sites (as seen in Figure 2.1) create lots of problems for the surroundings as these wastes are non-biodegradable. By realising the problem of limited natural fine aggregates for construction industry and surplus waste generated from stone industry, a good number of research works are available where attempts have been made to reduce the burden of such stone wastes by utilizing it in concrete and mortar mixes.



Figure 2.1 Dumping site near Shahpura, Jaipur

Granite is available in plenty and most widely used in flooring work by the construction sector. Most of the industries cut the block of granite and convert into small pieces suitable for flooring. During this process waste is generated in the form of boulders and fine particles slurry. The boulders are again converted into aggregate and used in construction activities. Slurry part keeps accumulating around the processing industry. It

contains major fraction as silica (refer Table 2.1) and has been attempted to be used by many researchers in cement concrete and cement mortar mixes.

Reference	1	2	3	7	12	14	15	18	19	22
CaO	3.69	4.90	4.10	3.27	1.82	4.80	-	3.26	7.56	8.04
MgO	1.70	2.50	0.23	0.36	0.71	0.57	0.83	1.82	0.99	0.96
Na ₂ O	3.62	-	3.30	4.69	3.69	5.92	4.21	2.68	1.46	1.39
Al ₂ O ₃	16.30	2.10	12.2	11.96	14.42	12.01	15.63	15.66	16.04	15.84
SiO ₂	61.40	85.5	62.0	58.17	72.04	64.50	72.57	63.22	71.48	69.17
P ₂ O ₅	-	-	-	0.41	0.05	0.07	-	-	-	-
SO ₃	0.05	1.80	0.10	0.39	-	-	-	0	-	-
Fe ₂ O ₃	3.66	0.40	9.30	13.35	1.22	5.77	-	4.47	-	1.16
K ₂ O	3.75	-	4.40	3.84	4.12	5.26	6.76	5.02	0.49	0.43
TiO ₂	-	-	-	0.37	0.30	0.67	-	-	-	-
MnO	-	-	-	-	0.12	0.39	-	-	-	-
Others	1.10	-	-	-	1.68	-	-	-	-	-
LOI	5.01	1.10	-	2.58	-	-	-	2.04	-	3.01
Cŀ	-	-	-	0.12	-	-	_	-	-	-

Table 2.1 Chemical composition of granite stone reported in reviewed literature

Many researchers have tried to use it as partial replacement of cement, fine aggregate in concrete and mortar mixes. Mechanical and durability properties have been evaluated as described in the following paragraphs.

2.2 Granite powder as cementitious material in concrete

Abukersh and Fairfield (2011)^{*1} have examined the potential of red granite dust (RGD) as partial replacement of cement in concrete. The RGD particles passing through 75 µm sieve were used. The substitution level for RGD was 20, 30, 40 and 50% by mass. The parameters like workability, compressive strength, flexural strength, split tensile strength, density, ultrasonic pulse velocity and elastic modulus were evaluated. Concrete prepared with 30% RGD was reported to possess improved fresh properties than control concrete. The increment in slump was due to availability of free water which was available because of less absorption capacity of RGD than cement. The compressive strength, flexural strength and split tensile strength decreased with addition of RGD. Density of concrete prepared with 30% RGD was slightly higher at 56 days and 90 days than that of control mortar. This behaviour was due to the gradual development of improved pore structure by continued cement hydration as reported by authors. The range for UPV observed by authors was between 4.65 to 5.00 km/s for all concrete mixes including control concrete which is termed as excellent as per specifications of standard followed.

Abd Elmoaty $(2013)^{*2}$ studied concrete modified with granite dust as cement replacement and addition. The ratio of control concrete was 1:1.2:1.2 which had 45 MPa strength after 28 days of curing. The reduction in w/c ratio as per BS 882 is shown in Figure 2.2.The addition and replacement of granite dust were 0, 5%, 7.5%, 10% and 15% by the weight of cement. During the replacement of cement, there was insignificant effect on initial, final setting times and expansion of cement paste. An increment of 8.2% in compressive strength was observed in concrete prepared with 5% replacement of cement by granite dust and it was maximum in all cases of replacement of cement in concrete. It was due to the filling effect by extremely fine granite dust. The pattern for tensile strength of concrete manufactured with granite dust as cement replacement was similar to the pattern obtained in compressive strength.

The compressive strength was increased with 10% addition of granite dust by 12% of its conventional strength. The tensile strength was also reported maximum with 10% addition

of granite dust in concrete. Beyond 10% addition of granite dust in concrete reduction in compressive and tensile strength was observed.

A negligible impact was reported on porosity of concrete up to 7.5% replacement or addition of granite dust. The change in corrosion cracking time was found to be insignificant with addition or replacement of cement by granite dust. The X-ray diffraction analysis showed no difference in hydration products of control concrete and concrete modified with granite dust. A good distribution of granite grains was noticed by the authors around the grains of cement in SEM images which may accelerate the presence of ettringite. Authors recommended 10% addition and 5% replacement of cement by granite dust in concrete without compromising the parameters.



Figure 2.2 Relation between compressive strength and water/cement ratio

Al-Hamaiedeh and Khushefati (2013)^{*3} used granite powder as replacement and addition by weight of cement in the range of 10% to 30% in concrete. The ratio of ingredients like cement, fine aggregate, and coarse aggregate was 1:1.44:2.52. The compressive strength of control concrete was 32MPa obtained after 28 days of curing. The parameters like normal consistency, soundness and setting time of cement were explored. With replacement or addition of granite powder from 10% to 30% in cement, enhancement of silica (SiO₂) from
23.73% to 29.61% and decrement of lime (CaO) from 58.29% to 49.95% was observed by XRF technique. This increment and decrement of silica and lime contents may have increased the possibility of change in behaviour of concrete and cement paste.

There was no effect on soundness of cement paste due to replacement or addition of granite powder. The addition of 10% percent granite powder in cement had increased its final setting time due to increase of water requirement for achieving consistency which supports in long distance casting.

For concrete two parameters like workability and compressive strength were evaluated. The slump value was found to be reduced with addition of granite powder in concrete due to the large specific area of particles of granite. The compressive strength showed positive trends with 20% addition of granite powder. Beyond 20% substitution reduction in compressive strength was observed due to less content of cement.

At 10% replacement level, slump and compressive strength of concrete were found similar to control mix due to filling effect of voids. The reason for increment in slump was fine granite particles which have acted as lubricant between the coarse aggregate. More than 10% replacement of cement by granite dust in concrete has shown negative effect on strength and workability due to large surface area of granite dust.

E. Bacarji et al. (2013)^{*4} used mix of marble and granite waste (MGR) in concrete as cement replacement. Total three type of MGR (named A, B and C) were used in this experimental study. The chemical analysis of all MGR's revealed that silica was maximum and approximately similar in percentage. The particle diameter of MGR used was $0.7-71\mu$ m (for A) and $0.7-90\mu$ m (for B and C). The substitution level was 5%, 10% and 20%. Mechanical properties i.e. compressive strength, elastic modulus and water absorption were determined. These mechanical properties remained unaltered when 5% substitution was done by MGR-A. A numerical analysis was also conducted by authors to evaluate the effect on the internal microstructure of concrete. The numericalanalysis showed that MGR acts as filler would not change the chemistry in concrete. Also there was no contribution of MGR in the pozzolanic reaction in concrete.

K.C. Reddy et al. (2015)^{*5} prepared M25 grade of concrete with granite fines as replacement of cement in four different proportions namely 2.5, 5, 7.5 and 10%. There are total four parameters such as workability (slump and compaction factor), compressive strength, flexural strength and splitting tensile strength were assessed. The workability of

blending mixes were decreased with addition of granite fines. The replacement of cement by granite fines up to 7.5% increases the mechanical properties. The compressive strength, split tensile strength and flexural strength were increased by 10%, 22% and 16%, respectively at 7.5% substitution of cement. The mechanical properties for blending mix was also comparable with control mortar at 10% replacement.

H. Li et al. (2016)^{*6} used granite dust as a supplementary cementitious material replacing fly ash in manufactured sand concrete. The replacement level was 10%, 20% and 30%. The parameters like slump (workability), compressive strength, bending strength, elastic modulus, chloride penetration, frost resistance and drying shrinkage were checked. With the replacement of fly ash by granite dust, workability of concrete was reduced because granite particles were of angular in shape than spherical particle of fly ash. The compressive and bending strength was found to be increased at 20% substitution of fly ash by granite dust due to compactness by granularity optimization. Long term drying shrinkage of concrete prepared with 30% granite dust was enhanced because of availability of free water which has evaporated due to less cement content. Concrete prepared with 20% granite dust had shown maximum reduction of 2.9% in dynamic modulus after 350 cycles of frost action. The resistance to chloride penetration was reduced with the substitution of fly ash by granite powder, but it was still in low permeability zone as per specifications of standard followed. The overall conclusion of this investigation was found that 20% granite dust can be successfully used in place of fly ash in concrete.

A.O. Mashaly et al. (2018)^{*7} used granite sludge by 10%, 20%, 30% and 40% of the total weight of cement. The physical properties like water absorption, oven dry density and apparent porosity, mechanical properties like compressive strength and flexural strength and durability properties like abrasion resistance, freeze–thaw resistance and sulphate resistance were evaluated. The increment of granite sludge in concrete was decreased the physical parameters but at 20% replacement physical parameters satisfy the requirements of the standardfollowed. The mechanical properties also decreased with the increment of granite sludge in concrete. Concrete with 20% granite sludge showed better performance against freeze-thaw and sulphate action. The resistance of abrasion of concrete prepared with granite sludge decreases with increment of replacement of cement due to availability of less binder.

Table 2.2 Effect on compressive strength of concrete when cement is substituted with granite

Reference	Variation	Results
Abukersh and Fairfield (2011)	10% increment from 20% to 50%.	Strength found to be decreased.
Abd Elmoaty (2013)	Replacement by 5%, 7.5%, 10% and 15%.	Maximum Strength at 5 % substitution.
Tiou Emioury (2015)	Addition by 5%, 7.5%, 10% and 15%.	Maximum strength at 10 % addition.
Al-Hamaiedeh and	Replacement by 10%, 20% and 30%.	Maximum strength achieved at 10% replacement.
Khushefati (2013)	Addition by 10%, 20% and 30%.	Maximum strength achieved at 20% addition.
K.C. Reddy et al. (2015)	Replacement by 2.5, 5, 7.5 and 10%.	Maximum strength achieved at 7.5% substitution.
H. Li et al. (2016)	Replacement by 10%, 20% and 30%.	Maximum strength achieved at 20% substitution.
A.O. Mashaly et al. (2018)	Replacement by 10%, 20%, 30% and 40%.	Strength found to be decreased.

fines

2.3 Granite Powder as fine aggregate in concrete

K. Williams C. et al. (2008)^{*8} prepared high performance concrete with granite powder as fine aggregate varying from 0 to 100%. Cement was also replaced with 10% fly ash, 7.5% silica fume, 10% slag and 1% superplasticizer at each replacement level of fine aggregate. The effect on compressive strength, split tensile strength, modulus of elasticity, drying shrinkage and water penetration were evaluated. The compressive strength, split tensile strength and modulus of elasticity were reported increased at 25% substitution of river sand by granite powder. Water penetration of concrete prepared with granite powder showed 5% less penetration than control mortar. The pattern and values for drying shrinkage of mixes

prepared with granite powder at all substitution level were similar to the control mix. The partial replacement of fine aggregate in concrete by granite powder has a beneficial effect on the mechanical properties of concrete.

S.N. Raman et al. (2011)^{*9} prepared high strength concrete (HSC) with rice husk ash (RHA). Initially RHA was used in place of cement from 10 to 30% in steps of 10%. After 28 days of curing, targeted strength was achieved with 10% RHA for HSC. Then the quarry dust (QD) obtained from granite crushing process was used in place of sand in the range of 10 to 40% in step of 10% with 10% rice husk ash (RHA). The parameters like slump and compressive strength were assessed for all the mixes prepared with RHA and QD. The 20% substitution of sand by QD was selected for further study in concrete on the basis of results of slump and compressive strength. At final stage, the properties like compressive strength, modulus of rupture, dynamic modulus of elasticity, ultrasonic pulse velocity and initial surface absorption were evaluated for four mixes i.e. control mortar, mortar with 10% RHA, mortar with 20% QD and mortar with 10% RHA & 20% QD. The best performance for above properties was obtained for concrete prepared with 10% RHA. It was due to the formation of additional C-S-H gel due to reaction of RHA with Ca(OH)₂. The second best performance was obtained for the mix prepared with 10% RHA and 20% QD. The negative effect of quarry dust was remunerated with RHA as mineral admixture as observed in mechanical properties of concrete.

Y. Divakar et al. (2012)^{*10} utilized granite fines as fine aggregate in five different proportions i.e. 5, 15, 25, 35 and 50%. The parameters like compressive strength, flexural strength and splitting tensile strength were investigated on M20 grade of concrete. This experimental investigation conclude that strength properties were increased by 28% when concrete prepared with 35% granite fines in place of natural fine aggregate. The micro filling effect of granite fines was the reason for such behaviour.

M. Vijayalakshmi et al. (2013)^{*11} experimentally investigate the suitability of granite powder waste as a substitute in concrete. This granite powder waste utilized in the range of 0 to 25% in the step of 5%. The effect of partial substitution of granite powder on various mechanical properties (compressive strength, split tensile strength, flexural strength, ultrasonic pulse velocity and elastic modulus) and durability properties (water permeability, rapid chloride penetration, carbonation depth, sulphate resistance and electricity resistivity) were assessed. The rough and angular texture of granite powder reduced the slump of

concrete. The early age strength for concretes prepared with 5%, 10% and 15% GP were better than that of control concrete. It might be because of nucleation sites developed by increased surface area and siliceous nature of granite powder. After the exposure of specimens to carbon-di-oxide for 365 days in concrete with 20% and 25% granite powder, the depth of carbonation was near to the cover of reinforcing bar which may cause corrosion in reinforcement. Concrete with granite powder at each substitution showed greater loss in compressive strength as compared to control concrete when subjected to sulphate attack. The maximum loss of 35% in compressive strength was observed with 25% granite powder in concrete. The presence of sulphate content in kerosene, diesel and wax added during the cutting and finishing of granite was transformed into sulphur trioxide due to enormous heat of cutting blade. This form of sulphur has increased the strength of sulphur solution which ultimately reduced the strength of concrete with granite waste. Overall 15% replacement of sand by granite powder waste was found well suited the concrete without compromising its mechanical and durability properties.

K. Kayathri et al. (2014)^{*12} utilized coper slag, fly ash and granite powder as fine aggregate in place of natural sand in M30 grade of concrete. The specifications of IS 10262 were used for the design of concrete. The percentage of replacement was 0, 25, 50 and 75%. The above three wastes were utilized in equal quantity, it means that if percentage of replacement is 75% then quantity of each waste is 25%. The compressive strength and split tensile strength were evaluated after 7, 14 and 28 days of curing. The compressive strength was obtained in case of split tensile strength as obtained in compressive strength. The results for above properties shows that copper slag, fly ash and granite powder have potential to be used as 75% part of fine aggregate in concrete.

R. Raghvendra et al. (2015)^{*13} utilized granite powder as fine aggregate in the range of 0 to 25% in the steps of 5%. In this experimental study authors also used 10% fly ash, 10% GGBS and 7.5% silica fume in place of cement. Two parameters like slump and compressive strength parameters were evaluated. The slump of concrete with granite found to be decreased by 18% with the increment of substitution of granite powder. The maximum compressive strength was achieved at 15% replacement level. It was increased by 25% after 28 days of curing.

S. Ghannam et al. (2016)^{*14} used granite powder a by-product developed from granite stone crushing and polishing industry as fine aggregate. Granite powder was used in concrete in 5, 10, 15 and 20% by weight of sand. All mixes with and without granite powder were designed for a slump of 80 mm and compaction factor of 0.95. Such workability was obtained by using extra admixture than additional water. The mechanical properties like compressive strength, flexural strength and splitting tensile strength were evaluated. The compressive strength as well as flexural strength was found to be increased with substitution of fine aggregate by granite powder. The maximum compressive strength and flexural strength, maximum strength was obtained 339 MPa which was 30% higher than split tensile strength of conventional concrete.

S. Singh et al. (2016)^{*15} studied the compatibility of using granite cutting waste (GCW) as a partial substitute of river sand in high strength concrete based on strength, durability and microstructural attributes. This analysis was carried out on 0.30, 0.35 and 0.40 w//c ratio by replacing 0, 10, 25, 40, 55 and 70% river sand by GCW. The parameters like workability, compressive strength, flexural strength, abrasive resistance, permeability, water absorption, carbonation, corrosion, microstructure with XRD and SEM were checked. The elements like O, Si, Al, K and Mg found similar in river sand and GCW. In all w/c ratio, the slump of concrete was decreased with incremental dose of GCW in place of river sand. It was due to internal friction developed by angular and rough texture of GCW. The maximum compressive strength was gained with 25% GCW concrete for all w/c ratios. The reason was filling of voids which makes the concrete denser. The least corrosion was observed for concrete with 40% GCW. The outcome of corrosion suggested that higher w/c ratio gives higher corrosion. The outcomes of above properties suggested that 25-40% partial substitution of river sand by GCW in concrete can be done successfully.

S. Singh et al. (2016)^{*16} investigate the concrete prepared with granite cutting waste (GCW) under adverse exposure conditions. The tests like carbonation, sulphate attack, chloride ion penetration, acid attack and elevated temperature were performed to check its suitability. This study was carried out on 0.30 and 0.40 w/c ratio. In this analysis river sand was partially replaced by GCW in proportions of 0, 10, 25, 40, 55 and 70%. The least carbonation and chloride penetration was observed in concrete with 0.3 w/c ratio. Concrete with 25% GCW showed lower carbonation and chloride penetration than that of control concrete due pore filling of finer GCW particles. In sulphuric acid solution, weight loss of

concrete was observed due to formation of ettringite (3CaO.Al₂O₃.3CaSO₄.32H₂O). The least weight loss was observed for concrete with 40% GCW. There was no spalling on surface was observed after exposure of magnesium sulphate solution for 210 days. After thermogravimetric analysis (TGA), least weight loss was observed for concrete with GCW due to higher value of CaO:SiO₂ ratio. The concrete prepared with 25% GCW performed better than conventional concrete in adverse conditions.

Reference	Variation	Results
K. Williams C. et al.(2008)	25, 50, 75 and 100%.	Maximum strength was obtained with 25% substitution.
S.N. Raman et al. (2011)	10, 20, 30 and 40%.	Maximum strength was found with 20% substitution.
Y. Divakar et al. (2012)	5, 15, 25, 35 and 50%.	Maximum strength was found with 35% substitution.
M. Vijayalakshmi et al. (2013)	5, 10, 15, 20 and 25%.	15% replacement showed strength without any compromise.
K. Kayathri et al. (2014)	25, 50 and 75%.	Maximum strength was found with 75% waste.
R. Raghvendra et al. (2015)	5, 10, 15, 20 and 25%.	Maximum strength was achieved at 15% replacement.
S. Ghannam et al. (2016)	5, 10, 15 and 20%.	Maximum strength was achieved at 10% replacement.
S. Singh et al. (2016)	0, 10, 25, 40, 55 and 70%.	Maximum strength was obtained with 25% waste.

 Table 2.3 Effect on compressive strength of concrete when fine aggregate is substituted with granite fines

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2.4 Granite powder as cementitious material in mortar

Marmol et al. (2010)^{*17} assessed the potential of granite sludge waste as structural components in cement mortar. The replacement level of cement was 5%, 10% and 20% in masonry mortar by granite sludge waste. The only parameter used in their study was compressive strength. Authors have proved that 10% use of granite sludge waste as cement replacement in masonry mortar has no negative effect on compressive strength after 7 days and 28 days of curing but further more replacement has decreased the compressive strength by 30%.

Ramos et al. (2013)*¹⁸ studied the effect of 5-10% granite powder (PG) and superfine granite powder (PGS) as replacement of cement in mortar. The value of D90 was 55.46µm and 13.34µm for PG and PGS, respectively. The parameters like workability, compressive strength, flexural strength, alkali silica reaction (ASR) and chloride ion diffusion were evaluated. The workability was found similar to that of control mortar with both granite waste. Introduction of both granite powders (PG and PGS) in mortar reduced its compressive strength. The reduction in compressive strength was 20% and 10% for PG mortar and PGS mortar, respectively due to higher fineness. There is no effect on ASR in PG mortar but expansion has reduced by 38% in PGS mortar. The chloride ion ingress has reduced in both i.e. PG mortar and PGS mortar but 70% increment was observed against chloride diffusion in PGS mortar.

Shankar and Mohan (2015)^{*19} partially substitute the cement with granite powder at 20%, 25% 30%, 35% and 40% in mortar. The durability tests such as fire resistance, sulphate resistance and corrosion resistance were performed on 1:3 mix proportion after 28 days of curing. The mortar with 30% granite content required 70 hours to get corrode than 52 hours of control mortar. The temperature of fire exposure was 200°C, 400°C, 600°C and 800°C used in this experimental study. The compressive strength was found to be maximum for mortar with 30% granite content after all temperatures of exposure. Authors have concluded after their experimental investigation that mortar prepared with 30% granite powder has shown better resistance against durability tests.

Li et al. $(2018)^{*20}$ investigated the durability and dimensional stability of mortar with substitution of cement by 5%, 10% and 15% replacement of cement by granite dust. Total three w/c ratio i.e. 0.40, 0.45, 0.50 and 0.55 were used in this study. The parameters like flow, compressive strength, carbonation, water absorption and drying shrinkage were

observed. All mixes were prepared for a range of flow of 200 mm to 300 mm. To achieve required flow in mortars, super plasticizer was used. The dose of admixture was increased by 54% to 85% with substitution of cement by granite dust. It was due to 75µm finer particles of granite dust. The compressive strength was increased by 10-12% with granite dust for all w/c ratio. The cement mortar with granite dust have improved the resistance against carbonation. It was due to dense structure of mortar by filling of voids by granite dust. The drying shrinkage also found to be reduced up to 38% with the presence of granite dust in place of cement. The drying shrinkage at higher w/c ratio was higher for all mortar mixes and at lower w/c ratio, the drying shrinkage was found to be least. The outcome of this experimental study revealed that granite dust enhanced the above properties due to improve in packing density of mortar.

Mashaly et al. (2018)^{*7} prepared mortar with substitution of cement in mortar by granite sludge from 0 to 40% by weight. The properties like water absorption, apparent porosity, compressive strength, flexural strength, abrasion resistance and sulphate resistance were evaluated. Water absorption and apparent porosity was found to be increased by 45% and 42% respectively for mortar with 40% substitution of cement by granite sludge. The water absorption value for all mixes satisfied the specifications of BS 4131:1973.The maximum reduction in compressive strength and flexural strength was 16% and 31% for mortar prepared with 40% granite sludge. The compressive strength and flexural strength of granite sludge. The abrasion depth for mortars with 40% granite sludge was found to be increased by 77%. It was due non-cementitious nature of granite sludge than cement. The results revealed that all above performances were decreased with an increase in granite sludge than to control mortar.

Table 2.4 Effect on compressive strength of mortar when cement is substituted with granite

Reference	Variation	Results
Marmol et al. (2010)	Replacement by 5%, 10% and 20%.	Strength found comparable with 10% substitution.
Ramos et al. (2013)	5% and 10% replacement.	Strength decreased with substitution.
Li et al. (2018)	5%, 10% and 15% replacement.	Maximum strength achieved with 15% replacement.
Mashaly et al. (2018)	Replacement from 10% to 40% in step of 10.	Comparable strength was obtained with 20% replacement.

fines

2.5 Granite powder as fine aggregate in mortar

Bonavetti and Irassar (1993)^{*21} used three types stone dust i.e. quartz, granite and limestone in 1:3 cement mortar. Natural sand was replaced by stone dust by 5, 10, 15 and 20% by weight. Parameters like water demand, compressive strength, flexural strength, porosity and drying shrinkage were investigated. As the dust content increased, water demand required for a constant flow was increased. The maximum compressive strength was achieved at 20%, 10% and 10% substitution of sand by quartz, granite and lime stone dust, respectively. The pattern for flexural strength was similar to compressive strength. The mortars prepared with quartz dust showed lower drying shrinkage than that of control mortar. The least drying shrinkage was achieved at 10% and 5% substitution of sand by granite and lime stone dust, respectively. The porosity also increased with an increase of dust in mortar.

Marmol et al. (2010)^{*17} used the granite sludge as filler and pigment in masonry and plaster mortar, respectively. A total of 10% CaCO₃ filler was used in masonry mortar which was replaced by dried granite sludge by 2%, 5% and 10%. The compressive strength was only parameter which was evaluated and compared with control mortar. The strength parameter found to be increased by 23% for masonry mortar mixes with substitution of CaCO₃ filler by 10% dried granite sludge. Authors also prepared granite sludge as pigment by heating it at 700-900°C for 4-24 hours in presence of KNO₃ mineralizer and using it for preparing colour plaster mortar in place of CaCO₃ filler. Due to the crystallizing of Fe₂O₃ present in granite at heat of 700-900°C, the plaster mortar prepared with granite sludge waste has shown middle intensity yellow orange colour with acceptable compressive strength.

Jeyaprabha et al. (2016)*22 prepared three mortar mixes i.e. mortar with 100% natural river sand (called RSM), mortar with 85% natural river sand and 15% granite powder (called G15M) and mortar with 100% manufactured sand (called MSM). The mortar mix of 1:3 with 0.5 w/c ratio was used in this experimental survey. The effect of direct exposure to fire (200°, 500°, 700° and 900°C) was evaluated on mechanical properties like compressive, flexural and split tensile strength. The compressive strength was 25, 36 and 38 MPa achieved after 28 days of curing for mortar prepared with natural river sand, granite powder and manufactured sand, respectively. After the fire exposure, compressive strength was reported decreased by about 21, 17 and 14% at 200°C, 43, 35 and 27% at 500°C, 62, 59 and 49% at 700°C for RSM, G15M and MSM mixes, respectively. At 900°C temperature, more than 75% strength loss was recorded for RSM and G15M specimens. The similar pattern was obtained in flexural and split tensile strength as obtained in compressive strength. The results obtained from mechanical properties were confirmed through micro-structure analysis i.e. TGA, XRD, SEM and FTIR of mixes. No change in chemistry of mortars prepared with granite powder (G15M) and manufactured sand (MSM) after direct exposure to fire were observed through micro-structure analysis. Authors concluded that mortars prepared with 15% granite powder (G15M) and manufactured sand (MSM) enhanced the mechanical properties as compared to mortar prepared with natural river sand (RSM).

Table	2.5	Effect	on	compressive	strength	of mortar	when	fine	aggregate	is	substituted	with
					gr	anite fines						

Reference	Variation	Results
Bonavetti and Irassar (1993)	5, 10, 15 and 20%.	Maximum strength was achieved at 10% replacement.
Marmol et al. (2010)	2%, 5% and 10%.	Maximum strength with 10% waste.
Jeyaprabha et al. (2016)	15%	Strength found to be increased with 15% waste.

2.6 Summary

From the above literature it can be concluded that limited study has been conducted on utilization of granite powder as fine aggregate in mortar. A study on adhesion of mortars with substrate and tensile bond strength of mortars prepared with granite powder has not yet been traced. The effect of adverse conditions like acid attack, sulphate attack and salt crystallization also requires to be investigated in mortar mixes containing granite powder as partial replacement of natural sand from durability point of view. Optimum percentage of utilization of the granite powder as fine aggregate needs to be found out for effective utilization of this waste. It is expected that cost of building infrastructure is likely to come down in the areas close to such granite processing industries. Finally, problems like disposing of granite powder around granite processing industries and dependency on costly natural fine aggregate can be resolved by using such waste in mortar production. This is in line with the government thrust on circular economy.

CHAPTER 3

MATERIALS AND METHODS

3.1 General

Properties of different constituents of mortar like cement, natural sand and granite powder have been presented in this chapter. Procedures of all the mechanical and durability tests performed during the experimental study have been discussed.

3.2 Constituents of mortar

The constituents like Portland pozzolana cement (PPC), natural river sand and granite powder were used for the production of mortar in this experimental study. Characterization of these materials are discussed in subsequent paragraphs.

3.2.1 Portland pozzolana cement

For the experimental program, Portland pozzolana cement (PPC) was utilized conforming to the specifications set by IS 1489 - Part 1 (1991). The physical properties are presented in Table 3.1. The scanning image of PPC is shown in Figure 3.1.

Property	Value
Specific gravity	2.9
Bulk density (kg/m3)	1100
Normal consistency (%)	33
Initial setting time (min)	129
Final setting time (min)	231
Compressive strength after 28th days of curing(MPa)	36

Table 3.1 Physical properties of PPC



Figure 3.1 Scanning Image of PPC

3.2.2 Natural river sand

Two types of conventional natural river sand were used: (i) Coarse river sand (CS), conforming to zone-II as per IS: 383 and (ii) Fine river sand (FS), conforming to IS: 2116-1980 (sand for masonry mortar) and IS: 1542-1992 (sand for plaster). Both conventional natural river sands were procured locally. Based on the particle size distribution which is represented in Figure 3.2, they were designated as coarse sand (CS) and fine sand (FS).

Physical properties of both sands are enlisted in Table 3.2. The SEM image of both sands are shown in Figures 3.3 and 3.4. From SEM image it is clearly observed that both natural sands have smooth and rounded surface.

Fine aggregate	Specific gravity	Water absorption (%)	Loose bulk density (kg/m ³)	Fineness Modulus
Coarse river sand (CS)	2.68	7.05	1597	2.65
Fine river sand (FS)	2.65	8.83	1545	1.65
Granite powder (GP)	2.46	15.29	1368	0.9

Table 3.2 Physical properties of CS, FS and GP



Figure 3.2 Particle size distribution of used fine aggregates



Figure 3.3 SEM image of coarse river sand (CS)



Figure 3.4 SEM image of fine river sand (FS)

The chemical analysis for both natural sands (CS and FS) is tabulated in Table 3.3 which was carried out using X – ray fluorescence (XRF) technique. From this table it can be seen that, the proportion of silica (SiO2) was approximately similar in both conventional natural sands.

Oxides	Coarse river sand (%)	Fine river sand (%)	Granite powder (%)
SiO ₂	75.82	73.46	74.39
Al ₂ O ₃	10.17	10.78	13.5
Fe ₂ O ₃	3.15	3.38	0.86
MnO	0.08	0.09	0.02
MgO	1.19	1.33	0.38
CaO	3.36	3.58	0.41
Na ₂ O	2.17	2.25	4.16
K ₂ O	1.9	2.06	4.79
TiO ₂	0.42	0.45	0.17
P ₂ O ₅	0.07	0.08	0.02

 Table 3.3 Chemical composition of river sands and granite powder

Figures 3.5 and 3.6 represent X-ray diffraction of CS and FS, respectively. The major components found in CS and FS is quartz at a diffraction angle of 26.63° and 26.88°, respectively.



Figure 3.5 X-ray diffraction spectrum of the coarse river sand (CS)



Figure 3.6 X-ray diffraction spectrum of the fine river sand (FS)

3.2.3 Granite Powder

Granite powder (GP) used to replace conventional sand in cement mortar was procured from nearby stone processing industry, in Jaipur, Rajasthan. GP was sun dried to remove the moisture before characterization and utilization in mortar production. On carrying out particle size analysis is shown in Figure 3.2. It was found that GP can be assigned to zone – IV category of IS 383 (2016). Physical properties like specific gravity, water absorption and bulk density of GP are detailed in Table 3.2.



Figure 3.7 SEM image of the granite powder (GP)



Figure 3.8 X-ray diffraction spectrum of the granite powder (GP)

The angular and rough surface of GP can be seen in the SEM image shown in Figure 3.7.The major components found in granite powder are quartz and k-feldspar (Orthoclase) at a diffraction angle of 26.68° and 27.95°, respectively as seen in Figure 3.8. The chemical composition of granite powder was evaluated using X-ray fluorescence (XRF) technique as shown in Table 3.3.

3.3 Research methodology

In line with the set objectives, river sand was replaced in different proportions by GP for the production of cement mortars. The experimental study was carried out on mortars

with mix proportions of 1:4 and 1:6. Two types of conventional natural river sands were used in two stages of this investigation.

3.3.1 Phase-I

In this stage, FS (zone – IV) conforming to IS 2116 (1980) and IS 1542 (1992) was used for the preparation of control mortar. This fine sand was replaced by GP from 0% to 100% in steps of 10% by volume. Quantities of materials required to produce one cubic meter of mortar is given in Table 3.4. Workability, fresh density, compressive strength, water absorption and permeable voids were evaluated and their results were compared with those of control mortar.

	1:4 Mix				1:6 Mix			
%	Cement	FS	GP	Water	Cement	FS	GP	Water
Replacement	(kg)	(kg)	(kg)	(kg)	(kg)	(kg)	(kg)	(kg)
0	273	1534	-	328	189	1591	-	337
10	269	1362	135	344	189	1432	141	344
20	266	1195	266	354	188	1268	281	348
30	268	1054	400	351	188	1108	420	351
40	271	913	539	347	187	948	559	354
50	271	760	674	349	186	784	694	361
60	270	607	804	354	185	624	829	366
70	268	453	933	359	183	464	958	374
80	265	297	1056	369	182	306	1085	382
90	261	146	1171	379	180	151	1206	391
100	258	-	1283	390	178	-	1331	398

Table 3.4 Quantities of materials to prepare one cum of mortar mixes

3.3.2 Phase-II

In this stage, CS (zone – II) and GP were combined such that the resulting composite fine aggregate mixture satisfied the gradation requirements stipulated by IS 2116 (1980) and IS 1542 (1992). Based on trials, the required gradation was achieved when 30% to 40% of CS was replaced by GP. Hence mortar mixes of the same two proportions were prepared in which GP constituted 30% and 40% of fine aggregate volume. Quantities of materials required to produce one cubic meter of mortar is given in Table 3.5. Initially parameters like workability, fresh density and compressive strength were evaluated. These results were compared with those of control mortar prepared by fine sand in Phase-I.

Good and satisfactory results were obtained for mortar mixes prepared with 30% and 40% GP in above three tests as compared with those of control mortar. Then other properties for mortars like adhesive strength, tensile bond strength and drying shrinkage were evaluated. Mortar was also checked for durability aspect to judge it's sustainability in adverse environmental conditions. The details of the specifications followed for conducting the tests on mortar mixes are tabulated in Table 3.6.

Mix	Cement (kg)	CS (kg)	FS (kg)	GP (kg)	Water (lit)
4FS	273	-	1534	-	328
4CS-30	279	1136	-	416	313
4CS-40	277	964	-	551	322
6FS	189	-	1591	-	337
6CS-30	192	1171	-	430	323
6CS-40	193	1009	-	575	327

Table 3.5 Quantities of materials to prepare one m³ of mortar mixes in second stage

Mix Notations:

1.	4FS	: 1:4 mortar with FS only.
2.	4CS-30	: 1:4 mortar with 70% CS & 30% GP.
3.	4CS-40	: 1:4 mortar with 60% CS & 40% GP.
4.	6FS	: 1:6 mortar with FS only.
5.	6CS-30	: 1:6 mortar with 70% CS & 30% GP.
6.	6CS-40	: 1:6 mortar with 60% CS & 40% GP.

S. No.	Test	Specification
1.	Consistency of mortar (Flow)	IS 2250 (1981)
2.	Fresh density	ASTM C 185 (2002)
3.	Compressive strength	IS 2250 (1981)
4.	Water absorption, dry bulk density, apparent density and % voids	ASTM C 642 (2006)
5.	Sorptivity	ASTM C 1403 (2006)
6.	Drying shrinkage	ASTM C 1148 (2002)
7.	Adhesive strength	BS EN 1015-12 (2000)
8.	Tensile bond strength	ASTM C 952 (2002)
9.	Ultra pulse velocity test	IS 13311 (1992)
10.	Dynamic modulus of elasticity	Pozo-Antonio (2015)
11.	Salt crystallization	BS EN 12370 (1999)
12.	Wet – dry cycles	BS EN 14066 (2013)
13.	Carbonation	RILEM CPC 18 (1988)
14.	Rapid freezing and thawing	ASTM C 666 (2003)
15.	Fire test	ISO 834
16.	Acid test	ASTM C 267 (2001)
17.	Sulphate test	ASTM C 267 (2001)

Table 3.6 Details of the tests performed

3.4 Experimental methodology

The procedure followed in tests tabulated in Table 3.6 to evaluate the physical, mechanical and durability properties of cement mortars are discussed below.

3.4.1 Flow table test

The flow table test was used to fix the water content of mortars to achieve the necessary workability in their fresh state. The test was performed on each mortar mix as per guidelines mentioned in IS 2250 (1981). For each mortar mix, water requirement was

expressed in terms of water cement ratio. Flow value for each mortar mix was kept constant in the range of 105 to 115% of diameter of base of standard frustum. The mould having top and bottom diameter of 70 and 100 mm, respectively and height of 50 mm was filled in two layers by the fresh mortar. Each layer was tamped 20 times. The mould was taken away from the mortar and the flow table was dropped immediately through a height of 12.5 mm, 25 times in 15 seconds.



Figure 3.9 Flow table test of control mortar

3.4.2 Compressive strength test

The compressive strength test was experimented on 7 and 28 days water cured mortar cubes as per IS 2250 (1981). For each selected curing period (7 and 28 days) four cubes of size 50 mm were tested using a compressive strength testing machine and the achieved values were recorded. The load was applied at a uniform rate of 2 to 6 N/mm² per minute on the specimen. The average of the four values was recorded as the compressive strength at the selected age.

Compressive strength in MPa = $\frac{\text{Load in Newton}}{\text{Area in mm}^2}$



Figure 3.10 Compressive strength of mortar

3.4.3 Ultra Pulse Velocity (UPV) Test and Dynamic Modulus of Elasticity

UPV is a popular in situ, non-destructive test which is globally used to access the quality of mix. The quality of a mix is represented in the terms of uniformity, strength, compactness, internal flaws, cracks etc. Three prisms of size 40 mm X 40 mm X 160 mm were caste for each mortar mix. If pulse velocity between transmitter and transducer across the specimen is high, then it indicates the matrix of mix is compact and possess less voids.



Figure 3.11 Experiment setup for UPV of mortar

Dynamic Young's modulus of elasticity of mortars were estimated by using the pulse velocity method. The expression to calculate dynamic Young's modulus of elasticity (MPa) from UPV as given by Pozo-Antonio (2015)

$$E_{dyn} = 0.001 \text{ x V}^2 \text{ x P}$$

Where,

V = Speed of the longitudinal elastic wave in m/s.

 $\mathbf{P} = \mathbf{Mortar} \ \mathbf{density} \ \mathbf{in} \ \mathbf{g/cm^3}.$

3.4.4 Tensile bond strength test

The tensile bond strength test was performed to determine the bond strength of masonry in tension by crossed-brick couplets method. This test followed the guidelines provided in ASTM C 952 (2002). A crossed brick couplet specimen was prepared using two bricks and bonded together using square mortar paste having each side of 92 mm and 13 mm depth. The formed specimen was compacted using a drop hammer of weight 900 g. The maximum tensile bond strength was calculated as:

Tensile bond strength in $N/m^2 = A/B$

Where,

A = Total applied load in N.

 $B = Cross-sectional area in bond in m^2$.



Figure 3.12 Casting of bricks couplet for bond test



Figure 3.13 Testing of bricks couplet for bond strength test

3.4.5 Adhesion test

This test was conducted to determine the adhesive strength of hardened mortar as plaster on a particular stratum as per the guidelines given in BS EN 1015-12 (2000). A layer of 10 mm fresh mortar was applied on the brick and the specimens were kept in air tight polythene bag at a temperature of 20° C for 7 days. Then these samples were taken out from the polythene bag and were further kept for 21 days in a room having controlled temperature of 20° C and relative humidity of 65%. A core of 50 mm diameter was drilled into the hardened layer of specimens and then the pull heads were attached to the core using a suitable adhesive. A tensile load was applied perpendicular to the test area through pull heads to measure the strength. The adhesive strength was calculated as:

Adhesive Strength in N/mm² = F_u/A

Where,

 F_u = Total tensile load in N.

 $A = Cross-sectional area in mm^2$.



Figure 3.14 Testing of adhesive strength of cement mortar

3.4.6 Test for water absorption and voids of mortar

This test was performed as per ASTM C 642 (2006) on hardened mortar. Specimens of 70 mm cube were oven dried at $110 \pm 5^{\circ}$ C for 24h and were weighed. The specimens were then dried again for 24h and weighed. The successive weighed values for samples were compared and if the value showed more than 0.5% variation, the samples were dried again in oven for 24h and the same procedure was followed till the target variation was achieved. This weighed value was termed as oven dry weight (A). Now the specimens were kept in water of temperature 21°C for 48h and weighed. The specimens were then kept in water for 24h more and weighed. The successive weighed values for samples were compared and if the value showed more than 0.5% variation, the samples were compared and if the value showed more than 0.5% variation, the samples were kept again in water for 24h and the same procedure was followed till the target increment was achieved. This was termed as surface dried weight (B). For apparent weight (C), the surface dried specimen was suspended in water through wire mesh and was weighed. Calculations were done using below formulae:

Water absorption after immersion in % = [(B-A)/A]*100

Permeable voids in % = [(B-A)/(B-C)]*100

Where,

- A = Oven dry weight
- B = Surface dry weight

C = Apparent weight

 $P = Density of water = 1g/cm^3$

3.4.7 Drying shrinkage test

This test was performed as per specifications given in ASTM C 1148 (2002). For measuring drying shrinkage, mortars of 25 mm x 25 mm x 285 mm prisms were casted. Total five specimens were cast for each mix of mortar. The gauge length between the gauge studs were kept 250 mm. The drying shrinkage of mortars was calculated for 7, 14, 21 and 28 days as follows:

Drying Shrinkage of Specimen in $\% = [(L_1-L)/L_0]*100$

Where,

 $L_o = Effective gauge length in mm.$

 L_1 = Initial measurement after removal from moist cure in mm.

L= Measurement after drying in mm.



Figure 3.15 Drying shrinkage of cement mortar

3.4.8 Fresh bulk density

The fresh bulk density was evaluated as per the specifications given in ASTM C 185 (2002). A standard metal container of 400 ml volume was filled by mortar whose empty weight was W_1 . The mortar was filled in four layers. Each layer was compacted by 20 times.

After compacting fourth layer the top surface of container was plain and then weight is noted as W₂.



Figure 3.16 Fresh density of cement mortar

The fresh bulk density of mortar is calculated as

Fresh Bulk Density in $gm/cc = (W_2-W_1)/V$

Where,

 $W_1 = Empty$ weight of container (gm)

 W_2 = Weight of container with mortar (gm)

V = Volume of container (ml)

3.4.9 Sorptivity test

To evaluate the water absorption by capillary action in mortar, sorptivity test was performed as per guidelines given in ASTM C 1403 (2006). In this test 3 cubes of 50 mm size were used for each mix. Initially cubes were oven dried at 100°C for 24h. After this the cubes were waxed by wax from five side and then weight recorded as W. One side is kept exposed to get in touch it with water for capillary rise. Exposed side was dip in water for depth of 3mm. The weight was noted down after 0.25h, 1h, 4h and 24h as $W_{0.25h}$, W_{1h} , W_{4h} and W_{24h} , respectively.



Figure 3.17 Sorptivity of cement mortar

The water absorption was calculated by following expression

Water Absorption after T time in $100 \text{gm/cm}^2 = (W_T - W)*10000/A$

Where,

 W_T = Weight of cube after exposure to water for 0.25h, 1h, 4h and 24 h, gm.

W = Weight of cube after oven dry and waxing, gm.

 $A = Area of exposed surface in mm^2$.

3.4.10 Salt crystallization test

To understand the effect of salt crystallization on mortar mixes with and without GP cubes of 70 mm and 50 mm were exposed to 15 cycles. One cycle includes 24 hours which was divided by 2 hours in 14% solution of sodium sulphate, one hour in cooling environment, 18 hours in oven at 60°C and 3 hours for room temperature. After such 15 cycles change in weight, appearance and water absorption was calculated by using 70 mm cubes. The compressive strength was calculated by using the cubes of 50 mm. The specifications given in BS EN 12370 (1999) were followed in this experiments. The following formulas are used for the change in weight, water absorption and compressive strength for all mortar mixes

Change in weight in percentage = $(W_i - W_{15c})*100/W_i$

Change in water absorption in percentage = $(Q_i - Q_{15c})*100/Q_i$

Change in compressive strength in percentage = $(C_i - C_{15c})*100/C_i$

Where,

Wi	= Weight of cube before salt crystallization test
W_{15c}	= Weight of cube after salt crystallization test
Qi	= Water absorption of cube before salt crystallization test
Q15c	= Water absorption of cube after salt crystallization test
Ci	= Compressive strength of cube after 28 days of curing
C15c	= Compressive strength of cube after 28 days of curing

3.4.11 Wet and dry cycle test

The guidelines of BS EN 14066 (2013) were followed for this test. Cubes of 70 mm and 50 mm of mortar mixes were subjected to 20 cycles of wetting and drying. Each cycle was of 24 hours in which 18 hours for wetting in water of 20°C and 6 hours for drying in oven at temperature of 60°C. After these 20 cycles change in weight, appearance, water absorption and compressive strength for all mixes were evaluated as evaluated in salt crystallization.

3.4.12 Carbonation

The specifications of RILEM CPC 18 (1988) were used for the carbonation of cement mortar mixes. The sample of size 40 x 40 x 160 mm was used to evaluate the resistance against carbonation. The samples were coated by epoxy paint from five side except one face of 40 x 40 mm. These sample were put in carbonation chamber which have 5% concentration of CO₂ (carbon di-oxide) with 50% relative humidity. Carbonation was measured after the exposure of CO₂ for 1, 7, 14, 21, 28, 56 and 84 days. The samples were split longitudinally and a solution of phenolphthalein was sprayed at end of each exposure period. The depth of change in colourless from pink colour for mortar specimens were measured as carbonated depth.



Figure 3.18 Specimen subjected to CO₂ in carbonation chamber

3.4.13 Rapid freezing and thawing test

Mortar specimens were subjected to 20 cycles of rapid freezing and thawing as per the specifications given in ASTM C 666 (2003). Each cycle constituted of 6 hours during which specimens were frozen to -18°C and then thawed to 4°C in succession. Change in weight, compressive strength and appearance were assessed as assessed in salt crystallization.



Figure 3.19 Specimen subjected to freeze and thaw

3.4.14 Effect of direct fire

To understand the effect of fire on mortar, specimens were exposed to real fire in a furnace. Standard fire curve as per ISO 834 was followed by furnace. Tests specimens were subjected to fire for the temperature of 200°C, 400°C, 600°C and 800°C. Weight loss and change in compressive strength of mortars after exposure to fire were calculated by the expression discussed in salt crystallization.



Figure 3.20 Experiment set up for direct fire

3.4.15 Acid attack

To understand the effect of acid on mortar, specimens were immersed in solution of 5% sulphuric acid. The specifications of ASTM C 267 (2001) were followed in this durability test of mortar. Cubes of 70 mm and 50 mm size were exposed to acidic medium and appearance, loss of weight and compressive strength were observed after 1, 7, 14, 28, 56 and 84 days of immersion in sulphuric acid solution. The loss in weight and compressive strength were evaluated by the formulas discussed in salt crystallization.



Figure 3.21 Specimens exposed to solution of sulphuric acid

3.4.16 Sulphate attack

The loss of weight and change in compressive strength were determined after exposing the specimens in a solution of 5% sodium sulphate as per the specifications of ASTM C 267 (2001). The performance was evaluated after the exposure of 1, 7, 14, 28, 84



Figure 3.22 Specimens subjected to solution of sodium sulphate

and 168 days in sodium sulphate solution. The change in weight and compressive strength were evaluated by the formulas discussed in salt crystallization.

3.5 Summary

All the materials were obtained from local dealers. Granite waste was collected from a granite processing industry located nearby the city of Jaipur. Thus complete emphasis was on best utilization of waste with the available materials locally. Experimental work was carried out as per relevant standards.

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 General

Properties of rich and lean cement mortar trial mixes prepared with air dried granite powder were evaluated and compared with those of reference mortar. After this a gradation as per BIS 1542 and BIS 2116 was attempted by mixing coarse river sand (CS) and granite powder (GP) in order to achieve the required specifications of fine aggregate in mortar. The recommended proportion was 70:30 and 60:40 for CS:GP mortar mixes. Mechanical and durability properties were evaluated for mortar mixes 1:4 and 1:6. The results and discussions are presented in detail in subsequent paragraphs.

4.2 Phase-I

In this stage, fine river sand (FS) conforming to BIS 1542 and BIS 2116 was replaced by granite powder (GP) for preparing cement mortar. This replacement was done in the range of 10 to 100% by volume. Two volumetric mix proportions i.e. 1:4 and 1:6 were studied. Properties like workability, fresh bulk density, compressive strength, water absorption and permeable voids were examined for above mentioned mixes. The findings of above results are discussed below.

4.2.1 Workability

The requirement of water for required flow as per BIS 2250 (1981) is shown in Figure 4.1. The required flow for mix 1:4 was achieved at 1.2 water-cement (w/c) ratio for reference mortar specimen and at 1.59 for mix containing 100% GP. An appreciable increase in w/c ratio was observed as 27% for maximum utilization of GP in mortar. For other mix 1:6 the increase in water demand was 22%.



Figure 4.1 Water-cement ratio of mortars

The rough and angular texture of GP (refer SEM in Figure 3.7) was reason behind this increment in water-cement ratio. Due to such texture of GP resistance to flow of mortar was increased which required more water to attain required flow. The greater water absorption by GP as compared to FS may also be another reason for increment in water demand.

4.2.2 Fresh bulk density

The variation in fresh bulk density of cement mortars with increase in GP utilisation has been shown in Figure 4.2. For all the mix proportions with the increase in GP as substitution of FS in cement mortars, density of mortar was found to be decreased. The maximum density was 2.066 gm/cc observed for references mix of 1:4 proportion and least was 1.882 gm/cc for cement mortar prepared with 100% GP in same proportion. The fresh bulk density decreased by 10% and 5% for mortars prepared with 100% GP in 1:4 and 1:6 mix proportions, respectively.

The main reason behind this reduction in density was lower specific gravity of GP. The specific gravity for FS and GP was observed as 2.65 and 2.46, respectively. Additionally, presence of greater amount of water in mixes with GP has also contributed to reduction of the same parameter. The reduction in bulk density was also reported by A.O.Mashley et al. (2018) when granite sludge of lower specific gravity was used in place of ordinary Portland cement of higher specific gravity in cement composite paste.


Figure 4.2 Fresh bulk density of mortars

4.2.3 Water absorption and permeable voids

The variations for water absorption and permeable voids in cement mortars with substitution of FS by GP are graphically represented in Figure 4.3 and Figure 4.4, respectively. From figure it can be seen that water absorption for mortars prepared with GP has increased with the increase in substitution of FS by GP in mortar. The water absorption is 10.98% for reference mix of 1:6 proportion which has raised to 18.72% for mortar with 100% GP. The hike in water absorption is 70%. The water absorption has risen by 50% for mortars with 100% GP in 1:4 mix proportion. The reason behind this increment was different absorption capacity of GP and FS. Water absorption capacity of GP and FS was 15.29% and 8.83%, respectively.



Figure 4.3 Water absorption of mortars



Figure 4.4 Permeable voids of mortars

The variation of permeable voids was found to be same as obtained in water absorption. With the substitution of FS by GP, voids of the mortar had increased. The uniform size of GP above 600 micron (refer Figure 3.2) is responsible for such behaviour. These uniform particles are in excessive amount which are unable to fill voids in mortars.

4.2.4 Compressive strength

The change in compressive strength after 7 days and 28 days of curing is plotted in Figure 4.5 and Figure 4.6, respectively. The compressive strength has decreased with the increased dose of GP in place of FS in cement mortars. The maximum compressive strength of control mixes observed after 28 days of curing was 7.92 MPa and 3.63 MPa for 1:4and 1:6 mix proportions, respectively.



Figure 4.5 Compressive strength after 7 days



Figure 4.6 Compressive strength after 28 days

The least compressive strength after 28 days of curing was 4.7 MPa and 1.81 MPa for 1:4 and 1:6 mix proportion, respectively which is obtained for mortar mixes prepared with 100% GP. The decrement behaviour in compressive strength of mortars prepared with GP was due to enhanced voids developed by excessive water in mortar matrix as discussed in section 4.2.3.

From the above tests on mortars with FS and GP, it was observed that performance of mixes with GP declined as compared to control mortar. The reduction in workability, fresh bulk density and compressive strength is due to presence of excessive finer fraction of granite powder in mortar mixes. Uniform size of GP (refer Figure 3.2) was unable to fill voids in mortars which are responsible for decline of performance. Therefore, to get the mix well graded and at the same time conforming to BIS:1542 and BIS:2116, it was planned to use coarse sand falling in zone-II in place of FS.

4.3 Phase-II

In order to improve performance parameters of mortars containing GP, it was decided to use coarse sand to negate the adverse effect of excessive fines. Mix ratio of 60:40 and 70:30 of CS:GP satisfied gradation recommended by BIS 1542 and BIS 2116 for plaster and masonry mortars respectively. Properties like compressive strength, tensile bond strength, adhesion, drying shrinkage and durability parameters were evaluated for most widely used mortar mixes 1:4 and 1:6 proportion in construction sector. The results have been discussed in following paragraphs.

4.3.1 Workability:

The variation in the water to cement ratio (w/c) has been plotted in Figure 4.7.Water cement ratio for mixes 4FS, 4CS30 and 4CS40 was found to be 1.2, 1.12 and 1.16, respectively. It was noticed that water demand was reduced to achieve required flow for 4CS30 and 4CS40 as compared to control mortar. Similar results have been observed for 1:6 mix proportions. This reduction in w/c ratio was due to filling of voids present in CS by GP which was earlier filled by water. The w/c ratio for 6CS30 and 6CS40 mixes were 1.68 and 1.69, respectively. This slight increase in w/c ratio in CS40 for desired workability is due to high content of GP which is irregular in shape and has rough surface texture as shown in Figure 3.7. On the other side natural fine aggregate have smooth and spherical particles (refer Figures 3.3 and 3.4) in control mortar, require less water content for same workability.





It is interesting to note that demand of water to achieve desired flow has decreased as compared to Phase-I. Reduction in water demand was 8% and 20% in 1:6 and 1:4 mix proportions, respectively. That is because water absorption by CS was less than that of FS and probably some voids in CS were filled by GP particles.

4.3.2 Fresh bulk Density:

The variation in fresh bulk density in mortars prepared with FS and CS:GP can be seen in Figure 4.8. The fresh bulk densities were 2.014 gm/cc and 1.987 gm/cc for control

mortars of 1:4 and 1:6 proportions, respectively. The density increased in mortars prepared with CS and GP. This increment in fresh bulk density is due to pore filling by GP particles. The maximum fresh bulk density was achieved for 70:30 proportion of CS:GP and its values were 2.051 gm/cc and 2.017 gm/cc for 1:4 and 1:6 mix proportions, respectively.





Figure 4.8 Fresh bulk density of mortar mixes

The density was observed reduced in 60:40 proportion mixes due to higher content of GP which has lower specific gravity. This density is still higher than density obtained in mortar prepared with FS (reference mortar) because of denser matrix obtained by filling of voids by GP as observed in SEM images (Refer Figures 4.23 to 4.25). No significant change was observed in these results when compared to mixes using FS in Phase-I.

4.3.3 Water absorption and permeable voids:

The capacity of water absorption in the mortar specimens links with the degree of porosity. The variation of water absorption and permeable voids are plotted in Figure 4.9 and 4.10, respectively. The water absorption of mortar mix 1:4 for CS30 and CS40 decreased by 6.6% and 6.1%, respectively as compared to control mortar (FS). The decrement in water absorption was due to decrease in the porosity of CS30 and CS40 mortar mixes as compared to control mortar (FS). The SEM images of different mortar mixes presented in Figures 4.23 to 4.25 reveal denser packing of particles with granite powder incorporated mix. The UPV test results of these mixes also show 27% improvement over control mortar tests results, indicating a denser structure (discussed in section 4.3.5).



Figure 4.9 Variation of water absorption of mortar mixes

Figure 4.10 Variation of % voids of mortar mixes

The permeable voids and water absorption for blended mortars in 1:6 mortar mix proportion was slightly higher than that of control mortar. The particles of GP above 600 micron size are almost uniform graded as shown in Figure 3.2. Higher content of such uniformly graded material in mix 1:6 give rise to more voids in the mix.

The voids in mixes prepared with CS and GP were found to be reduced by 5% and 6% than the mixes prepared with FS and GP for 1:4 and 1:6 mixes, respectively. But, a very little reduction was observed to water absorption for mixes prepared with CS than that of FS. Overall performance of combination of CS and GP improved with respect to voids and water absorption.

4.3.4 Compressive Strength:

The compressive strength of mortar mixes is shown in Figure 4.11 and 4.12. The maximum compressive strengths for CS40 mix at 28 days for 1:4 and 1:6 proportions were observed 8.21 N/mm² and 4.03 N/mm², respectively. It can be observed that compressive strength of mortar at 7 days and 28 days has slightly increased as compared to control mortar (FS).



Figure 4.11 Compressive strength of mortar mixes at 7 days



Figure 4.12 Compressive strength of mortar mixes at 28 days

This increment in compressive strength may be due to the decrement in w/c ratio as compared to control mortar. The angular shape of particles as seen in SEM images (Refer Figure 3.7) also enhances bond between aggregate thus resulting in higher strength. The observed compressive strength values for CS30 and CS40 mixes were higher than the minimum recommended values for respective mortars 3 MPa and 7.5 MPa as recommended by IS-2250:1981.

Mixes		1:4		1:6	
Properties	% GP	30%	40%	30%	40%
Compressive strength (MPa)	Phase-I	6.34	6.07	2.96	2.93
	Phase-II	7.98	8.21	3.92	4.03
	% Change	20.55	26.07	24.49	27.30

Table 4.1: Comparison of mortars prepared with CS and FS

It can be observed that compressive strength of mixes with 40% GP was found to be 26-27% higher than these observed with FS. The combine effect of reduction in water demand (discussed in section 4.3.1) and filing of voids (refer Figures 4.23-4.25) can be one reason. Enhancement of bond between particles of CS and rough particles of GP might also have contributed for increase in strength.

4.3.5 Ultrasonic pulse velocity and dynamic modulus of elasticity:

The UPV values for all the mixes were determined at the age of 28 days and is shown in Figure 4.13. In mortar mix proportion 1:4, the UPV value of CS30 and CS40 mixes are 13% and 27% higher than that of control mortar mix (FS), respectively which indicates better packing of particles. Similar pattern was also observed in UPV values of 1:6 mortar mix proportion. The UPV values for 6FS, 6CS30 and 6CS40 mixes were 2521, 2554 and 2655 m/s respectively. The reason behind this increment in UPV value was formation of denser CSH gel (as shown in SEM images refer Figures 4.23-4.25), imparting higher ultrasonic pulse velocity.



Figure 4.13 Variation of UPV of mortar mixes

The dynamic modulus of elasticity in mortar was calculated by the equation given by Pozo-Antonio (2015) and the variation in values is shown in Figure 4.14. The dynamic modulus of elasticity of 1:6 mortar mix proportion for CS30 and CS40 increased by 4% and 14% respectively as compared to control mortar (FS). This increment in modulus of elasticity clearly indicates that CS30 and CS40 mortar mixes will suffer lesser deformation for the same level of stress. The dynamic modulus of elasticity was also increased for mortars prepared with GP and CS than that of control mortar in 1:4 mix proportion. The decrement in w/c ratio is responsible for increment in dynamic modulus of elasticity. The variation pattern for the values of compressive strength and the dynamic modulus of elasticity is similar. Such

a linear variation between compressive strength and dynamic modulus of elasticity was also established by Han and Kim (2012).



Figure 4.14 Variation of dynamic of modulus elasticity of mortar mixes

4.3.6 Tensile bond strength:

The variation in tensile bond strength with the change in fine aggregate is shown in Figure 4.15. The tensile bond strength for cement mortar varied from 0.071 to 0.087 MPa and 0.018 to 0.025 MPa in 1:4 and 1:6 mix proportions, respectively. For CS30 mixes, the tensile bond strength increased by 23% and 39% for 1:4 and 1:6 mortar mixes, respectively, as compared to control mortar (FS). It could be due to lesser water-cement ratio than control mix which has led to the formation of a stronger bond between brick and mortar. The angular particle of granite powder also provide a good bond between fine aggregates.

In case of CS40 mixes, both proportions showed a slight decrement than CS30 but was higher than that of control mortar (FS). This decrement was 9% and 16% with respect to CS30 mixes for 1:4 and 1:6 mixes, respectively. This decrement in CS40 mix could be due to presence of excess of granite powder in the mix which get reduced coating of cement on their surface. This leads to a weaker interface and hence loss of bond strength.



Figure 4.15 Tensile bond strength of mortar mixes at 28 days

4.3.7 Adhesion test:

Variations in adhesive strength of test specimens are plotted in Figure 4.16. Increase of granite powder in mortar increased the adhesive strength of both proportions i.e. 1:4 and 1:6. For 1:4 mortar proportion, the adhesive strength increased by 21% and 23% for CS30 and CS40 mixes respectively as compared to control mortar (FS) in 1:4 mix proportion. The increment in 1:6 mortar proportion was 10% and 9% for CS30 and CS40 respectively. All mixes of 1:4 series failed at the interface between the brick and mortar (Figure 4.17(a)), whereas cohesive failure was observed in mortar mixes 1:6 (Figure 4.17(b)). Adhesive strength for mortars prepared with 30% and 40% granite powder for both mix proportions was higher than the conventional mortar. So it can be used for plastering purpose.

Normally cement mortars 1:6 are used for interior plaster work in buildings in the state of Rajasthan. Mortar of 1:4 are most commonly used for exterior plaster work in this region. Hence, use of GP as fine aggregate can be very well utilized for improved adhesion of plaster with background surface.



FS CS30 CS40

Figure 4.16 Adhesion of mortar mixes at 28 days



Figure 4.17 Images of (a) adhesive failure (b) cohesive failure

4.3.8 Drying shrinkage:

Drying shrinkage is the result of loss of water from mortar mix's matrix. The variation of drying shrinkage for mortar test specimens are plotted in Figure 4.18. From this figure it can be seen that, mortar mixes of proportion 1:4 have undergone more shrinkage than the mixes of 1:6 proportion, which is primarily due to excessive cement content in mixes 1:4. Apart from this, inclusion of granite powder has led to increase in drying shrinkage as compared to control mortars. This is despite the fact that, the mixes with granite powder have lesser water content. However, it can be noted that, all three mixes of a series have the same

particle size distribution as seen in Figure 3.2. The fineness of granite powder was the reason behind this behaviour of mortars prepared with granite powder. Granite powder being finer than natural fine aggregate, has more specific surface area exposed, resulting in increase in drying shrinkage.



Figure 4.18 Drying shrinkage of mortar mixes

4.3.9 Dry bulk density and apparent density:

The variation for dry bulk density and apparent density for hardened mortars with and without GP were plotted in Figures 4.19 and 4.20, respectively. The dry bulk density of mortar mixes prepared with granite powder was higher than that of control mortar for both mixes. The maximum dry density was achieved for CS30 mixes. The slight decrement was observed in dry density for CS40 mixes.



FS CS30 CS40



Figure 4.19 Dry Bulk Density of mortar mixes

Figure 4.20 Apparent density of mortar mixes

This behaviour was due to fineness of GP. Initially, GP fill the voids and increase the dry bulk density. In CS40 mixes, some voids are left unfilled due to excessive content of uniform size of GP particles. Water content was also responsible for this behaviour of dry bulk density. Water and dry density is inversely proportional with each other. The water demand was minimum for CS30 mixes which showed maximum density and water demand for control mixes was high resulting minimum density. The same pattern was observed for

apparent density.

4.3.10 Capillary water absorption (Sorptivity test)

The variation in water absorption by capillary suction is presented in Figures 4.21 and 4.22 for 1:4 and 1:6 mix proportion, respectively after time interval of 15 minute, 1 hour, 4 hours and 24 hours. In 1:4 mix proportion, the least water absorption was recorded in 60:40 (CS:GP) mortar. After 24 hours, the value of absorption is 95 gm/100cm². It is slightly lower than the observation obtained for control mortar. This behaviour was due to the pore filling of voids by GP in mortar which has reduced percentage of voids in mortar. Such behaviour of filling of voids by GP in mortar was discussed in Section 4.3.3.



Figure 4.21 Capillary water absorption of mortar mixes for 1:4 proportion



Figure 4.22 Capillary water absorption of mortar mixes for 1:6 proportion

In 1:6 mix proportion, absorption by capillary suction has increased in mortars prepared with GP. The maximum water absorption by capillary suction was 91 gm/100cm2 obtained for mortar prepared with 60:40 (CS:GP) after 24 hours. The voids for mortars prepared with GP in 1:6 mix was slightly higher than that of control mortar (as discussed in Section 4.3.3). The uniform graded particles of GP above 600 micron size (refer Figure 3.2) was found in excessive amount which give rise to higher voids in mix 1:6. The pattern for this result was similar to the results of water absorption after immersion.

4.4 Microstructure analysis

Microstructure analysis was carried out for elaborate discussion on the results obtained for various mortar mixes. Scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), Thermogravimetric analysis (TGA) and X-ray diffraction (XRD) techniques were carried out on selected samples to get clean picture of material distribution, calcium silicate hydrates (C-S-H) formation etc.

4.4.1 Scanning electron microscopy (SEM) analysis

SEM images of mixes 4FS, 4CS30 and 4CS40 are shown in Figures 4.23 to 4.25, respectively after 28 days of water curing. The SEM images of mortar containing 30% and 40% granite powder as shown in Figures 4.24 and 4.25 were relatively compact than that of control mortar (FS) as shown in Figure 4.23.



Figure 4.23 SEM image of control mortar (4FS)



Figure 4.24 SEM image of mortar containing 30% granite powder (4CS30)



Figure 4.25 SEM image of mortar containing 40% granite powder (4CS40)

As it can be clearly seen that voids are predominantly seen in image of control mortar (4FS), whereas widely spread presence of GP can be observed in other two mixes. It can be concluded from SEM images that both CS30 and CS40 mortar mixes are more compact and denser than control mixes. This compact and denser matrix of mortars with GP has improved its mechanical properties than that of control mortar.

4.4.2 X-ray diffraction (XRD) analysis

The X – ray diffractograms for 4FS, 4CS30 and 4CS40 mixes are shown in Figures 4.26-4.28, respectively. The products like C-S-H (Calcium silicate hydrate) and C-H (Portlandite) are formed from hydration of tri-calcium silicate (C₃S). Due to amorphous nature of C-S-H, its study using X-ray diffractograms is difficult. Hence in this we use the counts pertaining to a diffraction angle of 29° to estimate the consumption of C₃S to form C-S-H. The peaks of cement are 352.54 at 29.37°, 221.42 at 29.38° and 215.1 at 29.39° for 4FS, 4CS30 and 4CS40, respectively. The peak value of cement in granite containing mortars are lower than that of control mortar. So it can be concluded that granite containing mortars have greater hydration than control mortar.



Figure 4.26 XRD analysis of control mortar (4FS)



Figure 4.27 XRD analysis of mortar containing 30% granite powder (4CS30)



Figure 4.28 XRD analysis of mortar containing 40% granite powder (4CS40)

C-H formed on hydration of C₃S reacts with mullite in fly ash to form C-A-S-H (Calcium alumino silicate hydrate). This can be identified by the diffraction angle at 20°. The counts for 4FS, 4CS30 and 4CS40 are 82.86 at 20.85°, 183 at 20.85° and 120.15 at 20.81°, respectively. This implies that C-A-S-H is formed in greater quantity for mixes with granite. This higher formation of C-A-S-H shows Si-O-Si polymerization dominates Si-O-Al cross linking which ultimately help in improvement of mechanical properties for granite containing mortars than that of control mortar.

4.4.3 Fourier-transform infrared spectroscopy (FTIR) analysis

FTIR spectrum of the mixes 4FS, 4CS30 and 4CS40 are shown in Figures 4.29 to 4.31, respectively. The hydration of cement can be traced by the band at 1000 cm⁻¹. This band for the individual mixes 4FS, 4CS30 and 4CS40 can be seen at 968, 971 and 975 cm⁻¹ respectively.



Figure 4.29 FTIR of control mortar (4FS)



Figure 4.30 FTIR of mortar containing 30% granite powder (4CS30)

The band for the portlandite can be traced near 3400 cm⁻¹. The bands for the portlandite were available at 3416, 3413 and 3398 cm⁻¹ for 4FS, 4CS30 and 4CS40 mixes, respectively. From the FTIR spectrum it is indicated that slight variation occurs in hydration of cement clinker and portlandite with the introduction of GP by substituting natural fine aggregate.



Figure 4.31 FTIR of mortar containing 40% granite powder (4CS40)

However, the band occurring at around 1000 cm⁻¹ is seen to increase when GP is used instead of natural fine aggregate. Hence from this test it can be concluded that incorporation of granite modifies the hydration reaction (also proved in XRD) and helps in improvement of strength of the mortar mixes.

4.4.4 Thermo-gravimetric analysis (TGA)

The thermogravimetric analysis was performed to evaluate the effect of rising temperature at constant rate. TGA curve for different mortar mixes is shown in Figure 4.32. The loss of weight was observed with rising temperature. The percentage loss in weight was least in mortars prepared with GP and CS than the mortar prepared with FS only. This behaviour was due to the less availability of portlandite which was consumed by reacting mullite (fly ash) and forming C-A-S-H (discussed in Section 4.4.2). The effect of higher hydration depicted in results of mechanical properties for mortars with GP.



Figure 4.32 TGA curves for 4FS, 4CS30 and 4CS40 mortar mixes



Figure 4.33 DTG curvesfor4FS, 4CS30 and 4CS40 mortar mixes

The derivative thermo-graph (DTG) for mortar mixes is shown in Figure 4.33. The curve for each mix shows single major endothermic peak at 105 °C temperature. This peak is the indication of dehydration of calcium silicate hydrates (C-S-H) and minor amount of ettringite. The variation in TGA and DTG curves for mortars prepared with GP and CS was marginal than the curves for mortars prepared with FS only.

4.5 **Performance of Mortar Mixes under exposure to Aggressive Environments:**

In this section, behaviour of mortar samples when exposed to acid and sulphate solutions, salt crystallization, alternate wetting and drying cycles etc. have been discussed. The performance of test samples has been compared with that of reference mortar specimens.

4.5.1 Salt crystallization

To understand the effect of salt crystallization on mortar mixes with and without GP cubes of 70 mm and 50 mm were exposed to 15 cycles. One cycle includes 24 hours which has divided by 2 hours in 14% solution of sodium sulphate, one hour in cooling environment, 18 hours in oven at 60°C and 3 hours for room temperature. After such 15 cycles change in weight, appearance, water absorption and compressive strength are discussed below for all mortar mixes.

4.5.1.1 Change in weight and appearance

The variation in weight of mortars after 15 cycles of salt crystallization has been plotted in Figure 4.34. The maximum weight loss was observed for control mortars. The crystallization of sodium sulphate was probably responsible for the development of internal stresses in mortars. These stresses had promoted rupture which was ultimately responsible for deterioration of mortars leading to weight loss. After the cycles of salt crystallization change in appearance is shown in Figure 4.35 for both series of mixes. Appearance wise CS30 mixes showed less deterioration than the other mortar mixes. Least percentage of voids as discussed in Section 4.3.3 in CS30 mixes was the reason for such behaviour. Mortar mixes of 1:6 proportion had suffered more weight loss than that of 1:4 proportion. This might be because of higher water absorption capacity than that of 1:4 mix proportions.

 $Na_2SO_4 + H_2O = Na_2SO_4.10H_2O....(i)$ Thenardite Water Mirabalite



Figure 4.34 Change in weight of samples when subjected to cycles of salt crystallization



Figure. 4.35 Appearance of samples of mixes 1:4 and 1:6 when subjected to cycles of salt crystallization

4.5.1.2 Water absorption

The variation in water absorption for both series of mixes before and after salt crystallization are drawn in Figure 4.36. The water absorption after the salt crystallization was decreased as compared to water absorption before crystallization in mixes of 1:4 series. The reason behind this behaviour was filling of voids by sodium sulphate. The excessive fines of GP of almost uniform particles above 600 micron in 1:6 series was the reason for showing higher absorption than the control mix. The water absorption after salt

crystallization in mixes of 1:6 series was higher than that of 1:4 series. The increased weight loss by 16% to 22% in mixes of 1:6 series was the symbol of internal cracks which was ultimately the responsible for higher water absorption than that of mixes of 1:4 series.

The loss in weight and water absorption after salt crystallization was more in 1:6 mixes than the 1:4 mixes. The reason was conversion of thenardite (Na₂SO₄) into mirabalite (Na₂SO₄.10H₂O). This conversion occurred due to presence of moisture. The density of thenardite and mirabalite was 2.689 g/cc and 1.466 g/cc, respectively. Such large difference in density had promoted the volumetric expansion. In Section 4.3.10 capillary behaviour of mortars for both series were discussed. This section resulted that 1:6 mixes had higher capillary absorption than 1:4 mixes. That is why 1:6 mixes have higher volumetric expansion than 1:4 mixes which resulted more loss in weight and water absorption in 1:6 mixes.



After 28 days of water curing After 15 cycles of salt crysalization

Figure 4.36 Variation of water absorption after cycles of salt crystallization

4.5.1.3 Compressive strength

The compressive strength before and after conducting the salt crystallization for both series of mixes are shown in Figure 4.37 The maximum increase in compressive strength after the exposure of salt crystallization was found for CS30 mixes. It was increased in 28% and 36% for 4CS30 and 6CS30 mixes, respectively. The maximum increment in CS30 mixes were due to maximum density which is discussed in Section 4.3.9. For other mixes, it was raised by 18% to 28%. The reason for such behaviour was absorbed salt (crystallized sodium sulphate) which fill the voids.



28th day compressive strengthResidual compressive strength after 15 cycles of salt crysalization

Figure 4.37 Compressive strength of mortar mixes when subjected to cycles of salt crystallization

4.5.2 Wet and dry cycles

Cubes of 70 mm and 50 mm of mortar mixes were subjected to 20 cycles of wetting and drying. Each cycle was of 24 hours in which 18 hours for wetting in water of 20°C and 6 hours for drying in oven at temperature of 60°C. After these 20 cycles change in weight, appearance, water absorption and compressive strength for all mixes are discussed below.

4.5.2.1 Change in Weight

The variation in weight after conducting 20 wet and dry cycles are represented in Figure 4.38. No loss in weight was found for mixes prepared with GP in 1:4 mix proportion. The maximum loss in weight was observed at the end of the cycles for the control mix of 1:6 proportion. The loss in weight for all the mixes of both series were considered insignificant.



Figure 4.38 Change in weight of samples when subjected to wet and dry cycles

4.5.2.2 Appearance

The appearance of specimens were shown in Figure 4.39 for both series of mixes at the end of cycles. No change in appearance of mixes of 1:4 series were observed. A little chipping of surface layer was observed for mixes of 1:6 series.



Figure. 4.39 Appearance of samples of mixes 1:4 and 1:6 when subjected to wet and dry cycles

4.5.2.3 Water absorption

The variations in water absorption after 20 cycles of wet and dry cycles for all mixes of both series are shown in Figure 4.40. After conducting cycles, water absorption was increased insignificantly. The mixes of 1:6 proportion have shown slightly higher water absorption than that of 1:4 mix proportions. This was due to higher voids in series of 1:6 mixes.







4.5.2.4 Compressive strength

The variation in compressive strength for all mixes of both series were plotted in Figure 4.41. The mixes of 1:6 series have shown more reduction than that of 1:4 series. The reason was the high porosity of mixes of 1:6 series. The maximum reduction of 19% was observed for control mix of 1:6 series. The mixes prepared with GP in both series have relatively less declination in compressive strength than that of control mortars. The reduction in compressive strength was observed for all the mixes of both series after the cycles. The reduction was due to the cracking up of mortars on cycles of heating and cooling in water which endorse tensile stresses. That allows more water at deeper places and promote further cracking.



28th day compressive strengthResidual compressive strength after 20 cycles of wetting and drying

Figure 4.41 Compressive strength of mortar mixes when subjected to after wet and dry cycles

4.5.3 Rapid freezing and thawing

Mortar specimens were subjected to 20 cycles of rapid freezing and thawing. Each cycle constituted of 6 hours during which specimens were frozen to -18°C and then thawed to 4°C in succession. Change in weight, compressive strength and appearance were assessed. The results are discussed below.

4.5.3.1 Change in Weight

The variation in weight of mortars after 20 cycles of freezing and thawing has been plotted in Figure 4.42. The insignificant loss in weight was observed for all the mixes in both series. Lean mixes were highly effected by freezing and thawing due to less cement content than that of rich mixes. Due to less voids in mortars prepared with GP, very little effect was observed of freezing and thawing in terms of weight loss than that of control mortar.





4.5.3.2 Appearance

The appearance of specimens for both series of mixes at the end of rapid freezing and thawing are shown in Figure 4.43. Only deterioration of surface layer has been observed after rapid freezing and thawing. Mortars with GP were observed with less disintegration of surface due to less absorption capacity than that of control mortar.



Figure. 4.43 Appearance of samples of mixes 1:4 and 1:6 when subjected to cycles of freeze and thaw

4.5.3.3 Compressive strength

The variation in compressive strength after the 20 cycles of rapid freezing and thawing is shown in Figure 4.44. The reduction in compressive strength was observed at the end of rapid freezing and thawing. This reduction in compressive strength was due to the expansion of volume of water in freezing condition which introduces tensile stresses. Test specimens had shown the same behaviour as seen in alternative wet and dry cycles. Mortars mixes of 1:6 proportion suffered more reduction in compressive strength than that of 1:4 mixes. Due to less water absorption capacity of mortars prepared with GP, it has less reduction in compressive strength than that of control mortars.



Residual compressive strength after 20 cycles of freezing and thawing

Figure 4.44 Compressive strength of mortar mixes when subjected to cycles of freeze and thaw

4.5.4 Carbonation

The sample of size 40 x 40 x 160 mm was used to evaluate the resistance against carbonation. The samples were coated by epoxy paint from five side except one face of 40 x 40 mm. These sample were put in carbonation chamber which have 5% concentration of CO_2 (carbon di-oxide) with 50% relative humidity. Carbonation was measured after the exposure of CO_2 for 1, 7, 14, 21, 28, 56 and 84 days. The samples were split longitudinally and a solution of phenolphthalein was sprayed at end of each exposure period. The depth of change in colourless from pink colour for mortar specimens are shown in Figure 4.45 and 4.46.



Figure 4.45 Depth of change in colour after CO₂ exposure in 1:4 mixes



Figure 4.46 Depth of change in colour after CO₂ exposure in 1:6 mixes

From above figures, it was observed that mixes of 1:4 series had less penetration of CO_2 than that of 1:6 series. The reason might be less cement content in 1:6 series. The variation of carbonation in mixes was not significant.

The chemical changes occurred in carbonation chamber has been shown in terms of chemical equation below. CO_2 penetrates into the mortar specimen and react with moisture (H₂O) and generate the carbonic acid (H₂CO₃). This acid reduced its alkalinity and provide a medium in which portlandite [Ca(OH)₂] disintegrated into calcium carbonate (CaCO₃). This

reduction in alkalinity of mortar mixes was responsible for change in colour from pink to colourless.

$$CO_2 + H_2O = H_2CO_3....(ii)$$

 $Ca(OH)_2 + H_2CO_3 = CaCO_3 + H_2O....(iii)$

4.5.5 Effect of direct fire

To understand the effect of fire on mortar, specimens were exposed to real fire in a furnace. Temperatures as per standard fire curve as per ISO 834 was followed by furnace. Tests specimens were subjected to fire with exposure of maximum temperature 800°C. Weight loss and change in compressive strength of mortars after exposure to fire are discussed below.

4.5.5.1 Change in Weight

The variation in loss of weight of mortars are shown in Figure 4.47 and 4.48 for series 1:4 and 1:6, respectively for a temperature range of 200°- 800°C. The loss in weight was found to be less in mortars prepared with GP. This is because of quantity of portlandite [Ca(OH)₂] in mortars with GP was found to be less as it was converted into CASH as discussed in section 4.4.2. Portlandite when heated is disintegrated into lime (CaO) and water. Reduction of loss of water results less weight loss.



 $Ca(OH)_2$ =Heat= $CaO + H_2O....(iv)$

Figure 4.47 Loss in weight after fire exposure in 1:4 mixes



Figure 4.48 Loss in weight after fire exposure in 1:6 mix

The loss in weight of mixes of 1:6 series were slightly higher than that of 1:4 series. It might be due to less water-cement ratio for mixes of 1:4 series. Up to 300°C interlayer water and chemically bounded water were lost from mortar. That is why a little weight loss was found for mortars. When the temperature was more than 500°C, dehydration of cement paste started. This was the reason for rapid weight loss at 600°C and 800°C temperature.

4.5.5.2 Appearance

The variation observed in appearance of specimens after exposure of direct fire is represented in Figure 4.49. Major deterioration on surface was observed at temperature 800°C. Specimens were also exhibited reddish colour at 600°C and 800°C temperature. This might be due to the iron content present in GP.



Figure 4.49 Appearance of specimens after fire exposure

4.5.5.3 Compressive strength

The residual compressive strength of specimens after exposure to direct fire has been plotted in Figure 4.50 and 4.51 for mix 1:4 and 1:6, respectively. Residual compressive strength for mortars prepared with GP were approximately similar to that of control mortar in both series. Initially capillary water and physical water present in mixes were evaporated which accelerate the chemical reaction. That is why compressive strength was increased at temperature of 200°C.

After 3 to 5 minutes when the temperature reached at 400°C, fall in compressive strength was observed for all mixes. This reduction in compressive strength was due to internal pore pressure which was generated because of water vapour developed by heating of chemically bounded water. This reduction could also be due to partial cracking in siliceous aggregate at 350°C as reported in a study by I. Hager (2013).


Figure 4.50 Change in compressive strength after fire exposure in 1:4 mixes

When the temperature reached at 600°C, compressive strength was reduced by 57% to 62%. This decrement in compressive strength could be because of expansion of volume of quartz. A study by Mehta and Monteiro (2006) reported that the expansion of volume of quartz was due to conversion of its form from α to β at temperature of 573°C.



Figure 4.51 Change in compressive strength after fire exposure in 1:6 mixes

At temperature 800°C, decomposition of calcium silicate hydrates took place which ultimately reduced its strength. Horszczaruk et al. (2017) also concluded in their research that at this temperature rupture of ITZ took place due to the difference in the thermal expansion of cement paste and fine aggregate.

4.5.6 Acid Attack

To understand the effect of acid on mortar, specimens were immersed in solution of 5% sulphuric acid. Cubes of 70 mm and 50 mm size were exposed to acidic medium and appearance, loss of weight and compressive strength were observed after 1, 7, 14, 28, 56 and 84 days of immersion in sulphuric acid solution.

4.5.6.1 Change in Weight

The change in weight for mortars are plotted in Figure 4.52 and 4.53 for mix 1:4 and 1:6, respectively. The pattern in weight loss for all mixes prepared with GP has found similar to their control mortar after various days of acid exposure. When the specimens were subjected to acidic medium the loss in weight was observed from 0.72% to 4.79% for mixes of 1:4 proportion.

This loss in weight was due to formation of gypsum after reaction between portlandite and sulphuric acid. With the passage of time passes gypsum started filling the voids which has reduced its loss in weight till 14 days of exposure to acid in mixes of 1:4 series.

Ca(OH) ₂	+ H ₂ SO ₄	=	CaSO ₄ .2H ₂ O(v)
Portlandite	Sulphuric Acid		Gypsum

On continued exposure to acid, C-S-H and C-A-S-H/C-A--H were converted into basanite and ettringite. The product like ettringite has higher volume than C-S-H which reduced adhesion between particles of mortars. That is why weight loss of specimens were increased after 28 days of exposure to acid.



Figure 4.52 Loss in weight after acid exposure in 1:4 mixes

CaSiO₂.H₂O H_2SO_4 $CaSO_4 +$ $Si(OH)_4 + H_2O....(vi)$ += Sulphuric Acid Calcium Silicate Hydrate Basanite Amorphous Silica Water $3CaO.Al_2O_3.12H_2O + 3(CaSO_4.2H_2O) + 14H_2O = 3CaO.Al_2O_3.3CaSO_4.32 H_2O....(vii)$ Calcium Aluminate Hydrate Gypsum Water Ettringite

The weight loss varied from 4.04% to 10.18% for mixes of 1:6 series which has been plotted in Figure 4.51. The specimens for this series completely dismantled after 84 days of exposure to acidic medium. It might be due to less cement content. Trend of less of weight was same as that observed in mixes of 1:4.



Figure 4.53 Loss in weight after acid exposure in 1:6 mixes

4.5.6.2 Appearance

Change in appearance has been plotted in Figure 4.54. The significant surface deterioration of specimens were observed after 28 days of immersion in sulphuric acid. The changes on surface of mortars were observed due to formation of soluble salts of calcium after cation-exchange reaction between cement paste and sulphuric acid. After inspection of appearance of mortar specimens, mortars prepared with GP showed relatively less deformation of surface due to compact matrix of mortar than that of control mortar.



Figure 4.54 Appearance of specimens after exposure to sulphuric acid

4.5.6.3 Compressive strength

The residual compressive strength for mixes of 1:4 and 1:6 proportion are represented in Figure 4.55 and 4.56, respectively. The compressive strength was measured on cube of 50 mm size. For each exposure 3 specimens were used. No significant difference in compressive strength was observed for mixes prepared with GP than that of control mortars.



Figure 4.55 Change in compressive strength after acid exposure in 1:4 mixes

With the increase in exposure of acidic environment the compressive strength of specimens decreased. This decrement was observed due to formation of soluble salts of calcium like gypsum. At a later stage after 28 days exposure expansive ettringite formed which created internal cracks and reduced its compressive strength. The reduction in compressive strength was rapid and significant loss of compressive strength to about 50% was observed at the end of 84 days. The formation of these products are presented above in equations (v), (vi) and (vii).



Figure 4.56 Change in compressive strength after acid exposure in 1:6 mixes

4.5.7 Sulphate Attack

The loss of weight and change in compressive strength were determined after exposing the specimens in a solution of 5% sodium sulphate. The performance was evaluated after the exposure of 1, 7, 14, 28, 84 and 168 days in sodium sulphate solution. Observed data for weight and compressive strength are discussed below.

4.5.7.1 Change in weight

The loss of weight against exposure of sodium sulphate is represented in Figure 4.57 and 4.58. After the exposure of sulphate solution, no significant variation was observed in weight loss of mixes prepared with and without GP.

The formation of gypsum took place after the reaction between the sodium sulphate and portlandite as reported by Mehta and Monterio (2006). The reaction represented below:

$$Ca(OH)_2$$
 + Na_2SO_4 + $2H_2O$ = $CaSO_4.2H_2O$ + $2NaOH....(viii)$
Portlandite Sodium Sulphate Water Gypsum Sodium Hydroxide

Conversion of gypsum into ettringite took place when gypsum reacts with calcium silicate hydrate. This reaction was represented in previous section. Both product like gypsum and ettringite are of expansive nature.



Figure 4.57 Loss in weight after sulphate exposure in 1:4 mixes



Figure 4.58 Loss in weight after sulphate exposure in 1:6 mixes

The major effect of sodium sulphate was observed on mixes after the exposure of 84 days and 168 days. It was due to the formation of internal stresses developed by expansive gypsum and ettringite. The expansive nature of gypsum and ettringite in mortar mixes have reduced their cohesion which ultimately help the reduction of weight as reported by Mehta and Monterio (2006). Neville (2004) also reported the expansion caused due the formation of gypsum and ettringite in mortar mixes.

4.5.7.2 Compressive strength

The observed residual compressive strength after exposure of sodium sulphate is shown in Figure 4.59 and 4.60. This residual strength was average of three cubes of 50 mm. No significant change in compressive strength was observed in mortar mixes prepared with and without GP in both proportion.

Increment in compressive strength was observed up to 84 days exposure sodium sulphate solution for all mortar mixes. This increase in strength was 31% to 55% of original strength of mixes. It was because of formation of sodium hydroxide as by-product as shown in equation (viii). This by-product has provided alkaline environment to the mortar mixes. In this environment hydrated product like C-S-H has shown enhanced stability results in higher compressive strength as reported by Mehta and Monterio (2006).



Figure 4.59 Change in compressive strength after sulphate exposure in 1:4 mixes



Figure 4.60 Change in compressive strength after sulphate exposure in 1:6 mixes

After the exposure of sulphate solution for 168 days the compressive strength has reduced. Neville (2004) and Mehta and Monterio (2006) has reported that this reduction was due to the formation of gypsum and ettringite. This ettringite was of expansive nature which was responsible for internal stresses in mortar mixes. This effect was also observed in weight loss of mixes. Overall it can be observed that there was no adverse effect on compressive strength of mixes after exposure to sulphate solution.

CHAPTER 5

CONCLUSIONS and RECOMMENDATIONS

In the present study, an industrial waste product granite powder (GP) generated from cutting and finishing of granite stones was used as partial replacement of river sand in cement mortars of 1:4 and 1:6 mix proportions. Recommended gradation of fine aggregate as per BIS 1542 (1992) and BIS 2116 (1980) was achieved after mixing 30% and 40% part of granite powder in river sand.

Mechanical properties and durability parameters of the mixes were evaluated for mixes containing 30% and 40% GP. The results were compared with those of control mortar. The following conclusions have been drawn based on the study.

- 1. Water requirement to achieve required flow for mortars with GP slightly reduced as compared to that of control mortars.
- 2. Water absorption capacity and permeable voids of cement mortars with GP were found to be comparable to those of control mortar.
- 3. The marginal increase in compressive strength for cement mortars prepared with GP was observed due to reduction of water requirement. Angular shape of particles of GP might have enhanced the bond between aggregates.
- 4. The maximum hike up to 27% in UPV values was observed for mortar with GP when compared with control mortar. It was probably due to better packing of particles in mortar matrix.
- 5. Dynamic modulus of elasticity has also improved for mortars with GP due to less w/c ratio.
- Tensile bond strength of 1:4 and 1:6 mixes increased by 23% and 39%, respectively.
 Lesser water requirement in GP mortars has provided better bonding between brick and mortar that is why bond strength for such mortars was increased by 39%.
- 7. Adhesive strength has increased by 23% and 10% in 1:4 and 1:6 mix proportions, respectively. All mixes of 1:4 series failed at the interface between the brick and mortar, whereas cohesive failure was observed in mortar mixes 1:6.
- 8. Drying shrinkage values for 1:4 and 1:6 mix proportions were 10% and 77% higher than the drying shrinkage of their respective control mortars. The fineness of granite powder was the reason behind this behaviour of mortars mixes.

- 9. Water absorption by capillary action was found to be reduced due to pore filling of voids in 1:4 mortar mixes with GP. In 1:6 mix proportion, absorption by capillary suction has increased in mortars prepared with GP due to almost uniformly graded particles of GP above 600 micron size.
- 10. SEM images of mortars have shown that more compact and denser matrix were observed in mortars with GP than that of control mortar. This compact and denser matrix of mortars with GP has improved its mechanical properties than that of control mortar.
- 11. XRD analysis revealed that the peaks of C-A-S-H were found as 352.54 at 29.37°, 221.42 at 29.38° and 215.1 at 29.39° for 4FS, 4CS30 and 4CS40 mortar mixes, respectively. The peak value of C-A-S-H in granite containing mortars are lower than that of control mortar. So it can be concluded that granite containing mortars have undergone excessive hydration than that of control mortar. This ultimately help in improvement of mechanical properties for granite containing mortars as compared to control mortar.
- 12. Mortar mixes with granite waste has shown less weight loss and water absorption in rich mixes during process of salt crystallization. The water absorption for granite containing mortars was reduced by 19% to 71% due to filling of voids by sodium sulphate. The loss in weight and water absorption after salt crystallization was more in 1:6 mixes than the 1:4 mixes.
- 13. Insignificant change in weight loss and water absorption for all the mixes of both series were observed after conducting wet and dry cycles. The mixes of 1:6 series have shown more reduction in compressive strength than that of 1:4 series. The maximum reduction in compressive strength of 19% was observed for control mix of 1:6 series.
- 14. Carbon di-oxide penetration in all the mixes was almost same. However rich mixes1:4 had shown more resistance to CO₂ penetration as compared to lean 1:6 mixes.
- 15. The loss in weight after exposure to fire was found to be less in mortars prepared with GP because of less formation of portlandite as it was converted to C-A-S-H. Specimens also exhibited reddish colour at 600°C and 800°C temperature. This might be due to the iron content. When the temperature reached at 600°C, compressive strength reduced by 57% to 62%. This decrement in compressive strength could be because of expansion of volume of quartz.

- 16. No significant difference in residual compressive strength was observed for control mixes and those prepared with GP after exposure in 5% sulphuric acid solution.
- 17. The major weight loss was observed in mixes after the exposure to 168 days of sodium sulphate solution due to the formation of internal stresses developed by expansive gypsum and ettringite. Increment in compressive strength in the range of 31% to 55% was observed up to 84 days exposure in sodium sulphate solution for all mortar mixes.

Granite powder as partial substitute (30-40%) in cement mortar mixes has no adverse effect on mechanical properties. It was observed that water demand reduced to achieve the required workability. Compressive strength, adhesion and tensile bond strengths in mortar mixes were slightly improved as compared to those of reference mortar. Durability parameters like resistance to acid-sulphate solutions, alternative wetting and drying etc. improved. Drying shrinkage in lean mixes exceeded the normal value. Hence broadly 30-40% part of fine aggregate as GP can be used in mortar mixes in all the construction activities.

Recommendations for Future Work

1. A proper combination of granite powder and manufactured sand (M-sand) can be used for the production of cement mortar after achieving gradation recommended by standards.

2. The cement content also be partly replaced by GGBFS in above cement-sandgranite mortar.

3. Potential of granite powder to be used as replacement of surkhi can also be checked in lime-surkhi mortar.

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ANNEXURE

The cost benefit in cement mortars with granite powder is tabulated below:

MATERIAL	QUANTITY	UNITS	Rs./UNIT	AMOUNT
Cement	5.46	Bags	350.0	1911.0
Sand	0.99	m ³	900.0	891.0
Total Cost				2802.0

For 1:4 Control Cement Mortar

For 1:4 Cement Mortar with 30% Granite Powder

MATERIAL	QUANTITY	UNITS	Rs./UNIT	AMOUNT
Cement	5.58	Bags	350.0	1953.0
Sand	0.71	m ³	900.0	639.0
Granite Powder	0.30	m ³	0.0	0.0
Total Cost				2592.0

For 1:4 Cement Mortar with 40% Granite Powder

MATERIAL	QUANTITY	UNITS	Rs./UNIT	AMOUNT
Cement	5.54	Bags	350.0	1939.0
Sand	0.60	m ³	900.0	540.0
Granite Powder	0.40	m ³	0.0	0.0
Total Cost				2479.0

For 1:6 Control Cement Mortar

MATERIAL	QUANTITY	UNITS	Rs./UNIT	AMOUNT
Cement	3.78	Bags	350.0	1323.0
Sand	1.03	m ³	900.0	927.0
Total Cost				2250.0

For 1:6 Cement Mortar with 30% Granite Powder

MATERIAL	QUANTITY	UNITS	Rs./UNIT	AMOUNT
Cement	3.84	Bags	350.0	1344.0
Sand	0.73	m ³	900.0	657.0
Granite Powder	0.31	m ³	0.0	0.0
Total Cost				2001.0

For 1:6 Cement Mortar with 40% Granite Powder

MATERIAL	QUANTITY	UNITS	Rs./UNIT	AMOUNT
Cement	3.86	Bags	350.0	1351.0
Sand	0.63	m ³	900.0	567.0
Granite Powder	0.42	m ³	0.0	0.0
Total Cost				1924.0

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- L.K. Gupta, K.I.S.A. Kabeer, A.K. Vyas, "Effect on physical and mechanical properties of cement mortar prepared with waste granite powder as secondary aggregate" at UKIERI Concrete Congress 2019.

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Engineering)	University, Kota			
B.Tech. (Civil	Rajasthan Technical	2012	71.01 (Honours)	
Engineering)	University, Kota	2012		
XII Std	Board of Secondary	2008	79 54	
	Education, Rajasthan	2000		
X Std	Board of Secondary	2006	86 33	
	Education, Rajasthan	2000	00.55	