Experimental Study on Influence of Ceramic Waste and Fly ash as Part of Cement and Fine Aggregate on Microstructure and Mechanical Property of Cement Paste and Mortar

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Abstract : **Cement is mainly utilized for construction in present time because of its need but simultaneously its drawbacks to environment also require attention and also in the last 3 decades, the attention in consuming substitute material wastes in construction has grown continuously. An extensive amount of waste like ceramic waste powder and fly ash are being generated all around the world. These wastes are mostly sent to the landfill without considering recycling option, if we sent waste material directly to the environment, it might cause serious environmental issues. While Such ceramic waste powder and fly ash in fine particle forms has good potential in the infrastructure industry. In this study we can use waste materials Fly ash and ceramic waste as partially replacement of cement and fine aggregate for the purpose of use industrial by-product as supplementary cementnious material as well as fine aggregate and reduce waste product of industry as filling and decrease CO2 emission in environment. For that purpose make paste of different mixes and done pilot study on it and the strength and durability properties of a mortar and concrete comprising ceramic waste powder and fly ash were investigated. Properties studied include compressive strength,sorptivity,water absorption and carbonation and acid attack. Micro structure test scanning electron microscopy and X-ray diffraction are done on paste and mortar for crystalline structure and morphological characteristics.**

Keywords—Ceramic Waste powder, Fly Ash, Compressive Strength, Durability, Micro structural test

I. Introduction

For human beings creating durable Structures is very important. Be it for residential purpose or other purposes. Structures are must be strong and durable for healthy environment for its desire service. There are many different materials available for construction like concrete, brick, stone, and glass, steel etc. They must have some properties like strength, workability, durability and they need to be molded to any shape. Concrete is one of the materials which fulfills all the requirement for easy construction of any structure.[1]

Concrete is the most used construction material in the world. Cement is the main binding material in concrete. Over the past 3 decades, the production of cement has grown rapidly all over the world. The cement production in India is expected to grow three-folds by 2050. However, cement production has major environmental issues that are of concern worldwide. For every one tone of clinker manufactured, approximately one tone of CO_2 is released to the atmosphere, which contributes almost 5-7% of global anthropogenic carbon dioxide emissions. In the manufacturing process of cement, the main sources of gas emissions are combustion of fuels and decomposition of $CaCO_3$ to CaO and CO_2.[2]

India is the second largest cement producer in the world after China. India produced 280 million tones of cement in the year 2015, with an expected annual growth of approximately 8% to 10% annually in the coming years. As the pressure on the industry is expected to continue to grow, the prices of cement have been rising constantly due to increasing demand and costs of raw materials. Being relatively new, most of the cement plants in India utilize state-of-the-art technologies that are energy and resource efficient. It has been projected that the production of cement in India is expected to reach 550 MT by 2020[2]. So however environmental friendly consideration are increasing globally thus introducing alternative material which are partially replace of cement.

In the year 2012, the total production of ceramic tiles in the world was approximately 11.9 billion m2[8] the amount is increasing by 2.2% each year[3,6,7,9]. It was estimated that as much as 10–30% of the total ceramic production goes to waste[4,6,10] . Most of these ceramic wastes cannot be recycled, and thus present disposal issues at a later age[5,6]. As a result, stated that the use of ceramic waste in concrete contributes to cost and energy savings, ecological balance, and conservation of natural resources.[6]

As one of industrial by-products, fly ash has been commonly used as mineral admixtures to partially replace Portland cement in normal and high-strength concrete. Increased use of industrial by-products leads to a decrease of CO_2 emission and energy consumption[11,12]. The ecological and economic benefits provide incentives to study the influence of fly ash on the performance and durability of cementitious composites. When being incorporated in cementitious composites, fly ash can chemically react with CH (calcium hydroxide) to generate the secondary C-S-H (Calcium-Silicate-Hydrates). The process is well known as pozzolanic reaction[11,13]. So ceramic waste and fly ash both have good pozzolanic behavior ,hence use as cement and fine aggregate replacement.

So we can use Fly ash and ceramic waste as partially replacement of cement and fine aggregate for the purpose of we can use industrial by-product as supplementary cementnious material as well as fine aggregate and reduce waste product of industry at landfill or as filling and decrease CO2 emission in environment and also reduce use of natural available fine aggregate like river sand. It therefore leads to eco-friendly construction associated with disposing of waste materials.

II. SIGNIFICANCE OF THE WORK

In the present study the feasible application of ceramic waste powder and fly ash as partial cement and partial fine aggregate replace investigated. An extensive experiment research to consider the addition 0,10,20,30% of ceramic waste powder and 20% of fly ash as cement replacement was arranged for paste and done pilot study for different mixes to perform consistency test, fineness test, initial and final setting test, soundness test and best result take for mortar and 0,10,20,30% of ceramic waste replace with fine aggregate replace for mortar mixes. Then the done mechanical and durability properties for different mortar mixes. Best result of mortar apply for concrete and also perform mechanical and durability properties for different mixes. Best result from mortar and concrete mixes apply for micro structure test like XRD,SEM for paste and mortar sample.

III. MATERIALS

1. Cement

Cement used for our research work was OPC 53 grade confirming to IS12269[14]. The physical properties like specific gravity,consistency,initial and final setting time,soundness test,fineness test was carried out and results were obtained in table.

Physical Properties	ResultObtained	Requirements as per IS 12269[14]
Colour	Grey	--
Fineness modulus	4%	<10%
Standard consistency	30%	25-35%
Specific gravity	3.15	3.1-3.6
Soundness	2mm	10mm
Initial setting time(minutes)	100	Min. 30
final setting time(minutes)	270	Max. 600
Compressive strength(Mpa)		
3 days	28	Min 27(Mpa)
7 days	39	Min 37(Mpa)
28 days	55	Min 53(Mpa)

2. Ceramic waste powder

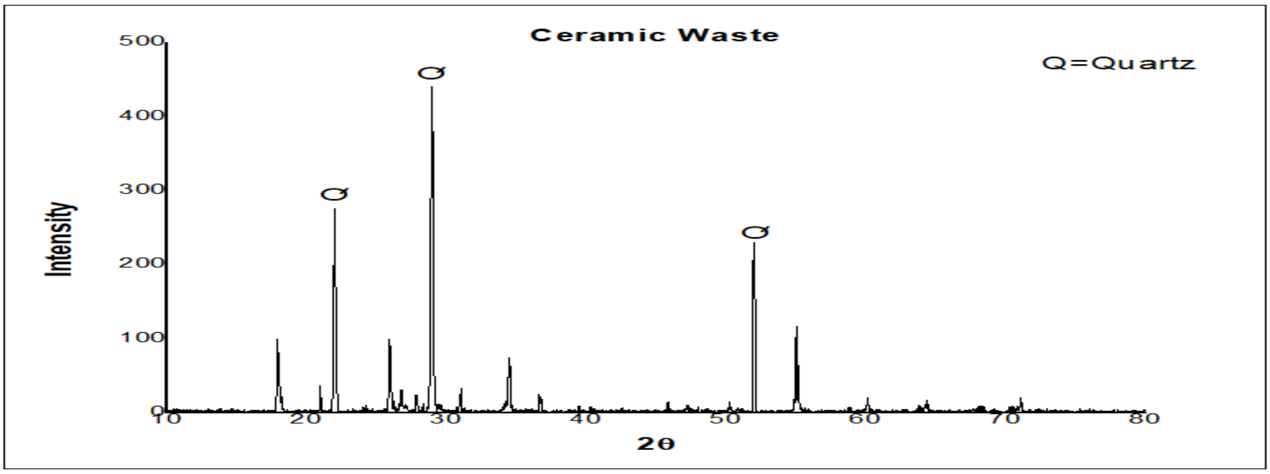
There are two different types of ceramic waste used as replacement of cement and sand. Both type of ceramic waste was collected from locally available dumping site. Also in order to find shape and angular particles of CWP XRD and SEM were investigated. SEM shows irregularly shaped particles with a smooth surface and sharp edges and mineralogical composition was determined by XRD. The data in XRD figure indicate that quartz is the major crystalline phase, with mullite, albite and diopside also present as minor constituents.

Results of physical property of waste ceramic sludge

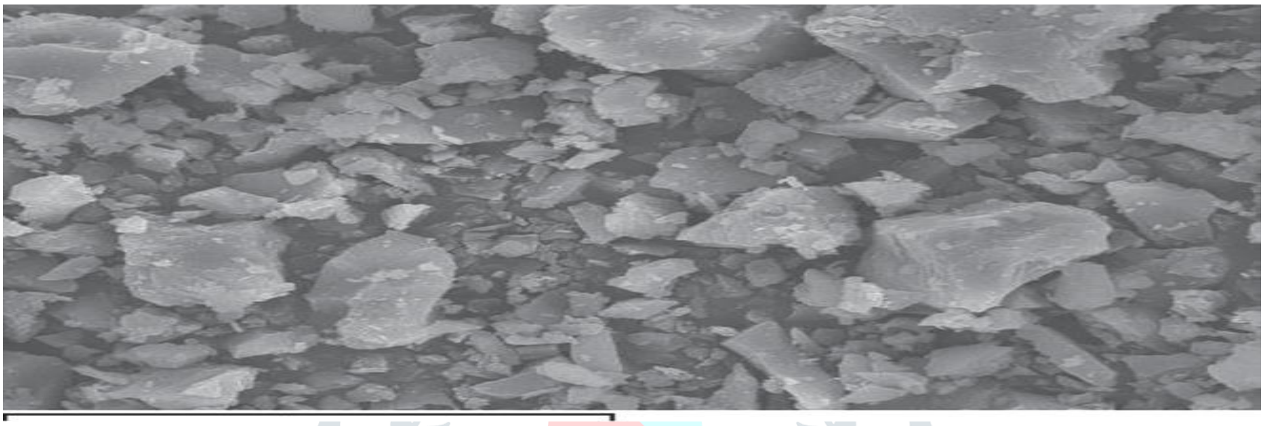
Partially replace with cement	**Specific gravity**	2.67	**Partially replace with sand**	**Specific gravity**	2.67
	Fineness modulus	8%		**Fineness modulus**	1.2%
	Colour	White		**Colour**	White

Chemical Composition of Ceramic Waste powder

Component	Weightage (%)
LOI	2.1
SiO2	53.6
Fe2O3	1.95
SO3	0.3
Al2O3	32.93
MgO	5.9



XRD of Ceramic waste



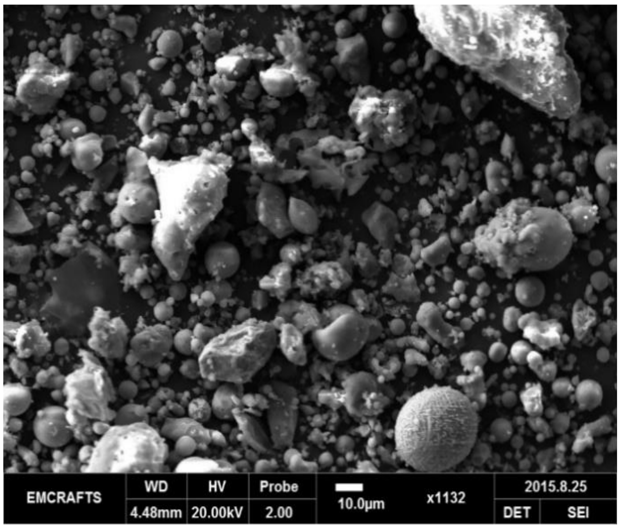
SEM OF CERAMIC WASTE[26]

3. FLY ASH

Fly ash receive from GTPS Gandhinagar, which is class F as per ASTM C618. Physical properties of fly ash is color of fly ash is grey, fineness modulus is 6% and specific gravity is 2.2. XRD patterns of unhydrated fly ashes are shown in Figure. Two types of phases can be seen in unhydrated fly ash: amorphous phase or glass phase, which is the actual reactive phase of the fly ash, and the main crystalline phases such as mullite, quartz and hematite were observed from diffractogram. XRD analysis of fly ash shows high intensity of quartz peaks. Figure show scanning electron micrographs(SEM) of the unhydrated fly ashes. Largely, the fly ashes contain spheres and cenospheres. Unburnt carbon, which depends on the characteristics of coal and its combustion process. SEM image of fly ash shows angular and irregular particles with smooth surface and sharp edges.

Chemical Composition of Fly ash

Component	Weightage (%)
LOI	0.90
SiO2	62.13
Fe2O3	0.40
SO3	0.88
Al2O3	32.14
MgO	0.95



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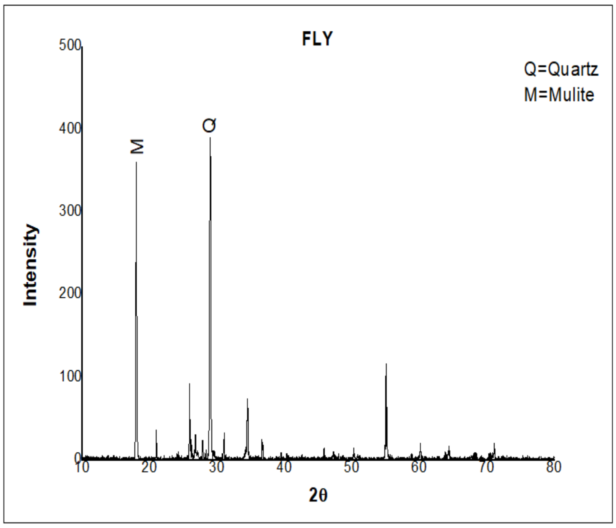


SEM and XRD of Fly ash

4. FINE AGGREGATE

River sand used in research work. Rounded aggregate provide good packing characteristics so we used river sand. Sand is replace with ceramic waste powder from 0 to 30% in range of 10%. For the fraction of particles which pass through 4.75 mm sieve and retained on 600 micron for fine aggregate. According to particle size the fine aggregate is divided into four zone as per IS: 383(1970)[15]. Ceramic waste powder screened through 4.75 mm sieve to remove larger particles.

Fine aggregate properties

Physical Properties	Result Obtained	Requirements
Sand Fall In Zone	II	-
Fineness Modulus	2.53	2-4
Water Absorption (%)	0.4	Max – 2%
Sp. Gravity	2.67	2.5-2.7
Bulk Density	1.65 gm/cm^3	1.52-1.68 gm/cm^3
Ceramic waste powder replace partially fine aggregate		
Specific gravity	2.67	2.5-2.7
Fineness modulus	1.2%	2-4
Colour	White	-

5. COARSE AGGREGATE

In this work the size of aggregate is used up to 20 mm. There are no any replacement for coarse aggregate. Grading of coarse aggregate conforming to IS 383:1970[15].

Results of coarse aggregate properties

Physical Properties	Result Obtained	Requirements
Fineness Modulus	7.31	6.50-8
Water Absorption (%)	0.8	Max – 2%
Sp. Gravity	2.70	2.5-2.7
Bulk Density	1.60 gm/cm3	1.52-1.68 gm/cm3

6. WATER

Water used for this work for mixing and curing was good quality and having Ph value within permissible limits (IS 456: 2007)[16]

IV. METHODOLOGY OF WORK

For pilot study different mixes of cement and paste are made and perform cement test for all mixes. Cement replace 0 to 30% with ceramic waste and 20% for fly ash. Also make cube of paste of size 70.6mmx70.6mmx70.6mm. for micro structure test.

Also done pilot study for ceramic waste which is replace fine aggregate from 0 to 50% and done compressive strength test for that. After result of pilot study for mortar fine aggregate replace 0 to 30% for new mixes of mortar and done mechanical and durability test on it. Make cube of size 70.6mmx70.6mmx70.6mm for compressive strength test and for durability perform water absorption, sorptivity, carbonation depth. After done compressive strength it is check for micro structure test.

From compressive strength result of mortar mixes different proportion of mortar apply for M35 grade of concrete in which coarse aggregate not replace by any waste. Make different mixes of concrete and perform mechanical and durability test like compressive strength, acid attack and sorptivity for that make cube of size 150mmx150mmx150mm.

V. RESULTS AND DISCUSSOIN

1. CEMENT PASTE RESULTS

PILOT STUDY RESULTS (CEMENT PASTE)

REMARK (cement+flyash+ceramic waste)	SA (100+0+0)	M1 (50+20+30)	M2 (60+20+20)	M3 (70+20+10)	M4 (80+20+0)	M5 (80+0+20)
Consistency test (%)[17]	30	32	32	31	31	31
FINENESS TEST(gm)[18]	4	6	6	4	4	4
SOUNDNESS TEST(mm)[19]	2	3	4	3	3	2
INITIAL SETTING TIME(minutes)[20]	100	115	110	105	105	120
FINAL SETTING TIME(minutes)[20]	270	260	245	255	285	260

From pilot study of cement paste take we take proportion of SA, M2 , M3 , M4 for mortar mixes.

2. CEMENT MORTAR RESULTS (PILOT STUDY)(SAND)

Pilot study results (sand)

Remark	Cement(%) +sand(%) +ceramic waste powder(%)	Average load (KN)	Average Compressive strength (N/mm^2)[21]
C0	100+100+0	270	54.16
C1	100+90+10	240	48.15
C2	100+80+20	250	50.15
C3	100+70+30	250	50.15
C4	100+60+40	210	42.13
C5	100+50+50	190	38.11

From pilot study we find out when we replace fine aggregate with ceramic waste than from 0 to 30% strength increase for further increase in ceramic waste strength is decrease. So for fine aggregate replace with ceramic waste from 0 to 30%.

3. CEMENT MORTAR RESULTS

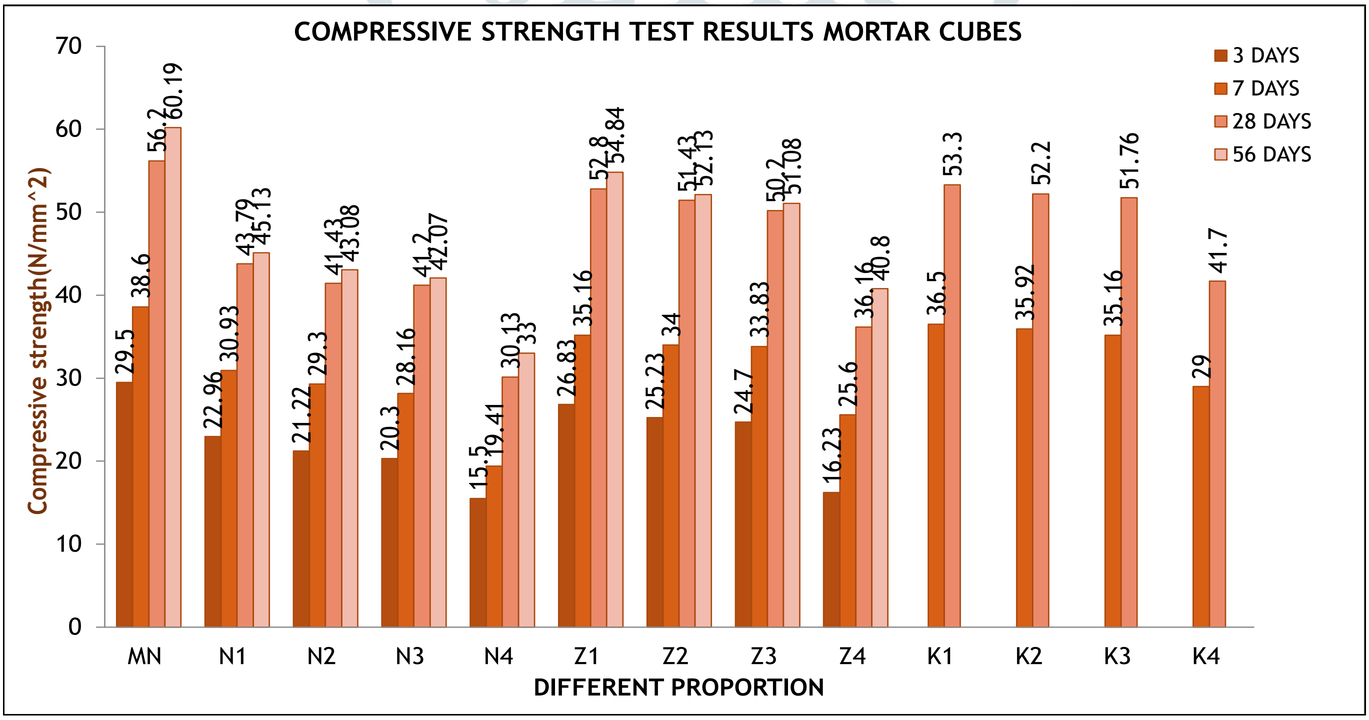
3.1 . COMPRESSIVE STRENGTH TEST

Compressive strength test was carried out as per IS 516:1956[21]. The test setup for compressive strength put in figure. Final strength of average three number of specimen were prepared. Test was carried out for 3,7,28,56 days. So it can be found that from test results when fine aggregate replace with ceramic waste up to 20% there is increase in strength but when further increase in ceramic waste strength is decrease. For K1,K2,K3,K4 test done only for 28 and 56 days.

Remark	Cement+flyash+ceramic waste+sand +ceramic waste (%)	Compressive strength (N/mm^2) (3,7,28 ,56days)			
MN	100+0+0+100+0	29.5	38.6	56.2	60.2
N1	60+20+20+100+0	22.96	30.93	43.79	45.1
N2	60+20+20+90+10	21.22	29.3	41.43	43.1
N3	60+20+20+80+20	20.3	28.16	41.2	42.1
N4	60+20+20+70+30	15.5	19.41	30.13	33
Z1	70+20+10+100+0	26.83	35.16	52.8	54.8
Z2	70+20+10+90+10	25.23	34	51.43	52.1
Z3	70+20+10+80+20	24.7	33.83	50.2	51.1
Z4	70+20+10+70+30	16.23	25.6	36.16	40.8
K1	80+20+00+100+0		36.5	53.3	
K2	80+20+00+90+10		35.92	52.2	

K3	**80+20+00+80+20**		35.16	51.76	
K4	**80+20+00+70+30**		29	41.7	

Compressive strength test perform



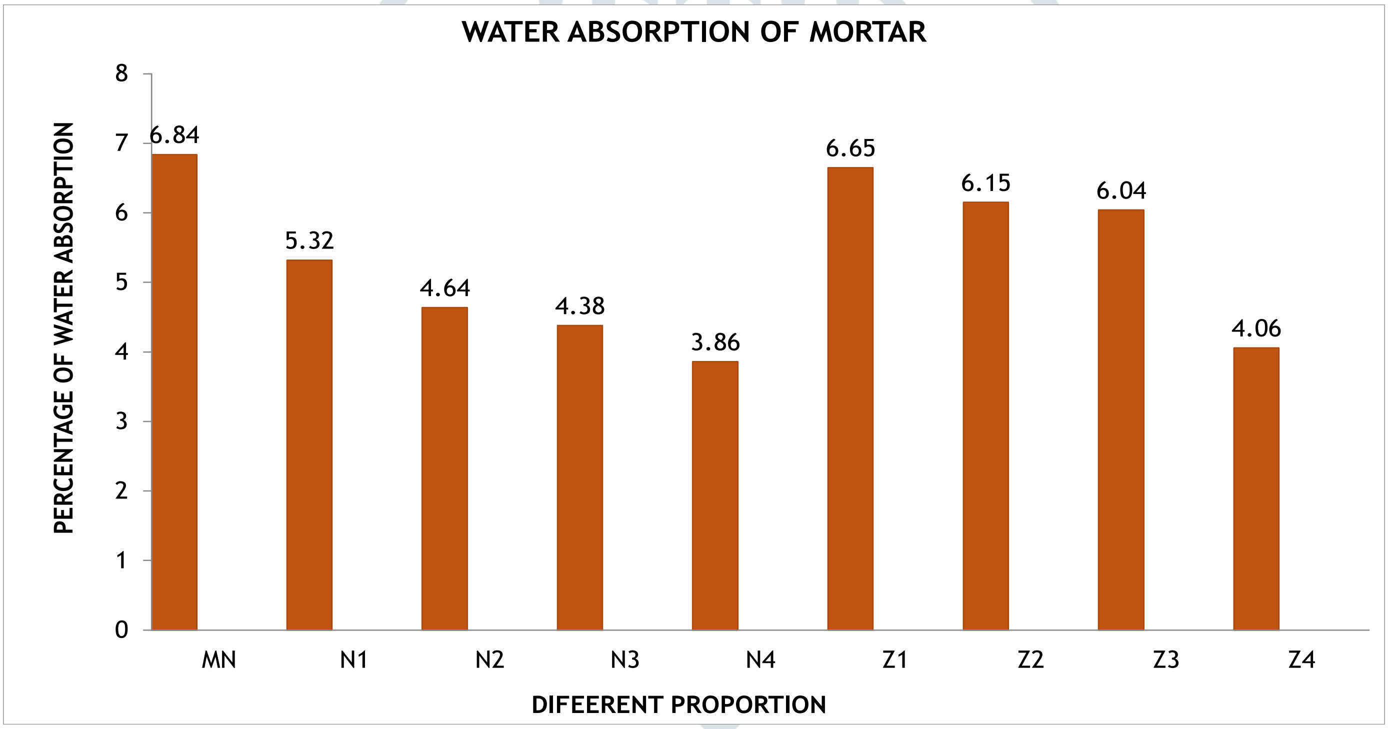
3.2 WATER ABSORPTION TEST

As per ASTM 1403-15[22] the specimen is dried in a ventilated oven at a temperature of 105 ^{0}C to 115 ^{0}C till it attained constant mass. The specimen cooled at room temperature and its mass is W1 noted. The completely dries specimen is immersed in clean water at temperature of 27 ^{0}C for 24 hours. The specimen is removed and wiped out of any traces of water with a damp cloth and weighted W2.

Remark	Cement+flyash+ceramic waste+sand +ceramic waste (%)	Dry weight in grams (W1)	Wet weight in grams (W2)	Water Absorption(%)
MN	**100+0+0+100+0**	760	812	6.84
N1	**60+20+20+100+0**	695	732	5.32
N2	**60+20+20+90+10**	732	766	4.64
N3	**60+20+20+80+20**	729	761	4.38
N4	**60+20+20+70+30**	761	790	3.86
Z1	**70+20+10+100+0**	691	737	6.65

Z2	**70+20+10+90+10**	731	776	6.15
Z3	**70+20+10+80+20**	711	754	6.04
Z4	**70+20+10+70+30**	665	692	4.06

Curing and drying mortar specimen



3.3 CARBONATION DEPTH TEST

Carbonation is a process by which carbon dioxide from the air penetrates into mortar through pores and reacts with calcium hydroxide to form calcium carbonates. It has been seen that the conversion of Ca(OH)2 in to Caco3 by the action of Co2 results in a small shrinkage. The measurement of carbonation depth using phenolphthalein solution. Spraying the indicator on surface of concrete cube. The solution become pink in carbonated concrete.

$$Ca(OH)_2 + Co_2 \Longrightarrow CaCo_3 + H_2O$$

Remark	Cement+flyash+ceramic waste+sand +ceramic waste (%)	Carbonation depth(mm)
MN	**100+0+0+100+0**	Nil
N1	**60+20+20+100+0**	Nil
N2	**60+20+20+90+10**	Nil
N3	**60+20+20+80+20**	1
N4	**60+20+20+70+30**	Nil
Z1	**70+20+10+100+0**	Nil
Z2	**70+20+10+90+10**	2
Z3	**70+20+10+80+20**	Nil

Z4	**70+20+10+70+30**	Nil

Carbonation test

3.4 SORPTIVITY TEST

As per ASTM C 1585-04[23]The sorptivity test is a simple and rapid test to determine the tendency of concrete or mortar to absorb water by capillary action.

The slope of the line of best fit of these points (ignoring the origin) is reported as the sorptivity.

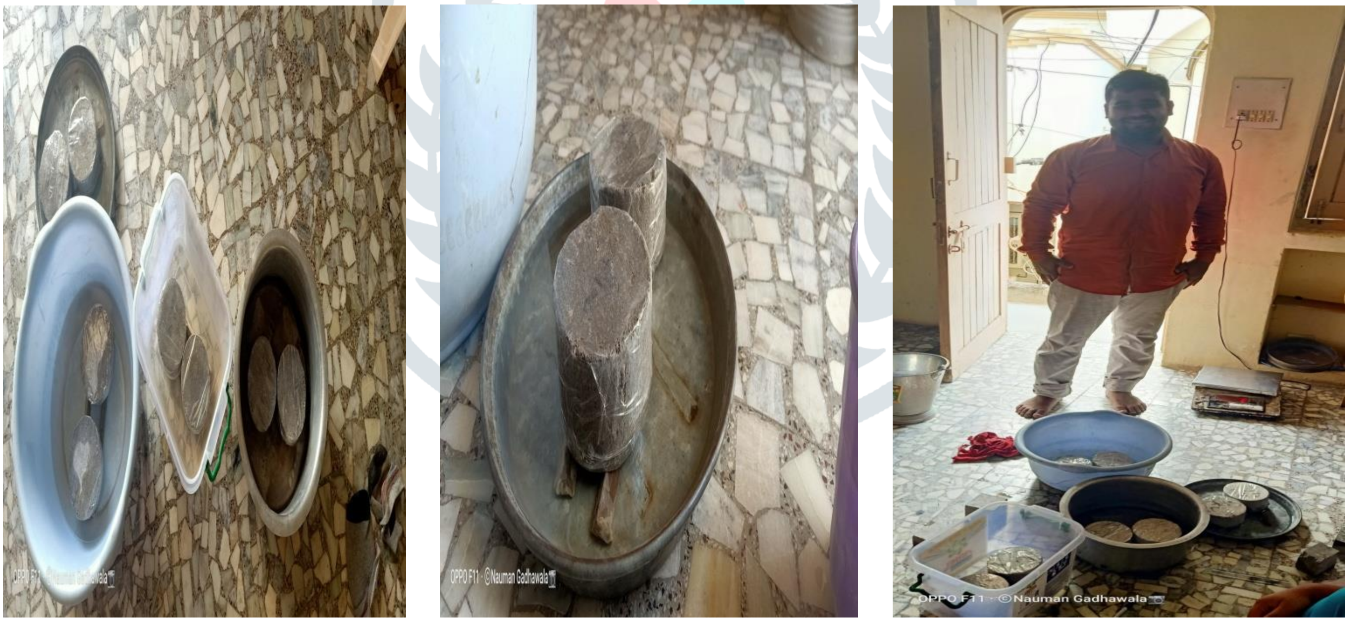
For one dimensional flow, it is written as:

i= st0.5

Where, i = cumulative water absorption per unit area of flow inflow surface.

s= sorptivity.

t= time elapsed.



Sorptivity test and results

MN	N1	N2	N3	N4	Z1	Z2	Z3	Z4	K1	K2	K3	K4
0	0	0	0	0	0	0	0	0	0	0	0	0
10.97	11.22	12.25	11.96	17.14	9.37	5.92	2.96	12.95	10.97	11.22	12.25	11.96
11.1	11.59	12.45	18.74	17.76	9.62	7.27	3.82	12.95	11.1	11.59	12.45	18.74
11.1	11.71	12.82	19.48	17.76	9.62	7.89	4.31	12.95	11.1	11.71	12.82	19.48
11.1	11.71	12.95	19.61	17.88	9.74	8.26	4.81	12.95	11.1	11.71	12.95	19.61
11.1	11.71	12.95	19.61	17.88	9.74	8.38	4.93	12.95	11.1	11.71	12.95	19.61
11.1	11.71	12.95	19.85	17.88	9.74	8.38	5.05	12.95	11.1	11.71	12.95	19.85
11.1	11.71	12.95	19.85	17.88	9.74	8.38	5.18	13.07	11.1	11.71	12.95	19.85
11.1	11.71	12.95	19.85	17.88	9.74	8.38	5.18	13.07	11.1	11.71	12.95	19.85
11.1	11.71	12.95	19.85	17.88	9.74	8.38	5.18	13.07	11.1	11.71	12.95	19.85
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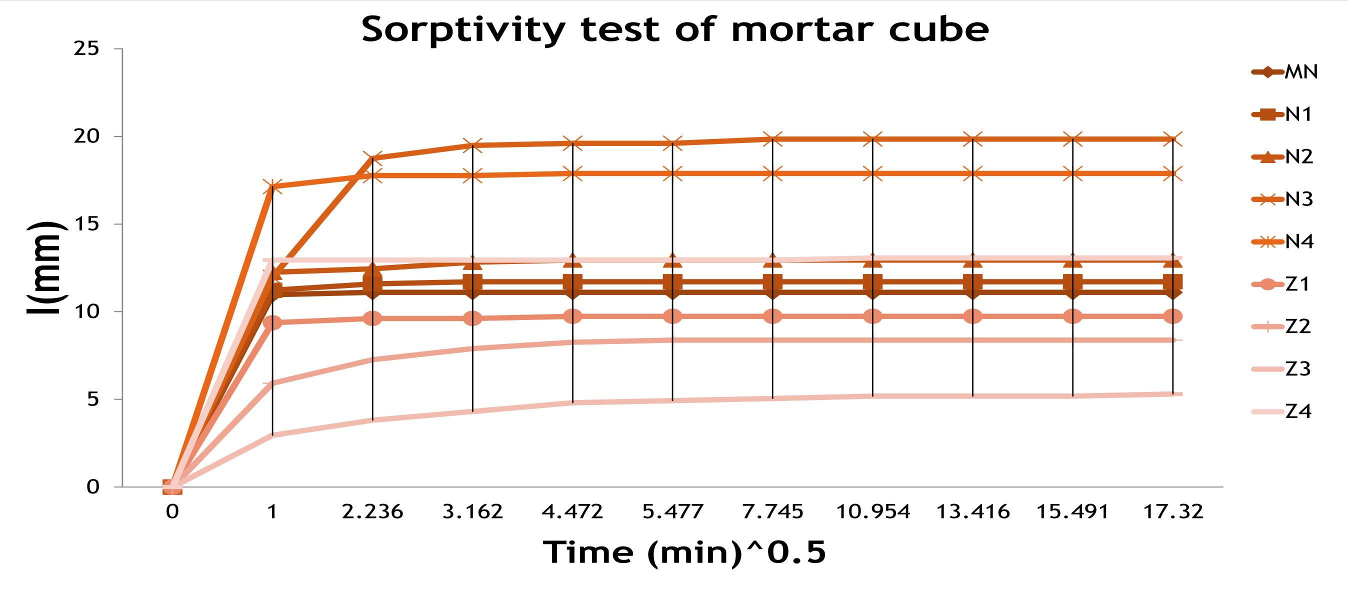
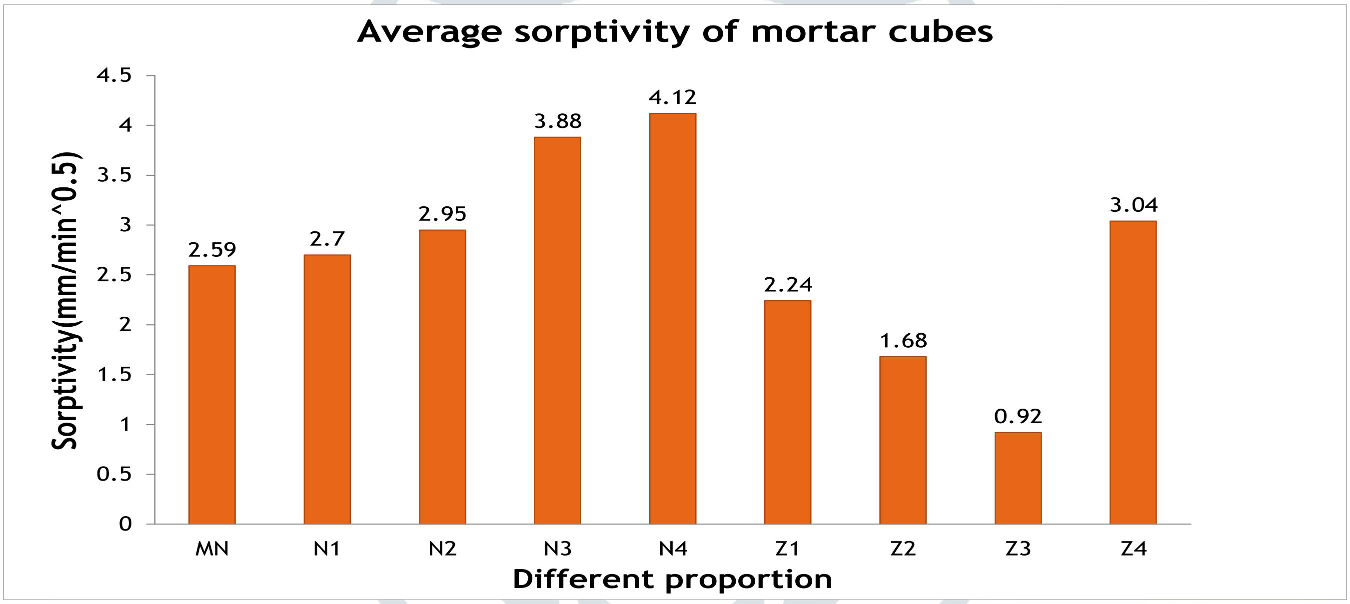




4. CONCRETE RESULTS

After done mechanical and durability test on different mortar mixes best result apply for M35 grade of concrete. There are no any replacement for coarse aggregate. Mix design is done for M35 grade of concrete. for $1m^3$ of concrete given mix design according to IS 10260:2019[24]. For M35 grade of concrete done mechanical and durability test like compressive strength ,acid attack and sorptivity.

Mix design proportion

Mix Proportions for M-35 grade					
Mix Ratio	W/C Ratio	Water	Cement	Sand	Aggregates
1:1.53 : 2.85	0.45	192	427	655	1220

MIX DESIGN PROPORTION

Remark (cement+fly ash+ceramic waste+sand+ceramic waste+coarse aggregate)	GA (100+0+0+ 100+0+10 0) (%)	GA1 (60+20+20 +70+30+10 0) (%)	GA2 (60+20+20 +80+20+10 0) (%)	GA3 (70+20+10 +70+30+10 0) (%)	GA4 (70+20+10 +80+20+10 0) (%)	GA5 (80+20+00 +70+30+10 0) (%)	GA6 (80+20+00 +80+20+10 0) (%)
Density (kg/m3)	2500	2500	2500	2500	2500	2500	2500
Cement (kg/m3)	451.35	270.81	270.81	315.5	315.5	361.08	361.08
C.A (kg/m3)	1291	1291	1291	1291	1291	1291	1291
F.A (kg/m3)	692	485	554	485	554	485	554
Fly ash(kg/m3)	0	90.3	90.3	90.3	85.4	90.3	90.3
C.W.P. (kg/m3)	0	90.3	90.3	45.13	45.13	0	0

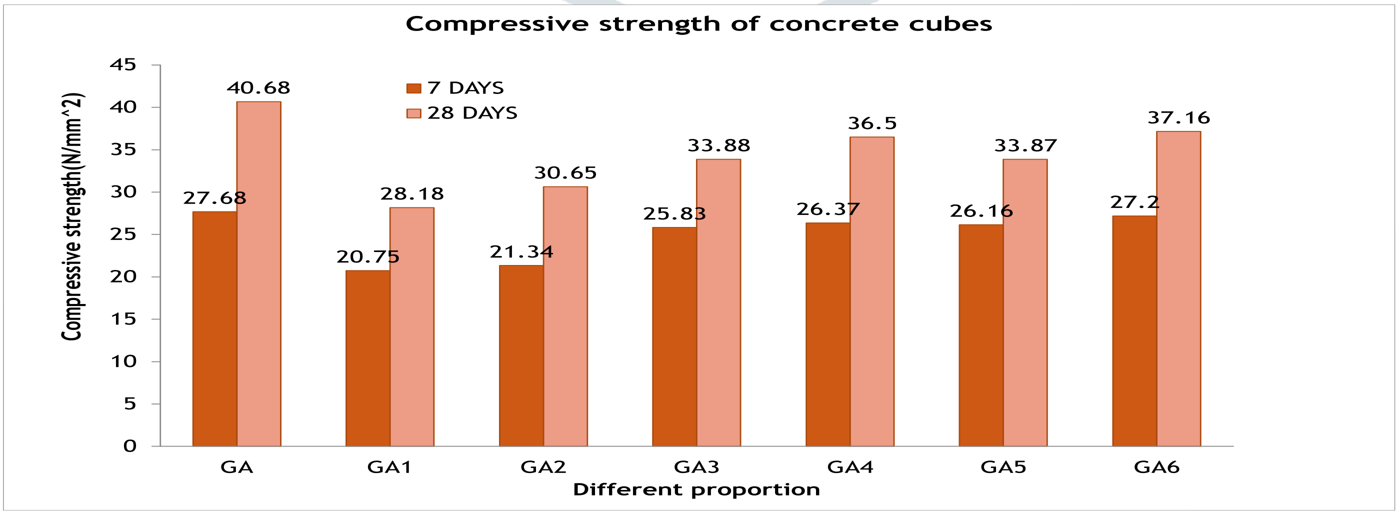
C.W. (kg/m3)	0	207.4	138	207.4	138	207.4	138
Ratio Of Mix Proportion	1:1.53:2.85	1:1.53:2.85	1:1.53:2.85	1:1.53:2.85	1:1.53:2.85	1:1.53:2.85	1:1.53:2.85

4.1 COMPRESSIVE STRENGTH TEST

Compressive strength test was carried out as per IS 516:1956[21]. The test setup for compressive strength put in figure. Final strength of average three number of specimen were prepared. Test was carried out for 7,28 days. So it can be found that from test results when fine aggregate replace with ceramic waste up to 20% there is increase in strength but when further increase in ceramic waste strength is decrease.

Compressive strength test perform

Remark	Cement+flyash+ceramic waste+sand +ceramic waste+coarse aggregate (%)	Compressive strength (N/mm^2) (7,28 days)	
GA	100+0+0+100+0+100	27.68	40.68
GA1	60+20+20+70+30+100	20.75	28.18
GA2	60+20+20+80+20+100	21.34	30.65
GA3	70+20+10+70+30+100	25.83	33.88
GA4	70+20+10+80+20+100	26.37	36.5
GA5	80+20+00+70+30+100	26.16	33.87
GA6	80+20+00+80+20+100	27.2	37.16



4.2 ACID ATTACK TEST

according to ASTM C 267-97[25] Concrete cube of size 150x150x150 mm are prepared. After that the Specimen are casted and curing in mould. After 24 hour all the specimens are demoulded and kept in tank for 7 days curing. After 7 day all specimen are kept in atmosphere for 2 days for constant weight, subsequently, the specimens are weighed and immersed in 5% hydrochloric acid (HCL) solution for 56 days. For preparing the sulfuric acid (HCL) solution, 95 gm of water and 5 gm of acid is added. pH value of the solution was checked after preparing of the solution. At every 15 days interval, pH was checked by measuring initial pH value at the time of preparing solution and it should be constant stable up to completion of the test. After 56 days of immersing, the specimens are taken out and washed in running water and kept in atmosphere for 2 days. After that specimens are weighted and from this loss of weight was calculated. Also check the change in compressive strength.

Acid attack results

Remark	Weight after water curing(kg)	Weight of cube after 56 days in acid(kg)	Initial strength (n/mm^2)	Strength of cube after 56days (n/mm^2)	Reduction instrength after 56 days in acid (n/mm^2)
GA	8.535	8.425	42.68	36.13	6.55
GA1	8.534	8.368	28.18	23.33	4.85
GA2	8.180	8.152	30.65	26.13	4.52
GA3	8.714	8.530	33.88	27.77	6.11
GA4	8.873	8.757	40.16	34.67	5.49

Acid attack test

4.3 SORPTIVITY TEST

According to ASTM C 1585-04[23] the sorptivity test is a simple and rapid test to determine the tendency of concrete or mortar to absorb water by capillary action.

The slope of the line of best fit of these points (ignoring the origin) is reported as the sorptivity.

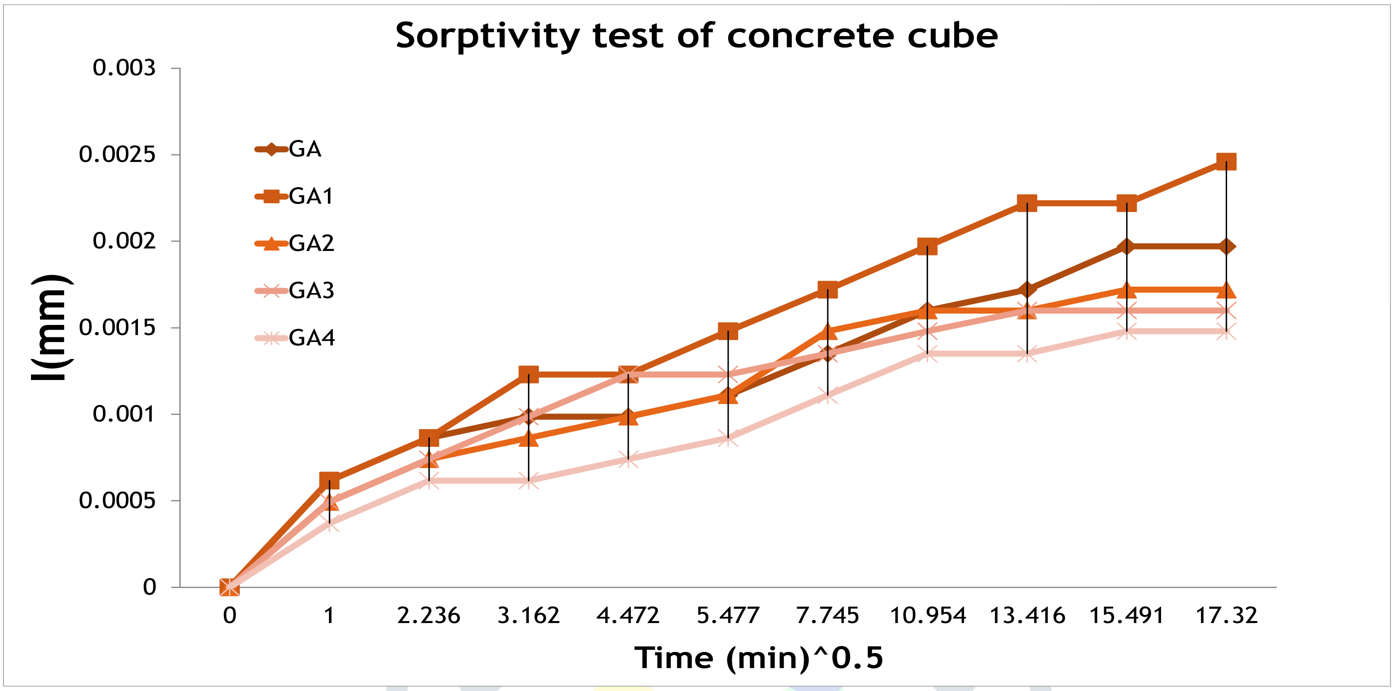
For one dimensional flow, it is written as:

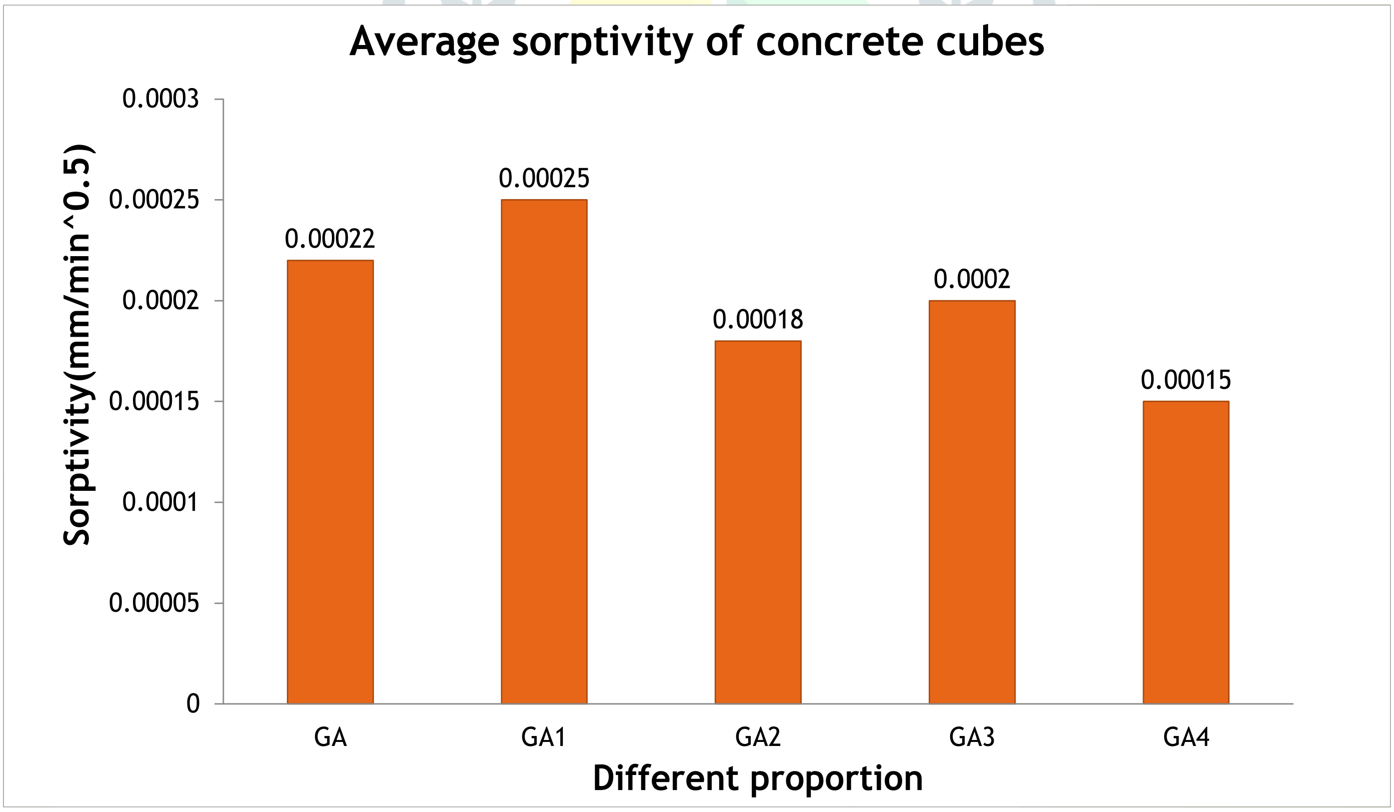
i= st0.5, Where, i = cumulative water absorption per unit area of flow inflow surface, s=sorptivity, t=time elapsed

Sorptivity test and results

GA	GA1	GA2	GA3	GA4
0	0	0	0	0
0.0006167	0.00037	0.0006167	0.000493	0.000493
0.000863	0.0006167	0.000385	0.00074	0.00074
0.000986	0.0006167	0.000388	0.000986	0.000863

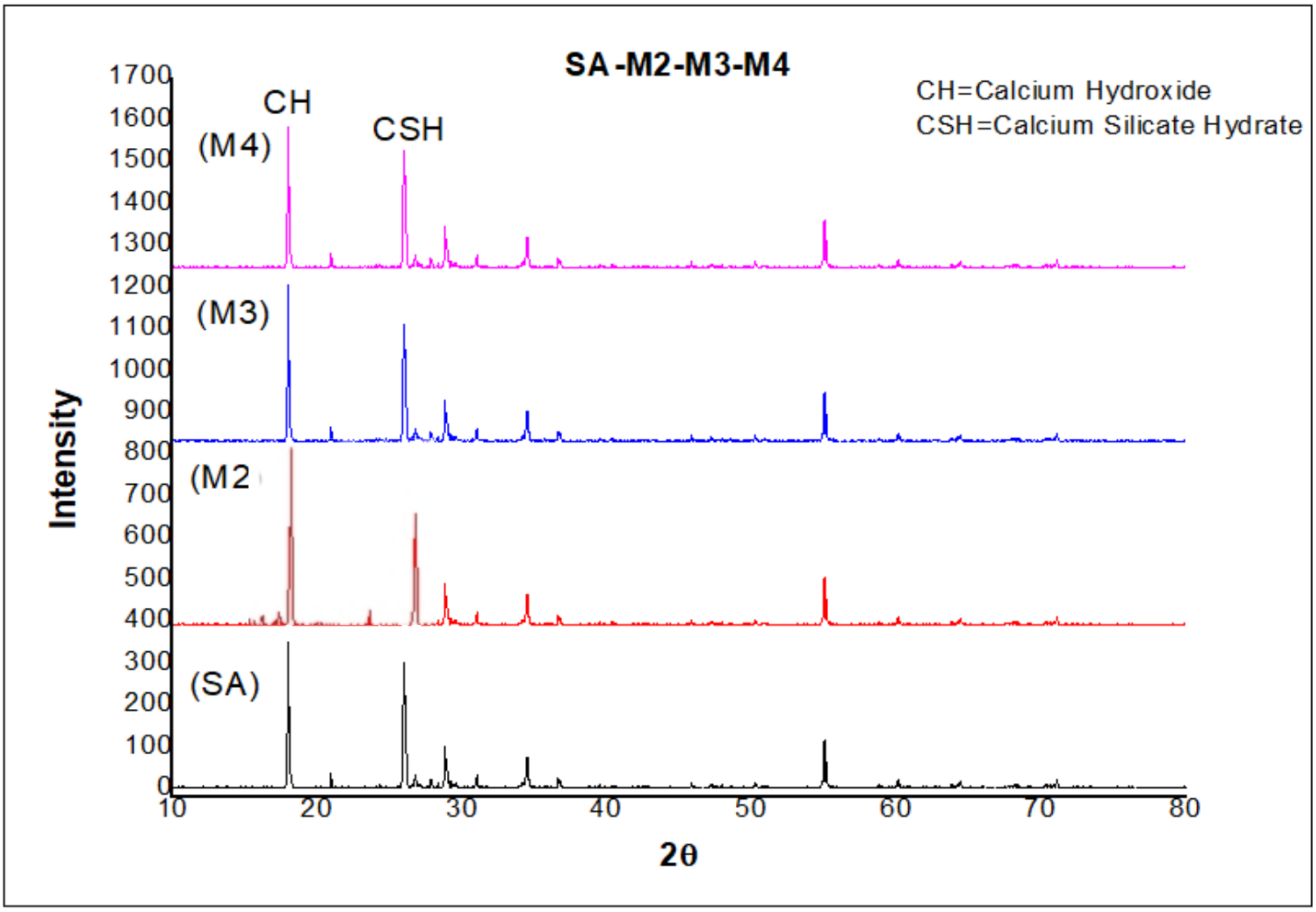
0.000986	0.00074	0.000275	0.00123	0.000986
0.00111	0.000863	0.00027	0.00123	0.00111
0.00135	0.00111	0.000222	0.00135	0.00148
0.0016	0.00135	0.000179	0.00148	0.0016
0.00172	0.00135	0.000165	0.0016	0.0016
0.00197	0.00148	0.000143	0.0016	0.00172
0.00197	0.00148	0.000142	0.0016	0.00172



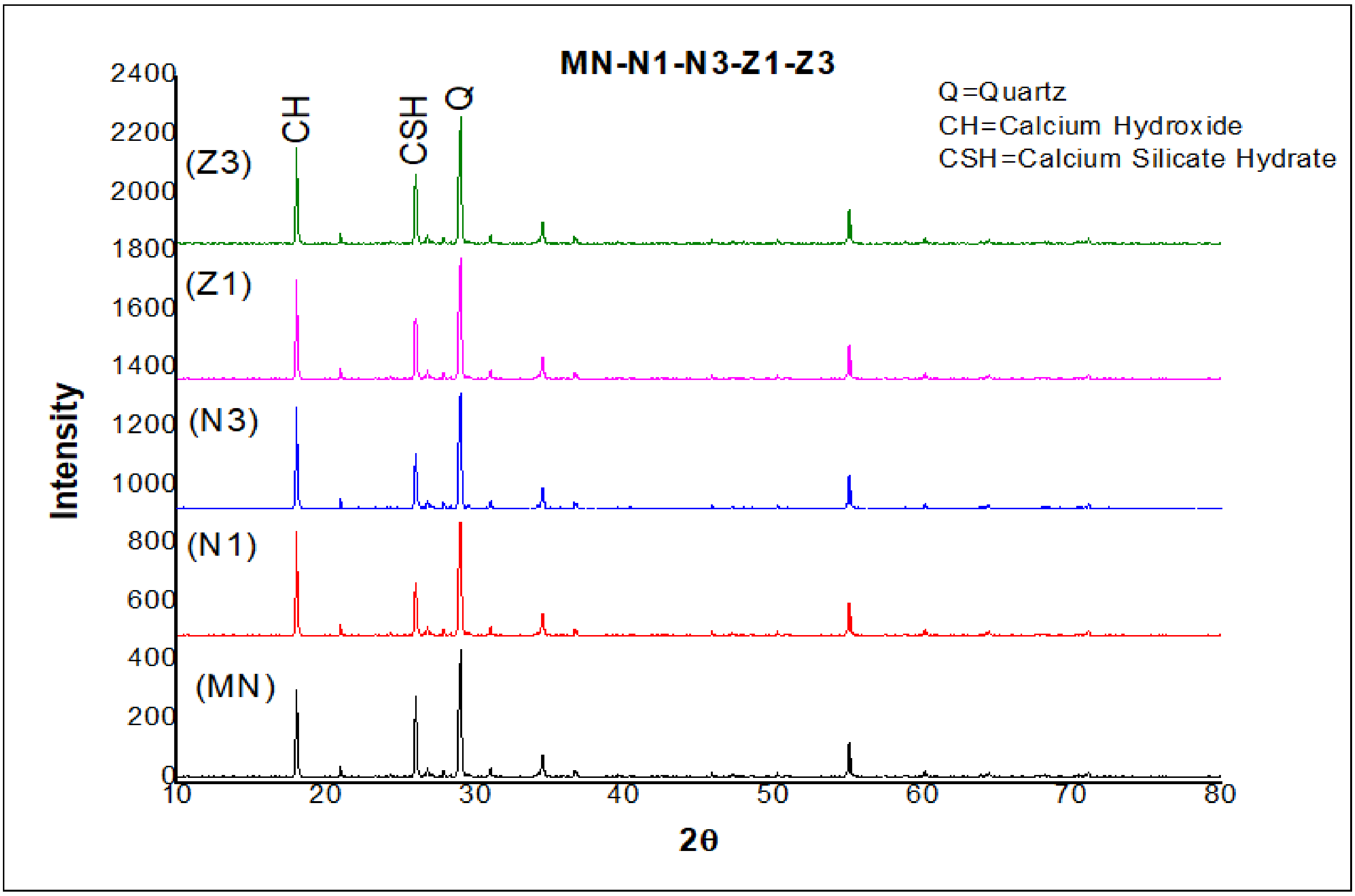


5. MICRO STRUCTURE RESULTS

XRD test for cement paste is done for SA,M2,M3,M4 mixes and for cement mortar done for MN,N1,N3,Z1,Z3 mixes.



XRD of paste mixes(SA,M2,M3,M4)



XRD of mortar mixes (MN,N1,N3,Z1,Z3)

6. Conclusion

- From study and research paper fly ash is replace 20% and ceramic waste powder is replace up to 30% in the range of 10% with cement.
- From pilot study fine aggregate replace with ceramic waste up to 30% in the range of 10%
- Compressive strength for mortar and concrete shows strength increase for ceramic waste replace with sand at 20% due to good bonding and further increase in ceramic waste strength decrease.
- Water absorption test shows when percentage of sand and cement increase there are increase in water absorption due to increase in fine particles. Example MN mortar mixes have highest water absorption than other mixes.
- Sorptivity test shows when ceramic waste replace from 0 to 20% with sand sorptivity decrease continuously and further increase in ceramic waste sorptivity increase.
- XRD tests result illustrated that addition of ceramic waste and fly ash improved the performance of the composite cement upto 20% replacement. It is due to the pozzolanic nature of ceramic waste and fly ash and formation of additional C-S-H gel contributed towards the reduction of $Ca(OH)2$, the pores fill up with hydration products and consequently enhanced the micro structural, strength and durability performance of mortar.

7. Future Scope

- For mechanical properties test only done compressive strength test for mortar so done flexural strength ,tensile strength test for future scope.
- Done other test of durability for mortar and concrete are chloride ion penetration , sulphate attack , water retentivity permeability,density,porosity etc.
- Not use admixture for mix design of concrete so use admixtures in mix design of concrete
- Mix design is done for higher grade of concrete must be required.Example M40,M50,M60 etc.
- Take other supplementary cementinious materials and waste materials and done same process for that.
- Partially or fully replace coarse aggregate with other materials.
- Separate collection system require to collect waste from the industries.

8. Referances

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