CHAPTER 3

Experimental Programme, Material and Methodology

3.1 Introduction

This chapter describes the materials used in the experiments, mix design and methodology of experimental procedure. Physical and chemical properties of the materials used for concrete samples have been elaborated in this chapter. Mix design used for concrete samples in the experiment has been tabulated, calculations for which have been separately shown in appendix. The experimental setup, the various types of tests adopted i.e. both conventional and advanced, have been detailed in the later sections.

3.2 Materials

Materials used in this experiment comprise of OPC, PPC 20 mm aggregate, 10 mm aggregate, sand, water, super-plasticizer, acrylic paint and mineral admixtures viz. micro silica, fly ash and GGBS. The source and properties of materials used in the experiment have been detailed in the following text.

3.2.1 OPC and PPC

OPC is the most common type of cement generally used across the globe as a basic ingredient of concrete, mortar etc. OPC contains 98% clinker and 2% gypsum. The properties of cement to Indian Standard IS 8112 (2013). The physical properties such as specific gravity and fineness comply with the code IS 4031 (1999). The physical and chemical properties of OPC are represented in Table 3.1 and 3.2 respectively.

PPC is a kind of blended Cement which is produced by inter-grinding of OPC clinker along with 15-35 present of pozzolanic materials.

The Pozzolanic materials material most commonly used for manufacture of PPC are fly ash (IS 1489:1991 part1)

Experiments have been conducted using OPC and PPC conforming to Indian Standard (IS: 269 and IS: 1489) codes, physical and chemical properties of which are presented in Table 3.1 and Table 3.2 respectively. Brand of OPC and PPC cement used is ACC.

Table 3.1: Physical Properties of OPC and PPC

Properties	**PPC**	**OPC**
Specific gravity	2.92	3.15
Fineness (%)	4	7
Consistency limit (%)	33	32
Initial setting time (min)	120	104
Final setting time (min)	170	155
Soundness (Le-Chatelier Expansion) (mm)	1.0	0.01
Soundness (Auto-Clave Expansion) (%)	0.043	0.023
Surface area(m^2/kg)	382	265
Compressive strength in MPa (28 days)	51.2	54.5

Table 3.2: Chemical Properties of OPC and PPC

Composition (%)	**OPC**	**PPC**
Magnesia (MgO)	1.9	1.61
Sulphuric Anhydride (SO_3)	2.1	2.65
SiO_2	22.9	21.5
AL_2O_3	4.9	9.9

CaO	62.5	45
Fe_2O_3	2.9	2.04
Total alkali	0.59	0.71
Chloride Content	0.017	0.01
Insoluble Residue	2.1	16.73
Loss on Ignition	2.1	1.23
FA content	NA	33

3.2.2 Aggregate

Aggregates constitute the mass and volume to concrete, reduce shrinkage and affect economy. Aggregates constitute nearly 70-80% volume of the concrete. They bear considerable impact on various characteristics and properties of concrete. Two types of aggregates are used i.e. coarse aggregates and fine aggregates. The latter fills the voids in the former.

3.2.2 (a) Coarse Aggregate

Crushed basalt with a maximum size of 20 mm has been used as coarse aggregate. The aggregates retained on 4.75 mm sieve are classified as coarse aggregate. They are granular materials such as gravel or crushed stone, and are used with fine aggregate and cementing material or binder to produce concrete. Coarse aggregate have conspicuous influence upon the flowing ability, segregation resistance and strength of concrete.Sieve analysis and physical properties of 20 mm and 10 mm size aggregate are given in Table 3.3 below. Coarse aggregates used in our experiments are obtained from Gaya and confirm to IS 383:1970.The water absorption and specific gravity tests conducted on the aggregates are in accordance with IS 2386-1963.

3.2.2 (b) Fine Aggregate (Sand)

Sand is the most commonly used fine aggregate for concrete. Fine aggregates are the second most vital ingredient of aggregate phase. Fine aggregates are defined as those aggregates which pass the 4.75 mm sieve but are retained on 75 μm sieve (ASTM C125, 2004). Fine aggregate increases the flow ability and segregation resistance when used at optimum content. Additionally, they modify the strength of concrete when used in varying proportion with cement and coarse aggregate. Sand used in our experiments are obtained from the banks of Son River and pertain to Zone II and confirm to IS 383:1970.

Table 3.3. Sieve Analysis of 20 mm Nominal Size Aggregate

Sample taken - 5 Kg.

IS Sieve Size ‘mm’	Mass Retained ‘gm’	Cum. Mass ‘gm’	Retained ‘%’	Passing ‘%’	Passing as per IS – 383 ‘%’
40	0	0	0.00	100.00	100
20	223	223	4.46	95.54	85 - 100
10	4757	4980	99.60	0.40	0 - 20
4.75	10	4990	99.80	0.20	0 - 5
2.36	2	4992	99.84	0.16	0 - 5
Pan	8	5000	100.00	0.00	0

Table 3.4 Sieve Analysis of 10 mm Nominal Size Aggregate

Sample taken - 5 Kg.

IS Sieve Size ‘mm’	Mass Retained ‘gm’	Cum. Mass ‘gm’	Retained ‘%’	Passing ‘%’	Passing as per IS – 383 ‘%’
40	0	0	0.00	100.00	100
20	0	0	0.00	100.00	100
10	270	270	5.40	94.60	85 - 100
4.75	4560	4830	96.60	3.40	0 - 20
2.36	88	4918	98.36	1.64	0 - 5
1.18	15	4933	98.66	1.34	0 - 5
Pan	67	5000	100.00	0.00	

Table 3.5 Sieve Analysis of Sand

Sample taken - 1 Kg.

IS Sieve Size 'mm'	Mass Retained 'gm'	Cum. Mass 'gm'	Retained '%'	Passing '%'	Passing as per IS – 383 for Zone II '%'
20	0	0	0.00	100.00	100
10	0	0	0.00	100.00	100
4.75	28	28	2.80	97.20	90 - 100
2.36	50	78	7.80	92.20	75 - 100
1.18	98	176	17.60	82.40	55 - 90
600 Micron	278	454	45.40	54.60	35 - 59
300 Micron	448	902	90.20	9.80	8 - 30
150 Micron	91	993	99.30	0.70	0 - 10
Pan	7	1000	100.00	0.00	

Table 3.6 Physical Properties of Aggregates

Sl. No.	Physical property	CA-20 mm	CA-10 mm	FA(Sand)
1	Bulk Density(Kg/m^3)	1670	1570	1750
2	Specific Gravity	2.82	2.73	2.64
3	Water absorption	0.15%	0.25%	1.77%

3.2.3 Super Plasticizer

Super plasticizers are water-soluble organic polymers. They dissolve in water and form long molecules by polymerization reaction. These molecules having negative charges and they can be adsorbed on the surface of the cement particles which having opposite charges. As a result of the dispersion of cement particles by electrical repulsion between these negative charges, the combined water between the cement agglomerations are freed.The locally available SIKAPLAST super plasticizer has been used in the experiments. It is based on Poly- carboxylic ether composition. Its advantages are reduction in required water content in concrete. The optimum amount of the concrete mix after trial is taken as 0.6% by weight of cement. Specific Gravity of super plasticizer is 1.09.

Table 3.7: Properties of Super Plasticizer

Parameter	Observed value
Relative Density	1.058
Dry material content	12.03 %
pH value	6.7
Ash content	0.63 %
Chloride ion content	0.01 %

3.2.4 Mineral Admixtures

3.2.4.1 Micro Silica or Silica Fume

Silica fume is a by-product of the manufacture of Silicon metals from Ferro silicon alloys in the electric arc furnace. Micro silica is a mineral admixture composed of very fine solid glassy spheres of silicon dioxide. Most micro silica particles are less than 1 micron (0.00004 inch) in diameter, generally 50 to 100 times finer than average cement or fly ash particles. The specific gravity of micro silica is 2.22.Silica fume was procured from Quebec Analytica, Varanasi, India. It confirms to IS 15388 (2003). The physical and chemical properties of SF are tabulated in Table 3.8 below.

Table 3.8 Physical and Chemical Properties of SF

Properties	Value
SiO_2(%)	92
Fe_2O_3(%)	1.48
Al_2O_3(%)	1.12
CaO(%)	.6
MgO(%)	.5
SO_3 (%)	.1
Loss on Ignition(%)	1.85
Specific gravity	2.22
Surface area(m^2/kg)	24000
Compressive strength in MPa (28 days)	82

3.2.4.2 Fly Ash

Fly ash is a coal combustion product that is composed of the particulates (fine particles of burned fuel) that are driven out of coal-fired boilers together with the flue gases. Depending upon the source and composition of the coal being burnt, the components of fly ash vary considerably, but all fly ash includes substantial amounts of silicon dioxide (SiO_2) (both amorphous and crystalline), aluminium oxide (Al_2O_3) and calcium oxide (CaO). Fly ash is generally spherical in shape and range in size from 0.5 µm to 300µm. Fly ash being a pozzolanic material has been used as a mineral admixture, partially replacing the cement. Fly ash used for this experimental study is sourced from NTPC, Singarauli and confirms to IS 3812 (Part 1) 2003. The physical and chemical properties of Fly Ash are tabulated in Table 3.9.

Table 3.9 Physical and chemical properties of Fly Ash

Properties	Value
SiO_2(%)	51
Fe_2O_3(%)	6.9
$Al_2O_{3(\%)}$	25
CaO(%)	8.7
MgO(%)	1.8
$SO_{3\ (\%)}$	0.6
Loss on Ignition(%)	1.82
Specific gravity	2.17
Surface area(m^2/kg)	250
Compressive strength in MPa (28 days)	88

3.2.4.3 Ground Granulated Blast-furnace Slag (GGBS)

Ground-granulated blast-furnace slag (GGBS) is obtained by quenching molten iron slag (a by-product of iron and steel-making) from a blast furnace in water or steam, to produce a glassy, granular product that is then dried and ground into a fine powder. Ground Granulated blast furnace slag (GGBS) is a mineral admixture with both Cementitious and pozzolanic properties. The blast furnace slag used in the present study was obtained from SAIL Bokaro India and complies with IS 12089–1987 (2004). The physical and chemical properties of GGBS are tabulated in Table 3.10.

Table 3.10: Physical and Chemical Properties of GGBS

Properties	**GGBS**
SiO_2(%)	34
Fe_2O_3(%)	-
$Al_2O_{3(\%)}$	13
CaO(%)	41
MgO(%)	78
$SO_{3\ (\%)}$	.11
Loss on Ignition(%)	1.8
Specific gravity	2.85
Surface area(m^2/kg)	280
Compressive strength in MPa (28 days)	92

3.2.5 Water

Clear portable water, free from injurious oils, acids, alkalis, organic matter, soluble silts or other deleterious substances which may cause corrosion, dis-coloration, efflorescence etc. is found suitable for use in concrete. IS: 3550 is followed for routine tests, while acceptance tests for water are conducted as per IS: 3025, and Table-1 of IS: 456. The pH value of water shall generally be not less than 6.

3.2.6 Paint

Cubes were coated by Nerolac acrylic paint which consists of acrylic resin. This paint is non-breathable and can help in prevention of CO_2 impregnation. An even thin layer coating is applied in on all the sides of cubes.

3.3 Mix Design

The design of concrete mix proportions has been based on many parameters, such as water binder ratio, type of cement, super plasticizers, replacement ratios of cement and fine aggregate with mineral admixtures such as, SF, FA and GGBS. The details of calculation for deriving the mix proportions are mentioned in the Appendix 1, given at the end of the thesis. The design mix proportions are in accordance with IS 10262 (2009).

The combined grading of all in aggregates used in the experiments are as follows:

i) For design mix corresponding to W/C = 0.4; from the following table and graph, it is concluded that 39 % of coarse aggregate 20 mm, 25 % of coarse aggregate 10 mm and 36 % of sand is used.

Table 3.11 - ALL IN AGGREGATE AS PER IS-383, Table 5 for W/C 0.4

Percentage	39	25	36	**All in aggregate grading**	**IS LIMIT**		**Remarks**
Aggregate size	**20 mm CA**	**10.0 mm CA**	**SAND**		**Lower limit**	**Upper limit**	
Sieve size	**% of Passing**	**% of Passing**	**% of Passing**				
80 mm	–	–	–	–	–	–	See Grading graph
40 mm	39.00	25.00	36.00	100.00	100	100	
20 mm	37.26	25.00	36.00	98.26	95	100	
4.75 mm	0.08	0.85	34.99	35.92	30	50	
600 μ	0.00	0.00	19.66	19.66	10	35	
150 μ	0.00	0.00	0.25	0.25	0	6	

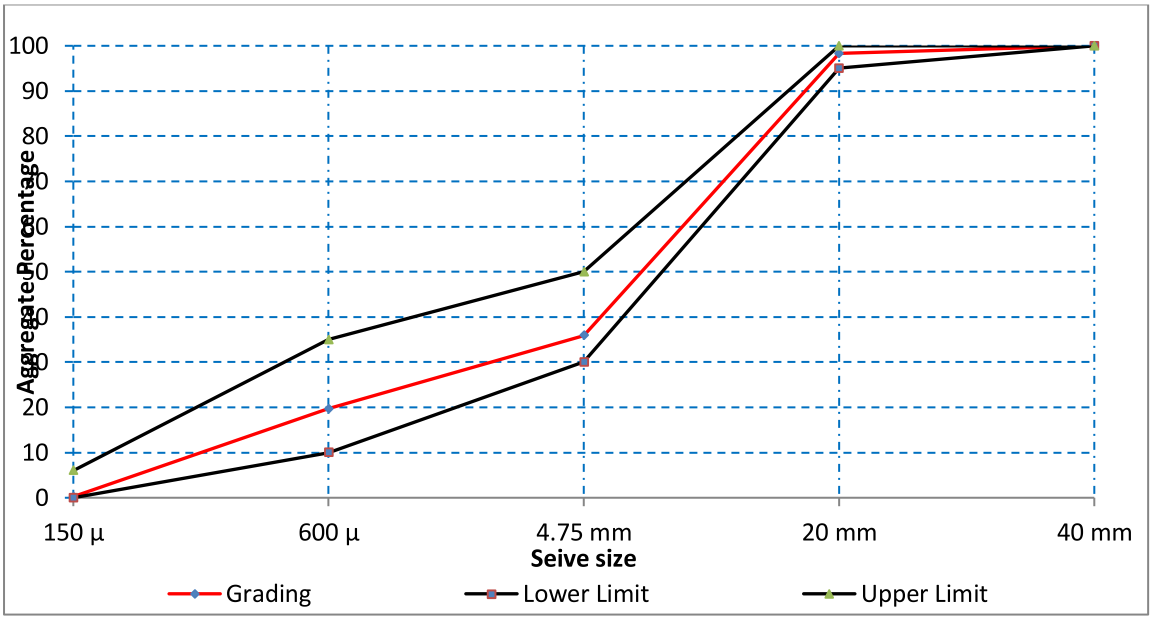


Fig 3.1 - All in Aggregate as per IS-383, Table 5 for W/C 0.4

ii) For design mix corresponding to W/C = 0.45; from the following table and graph, it is concluded that 38 % of coarse aggregate 20 mm, 25 % of coarse aggregate 10 mm and 37 % of sand is used.

Table 3.12 - ALL IN AGGREGATE AS PER IS-383, Table 5 for W/C 0.45

ALL IN AGGREGATE AS PER IS-383, Table 5							
Percentage	38	25	37	**All in aggregate grading**	**IS LIMIT**		**Remarks**
Aggregate size	**20 mm C/A**	**12.5 mm C/A**	**SAND**		**Lower limit**	**Upper limit**	
Sieve size	**% of Passing**	**% of Passing**	**% of Passing**				
80 mm	–	–	–	–	–	–	Refer Grading graph
40 mm	38.00	25.00	37.00	100.00	100	100	
20 mm	36.31	25.00	37.00	98.31	95	100	
4.75 mm	0.08	0.85	35.96	36.89	30	50	
600 µ	0.00	0.00	20.20	20.20	10	35	
150 µ	0.00	0.00	0.26	0.26	0	6	

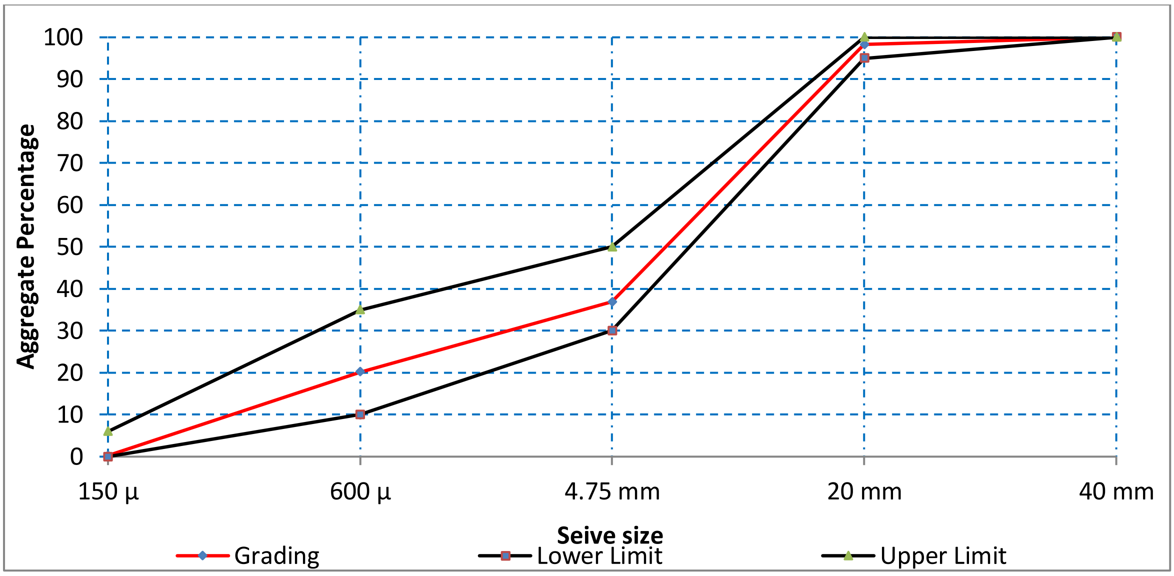


Fig 3.2 - All in Aggregate as per IS-383, Table 5 for W/C 0.45

iii) For design mix corresponding to W/C = 0.4; from the following table and graph, it is concluded that 37 % of coarse aggregate 20 mm, 25 % of coarse aggregate 10 mm and 38 % of sand is used.

Table 3.13 - ALL IN AGGREGATE AS PER IS-383, Table 5 for W/C 0.5

ALL IN AGGREGATE AS PER IS-383, Table 5							
Percentage	37	25	38	All in aggregate grading	IS LIMIT		Remarks
Aggregate size	20 mm C/A	12.5 mm C/A	SAND		Lower limit	Upper limit	
Sieve size	% of Passing	% of Passing	% of Passing				
80 mm	–	–	–	–	–	–	See Grading graph
40 mm	37.00	25.00	38.00	100.00	100	100	
20 mm	35.35	25.00	38.00	98.35	95	100	
4.75 mm	0.07	0.85	36.94	37.86	30	50	
600 µ	0.00	0.00	20.75	20.75	10	35	
150 µ	0.00	0.00	0.27	0.27	0	6	

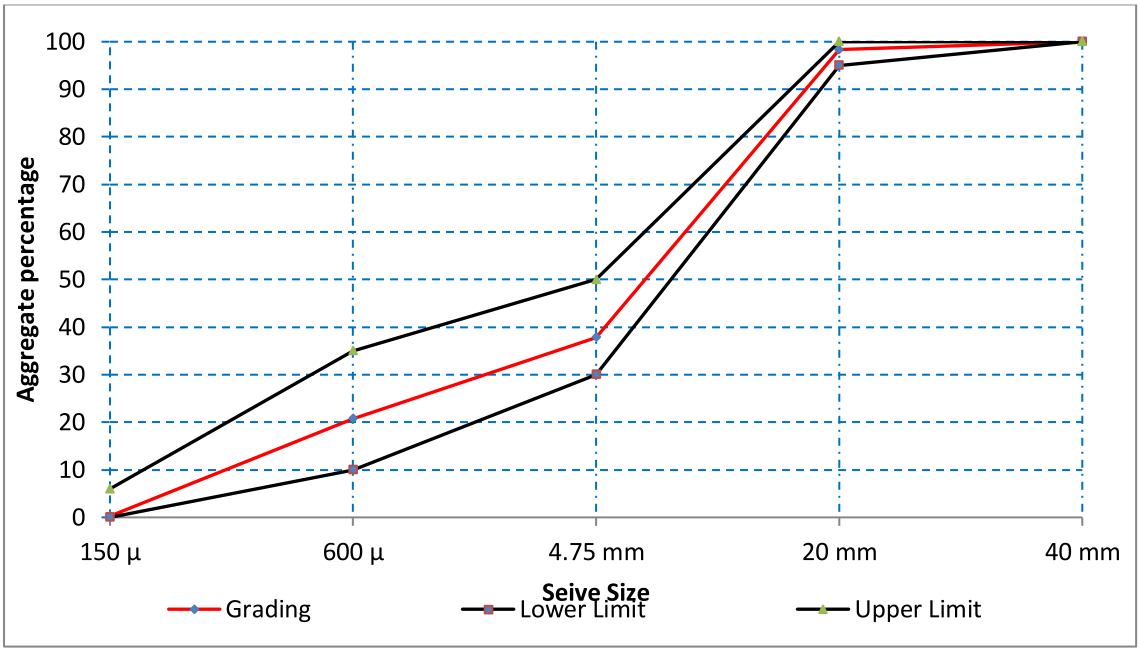


Fig 3.3 - All in Aggregate as per IS-383, Table 5 for W/C 0.5

Concrete mixes have been prepared using the following combinations:

- OPC with variable W/C of 0.4,0.45 and 0.5
- PPC with variable W/C of 0.4,0.45 and 0.5
- Addition of super plasticizers to OPC and PPC mixes at variable W/C of 0.4,0.45 and 0.5
- Replacement of 10, 30 , 50 and 70% by weight of cement with mineral admixtures such as SF, FA and GGBS for OPC combinations
- Replacement of 10, 30, 50 and 70% by weight of cement with mineral admixtures such as SF, FA and GGBS for PPC combinations
- Replacement of 10, 30 , 50 and 70% by weight of fine aggregate with mineral admixtures such as SF, FA and GGBS for OPC combinations
- Replacement of 10, 30 , 50 and 70% by weight of fine aggregate with mineral admixtures such as SF, FA and GGBS for PPC combinations

Design mix proportions for various combinations with notations are tabulated below:

Table 3.14: Table for Mix Design of Various Concrete Samples Prepared for the Experimental Study

Mix Proportions (in kg) for 1 Cu.m Concrete											
Mix	**W/C**	**W**	**C**	**fa**	**Ca_{10}**	**Ca_{20}**	**Fly Ash**	**MS**	**GGBS**	**Super**	**Total wt**(Kg)
Concrete mix with OPC											
O_1	0.5	197	394	672	442	654					2359
O_2	0.45	197	438	727	398	606					2366
O_3	0.4	197	493	613	426	664					2393
OPC Concrete mix with super plasticizer											
OS_1	0.5	169.4	394	698	459	680				2.36	2402.76
OS_2	0.45	169.4	376	778	426	649				2.63	2410.03
OS_3	0.4	169.4	339	717	471	698				2.96	2426.00
OPC Concrete mix with with 10 % replacement of cement with Fly ash											
OF_1	0.5	197	355	666	438	649	39				2344
OF_2	0.45	197	394	720	394	600	44				2349
OF_3	0.4	197	444	606	421	656	49				2373
OPC Concrete mix with 30 % replacement of cement with Fly ash											
OF_4	0.5	197	276	655	431	637	118				2314
OF_5	0.45	197	306	706	386	588	131				2314
OF_6	0.4	197	345	592	411	641	148				2334
OPC Concrete mix with 50 % replacement of cement with Fly ash											
OF_7	0.5	197	197	643	423	626	197				2283
OF_8	0.45	197	219	691	379	576	219				2281
OF_9	0.4	197	247	578	402	626	247				2297
OPC Concrete mix with 70 % replacement of cement with Fly ash											
OF_{10}	0.5	197	276	631	415	615	276				2410
OF_{11}	0.45	197	131	677	371	564	306				2246
OF_{12}	0.4	197	148	564	392	611	345				2257
OPC Concrete mix with 10 % replacement of cement with Micro silica											
OM_1	0.5	197	355	667	439	649		39			2346
OM_2	0.45	197	394	720	395	600		44			2350
OM_3	0.4	197	444	606	421	657		49			2374

			OPC Concrete mix with 30 % replacement of cement with Micro silica							
OM4	0.5	197	276	656	432	639		118		2318
OM5	0.45	197	306	707	387	589		131		2317
OM6	0.4	197	345	594	412	643		148		2339
			OPC Concrete mix with 50 % replacement of cement with Micro silica							
OM7	0.5	197	197	645	424	628		197		2288
OM8	0.45	197	219	694	380	578		219		2287
OM9	0.4	197	247	581	403	629		247		2304
			OPC Concrete mix with 70 % replacement of cement with Micro silica							
OM10	0.5	197	118	634	417	618		276		2260
OM11	0.45	197	131	681	33	567		306		1915
OM12	0.4	197	148	568	394	615		345		2267
			OPC concrete mix with 10 % replacement of cement with GGBS							
OG1	0.5	197	355	662	435	654			39	2342
OG2	0.45	197	394	725	397	605			44	2362
OG3	0.4	197	444	600	417	650			49	2357
			OPC concrete mix with 30 % replacement of cement with GGBS							
OG4	0.5	197	276	668	440	650			118	2349
OG5	0.45	197	306	713	391	594			131	2332
OG6	0.4	197	345	599	416	649			148	2354
			OPC concrete mix with 50 % replacement of cement with GGBS							
OG7	0.5	197	197	660	434	643			197	2328
OG8	0.45	197	219	713	390	594			219	2332
OG9	0.4	197	247	599	416	649			247	2355
			OPC concrete mix with 70 % replacement of cement with GGBS							
OG10	0.5	197	118	660	434	462			276	2147
OG11	0.45	197	131	712	390	593			306	2329
OG12	0.4	197	148	598	415	648			345	2351
			OPC Concrete mix with 10 % replacement of fine aggregate with Fly ash							
OFF1	0.5	197	354.6	604.8	442	654	67.2			2319.6
OFF2	0.45	197	394.2	654.3	398	606	72.7			2322.2
OFF3	0.4	197	443.7	551.7	426	664	61.3			2343.7

OPC Concrete mix with 30 % replacement of fine aggregate with Fly ash											
OFF_4	0.5	197	354.6	470.4	442	654	201.6				2319.6
OFF_5	0.45	197	394.2	508.9	398	606	218.1				2322.2
OFF_6	0.4	197	443.7	429.1	426	664	183.9				2343.7
OPC Concrete mix with 50 % replacement of fine aggregate with Fly ash											
OFF_7	0.5	197	354.6	336	442	654	336				2319.6
OFF_8	0.45	197	394.2	363.5	398	606	363.5				2322.2
OFF_9	0.4	197	443.7	306.5	426	664	306.5				2343.7
OPC Concrete mix with 70 % replacement of fine aggregate with Fly ash											
OFF_{10}	0.5	197	354.6	201.6	442	654	470.4				2319.6
OFF_{11}	0.45	197	394.2	218.1	398	606	508.9				2322.2
OFF_{12}	0.4	197	443.7	183.9	426	664	429.1				2343.7
OPC concrete mix with 10 % replacement of fine aggregate with GGBS											
OFG_1	0.5	197	354.6	604.8	442	654			67.2		2319.6
OFG_2	0.45	197	394.2	654.3	398	606			72.7		2322.2
OFG_3	0.4	197	443.7	551.7	426	664			61.3		2343.7
OPC concrete mix with 30 % replacement of fine aggregate with GGBS											
OFG_4	0.5	197	354.6	470.4	442	654			201.6		2319.6
OFG_5	0.45	197	394.2	508.9	398	606			218.1		2322.2
OFG_6	0.4	197	443.7	429.1	426	664			183.9		2343.7
OPC concrete mix with 50 % replacement of fine aggregate with GGBS											
OFG_7	0.5	197	354.6	336	442	654			336		2319.6
OFG_8	0.45	197	394.2	363.5	398	606			363.5		2322.2
OFG_9	0.4	197	443.7	306.5	426	664			306.5		2343.7
OPC concrete mix with 70 % replacement of fine aggregate with GGBS											
OFG_{10}	0.5	197	354.6	201.6	442	654			470.4		2319.6
OFG_{11}	0.45	197	394.2	218.1	398	606			508.9		2322.2
OFG_{12}	0.4	197	443.7	183.9	426	664			429.1		2343.7
Concrete mix with PPC											
P_1	0.5	197	394	662	436	645					2334
P_2	0.45	197	438	715	391	595					2336
P_3	0.4	197	493	601	417	651					2359
PPC Concrete mix with super plasticizer											
PS_1	0.43	169.4	424	648	450	703				2.36	2376.76
PS_2	0.39	169.4	376	768	420	640				2.63	2379.03
PS_3	0.4	169.4	339	708	466	689				2.96	2401.36

PPC concrete mix with 10 % replacement of Cement with Fly ash										
PF_1	0.5	197	355	657	432	640	39			2320
PF_2	0.45	197	394	709	388	591	44			2323
PF_3	0.4	197	444	595	413	645	49			2343
PPC concrete mix with 30 % replacement of Cement with Fly ash										
PF_4	0.5	197	276	655	431	637	118			2314
PF_5	0.45	197	306	697	382	581	131			2294
PF_6	0.4	197	345	584	405	632	148			2311
PPC concrete mix with 50 % replacement of Cement with Fly ash										
PF_7	0.5	197	197	638	420	621	197			2270
PF_8	0.45	197	219	685	375	571	219			2266
PF_9	0.4	197	247	572	397	620	247			2280
PPC concrete mix with 70 % replacement of Cement with Fly ash										
PF_{10}	0.5	197	118	628	413	612	276			2244
PF_{11}	0.45	197	131	673	369	561	306			2237
PF_{12}	0.4	197	148	561	389	607	345			2247
PPC concrete mix with 10 % replacement of Cement with Micro silica										
PM_1	0.5	197	355	658	433	640		39		2322
PM_2	0.45	197	394	709	388	591		44		2323
PM_3	0.4	197	444	595	414	645		49		2344
PPC concrete mix with 30 % replacement of Cement with Micro silica										
PM_4	0.5	197	276	649	427	632		118		2299
PM_5	0.45	197	306	698	382	582		131		2296
PM_6	0.4	197	345	585	406	634		148		2315
PPC concrete mix with 50 % replacement of Cement with Micro silica										
PM_7	0.5	197	197	640	421	623		197		2275
PM_8	0.45	197	219	688	377	573		219		2273
PM_9	0.4	197	247	575	399	623		247		2288
PPC concrete mix with 70 % replacement of Cement with Micro silica										
PM_{10}	0.5	197	118	634	417	618		276		2260
PM_{11}	0.45	197	131	681	373	567		306		2255
PM_{12}	0.4	197	148	564	392	611		345		2257
PPC concrete mix with 10 % replacement of cement with GGBS										
PG_1	0.5	197	355	662	435	644			39	2332
PG_2	0.45	197	394	725	397	605			44	2362
PG_3	0.4	197	444	600	417	650			49	2357

PPC concrete mix with 30 % replacement of cement with GGBS									
PG_4	0.5	197	276	668	440	650		118	2349
PG_5	0.45	197	306	713	391	594		131	2332
PG_6	0.4	197	345	599	416	649		148	2354
PPC concrete mix with 50 % replacement of cement with GGBS									
PG_7	0.5	197	197	660	434	643		197	2328
PG_8	0.45	197	219	713	390	594		219	2332
PG_9	0.4	197	247	599	416	649		247	2355
PPC concrete mix with 70 % replacement of cement with GGBS									
PF_{10}	0.5	197	118	660	434	642		276	2327
PF_{11}	0.45	197	131	712	390	593		306	2329
PF_{12}	0.4	197	148	598	415	648		345	2351
PPC concrete mix with 10 % replacement of Fine aggregate with Fly ash									
PFF_1	0.5	197	394	595.8	436	645	66.2		2334
PFF_2	0.45	197	438	643.5	391	595	71.5		2336
PFF_3	0.4	197	493	540.9	417	651	60.1		2359
PPC concrete mix with 30 % replacement of Fine aggregate with Fly ash									
PFF_4	0.5	197	394	463.4	436	645	198.6		2334
PFF_5	0.45	197	438	500.5	391	595	214.5		2336
PFF_6	0.4	197	493	420.7	417	651	180.3		2359
PPC concrete mix with 50 % replacement of Fine aggregate with Fly ash									
PFF_7	0.5	197	394	331	436	645	331		2334
PFF_8	0.45	197	438	357.5	391	595	357.5		2336
PFF_9	0.4	197	493	300.5	41	651	300.5		1983
PPC concrete mix with 70 % replacement of Fine aggregate with Fly ash									
PFF_{10}	0.5	197	394	198.6	436	645	463.4		2334
PFF_{11}	0.45	197	438	214.5	391	595	500.5		2336
PFF_{12}	0.4	197	493	180.3	417	651	420.7		2359
PPC concrete mix with 10 % replacement of fine aggregate with GGBS									
PFF_1	0.5	197	394	595.8	436	645		66.2	2334
PFF_2	0.45	197	438	643.5	391	595		71.5	2336
PFF_3	0.4	197	493	540.9	417	651		60.1	2359

PPC concrete mix with 30 % replacement of fine aggregate with GGBS								
PFF$_4$	0.5	197	394	463.4	436	645	198.6	2334
PFF$_5$	0.45	197	438	500.5	391	595	214.5	2336
PFF$_6$	0.4	197	493	420.7	417	651	180.3	2359
PPC concrete mix with 50 % replacement of fine aggregate with GGBS								
PFF$_7$	0.5	197	394	331	436	645	331	2334
PFF$_8$	0.45	197	438	357.5	391	595	357.5	2336
PFF$_9$	0.4	197	493	300.5	417	651	300.5	2359
PPC concrete mix with 70 % replacement of fine aggregate with GGBS								
PFF$_{10}$	0.5	197	394	198.6	436	645	463.4	2334
PFF$_{11}$	0.45	197	438	214.5	391	595	500.5	2336
PFF$_{12}$	0.4	197	493	180.3	417	651	420.7	2359

3.4 Experimental Programme

The purpose of this experimental study is to analyse the impact of various factors on the rate and depth of carbonation in concrete. Initially the impact of variation of external factors such as curing period, CO_2 concentration, relative humidity and exposure period has been studied. Concrete carbonation depths have been determined for curing periods of 3, 7, 21, 28 and 56 days keeping CO_2 concentration, relative humidity and exposure conditions fixed. Subsequently the depths of carbonation have been studied by varying exposure to 20, 60, 90, 120 and 150 days, keeping the curing and other conditions fixed. Carbonation depths have also been examined for variable percentages of CO_2 concentration i.e. 4, 10, 20, 30 and 50% at fixed curing and RH conditions. Thereafter the impact of variation of relative humidity at experimental values of 40, 50, 65, 90 and 100% have been studied keeping CO_2 concentration and curing fixed. The impact of external factors like paint and plaster have also been tested for carbonation depth by examining depths of carbonation on plastered and painted concrete samples. Each of the above tests by variation of external factors has been conducted on both OPC and PPC to analyse the effect of carbonation on type of cement. The impact of various internal factors has been experimentally examined by applying the following variations at fixed parameters of 65 % RH and 4% CO_2 for 3 days curing conditions:

- Carbonation depths for OPC and PPC concrete without super plasticizers
- Carbonation depths for OPC and PPC concrete with super plasticizers
- Carbonation depths for OPC and PPC concrete samples with replacement of 10, 30, 50 and 70% by weight of cement with Micro silica
- Carbonation depths for OPC and PPC concrete samples with replacement of 10, 30, 50 and 70% by weight of cement with FA
- Carbonation depths for OPC and PPC concrete samples with replacement of 10, 30, 50 and 70% by weight of cement with GGBS
- Carbonation depths for OPC and PPC concrete samples with replacement of 10, 30, 50 and 70% by weight of fine aggregate with FA
- Carbonation depths for OPC and PPC concrete samples with replacement of 10, 30, 50 and 70% by weight of fine aggregate with GGBS

All of the above tests have been conducted by conventional phenolphthalein indicator method. However, carbonation depths for OPC and PPC concrete samples have also been tested by advanced methods such as XRD, FTIR and SEM analysis to determine the accuracy of test results by various methods.

Last but not the least, the compressive strength of carbonated and non-carbonated samples have been tested and compared to analyse the impact of carbonation on compressive strength of concrete.

Casting of Cubes

The ingredients of the concrete mix are measured by weight in a steel pan and mixed in mechanical mixer. Water is added to the mixer after all the cement and aggregates constituting the batch are already in the drum andmixed for at least one minute. Mixing of each batch is continued until there is a uniform distribution of the materials and the mass is uniform in colour and consistency. The entire contents of the drum are discharged before the ingredients for the next batch are fed into the drum. Each time the work stops, the mixer is thoroughly cleaned.

The fresh concrete properties are measured as per the guidelines in IS 1199-1959 (2004). Slump of concrete mix measured with the help of slump cone test. Moulds of size 100x100x100mm were used for casting of cubes. Then concrete moulds placed on table vibrator for compaction of concrete. Concrete cubes removed from moulds after 24 hours and submerged in water for various curing days.



Fig 3.4 Preparation of Concrete Cubes and Placing of Samples in Carbonation Chamber-

Departmental Laboratory, IIT (BHU), Varanasi

3.5 Pre- Conditioning Parameters

CO_2 Concentration: A CO_2 concentration of 4 % is maintained in the chamber with the help of a CO_2 cylinder attached to the chamber. However, some samples have been tested at 4, 10, 20 and 60%percentage to investigate the impact of CO_2 concentration on carbonation. Since carbonation chamber used in the experiment can be used for variation of CO2 concentration upto 4 % only, an alternate arrangement has been made for increase of CO2 percentage upto 60 % for experimental purpose only.

Temperature: Temperature of the apparatus is maintained at 25℃ ± 5 ℃

Relative Humidity: A humidity of 65%, and pressure of 1 atm is maintained in the carbonation chamber an RH of 65% was chosen because it was the recommended value given by RILEM. (51). However, some samples have been tested at 40, 50, 65, 85 and 100% RH to investigate the impact of relative humidity on carbonation

W/C Ratio: Water cement ratio has been varied at 0.5, 0.45 and 0.4 in various samples for experimental purpose.

Cement Consumption: As per design mix tabulated above from table 3.6 to table 3.37.OPC and PPC cement have been used.

Curing: Samples have been tested at 3 days curing for experimental purpose. However, some samples have been tested at 3, 7, 21 and 28 days curing to investigate the impact of curing on carbonation.

Set-up for Carbonation of Concrete

Carbonation of concrete is a long-term process. Therefore, an accelerated carbonation system has been used to accelerate the carbonation of concrete in short time. CO_2concentration can be regulated from 0 – 4% within the chamber used while RH can be regulated from 0-100%.

The accelerated carbonation chamber was used to retain concrete samples in controlled environment. There was constant concentration of CO_2 of 4%, humidity65 % , temperature 25 +/- 5 degree celcius and pressure 1atm.$CO_2$is supplied to the chamber from a CO_2 cylinder. Water is kept in container over the apparatus and is connected to the same through an inlet pipe to maintain desired humidity. Concrete cubes are placed in carbonation chamber for 20 days. Carbonation chambers is designed and manufactured by Ferrotekeuipments, Ghaziabad, India.

Fig 3.5 Carbonation Chamber - Departmental Laboratory, IIT (BHU), Varanasi

Properties of Fresh Concrete

Slump observed for OPC concrete and PPC concrete is 100 mm and 110 mm respectively.

Upon replacement of cement with mineral admixture, slump is observed to increase for both OPC and PPC concrete.

Upon replacement of fine aggregate with mineral admixture, slump is observed to decrease for both OPC and PPC concrete.

3.6 Tests Conducted

To study the durability of carbonated concrete, the concrete specimens are subjected to tests that determine the depth of carbonation. Also, to study the mechanical properties of carbonated concrete, the concrete specimens after having undergone accelerated carbonation are also tested for their strength in compression.

3.7 Compressive Strength

Compressive strength is the maximum compressive stress that a solid material can sustain without fracture under a gradually applied load. This test was performed in accordance with IS 516- 1959 (2004) on concrete cubes of 150 mm size. The cubes are placed in such a manner that the load is applied to opposite sides of the cubes as cast where the casting face and the testing face are perpendicular to each other. The load is axially applied without any shock and increased continuously at a rate of approximately 140 kg/cm2/min. The maximum load sustained by the specimen is recorded and divided by cross-sectional area to obtain the compressive strength of the specimen. In the present study, compressive strength were tested for 7 days curing concrete and 20 days carbonated concrete.

3.8 Measurement of Depth of Carbonation by Various Methods

3.8.1 Phenolphthalein Indicator

Phenolphthalein (Hph) is prepared by the reaction of phallic anhydride and phenol in presence of concentrated H_2SO_4. Phenolphthalein is a weak acid and is used as an acid base indicator. Hph dissociates to H^+ and Ph^- and remains in equilibrium condition. The un-dissociated form of Phenolphthalein (Hph) is colourless whereas the colour of Ph- is pink. In acidic medium Phenolphthalein remains un-dissociated, hence appears colourless while in basic medium, the H+ is consumed to increase the concentration of Ph-, thereby indicating the colour pink. The pH range of Phenolphthalein indicator is 8.2 to 10.

The pH of concrete medium ranges between12 to 13 (basic), hence the colour of Phenolphthalein is pink in concrete. When the pH drops below 9.5 upon carbonation in concrete the indicator turns colourless.

On completion of carbonation in chamber, the cubes were drawn off from the chamber and sliced into pieces. The sliced parts were cleaned and phenolphthalein pH indicator, prepared by diluting 1 gram phenolphthalein with 90 ml ethanol in water up to 100 ml, was sprayed on their surface. Carbonated parts were identified to be colourless whereas the non-carbonated parts of the samples turned pink on spraying the indicator. Carbonation depth was averaged for multiple readings of colourless zones (A1, A2, A3, A4, A5, A6, A7& A8) from the edge taken after five minutes of spraying.

The main limitation of this test is that the procedure will cause localised surface damage and this method provides only an indication of the extent of carbonation.

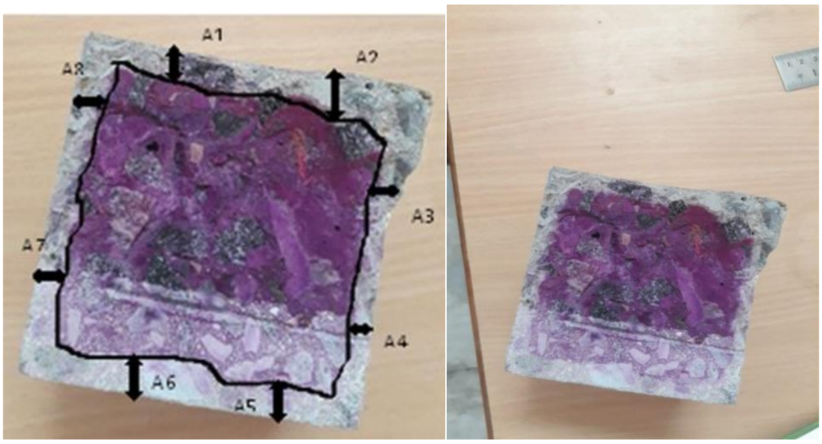


Fig. 3.6 Concrete Sample Tested with Phenolphthalein Indicator

3.8.2 FTIR Method

The Fourier Transform Infrared Spectroscopy (FTIR) method is a technique to determine the functional group present in a molecule. Every functional group vibrates in two directions, one along the bond (stretching frequency) while the other in the direction perpendicular to the bond (wagging frequency). Further, every atom possesses a unique stretching and wagging frequency. This helps to identify the specific atom/functional group present in a molecule. When the Infrared rays pass through the molecule, the functional group present in the molecule absorbs the IR rays of matching frequency. The FTIR spectrum is graphically represented as percentage transmittance (%T) or percentage absorbance (%A) versus wavenumber. The type of bond/functional group is indicated by the wave number of the peak. The graph falls in the transmittance curve while it peaks in the absorbance curve at specific wave number or frequency which is indicative of carbonation depth. When applied to concrete, infrared spectroscopy has been used to detect the spectral changes associated with the presence of $Ca(OH)_2$ at the interface. The process of carbonation includes the conversion of C=O bonds of carbon dioxide into C–O bonds in the calcium carbonate having wave number in range of 1410–1510 cm-1. Hence, the wave number of the C-O bond indicates the carbonation in concrete.

Fig 3.7 FTIR Instrument - Departmental laboratory, IIT (BHU), Varanasi

3.8.3 X-Ray Diffraction Analysis

In the single and double slit experiments, visible light is used to create diffraction pattern since the wavelength of visible light (10^{-6}m) is of the order of the width of the slit. The same experiment cannot be used in crustal lattice structures since the atomic spacing is much smaller i.e. of the order 10-10m. Hence X-rays having wavelength of the same order are used for diffraction.

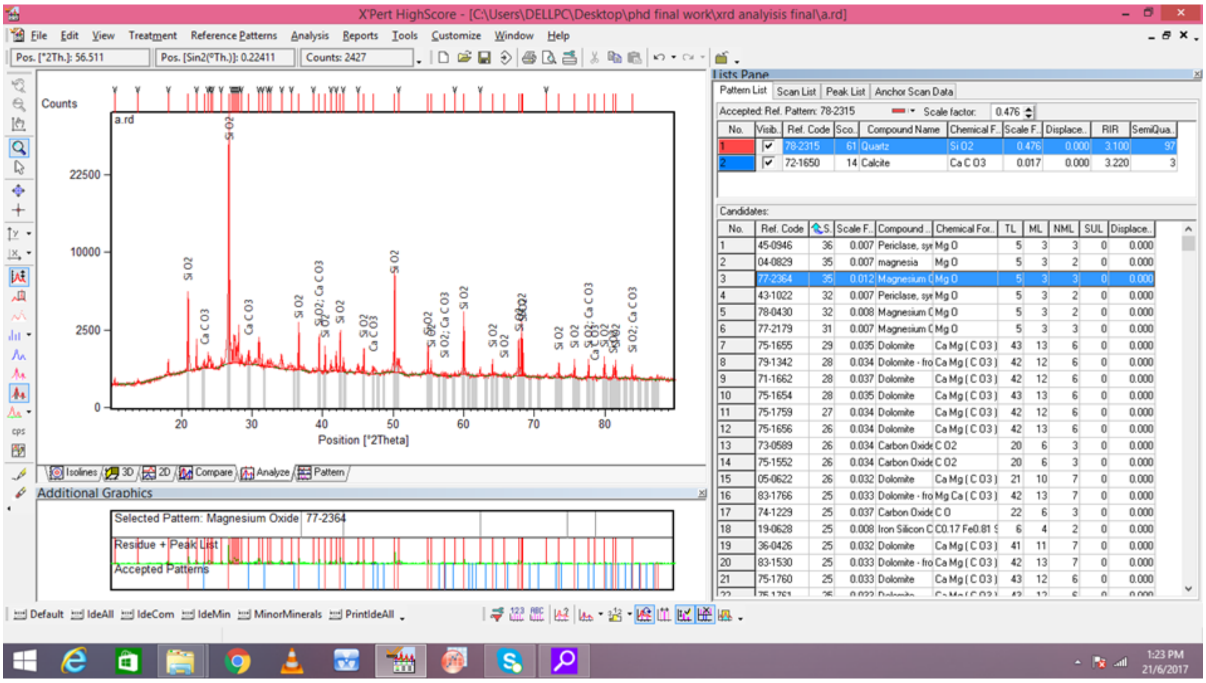
The X-Ray Diffraction method operates on the principle of phase identification of a crystalline material, providing data on unit cell dimension. X-ray diffraction method is used for recognizing the atomic and molecular structure of a crystal.

A cathode ray tube is used to throw Monochromatic X-rays on a crystalline sample resulting in constructive interference to produce diffraction pattern in accordance with Bragg’s law ($n\lambda = 2d \sin \theta$). where n , λ ,d and θ are a positive integer , wavelength of the incident wave , scattering and inter-planar distance respectively. All possible diffraction directions of the lattice should be attained due to the random orientation of the powdered material.

The XRD analysis was conducted with model Mini Flex 600. The concrete cube samples after were split and powder from other side of the cube is crushed at every 5mm from the top surface and few mg of the sample is taken. This is sieved through 75 μm sieve. The fine powder after sieving is placed on spatula and kept on the XRD machine. An operating condition with radiation (40 kV, 15 mA) and scan rate: 10 degree/minis maintained and the samples are observed for 2θ values ranging from 10–90 degree. Separate peak values of 2θ are observed for specific chemical constituents of the sample i.e. 2θ peak values shall be derived for $CaCO_3$ and $Ca(OH)_2$etc.Multiple crests against 2θ values for individual constituents may be observed. However the highest peak value at a given value of 2θ corresponds to the highest availability of the constituent within the sample. As such, the peak values of $CaCO_3$ and Ca(OH)2 are used to determine the extent of carbonation in concrete. For example, the existence of a peak for $CaCO_3$ exists and no peak for $Ca(OH)_2$ indicates that the sample is fully carbonated, while a peak for $Ca(OH)_2$ and no peak for $CaCO_3$ indicates that the specimen is non-carbonated. The existence of both the peaks denotes that the specimen is partially carbonated.

Fig 3.8 XRD Apparatus - Departmental Laboratory, IIT(BHU),Varanasi

Phase identification analysis was carried out by analysing the test results of XRD patternsusing Philip's X'PERT High Scorer software wherein the serial number of $CaCO_3$ was found to be 72-1650, 72-1651 and 72-1652 and serial number of SiO_2 was found to be 78-2315. The presence of $CaCO_3$ was observed at several values of 2ϴ, however the highest peak was observed at a 2ϴ value of (29.37). The samealso confirms to JCPDS pdf no. 72-1937. Following pictures (Fig. 3.9) are sample representation of XRD data analysed through Philip's X'PERT High Scorer software. Further 2ϴ vs. intensity graphs at 3 random values in range were plotted in ORIGIN software to compare $CaCO_3$ peaks and thus carbonation depths were derived from the graphs.



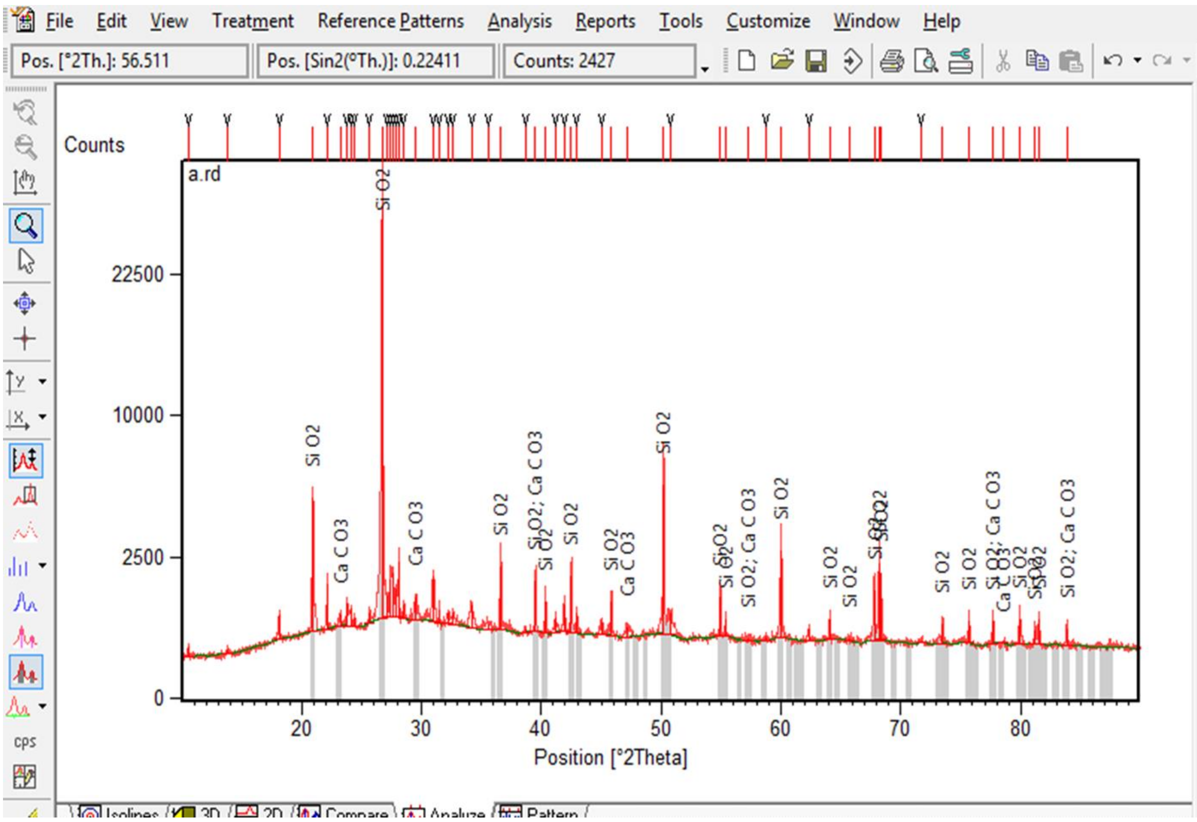


Fig.3.9 Analysis of Test Data on Philip’s X’PERT High Scorer Software

3.8.4 SEM

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. Part of these electrons are reflected, some are transmitted while some are absorbed in the surface. Some electrons and X-rays etc. are emitted from surface of the specimen also. The latter are called secondary electrons which are detected in this method, the nature of which determines the image of the compound formed. The EDX analysis of this indicates the element present in the compound. The EVO 18, Zeiss4.0 high resolution scanning electron microscope(SEM) with energy dispersive X-Ray Spectroscope detector was used.

Fig 3.10 SEM Instrument- Departmental Laboratory, IIT (BHU), Varanasi

In order to examine the microstructural changes in concrete cube samples during the process of carbonation, samples were taken at every 5mm from the carbon dioxide exposed face of the cubes. These samples were examined through SEM for the possible existence of calcites or any of the hydration products of cement which indicate extent of carbonation.

3.9 Conclusions

The sieve analysis of sand indicates that it falls within zone II. The coarse aggregate are classified as per sieve analysis as 20 mm and 10 mm aggregates. All in aggregate graph has also been prepared to calculate the percentage in composition of fine and coarse aggregate. Mix design with both OPC and PPC have been calculated and finalized in line with the above. The mass of mix constituents have also been calculated for replacement percentages and accordingly mix design have been finalised to prepare cube samples for testing purpose. Test cubes have been tested for compressive strength as well as carbonation depths through various methods, as mentioned in this chapter. Results of carbonation depth obtained from conventional method, as well as advanced methods, are discussed in chapter 4 and results of compressive strength of concrete used in this study are discussed in chapter 5.