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PERFORMANCE OF MULTI BLEND CEMENT CONTAINING FLY ASH, GRANULATED BLAST FURNACE SLAG AND LIMESTONE

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ABSTRACT: Investigations have been taken up for performance evaluation and hydration mechanism involved in mortar samples prepared using multi blend cements containing fly ash, GBFS and low grade limestone. The detailed investigations carried out revealed that the multi blend cements containing fly ash, GBFS and low grade limestone have performance particularly strength development characteristics comparable to the of control cement. The papers highlights the performance results of mortar samples prepared using multi blend cements, the reaction mechanism involved vis-à-vis scope for simultaneous use of these materials in cement. The results indicated that in a multi blend of fly ash, GBFS, low grade limestone and clinker, up to 65 % of clinker can be replaced gainfully without affecting the performance in a mortar sample. The investigations further revealed that the quality of limestone particularly its Calcium carbonate content does not affect the performance of multi blend cement. Further, experimentation on concrete and its durability are continuing.

Keywords: Multi blend cement, Low grade limestone, Fly ash, Granulated blast furnace slag

1. INTRODUCTION

Hydraulic cements specified by Bureau of Indian Standards (BIS) include OPC (33, 43 & 53 Grades), PPC, PSC, recently approved composite cement and various other special purpose cements. Fly ash conforming to IS 3812 (Part 1): 2013 and granulated blast furnace slag conforming to IS 12089: 1987 are used in the manufacture of PPC, PSC and composite cements respectively. Manufacture of multi blend cements will permit greater utilization of industrial wastes and enable production of cements with lower clinker factor leading to resource conservation, enhanced waste utilization and greater sustainability in cement manufacture. Replacement of clinker by slag and fly ash improves the performance of concrete by increasing the resistance to chloride penetration. It was reported that the addition of slag results in better performance of concrete containing class F fly ash than the one containing class C fly ash[2]. The development of compressive strength in composite cements containing fly ash improves at later ages and is roughly proportional to the content of active silica.

European standards (EN 197-1: 2000) already have specifications on Portland Composite Cement (Cement Type II) permitting simultaneous use of various mineral admixtures, with total additions in the range of 6-20% (CEM II/A-M) and 21-35 % (CEM II/B-M). European Standards also specify Composite Cement (Type V - CEM V/A and CEM V/B) where simultaneous use of only granulated blast furnace slag and pozzolana/ fly ash is permitted. ASTM too has introduced performance-based specifications for hydraulic cements (C 1157-00) with no restrictions on composition of the cement.

The physical and chemical properties of multi blend cement as specified in European Standards or ASTM are based on material characteristics, testing procedures and product requirement, which are different from those prevailing in India and/or specified by BIS. The values of various properties specified in EN or ASTM standards for different types of cements, especially the compressive strength values, are not directly comparable to the values mentioned in Indian Standards for similar cements, on account of different test procedures. Hence performance data on multi blend cement containing Indian fly ashes, GBFS and low grade limestone, tested as per BIS procedures, is not available and need thorough and systematic investigations [1]-[30].

The investigations are taken up for simultaneous use of fly ash, GBFS and low grade limestone in multi blend cements. The results obtained indicated that fly ash and GBFS could be added simultaneously as blending material and the most optimum performance was obtained at the addition level of 15% fly ash and 40% GBFS. In order to further explore utilization of low grade limestone in the above binary blend, addition of low grade limestone as well as cement grade limestone was done in the quantum of 5, 10 and 15% by weight. The performance of such multi blend cements indicated that low grade limestone could be added upto 10% and the performance was comparable to that of control cement containing 15% fly ash and 40% GBFS. The investigation further revealed that the effect of quality of limestone does not play significant role and low grade limestone can be utilized gainfully up to 10%. The properties of multi blend cements were found to depend on the quality of clinker and mineral Distribution. Further work on performance evaluation of multi blend cements including their durability characteristics in concrete are continuing.

2. MATRIALS & EXPERIMENTATION

2.1 Characterisation of Materials

The clinker, fly ash, granulated blast furnace slag (GBFS), low and cement grade Limestone and gypsum samples were collected from cement, power and steel plants in India and characterized. Fly ash sample was also characterized for physical properties as per requirements in terms of fineness, lime reactivity, cement index and residue on 45 µ sieve as mentioned in IS:3812. The samples of fly ash and hydrated cements were evaluated for mineralogy using X-ray Diffractometer (XRD). XRD studies were carried out using Rigaku International, D-Max 2200V/PC, Cu Kα radiation, additions, besides fineness and particle size λ=1.5405 Å. The samples were scanned from 2θ= 5 to 65^0, with increments of 0.02^0. X-ray tube was operated at an accelerated voltage of 40 kV and current of 30 mA.

The results obtained indicated that Fly ash was having fineness of 270 m^2/kg and 4.6 N/mm^2 lime reactivity. The glass content in fly ash and GBF slag, estimated by Optical Microscopy, was 43 and 94 percent respectively. The chemical analysis of the above materials is given in Table 1. The purity of gypsum worked out to be 86.4 %. XRD studies of fly ash indicated the presence of quartz, mullite, hematite phases along with amorphous content. JEOL 6510 SEM, in the secondary electron mode, was used to carry out morphological studies of fly ash at an accelerating voltage of 15 kV. SEM studies indicated that most of the fly ash particles were in the spherical shape and in agglomerated form besides angular to irregular shaped quartz and hematite particles [31].

Table 1 : Chemical Analysis of Materials

Oxides (%)	Clinker	Fly ash	GBF Sag	Cement grade Limestone	Low grade Limestone	Gypsum
LOI	1.59	4.67	0.56	35.67	27.23	17.16 *
SiO_2	20.06	60.08	33.86	11.85	23.91	12.44 #
Fe_2O_3	3.55	6.85	0.72	1.94	3.82	0.40
Al_2O_3	5.97	23.89	19.03	2.95	7.15	0.51
CaO	63.05	1.43	35.77	44.50	33.40	28.28
MgO	2.73	1.12	7.80	1.06	2.25	0.74
SO_3	1.39	0.10	0.15	--	--	40.19
Na_2O	0.59	0.18	0.29	1.32	1.58	0.05
K_2O	0.64	1.12	0.40	0.40	0.38	0.04
Sulfide sulphur	-	-	0.51	--	--	-

* combined water, # SiO_2 + acid insoluble

2.2 Preparation of Cement Blends containing Fly ash and GBFS

Control Ordinary Portland cement (OPC), Portland pozzolana cement (PPC) and Portland slag cement (PSC) were prepared along with cement blends containing OPC clinker (40-60% by wt.) with different doses of fly ash and GBFS (33-55%) in various combinations and fineness was maintained at 340±10 m^2/kg. The blend compositions are given in Table 2. Based on the performance results of above samples, portland slag cement containing 40% GBFS and composite cement blend containing 40% GBFS and 15% fly ash were considered optimum. Multi blend cements were then prepared by addition of cement and low grade limestone to the optimized blend containing 40% GBFS and 15% fly ash in laboratory ball mill by inter grinding. The fineness was maintained at 340±10 m^2/kg. The blend compositions are given in Table 3.

Table 2: Composition of cement blends prepared by varying wt composition of fly ash and GBF slag

Sl No	Cement	Wt. proportions (%)			
		Clinker	Fly ash	BF Slag	Gypsum
1	OPC-Cont.	95.0	-	-	5.0
2	PPC-Cont.	70.0	25.0	-	5.0
3	PSC-Cont.	55.0	-	40.0	5.0
4	COM-1	40.0	5.0	50.0	5.0
5	COM-2	40.0	15.0	40.0	5.0
6	COM-3	40.0	25.0	30.0	5.0
7	COM-4	40.0	35.0	20.0	5.0
8	COM-5	40.0	45.0	10.0	5.0
9	COM-6	50.0	5.0	40.0	5.0
10	COM-7	50.0	15.0	30.0	5.0
11	COM-8	50.0	20.0	25.0	5.0
12	COM-9	50.0	25.0	20.0	5.0
13	COM-10	50.0	35.0	10.0	5.0
14	COM-11	60.0	5.0	30.0	5.0
15	COM-12	60.0	15.0	20.0	5.0
16	COM-13	60.0	25.0	10.0	5.0
17	COM-14	30.0	25.0	40.0	5.0

Table 3: Composition of cement blends prepared by varying wt composition of fly ash, GBF slag and Limestone

Cement	Wt. proportions (%)			
	Clinker-1	Limestone	Fly ash-1	GBF Slag
CON-PSC(N)	55.25	-	-	40.0
COM-2(N)	40.25	-	15.0	40.0
COM-2-CLS-5	35.25	5.0	15.0	40.0
COM-2-CLS-10	30.25	10.0	15.0	40.0
COM-2-CLS-15	25.25	15.0	15.0	40.0
COM-2-LLS-5	35.25	5.0	15.0	40.0
COM-2-LLS-10	30.25	10.0	15.0	40.0
COM-2-LLS-15	25.25	15.0	15.0	40.0

3. RESULTS AND DISCUSSIONS

Physical performance evaluation of composite cement blends containing fly ash and GBF slag was carried out up to 360 days as per relevant Indian Standard and results are given in **Table 4**. Composite cement blends have shown variation in setting time values, particularly in the initial setting time. However, there was no definite trend observed with respect to increase in fly ash or GBF slag contents. The normal consistency found to be increased with increasing fly ash content in the composite cement blends. The increase in normal consistency is attributed to the presence of finer fraction in fly ash, which dominated the ball bearing effect of spherical fly ash particles.

A comparison of the compressive strength values of composite cement samples containing fixed fly ash content of 25% and varying GBF slag and clinker content is drawn, given in Fig. 1. PPC containing 25% fly ash content was taken as reference. Reduction in the compressive strength was observed with decrease in clinker content. COM-13 containing 60 % clinker has shown marginally lower compressive strength compared to reference PPC at initial ages but at later ages the performance is comparable with reference sample. At 50% clinker content, COM-9, significant decrease in the compressive strength at all ages is observed. Further reduction in clinker, COM-3 containing 40% clinker, has not resulted in considerable decrease in compressive strength from compressive strength of COM-9.

A similar comparison is drawn for the compressive strength of composite cement samples containing fixed GBF slag content of 40 % and varying fly ash and clinker contents, shown in Fig. 2. PSC containing 40 % GBF slag was considered as reference. COM-6, containing 50% clinker and

5% fly ash, showed better compressive strength development as compared to reference sample at all ages. Further reduction in clinker content resulted in decrease in compressive strength. Though the compressive strength development in composite cement samples at initial ages was lower as compared to reference PPC and PSC, there is improvement in the compressive strength at later ages. The reduction in compressive strength values at initial ages is attributed to dilution effect, where as the improvement in strength values at later ages can be due to synergetic effect of addition of fly ash and GBF slag together.

Table 4: Physical performance of composite cement blends containing fly ash and GBFS

Sl No	Cement Sample No.	Setting Time (Min.)		Compressive Strength (MPa)						
		IST	FST	3 d	7 d	28 d	90 d	120 d	180 d	360 d
1	OPC-Cont.	190	270	35.5	48.0	57.5	58.50	60.50	61.00	62.00
2	PPC-Cont.	210	290	26.0	35.5	48.0	65.00	68.00	68.50	70.00
3	PSC-Cont.	215	285	26.0	40.5	50.5	57.00	60.00	61.00	63.00
4	COM-1	210	295	28.0	39.5	51.5	57.50	62.50	63.00	65.00
5	COM-2	210	300	22.5	32.0	45.0	54.00	58.00	58.00	60.50
6	COM-3	205	300	18.5	27.5	40.5	52.00	55.00	56.50	58.50
7	COM-4	200	300	17.0	25.5	37.0	48.00	51.00	52.00	54.00
8	COM-5	190	310	13.0	19.0	35.0	39.00	44.00	45.00	46.50
9	COM-6	170	275	29.0	41.0	52.0	58.00	63.00	65.00	67.50
10	COM-7	190	275	25.0	36.0	49.0	54.50	60.50	62.00	64.50
11	COM-8	175	295	23.5	34.5	48.0	62.00	63.00	63.50	65.00
12	COM-9	205	295	20.0	32.0	45.5	52.00	56.00	57.00	59.50
13	COM-10	170	260	15.5	22.0	41.0	45.00	51.00	52.50	55.00
14	COM-11	195	285	30.0	44.0	53.0	60.00	66.00	67.00	68.00
15	COM-12	170	255	29.0	39.5	52.0	60.00	65.00	66.00	67.50
16	COM-13	205	290	25.0	33.0	46.0	57.00	64.50	65.00	68.00
17	COM-14	210	298	16.0	23.0	37.0	49.0	52.00	52.50	54.50

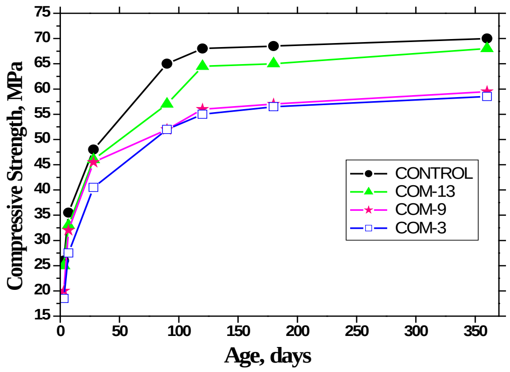


Fig 1: Compressive Strength developments of cement blends

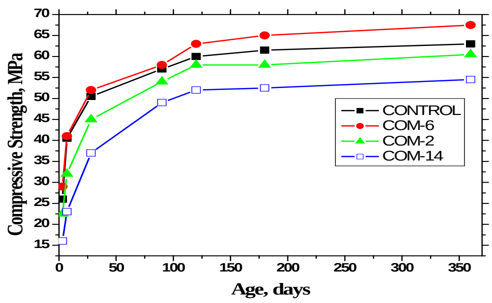


Fig 2: Compressive Strength developments of cement blends

XRD studies of the hydrated samples of OPC, PPC and composite cement blend COM-2, shown in Fig. 3, indicated that the portlandite (CH) content was less in composite samples as compared to OPC and PPC at 28 days of hydration. The factors affecting the CH content in the hydrated samples are clinker content and paozzolanic activity of fly ash. Broad hump in the XRD patterns of PPC and COM-2 indicated the presence of amorphous content in the samples. The presence of portlandite crystals in 28 day hydrated samples of PPC and COM-2, studied using SEM, is shown in Fig. 4. In PPC, portlandite crystals were present as pack of parallel plate like structures with preferred orientation.

SEM images of fly ash particles at different stages of reaction are shown in Fig. 5. Fly ash consists of spherical particles of amorphous nature along with quartz and hematite particles, shown in Fig. 5a. Fig. 5b is showing the partially reacted fly ash, in which the surface of the particle is reacted and needle like mullite crystals are visible. Completely reacted fly ash particle is shown in Fig.

5c, the spherical particle is totally consumed leaving a void space.

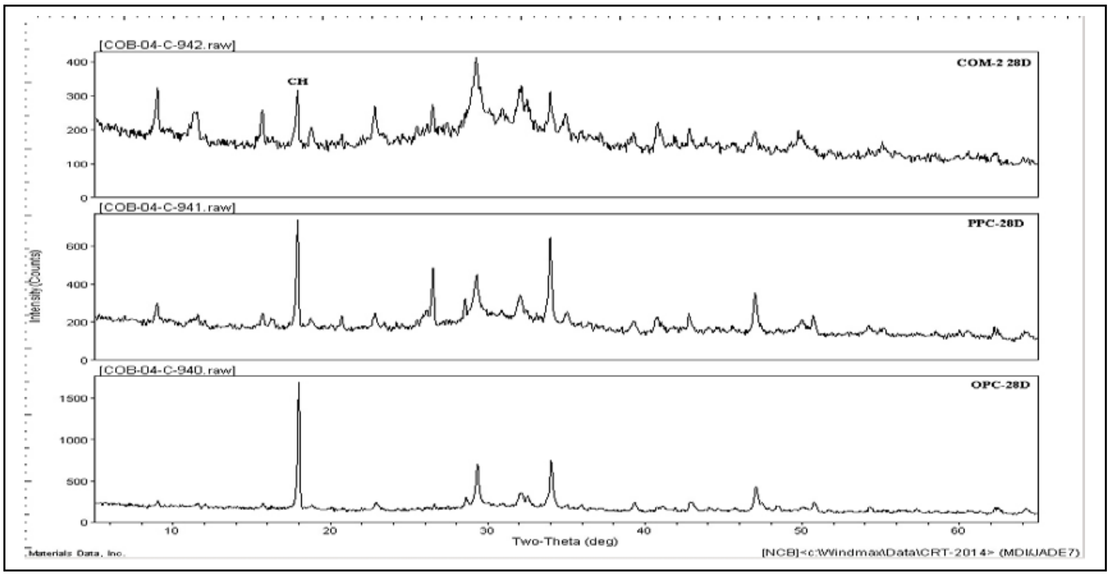
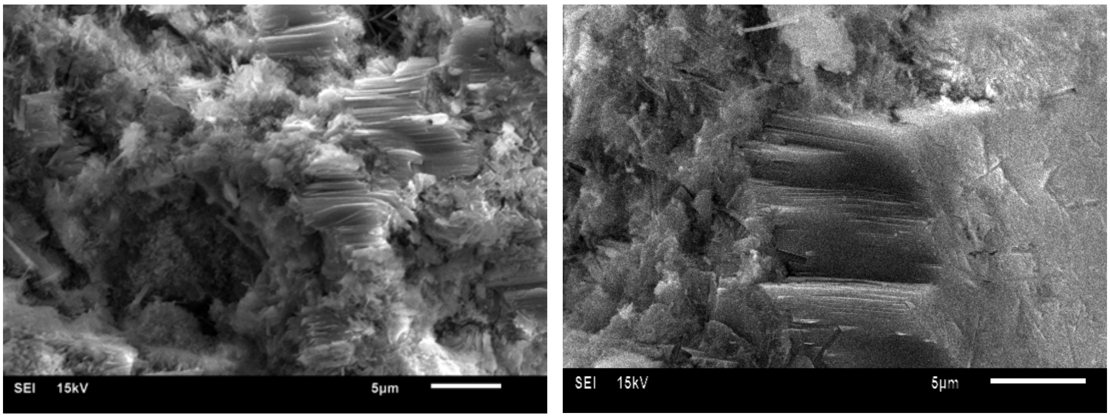
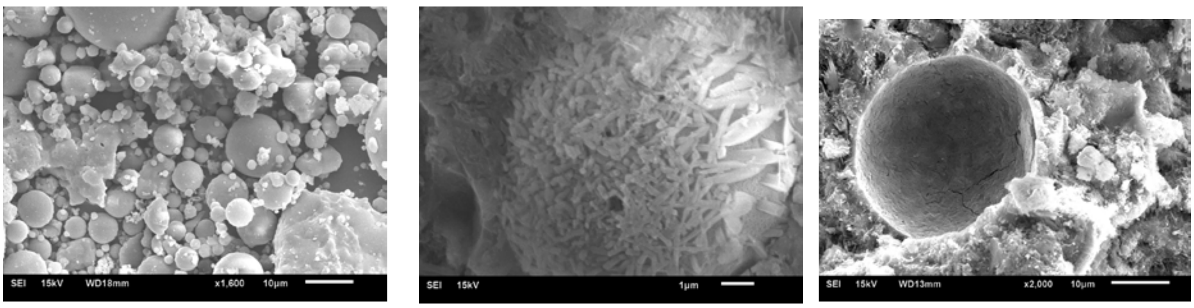


Fig. 3: XRD of 28 days Hydrated pastes of COM-2 (Fly ash 15% & GBFS 40%), PPC & OPC



(a) (b)

Fig. 4: SEM images of 28 day hydrated (a) PPC and (b) COM-2



(a) (b) (c)

Fig. 5: SEM images of (a) fly ash, (b) partially reacted fly ash particle and (c) Completely reacted fly ash

Dense CSH gel is present around the reacted fly ash particle. The partially or un-reacted particles contributing to filler effect where as the reacted particles are contributing to formation of additional CSH. Based on the above investigations, cement blend containing 40% GBFS and 15% fly ash was considered most optimum for further investigations.

Physical performance evaluation of the multi blend cement containing 40% GBFS, 15% Fly ash and varying dosages of low and cement grade limestone was carried out up to 28 days as per relevant Indian Standard and results are given in **Table 5**. Multi blend cement samples have shown variation in setting time values, particularly in the initial setting time. A definite trend was observed with respect to increase in limestone contents. The normal consistency was also found to increase with increasing limestone content of both types. The increase in normal consistency is attributed to the presence of finer fraction in fly ash as well as limestone.

Interpretation of the compressive strength values of multi blend cement containing fixed fly ash content of 15% and 40% GBF slag and varying content of low and cement grade limestone and clinker content indicated that the performance of multi cement blend containing 10% limestone of both types was comparable with that of control cement COM-2(N).

XRD studies of the hydrated samples of COM-2 and multi blend cement samples are shown in Fig. 6 and 7. The results obtained indicated that at one day of hydration, there is an increase of ettringite formation in multi blend cements as compared to that of com-2(N). This Increase in ettrengite formation is bacause of the increased hydration. At one day of hydration, the interpretation of XRD results indicated initian of a newer phase formation containing compounds of calcium and aluminium. The results of 3 days hydration further confirmed the observations of one day hydration with increased formation ettrengite and complex compound of calcium and aluminium. and aluminium. This additional hydrated complex compound of calcium and aluminium is termed as calcium carbo aluminate hydrate (CCA).

Table 5: Physical Performance of Multi Blend Cements

Cement Sample	Fineness (m2/kg)	Normal consistency (%)	Setting Time (Min.)		Comp. Strength (MPa)		
			IST	FST	3 d	7 d	28 d
CON-PSC(N)	340	26.5	180	240	27.0	42.5	50.0
COM-2(N)	363	28.0	190	255	24.0	33.0	47.5
COM-2-CLS-5	341	28.5	215	265	20.0	30.5	40.0
COM-2-CLS-10	353	27.5	220	305	17.0	26.0	36.0
COM-2-CLS-15	349	27.5	220	310	18.0	25.0	30.0
COM-2-LLS-5	346	27.5	210	285	22.0	32.0	42.0
COM-2-LLS-10	343	28.5	230	300	20.0	26.0	38.0
COM-2-LLS-15	334	28.8	250	310	17.0	24.0	28.0

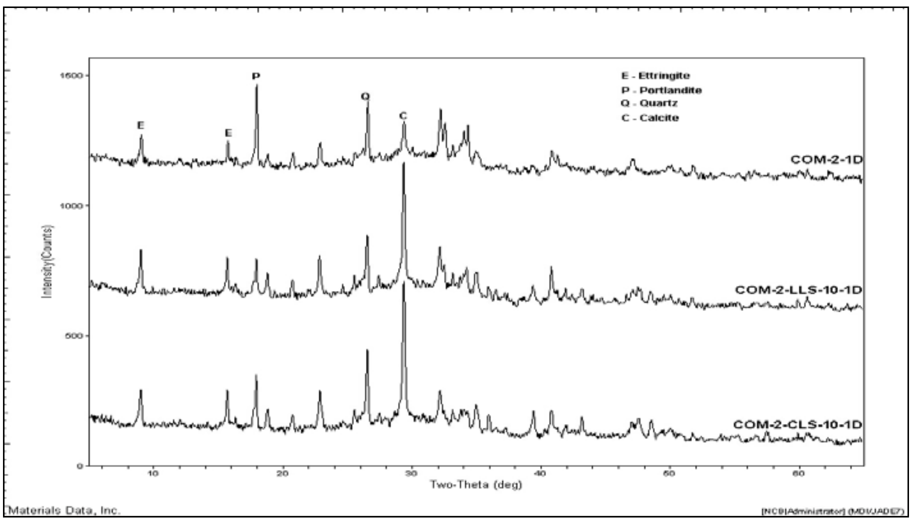


Fig. 6: XRD of 1 day Hydrated pastes of COM-2 (N) and multi blend cement samples containing low and cement grade limestone

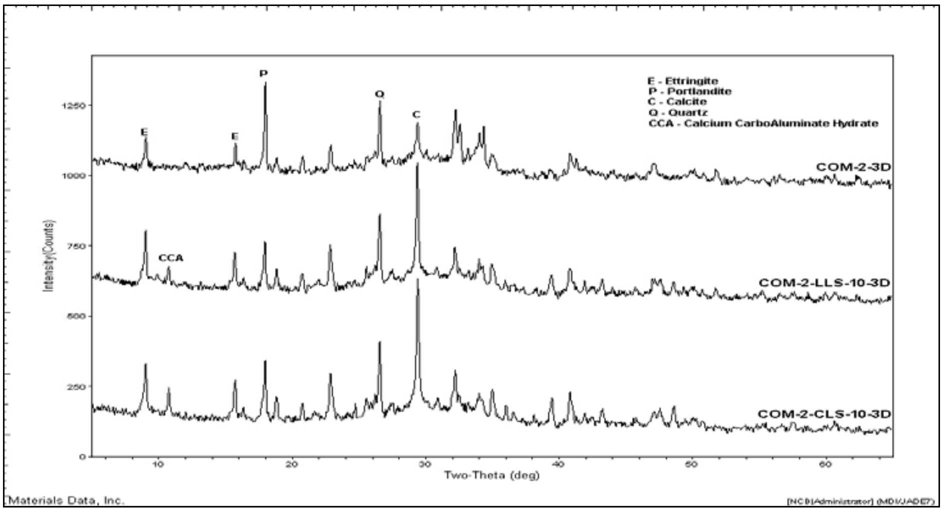


Fig. 7: XRD of 3 days Hydrated pastes of COM-2 (N) and multi blend cement samples containing low and cement grade limestone

4. CONCLUSIONS

The compressive strength values of Cement blends containing GBFS and fly ash were marginally lower at the initial ages and improved at later ages compared to the that of control cement samples. The reduction in compressive strength at initial ages is attributed to dilution effect and the improvement in strength at later ages was due to synergetic effect of addition of fly ash and GBF slag. XRD and SEM studies of hydrated samples indicated that the portlandite content was less in the cement blends containing GBFS and fly ash compared to control cements. In case of multi blend cement, the results obtained clearly indicated that low grade limestone could be added upto 10% and the performance was comparable to that of control cement containing 15% fly ash and 40% GBFS. The investigation further revealed that the effect of quality of limestone does not play significant role and low grade limestone can be utilized gainfully up to 10%. The properties of multi blend cements were found to depend on the quality of clinker and mineral additions, besides fineness and particle size distribution. XRD studies of hydrated samplesof multi blend cement indicated increased formation ettrengite and complex compound of calcium and aluminium. and aluminium at 3 days as compared to that of 1 day which is considered for further contributing to compressive strength development in the system.

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