

STUDY THE CORRELATION OF CLINKER QUALITY, RESIDE, PSD ON THE PERFORMANCE OF PORTLAND CEMENT

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ABSTRACT

This research establishes the correlation between clinker quality, residue, particle size distribution and performance behaviour of cement. Different qualities of clinker were identified to understand the influence of mineralogy on the performance of cement. Chemical and mineralogical evaluation of clinker and gypsum were carried out as per the Indian specified standards, XRD and Optical microscopy. Cement samples were prepared by inter mixing of clinker and gypsum with 95 and 5% respectively for different fineness zones such as 225, 250, 275, 300, 325 and 350 m²/kg. The resultant samples were studied for residue, particle size distribution and performance evaluation as per IS 4031. It was observed that clinker quality plays an important role to achieve the desired performance characteristics in addition to the residue and particle size distribution of the cements.

Key words: Cement, residue, particle size distribution and strength.

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1. INTRODUCTION

It is known that the quality and type of cement are significant factors to determine the strength development of concrete, which is specifically affected by the fineness and mineral composition of cement. The fineness of cement also affects its reactivity with water. Generally, the finer the cement, the more rapidly it will react [1,2]. However, the cost of grinding and the heat evolved on hydration set some limits on fineness.

It is reported in the study that the rate of reactivity and the strength development can be enhanced by finer grinding of cements. It is generally agreed that cement particles larger than 45×10^{-6} m are difficult to hydrate and those larger than 75×10^{-6} m could never hydrate completely. However, an estimate of the relative rates of reactivity for similar cement composition cannot be made without knowing the complete particle-size distribution by sedimentation methods [3–5].

Fineness of grinding constitutes (clinker and gypsum) an important variable in the behavior of portland cement. Everyone is well aware of the opinions that while increasing the fineness of the cement sample increases the strength characteristics [6-19]. Unfortunately, the substantial data is not available to correlate the percentage of performance of improvement while increasing the fineness of the cement. Particularly how much percentage of compressive strength of the cement enhances while increasing the fineness and what is optimum level of fineness for obtaining the better properties. In addition to this, no substantial data were available regarding the parameters which influences the fineness of the material and how the mineralogical aspects influence on the residue or particle size distribution while increasing the fineness of the cement. The behavior of particles of different sizes has a significant effect on such obvious properties of paste as consistency, strength, setting time and durability. In view of the above limitations of the supporting data and to justify the observed findings on the evaluated product, this research work planned systematically for understands the correlation between fineness & performance characteristics and optimum level of OPC. In addition to this, correlation between the residue and particle size distribution on the fineness of the product. It is the aim of this paper to show in a brief way how clinker quality influences residue and particle size distribution and their reactivity and further, to indicate which chemical changes are occurring. This work at present merely touches on the fringes of some significant developments that are increasing our understanding of portland cement and its behavior. Although this work may appear to lack massive amounts of supporting evidence, time and further experimentation will correct this defect.

2. EXPERIMENTAL

Two qualities of clinker samples were collected from the cement plants to establish the correlation between the mineralogical aspects on the performance and residue. Initially both the industrial clinkers were crushed using jaw crusher by passing 100% through 2.8 mm sieve. 8 kg of different cement samples were prepared in laboratory ball by stchiometrically weightment of clinker and gypsum for the target fineness and the details are given in table 1. The obtained cement samples were evaluated for chemical analysis, mineralogical analysis and performance evaluation of setting time, normal consistency and compressive strength as per the relevant Indian standards.

Table 1 Designed proportions of clinker and gypsum along with target fineness

	% of Clinker	% of Gypsum	Target fineness, m2/kg
SOPC 1	95	5	225
SOPC 2	95	5	250
SOPC 3	95	5	275
SOPC 4	95	5	300
SOPC 5	95	5	325
SOPC 6	95	5	350
COPC 1	95	5	225
COPC 2	95	5	250
COPC 3	95	5	275
COPC 4	95	5	300
COPC 5	95	5	325
COPC 6	95	5	350

2.1. Characterization

Chemical analysis of the clinker and cement samples were carried out as per IS 4032-1985 [20]. The room temperature powder X-ray diffractograms were recorded on Rigaku smart lab XE X-ray diffractometer using Nickel filtered Cu-K α radiation of wavelength 1.5406 Å for phase confirmation in the 2 θ range 10 – 80°, with step size (2 θ) of 0.02° and scan step time of 0.15 s. The unit cell parameters and reitviled refinemnt were carried out using JADE software. Morphology and granulometry of clinkers was investigated on their polished sections using NIKON POL 600 reflected light microscope equipped with image analyser. Seive analysis were carried out by using 212, 90, 75, 63, 53 and 45 sieves manually. Particle size analysis of the samples were carried out using

3. RESULTS AND DISCUSSION

3.1. Chemical Analysis

The chemical analysis of Portland clinker samples CL-1 and CL-4, collected from different cement plants showed presence of usual oxide constituents with free lime content in the range of 0.91-1.77 indicating well burnt clinker samples (Table 1). The moduli value such as Lime Saturation Factor (LSF), Silica Modulus (SM), Alumina Modulus (AM) and potential phases C₃S, C₂S, C₃A and C₄AF as determined on the basis of Bogue' calculations are given in Table 2. The chemical analysis of clinker samples showed their conformity to Indian standard IS:16353-2015 specified for Portland clinker.

Table 2 Chemical anlysis of clinker, gypsum and cement samples

Constituents	CCL-1	SCL-2	Gyp	SOPC-1	COPC 2
LOI	0.86	0.6	21.76	1.66	1.91
CaO	62.3	62.65	30.89	61.06	60.73
SiO ₂	20.9	19.8	4.31	19.03	20.07
Al ₂ O ₃	5.99	4.94	0.77	4.73	5.73
Fe ₂ O ₃	3.91	3.36	0.56	3.22	3.74
MgO	2.32	4.25	0.56	4.07	2.23
SO ₃	2.33	0.87	44.18	3.04	4.42
Na ₂ O	0.45	0.21	0.03	0.20	0.43
K ₂ O	0.57	0.29	0.03	0.28	0.54
IR	0.41	0.56		0.53	0.39
Cl	0.02	0.005	0.015	0.01	0.02
f-CaO	1.77	0.91		0.86	1.68
CW			18.61	0.93	0.93

*Silica & acid insoluble, IR: Insoluble residue, CW: combined water

3.2. X-ray Diffraction

XRD analysis of Portland clinker samples showed presence of usual clinker mineral phases; alite [Ca₅₄MgAl₂Si₆₀O₉₀], belite [Ca₂SiO₄], calcium aluminate [Ca₃Al₂O₆] and calcium alumina ferrite [Ca₂(Al,Fe)₂O₅] along with periclase [MgO] in small amount as shown in Figures 1 and 2. Semi-quantitative estimation of clinker mineral phases, in clinker samples CCL-1 and SCL-2, carried out by considering the integral intensity of diffraction line d=1.76Å corresponding to alite and intensities of lines d=2.70 and 7.24Å corresponding to C₃A and C₄AF. Alite and belite contents of clinker samples CCL-1 and SCL-2 were found to be in between 51 and 55 % and 26 and 22 % respectively as given in Table 3 indicating their good quality.

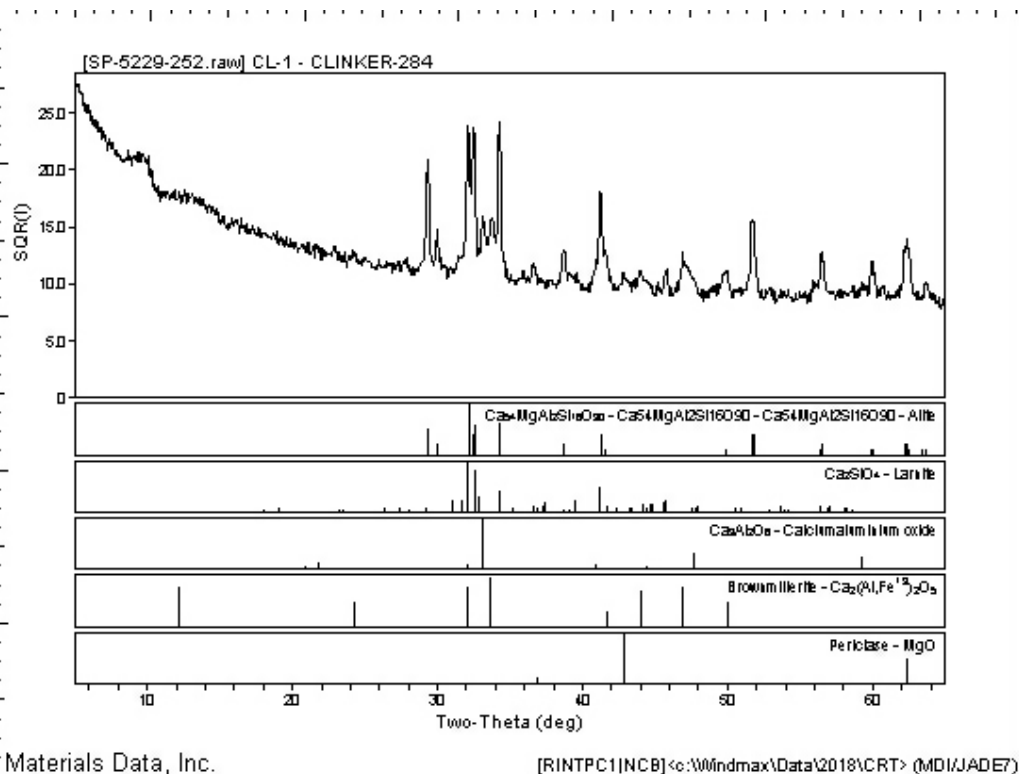


Figure 1 X-ray diffractogram of CCL-1

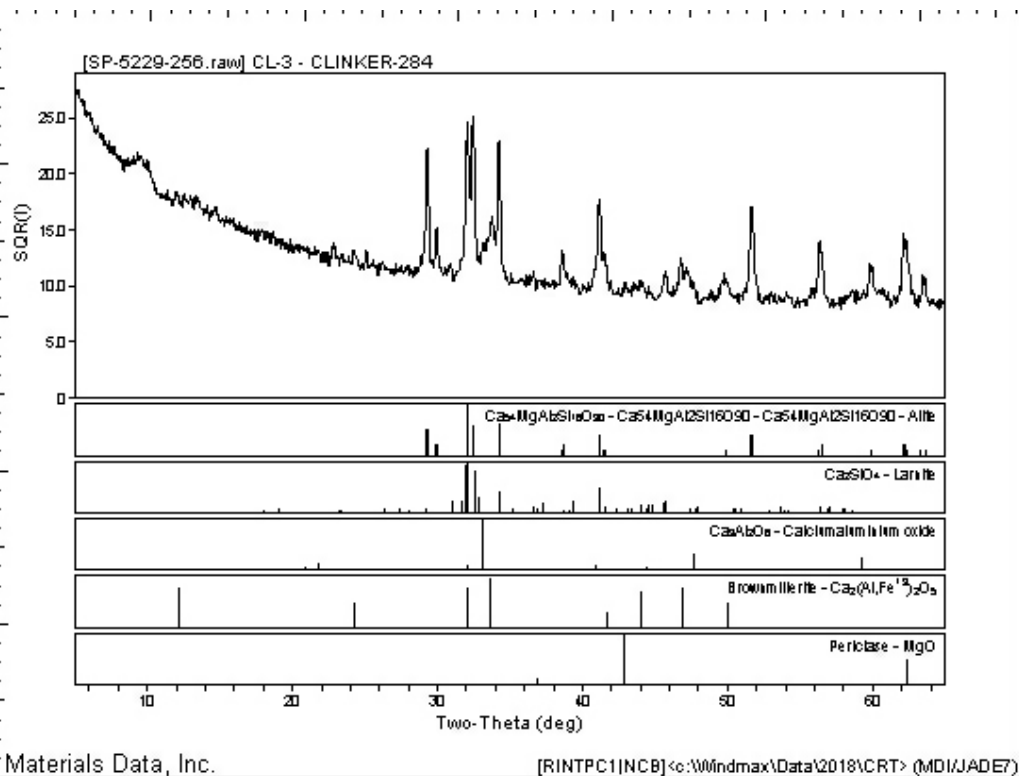


Figure 2 X-ray diffractogram of SCL-2

Table 3 Potential mineral phase by Boague method and semi quantitative mineral phases by XRD

Potential clinker minerals			Obtained clinker minerals by XRD	
Minerals	CCL-1	SCL-2	CCL-1	SCL-2
C3S	42.0	59.97	51.0	55.0
C2S	28.0	11.53	26.0	22.0
C3A	9.0	7.41	5.0	4.0
C4AF	12.0	10.23	13.0	14.0
Others	-	-	5.0	5.0
LSF	0.89	0.97		
AM	2.11	2.38		
SR	1.53	1.47		

4. MICROSTRUCTURE

Clinker CCL-1

Clinker phases were moderately developed and in homogeneously distributed.

Majority of alite grains are pseudo-hexagonal to hexagonal in shape with sharp grain margins along with development of very fine alite grains on the margins of alite phenocrysts. Few alite grains were partially broken on the margins. Bleb shaped belite grains were also observed in the alite grains. Fused alite grains were also observed in clinker. Transformation of belite into alite did not reach equilibrium conditions. Majority of alite grains were in the size range of 25 to 35 μm . Most of belite grains were sub rounded to rounded in shape with corroded margins. Radiating needles and one and two set twinning was observed in belite grains. The Majority of belite grains were in the size range of 15 to 20 μm . Porosity of the clinker was high with pores of various shapes and sizes (20-680 μm). The average grain size of alite and belite grains were found to be 32 and 16- μm respectively. The percentage of alite and belite were 53 and 28% respectively with 19% interstitial matter.

Clinker SCL-2

The clinker phases were moderately developed and in-homogeneously distributed. Majority of alite grains were pseudo-hexagonal to hexagonal in shape with sharp grain margins. Bleb shaped belite grains were detected in the alite grains. Fused alite grains were also very commonly developed in the clinker. Grain size variation in alite is too large. Transformation of belite into alite did not reach equilibrium conditions. Majority of alite grains were in the size range of 30 to 40 μm . Most of the belite grains were sub rounded in shape with corroded margins. Radiating needles were also observed in the belite grains. Majority of belite grains are in the size range of 20 to 30 μm . Porosity of the clinker was high with pores of various shapes and sizes (40 to 690- μm) were uniformly distributed. The percentage of alite and belite were 59 and 25% respectively with 16% interstitial matter. The average grain size of alite and belite grains were found to be 37 and 26- μm respectively.

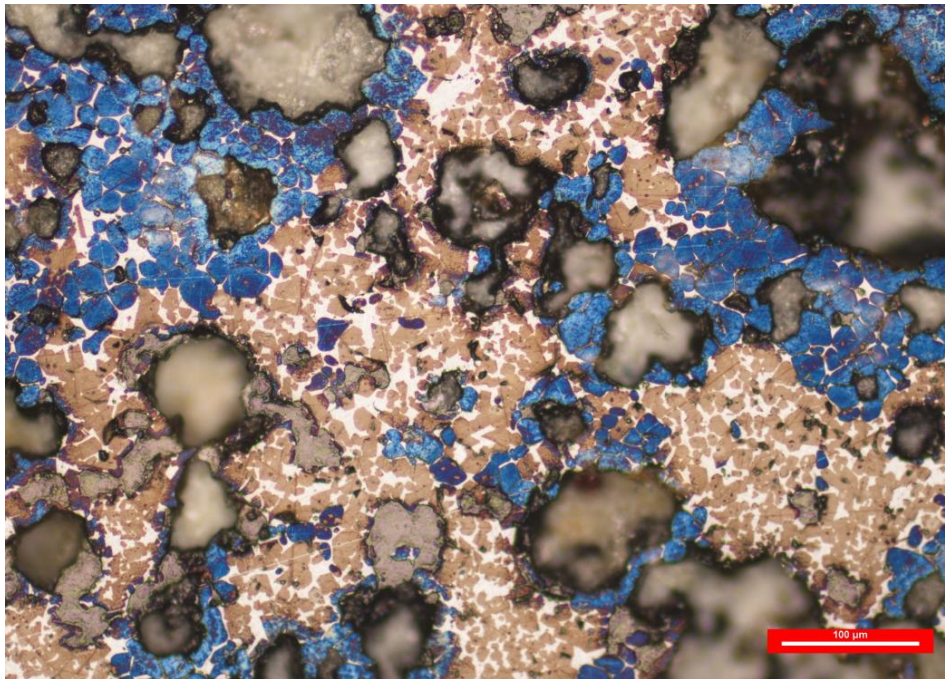


Figure 3 Distribution of alite and belite grains in the clinker (CCL-1). (10X)

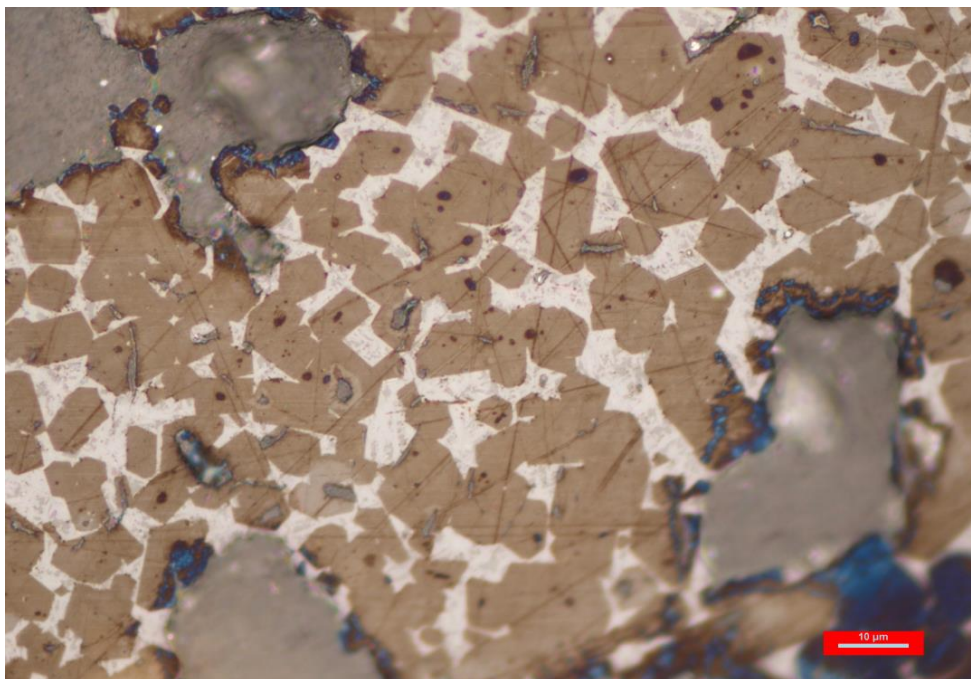


Figure 4 Development of hexagonal to pseudo hexagonal alite grains in the clinker (CCL-1). (50X)

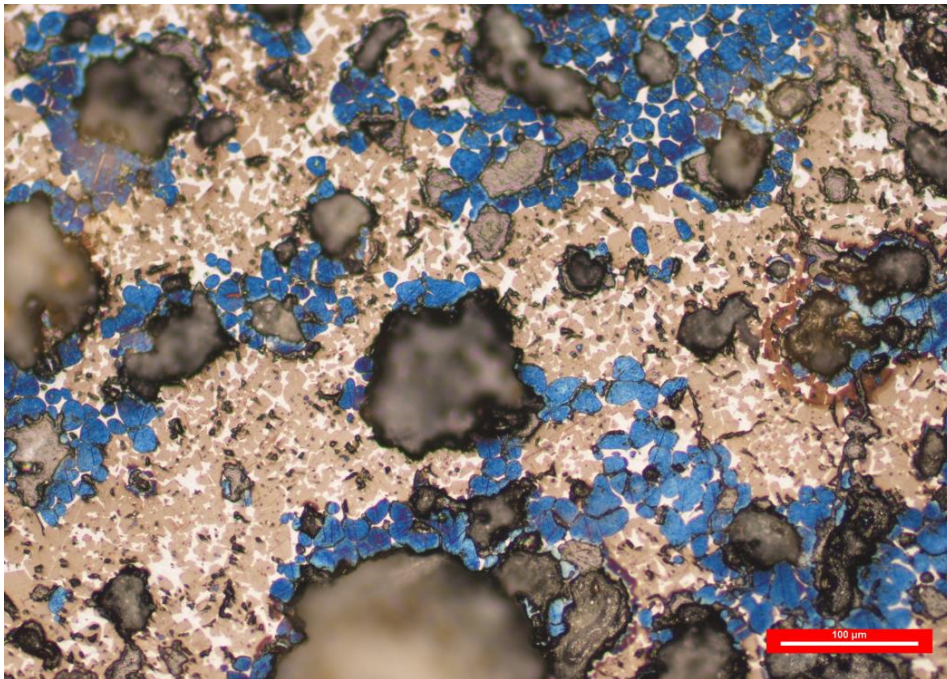


Figure 5 Distribution of alite and belite grains in the clinker (SCL-2). (10X)

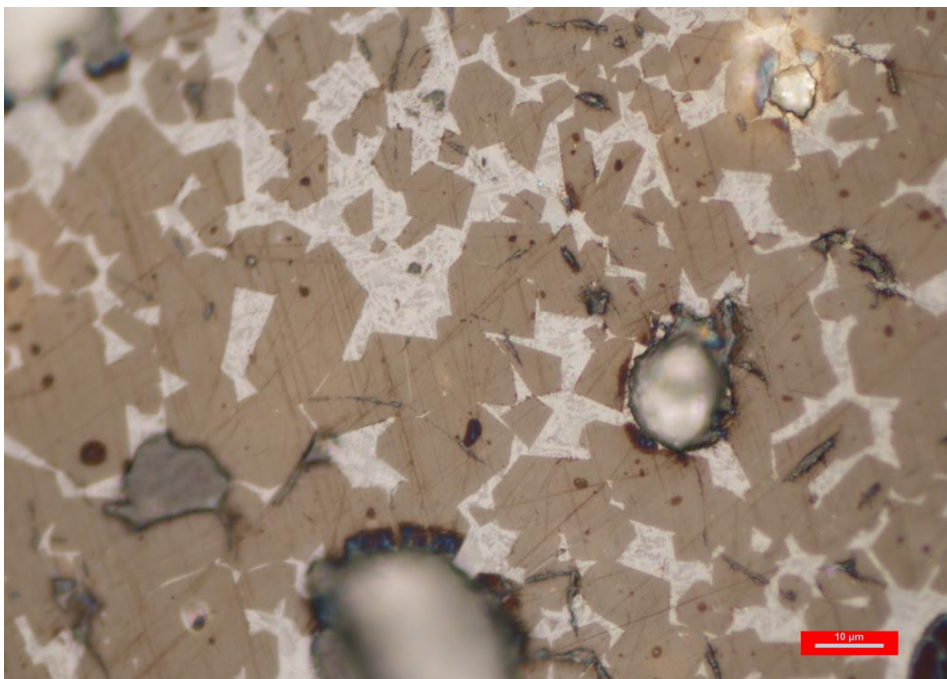


Figure 6 Distribution of alite grains in the clinker (SCL-2). (50X)

5. RESIDUE

Residue of all the cement samples were carried out using 212, 90, 75, 63, 53 and 45 sieve manually. The obtained results are given in table 4. While increasing the fineness of cement samples from 225 to 350 m²/kg, residue on 212 microns decreases slightly from 0.97 to 0.36 and then the remaining samples shows 99.9% passing. The remaining sieve analysis shows decreases in the residue while increase the fineness of cement samples. Significant changes with

all the sieves interms of residue was observed of samples contains the fineness level of 250 and 275 m²/Kg sample. Clinker 1 containing samples shows significant difference around 50% of the residue with all the sieves between the fineness level of 225 to 250 m²/kg. After that gradual decrement was observed of the residue with all the sieves. Clinker 2 containing samples shows significant difference around 50% of the residue with all the sieves between the fineness level of 250 to 275 m²/kg. It is observed that the percentage of passing of SOPC samples was significantly more at all the sieves compared to COPC sample. It may be due to the more percentage of C₂S percent in the CCL-1 than the SCL-2.

It was observed that while increasing the fineness from 225 to 350 m²/kg, more number of more than 63 microns size particles were ground about 85% of 90 microns, 81% of 73 microns, 71% of 63 microns, 61% of 53 microns and 50% of 45 microns were ground.

Table 4 Residue of all the OPC samples

Sample	+212 μ	+90 μ	+75 μ	+63 μ	+53 μ	+45 μ	-45 μ
COPC 1	0.85	12.35	19.99	28.37	34.68	40.43	59.57
COPC 2	0.15	4.98	10.61	18.52	25.91	32.46	67.54
COPC 3	0.13	4.41	9.57	17.14	24.42	30.97	69.03
COPC 4	0.12	3.16	7.05	13.75	20.28	27.01	72.99
COPC 5	0.13	2.38	5.82	12.30	18.64	25.02	74.80
COPC 6	0.10	1.82	3.61	7.70	13.35	20.20	79.80
SOPC 1	0.97	13.89	21.43	29.57	35.16	40.70	59.30
SOPC 2	0.37	11.27	18.30	26.28	32.43	37.90	62.10
SOPC 3	0.12	5.35	11.42	19.08	25.02	31.02	68.98
SOPC 4	0.11	3.46	8.09	14.89	21.17	27.14	72.86
SOPC 5	0.10	2.34	5.89	12.26	17.96	24.12	75.88

5.1. Particle size distribution

Particle size analysis of all the cement samples were carried out by using microtrac S3500 and the obtained results are given in the below table. Portland cements COPC1 and SOPC 1 of Blaine fineness about 225 m²/kg respectively contains about 52 and 50 percent on + 33 μ particles and 11.32 and 15.37 percent of - 10 μ particles respectively. While increasing the fineness from 225 to 350 m²/kg, residue on 33 microns decreased from 52% to 40%, 37%, 37% and 33% of COPC 2, COPC 3, COPC 4, COPC 5 and COPC 6 respectively. Similarly, increasing the fineness to 350 m²/kg increases the - 10 μ from 11.32 to 15.52, 16.12, 16.86, 18.76 and 21.45 of COPC 2, COPC 3, COPC 4, COPC 5 and COPC 6 respectively. Average mean size of the cement particles of COPC 2, COPC 3, COPC 4, COPC 5 and COPC 6 are 21.24, 17.57, 18.07, 16.68, 15.86 and 14.76 respectively.

SOPC samples also show the similar characteristics by increasing the fineness of the samples from 225 to 350 m²/kg. Residue on 33 microns decreased from 50% to 49%, 43%, 39% and 37% of SOPC 2, SOPC 3, SOPC 4 and SOPC 5 respectively. Similarly, increasing the fineness to 350 m²/kg increases the - 10 μ from 15.37 to 16.10, 17.32, 19.01 and 21.35 % of SOPC 2, SOPC 3, SOPC 4 and SOPC 5 respectively. Average mean size of the cement particles are 18.01, 17.14, 16.77, 15.80 and 14.88 of SOPC 2, SOPC 3, SOPC 4 and SOPC 5 respectively.

Table 5 PSD studies of COPC samples

	COPC 1	COPC 2	COPC 3	COPC 4	COPC 5	COPC 6
%Tile	Size(um)	Size(um)	Size(um)	Size(um)	Size(um)	Size(um)
1.0	4.11	3.89	3.85	3.80	3.40	3.30
3.0	5.76	5.22	5.22	5.06	4.65	4.43
5.0	6.95	6.17	6.21	5.97	5.53	5.23
10.0	9.40	8.08	7.89	7.78	7.27	6.78
50.0	34.97	26.15	25.46	24.05	23.95	21.75
60.0	44.30	33.65	34.85	30.42	30.62	27.96
70.0	54.74	42.02	43.38	37.90	38.10	35.23
80.0	68.23	51.99	54.12	47.45	47.49	44.32
90.0	91.71	66.50	72.61	63.39	63.26	59.68
95.0	119.1	80.17	93.36	80.95	81.97	77.94
MV(um):	45.11	32.83	35.65	31.31	31.53	29.46
MN(um):	6.46	6.37	6.28	6.29	5.50	5.41
MA(um):	21.24	17.57	18.07	16.68	15.86	14.76

Table 6 PSD studies of COPC samples

	SOPC 1	SOPC 2	SOPC 3	SOPC 4	SOPC 5
%Tile	Size(um)	Size(um)	Size(um)	Size(um)	Size(um)
1.0	2.928	3.48	3.34	3.21	3.17
3.0	4.26	4.90	4.58	4.36	4.25
5.0	5.30	5.97	5.50	5.21	5.04
10.0	7.57	8.22	7.40	6.98	6.63
50.0	33.27	32.74	27.34	24.63	22.80
60.0	43.45	43.20	35.04	32.05	29.96
70.0	55.10	55.20	43.56	40.91	38.29
80.0	70.28	70.65	54.15	52.3	48.72
90.0	96.48	95.48	72.22	72.32	65.83
95.0	126.6	119.7	92.22	96.02	85.44
MV(um):	45.25	44.11	35.19	34.53	31.51
MN(um):	4.35	5.34	5.30	5.17	5.17
MA(um):	18.01	19.14	16.77	15.80	14.88

5.2. Performance Evaluation

The Blaine's fineness of cement blends, determined by air permeability (Blaine's apparatus) method, as per IS: 4031(2)-1999. The water requirement to prepare cement paste of normal consistency was measured according to IS: 4031(4)-1998. The initial and final setting times, determined as per IS: 4031(5)-1998 of cement blends. The compressive strengths at different aging, measured as per IS: 4031(6)-1989 on 1:3 cement-sand mortar of cement blends [21]. The obtained results of COPC and SOPC are given in Table 8 and 9 respectively. The strength of cement is the most important of all the cement properties. The compressive strength of cement cube specimens of COPC and SOPC samples in Figure 6. From the obtained results, it can be observed that an increase in cement fineness of both the cements reduces the initial and final setting times as well as increases in the initial age compressive strength of the cement mortar. This is due to the fast kinetics of hydration of the mineral C3S (tricalcium silicate) and C2S (dicalcium silicate). These two mineral phases are the two principal constituents in the cement which ensure the development of the strength. Thus, it can be concluded that the fineness of cement is a significant characteristic during the hydration of the mix, more the particles are fine, more the cement surface in contact with water is large and more the hydration is fast and

complete (shortening of set times). Generally, most of the literature reported that higher alkali content, present in the form of hydroxide or sulfate, result in quick setting.

In the case of COPC samples, strength increase due to the increase of the reactivity of C_3A and C_3S with the presence of alkali content in the cement. Generally, the evidence points out to the increase of initial hydration. However, it is possible that the nature of the reaction products is impacted rather than just the reaction rate itself; also, the role of alkalis on the hydration process greatly depends on the sulfate levels in cement. It is very clear from the literature that alkalis impact the hydration and kinetics of hydration of Portland cements. That is, the dependence of the hydration process on many factors, especially sulfate content, and the dependence of this on alkali content as well as the cement composition appear not to be clearly considered.

The later strength development of COPC samples shows different results. Up to 250 m²/kg fineness, samples show strength gain from 7 Days to 28 days. But, beyond the 250 m²/kg fineness level samples don't show strength improvement from 7D to 28 D. This may be due to the presence of alkalis in the clinker (CCLK-1) samples as well as average belite size is lower comparing to SCLK-2 (i.e. 16 micro meters). The alkali hydroxide favors the formation of crystalline monophase around the C_3A grains, and the sulfate, mostly in the form of potassium, leads to the precipitation of syngenite. These effects are strongly dependent on the content of these ions. For strength development, most of the evidence pointed to an increase in the early strength and a decrease in the 28-day strength. This is due to lowering of the gel in the presence of alkalis compared to that of calcium which enhances the gelatinous properties of the hydrates. It was concluded that the effect of the alkalis on late strength is caused by something happened during the early hydration and not governed by the conditions of the pore liquid at later ages.

Residue on 45 microns and passing percentage of COPC samples at different fineness respectively low and more compare to SOPC samples. Strength development at later ages depends mainly on the particle size between 10 to 30 microns. The 50 passing particle size in the case of COPC samples is lower than the SOPC samples. Based on the above observations, in addition to the sulfate optimization according to the fineness for optimum performance characteristics, mineralogy of the clinker plays an important role for optimizing the fineness, residue and PSD for desired performance cement characteristics.

Table 7 Performance characteristics of OPC prepared by CCL-1 at different fineness levels

Name	N.C	Fineness	Setting Time, min		Compressive Strength, Mpa			
		m ² /kg	IST	FST	1D	3D	7D	28D
COPC-1	25.0	225.0	175.0	220.0	16.5	35.5	41.5	49.0
COPC-2	25.2	247.0	165.0	215.0	17.5	38.0	43.0	49.0
COPC-3	25.5	267.0	140.0	180.0	25.5	42.0	52.0	52.0
COPC-4	25.8	308.0	130.0	170.0	29.0	46.0	54.0	50.0
COPC-5	26.0	321.0	110.0	160.0	28.5	48.0	52.5	49.5

Table 8 Performance characteristics of OPC prepared by CCL-1 at different fineness levels

Name	N.C	Fineness	Setting Time, min		Compressive Strength, Mpa			
		m ² /kg	IST	FST	1D	3D	7D	28D
SOPC-1	25.0	225.0	150.0	215.0	22.5	33.5	42.5	53.0
SOPC-2	25.2	256.0	145.0	210.0	23.5	33.5	43.0	52.5
SOPC-3	25.5	285.0	130.0	185.0	28.5	34.5	45.0	55.0
SOPC-4	25.8	307.0	125.0	180.0	28.5	35.0	42.5	54.0
SOPC-5	26.0	335.0	110.0	170.0	32.0	35.0	43.5	52.5

6. CONCLUSIONS

Clinker quality is an important parameter to consider the set points for target fineness. Quality of the clinker plays an important role on the residue and particle size distribution of the cements. Clinker (CCL-1) contains high C₂S shows drastic difference on the residue while increasing the fineness from 225 to 250 m²/kg. SCL-2 shows significant difference on the residue at the fineness level of 275 m²/kg. Particle size distribution studies shows increasing the percentage of pass after 33 microns while increasing the fineness from 225 to 350 m²/kg. Clinker contains more C₂S around 26% shows significant passing percentage difference between 225 to 250 m²/kg as 90 percentage on 91.71 microns to 90 percent on 66.50 microns. Clinker contains more C₂S around 22% shows significant passing percentage difference between 250 to 275 m²/kg as 90 percentage on 95.48 microns to 90 percent on 72.22 microns. The setting times are shortened as the cement particle fineness increases. This is due to the fact that the coarse cement particles require less hydration to achieve equivalent set than a finer cement particle. The compressive of cement increases as cement particles increase in fineness from 225 to 350 m²/kg. Also, the finer the cement, the higher the reactivity of its particle. Based on the above observations, in addition to the sulfate optimization according to the fineness for optimum performance characteristics, mineralogy of the clinker plays an important role for optimizing the fineness, residue and PSD for desired performance cement characteristics.

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