MICROSTRUCTURE STUDY AND STRENGTH BEHAVIOR OF CEMENT MORTAR INCORPORATING POND ASH

[1]Dhirajkumar Lal, [2]Arunkumar Dwivedi, [3]Aniruddha Chatterjee
[1]Assistant Professor, Department of Civil Engineering, PG MOZE College of Engineering, Savitribai Phule Pune University, (INDIA).
[2]Professor & Dean (Academics) Sandip University, NASHIK, (INDIA)
[3]Assistant Professor, Department of Plastic and Polymer Engineering, Maharashtra Institute of Technology, Aurangabad

ABSTRACT

This paper explains experimental studies carried out to investigate strength behavior of cement mortar incorporating pond ash replaced with cement and sand in the mortar formations. It is studied through mechanical, structural, surface morphological and composition properties. The microstructure properties are studied by x-ray diffraction techniques and SEM. Results shows that crystalline nature for the mortar sample with replacement of sand increases as compared to the mortar sample replaced with cement. XRD studies suggest that the grain size is less when pond ash replaced with sand while grain size found to be more when pond ash replaced with cement. Compressive strength of cement mortar block incorporating pond ash in sand replacement observed to be more due to finer grain size. While in cement replacement it is found to be less due to larger grain size.

Keywords – Cement mortar, Compressive strength, Pond ash, SEM, XRD.

I. INTRODUCTION

Fly ash and bottom ash are the two major residual by product after coal combustion in the thermal power generation plants. Enormous research on the utilization of fly ash is in progress these days due to its pozzolanic properties and fineness. Pond ash which is also one of the byproduct after coal combustion obtain from burning which comprises of ply ash, bottom and water to form slurry which later disposed off in the pond. Efforts are made in this research to utilize pond ash in the cement mortar with sand and cement and to investigate its behavior through some of the mechanical as well as chemical tests. Cement mortar incorporating pond ash in two set (first sand replaced with pond ash & second cement replaced with pond ash) of mortar cube specimen with replacement percentage varying from 0% to 100% prepared and cured for 28 days. As per literature available, pond ash in mortar and concrete were utilized as a fine aggregate or cement without correlating its mechanical properties with chemical properties such as SEM, FESEM, XRD and XRF [1, 2].

II. LITERATURE SURVEY

Table 01 below gives details of aim, methodologies and conclusion of various researches done previously [3-13]. Cement with various proportions was replaced with various alternate/ industrial waste materials to study the microstructure of replacement binder material.

Table 01: Literature survey for the various replacement binder materials referred for XRD and SEM analysis.

Authors	Year	Aim	Methods	Main Findings
[3] Nuruddin, M.F. *et al.*	March 2010	Development of **Sidoarjo Mud** as cement replacement material	Compression, tensile, porosity and Ultrasonic Pulse Velocity (UPV) (XRD), (XRF), and (SEM-EDX)	Mud can be used as a cementitious material with optimum (OPC) replacement at 10%.
[4] Demir I. *et al.*	2011	Prepared mortar using ground waste brick **(GWB)** as a pozzolanic partial replacement for cement.	Compressive strength (SEM) and (EDX)	Highest compressive strength values were found for the samples with 10% replacement ratio.
[5] Ozgunler and Ozgunler	**2011**	Study the chemical properties and pozzolanic activity of Karamursel-Kaytazdere**volcanic tuffs**	XRF, XRD and pozzolanic activity water absorption, density, specific gravity, Compressive and tensile strength.	Physical and mechanical properties of the repair mortars were compatible with the original samples.
[6] O. Cizer*et al.*	**2008**	Performance of cement mortar replacing it **with lime hydrate** and **lime putty** at 30%, 50% and 70% by mass.	Compressive, flexural strength, Bulk density, porosity, DTG, SEM	Long-term compressive strength development has been achieved after 180 days while
[7]Chai and Cheerarot	**2003**	Replacement of cement with **bottom ashes** before and after being ground	Compressive strengths, Los Angeles abrasion test, Normal consistency and setting times of cement, Specific Gravity and Fineness, PSA SEM	Compressive strengths of mortar containing 20–30% of bottom ash as cement replacement were less than cement mortar at all ages, but shows higher compressive strength than the cement mortar when it was grounded.

[8] I. Vegas *et al.*	**2009**	Effect of **thermally-treated paper sludge** on behaviour of cement mortar	XRF, XRD, Setting times, Compressive strength	Metakaolinite and calcite influence on physical-mechanical properties of blended cements. enhancement in compressive strengths with replacement greater than 10% and increase in drying shrinkage of cement based materials.
[9] D.A. Pereira *et al.*	**2000**	Partial replacements of either sand or cement with **salt slag** which is a byproduct from **aluminum scrap.**	Compressive strength Flexural strength, XRD, XRF and SEM,	Up to 10 % of salt slag can be replaced by cement to achieve the significant strength of mortar. Sand substitution by slag gives better performance while replaced at 30-50 % replacement levels.
[10] Valeria Corinaldesi*et al*.	**2010**	Utilization of ash from burning of **paper mill sludge** as a supplementary cementitious material in mortar and concrete manufacturing. Replaced by 5 to 10% of Portland cement.	Thermo-gravimetric (TG) and differential thermal analysis (DTA), XRD, SEM, and Compressive Strength test.	The mortars containing 5% PA exhibited a compressive strength higher that of mortar made from OPC at 28 days.
[11] C.M.A. Fontes*et al.*	**2004**	Utilization of **sewage sludge ash** as mineral additive in cement mortars.	Thermal differential (DTA), thermogravimetric (TGA) and differencial thermogravimetric (DTG), PSA, XRD, Compressive strength, Total porosity and absortivity.	The results of the study indicate that high strength mortar and concretes can be obtained using sewage sludge ash as mineral additive.
[12] B. Liu *et al.*	**2007**	Studied effect of replacement of **alumina-silica rich fired clay brick waste** fines with cement in mortar.	XRF, XRD, Compressive strength, DTA, SEM.	The incorporation of CB waste fines with OPC-quartz was found to be beneficial in terms of strength development up to 15%. The addition of MgO to OPC-quartz caused a decrease in proportion of PC results decrease in compressive strength.
[13] HanifiBinici *et al.*	**2008**	Experimental investigation on the effect of ternary blending on the various properties of cement paste and mortar.	Compressive strength, Flexural Strength, XRD, SEM.	Basaltic pumice and slag had more than 61 % of major chemical components (SiO_2 $+Al_2O_3$ $+Fe_2O_3$) also leads to increase in the amount of CSH gel in the mortar.

III. EXPERIMENTAL DETAILS

2.1 MATERIALS

Ordinary Portland cement conforming to BIS: 8112 and ASTM C-150 was used throughout the research work. Sand confirms to BIS: 383(1970) and IS: 2116-1980, free from natural resources or crushed stone sand, not contained any appreciable amount of clay balls or pallets, not contained any harmful impurities have been used. The sand was properly graded and of medium sized with a fineness modulus of 2.0 to 2.2. Pond ash as per IS: 3812:2003, the generic name of the waste product due to burning of coal or lignite in the boiler of a thermal power plant is pulverized fuel ash (PFA). PFA can be fly ash, bottom ash, pond ash or mound ash. Fly ash is the pulverized fuel ash extracted from the flue gases by any suitable process like cyclone separation or electrostatic precipitation. PFA collected from the bottom of boilers by any suitable process is termed as bottom ash.

2.2 TEST METHODS

All laboratory experiments were conducted in general accordance with the applicable procedures outlined by the European Norm EN 1015-10 (1999), ASTM C 270, ASTM C780. To evaluate the performance of cement mortar incorporating pond ash, compressive strength, XRD and SEM tests were conducted for assessing the performance of mortar incorporating pond ash. The reference mortar taken was a standard mortar without replacement of pond ash.

- *Compressive strength*

Compressive strength of cement mortar specimens were conducted on cube measuring 70.7 x 70.7 x 70.7 mm. The cubes for series I and II were casted as per mix proportion for cement to sand ratio of 1:4 and. The cubes were tested for compressive strength for the curing period of 28 days. The results are shown in Table 02.

- *X-ray Diffraction*

To analyze the crystalline properties of a material, X-Ray Diffraction (XRD) technique was used. A graph pattern of XRD analysis suggests whether the material is crystalline, partially crystalline or amorphous [07]. Tests were conducted in the research by using Bruker D-8 Advance diffracto-meter with CuKα1 radiation.

- *Scanning Electron Microscopy*

(SEM) of different samples were also investigated by using Hitachi S-4800-Type-II (Hitachi High Technology Corporation). SEM images were taken at 10 µ M, 20 µ M and 200 µ M magnifications for pond ash.

2.3 MIX PROPORTIONS

In the present study, total of 2 mixes in 1:4 fractions is prepared. In the first series (series-I) of mixes one part of cement and 4 parts of sand & pond ash is taken with pond ash replacement in sand varying from 0 to 100%. In the second series (series-II) of mixes one part of cement & pond ash (PA) and 4 parts of sand is taken with pond ash replacement in cement varying from 0 to 100%. All mixes were prepared at room temperature. [As per BIS-3812 (Part-1):2003]

IV. RESULTS AND DISCUSSION

4.1 COMPRESSIVE STRENGTH

The compressive strength of cement mortar of series-I and series-II are plotted in figure 1. In case of series-I, the compressive strength containing 30 % pond ash replacement is shown in table 2 is higher than standards cement mortar for 28 days curing period. Incorporating the 40 % and above, the decrease in compressive strength after 28 days curing was reported. It is observed that compressive strength for series-II (Cement Replacement), decreases with increased rate of replacement incorporating pond ash percentage.

. 3.2 X-RAY DIFFRACTION (XRD) STUDIES

The structural property was investigated through-X-ray diffraction (XRD) technique using Bruker D8 Advance diffracto-meter with CuKα1 radiation. XRD patterns were recorded in the 2θ range 20^0 to 80^0 and 2θ/θ coupling linkage method was applied to study the crystalline nature of prepared cement mortar cured up to 28 days. The structural properties are studied by x-ray diffraction techniques and results show that crystalline nature for the mortar sample with replacement of sand increases as compared to the mortar sample replaced with cement.

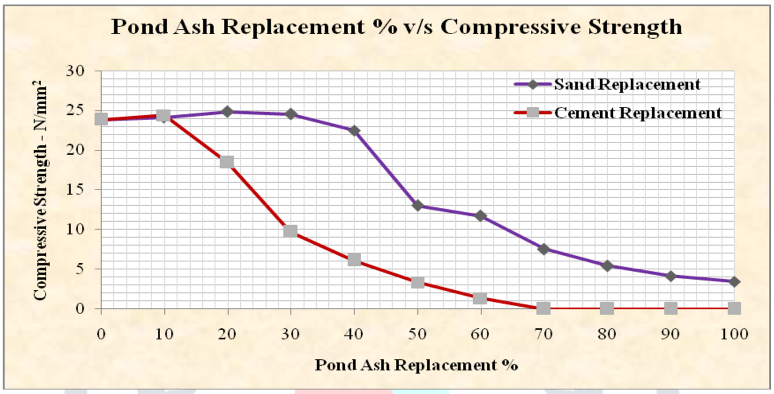


Figure 1. Compressive Strength of Series-1 & 2 Mortar specimens

Compressive strength of cement mortar block incorporating pond ash in sand replacement observed to be more due to finer grain size. While in cement replacement it is found to be less due to larger grain size

Table 2. Compressive Strength of cement mortar for sand and cement Replacement

Sr No	Pond Ash % Replacement Proportion	Sand Replacement [Series-1]	Cement Replacement [Series-2]
1	0	23.81	23.81
2	10	24.15	24.37
3	20	24.82	18.41
4	30	24.51	9.71
5	40	22.48	6.14
6	50	13.04	3.40
7	60	11.73	1.38
8	70	7.56	0.00
9	80	5.45	0.00
10	90	4.17	0.00
11	100	3.47	0.00

All the major diffraction peaks for all mortar samples were chosen to determine the average size of the crystallites by using Debye Scherer's equation 1. [14]

$$D=0.94\lambda / \beta \cos \theta \quad ... (1)$$

Where,
λ = wavelength of X-ray used
β = Full width at half maximum in radian and
θ = Bragg's diffraction angle.

Diffraction pattern of all the mortar samples are presented in the figure 2, 3 and 4 illustrates the difference between each mortar samples and size fraction. Differences in peak intensity reflect phase concentration of fine and course mortar fractions. The JCPDS is referred from the peak intensities at different angles with a database of various compounds and minerals' by matching 2 theta angles. The compounds present in the samples are determined by matching the 2e values with maximum peak intensities [15].

Considering the strength behavior in presence of pond ash in both the replacement, the particle size play a vital role. The grain sizes for various replacement levels are determined by XRD peaks and Debye Scherer's equation and in accordance with [17-18]. The results shows increase in the compressive strength with decrease in the grain size from table 03 for sand replacement level from 20 % to 60%.

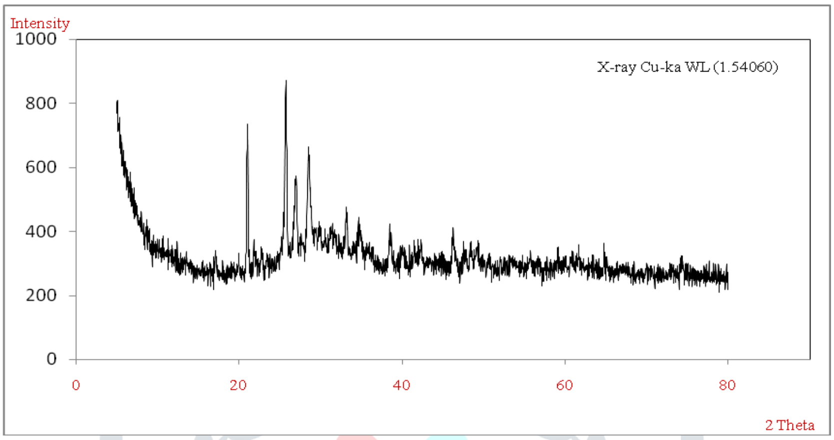


Figure 2. X-ray diffraction of Mortar (0 % Replacement)

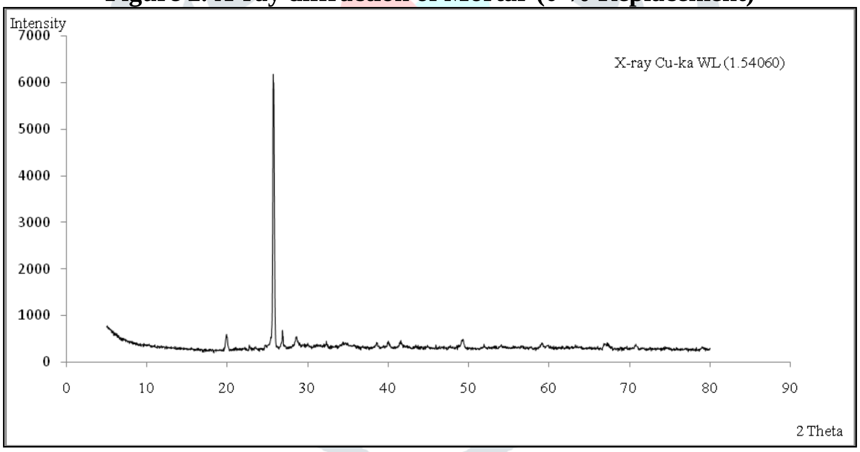


Figure 3. X-ray diffraction of Mortar (60 % Cement Replacement with Pond Ash cement)

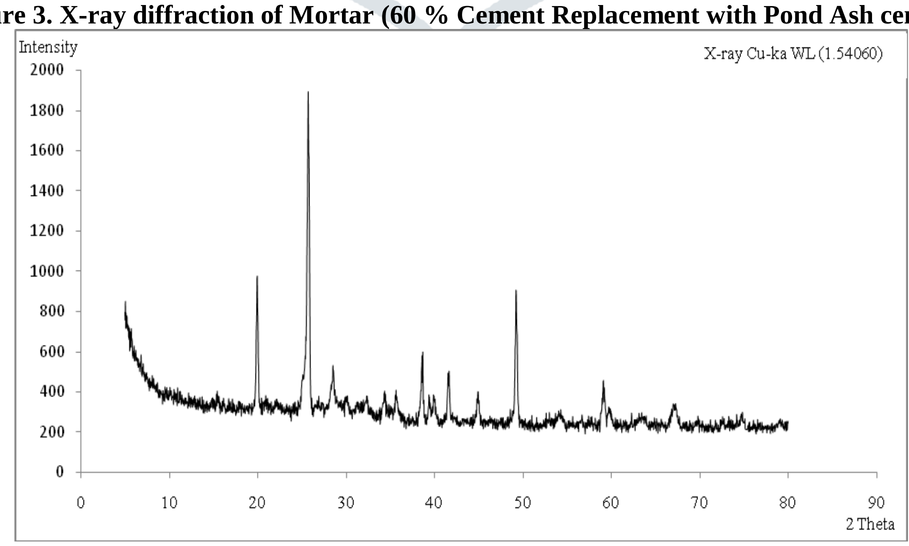
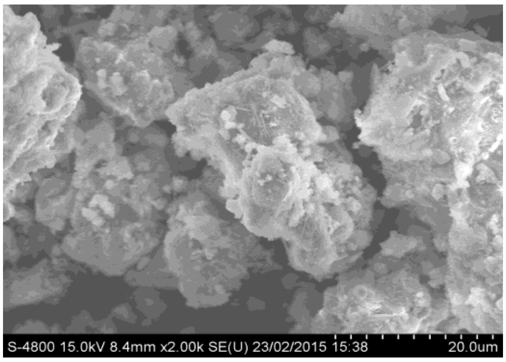


Figure 3. X-ray diffraction of Mortar (60 % Sand Replacement with Pond Ash)

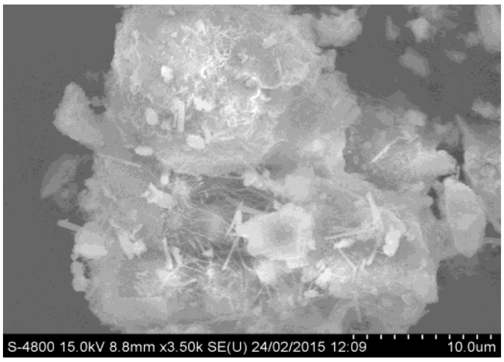
Table 03. Average grain size for prepared cement mortar samples for various replacement percentages

Mix Proportion	2θ (Deg)	d (A^0) (Standard)	d (A^0) (Observed)	Grain Size nm
0%	21.055	4.21594	4.216029898	31.69
	25.757	3.45609	3.456052816	
	26.963	3.30411	3.304145398	
	28.578	3.12099	3.120986707	
	33.196	2.69662	2.696610345	
C-40	19.963	4.44403	4.444119	22.77
	25.76	3.45569	3.455657	
	28.519	3.12729	3.127309	
	38.561	2.33287	2.332876	
	49.139	1.85258	1.852587	
C-50	19.911	4.4556	4.455608	10.45
	25.698	3.46379	3.463854	
	28.502	3.12911	3.129136	
	39.947	2.25507	2.255072	
	74.823	1.26792	1.26791	
C-60	19.905	4.4569	4.456937485	21.50
	25.752	3.45672	3.456712504	
	26.841	3.31891	3.318886139	
	28.582	3.12055	3.120559024	
	49.223	1.84962	1.849622856	
C-90	26.571	3.35194	3.351995	8.78
	40.282	2.23707	2.237194	
	50.055	1.8208	1.820641	
	57.414	1.60369	1.603789	
	68.156	1.37475	1.374854	
S-20	19.999	4.43611	4.4362	13.49
	25.698	3.46392	3.463854	
	35.699	2.51305	2.513068	
	41.602	2.16909	2.169106	
	49.277	1.84773	1.847722	
S-40	19.938	4.44968	4.449635	12.24
	25.706	3.46274	3.462794	
	28.52	3.12724	3.127202	
	38.548	2.33365	2.333634	
	49.188	1.85084	1.850857	
S-50	19.917	4.45438	4.449193	7.05
	25.692	3.46462	3.467568	
	28.485	3.13092	3.123984	
	49.18	1.85114	1.850786	
	59.055	1.56299	1.56358	
S-60	19.94	…	4.449193286	24.44
	25.73	…	3.459618206	
	28.52	…	3.127201738	
	38.6	…	2.33060955	
	49.22	…	1.849728572	

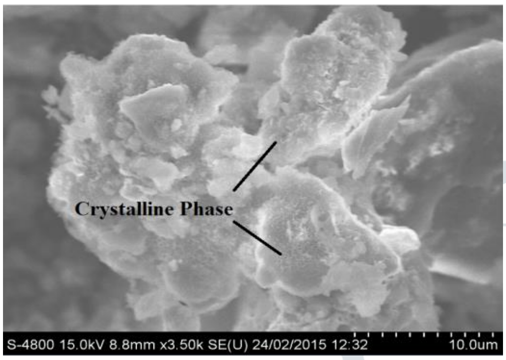
Microstructure of the sample supports the strength properties due to their shape and size differences [16]. The presence of pond ash with rough surface leads to increase in the bondage between the particles results in enhanced compressive strength.



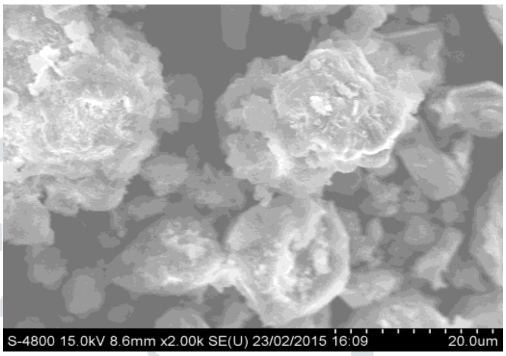
(a) 0 %



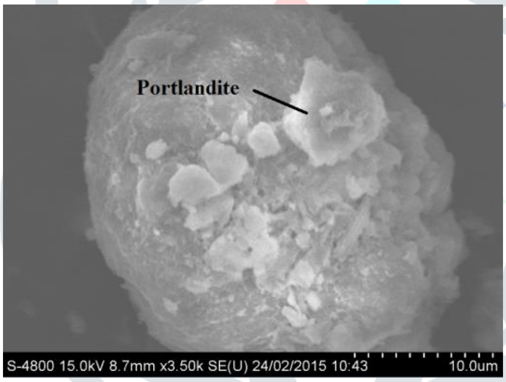
(b) C-50



(d) S-50



(c) C-90



(e) S-60

Figure 5. SEM Images of powdered cement mortar sample of replacement of
(a) 0 %, (b) C-50, (c) C-90, (d) S-50and (e) S-60
(b)

Figure 5 shows the SEM Crystal formation of hydrated silicates of calcium with calcium hydroxide and calcium aluminates were observed in SEM of S-50 in figure 5(d). The honeycombed structures with fiber or needle like structure of ettringite were also observed in figure 5 (b) in the SEM for mortar mix C-50.

V. CONCLUSION

The mortar sample with sand replacement (series-I) comprises of higher compressive strength for replacement percentage of up to 30- to 40. Beyond that strength goes on decreasing with increase in pond ash. For series-II mortar sample, strength found goes on decreasing with increase in replacement percentages. The compressive strength containing 30 % pond ash replacement with sand is higher than standards cement mortar for 28 days curing period. Incorporating the 40 % and above, the decrease in compressive strength after 28 days curing was reported. With the increase in pond ash replacement, grain size obtained by Debye Scherer's equation represents to be higher in sand replacement as compared to cement replacement. The results shows increase in the compressive strength with decrease in the grain size from table 03 for sand replacement level from 20 % to 60%. The presence of pond ash with rough surface leads to increase in the bondage between the particles results in enhanced compressive strength. With the inclusion of pond ash in mortar we can open one way of utilizing it in major quantity to reduce the amount of CO_2 emission in the environment with decreasing use of cement and unnecessary quarrying of river sand.

REFERENCES

[1.] Chunet al.,
"A Study on the Fundamental Properties of Concrete Incorporating Pond-Ash in Korea", The 3rd ACF International Conference-ACF/VCA 2008, pp.701-408.
[2.] Bapat et al.,
"Eco-friendly Concrete with High Volume of Lagoon Ash", Journal of Materials in Civil Engineering, Vol. 18, No. 3, June 1, 2006, ASCE,ISSN 0899-1561/2006/3-453–461).
[3.] Nuruddin, M.F. *et al*,
"Sidoarjo Mud: A Potential Cement Replacement Material", Journal of Civil Engineering Dimension, Vol. 12, No. 1, March 2010, pp. 18–22.
[4.] Demir I.et al.,
"Performance of cement mortars replaced by ground waste brick in different aggressive conditions", Ceramics – Silikáty 55 (3), 2011, pp. 268-275.
[5.] Ozgunler and Ozgunler,
"A research on Karamursel region volcanic tuff as a pozzolanic additive in repair mortars used for historical buildings" Scientific Research and Essays Vol. 6(3), 4 February, 2011, pp. 641-647.
[6.] O. Cizeret al.
"Blended lime-cement mortars for conservation purposes: Microstructure and strength development"Journal of Structural Analysis of Historic Construction – D'Ayala&Fodde (eds)2008 Taylor & Francis Group, London, ISBN 978-0-415-46872-5, pp. 965-972.
[7.] Chai and Cheerarot,
"Development of Bottom Ash as Pozzolanic Material", Journal Of Materials In Civil Engineering, ASCE, January/February 2003, pp. 48-53.
[8.] I.Vegas et al.
"Engineering Properties of Cement Mortars Containing Thermally Activated Paper Sludge"WASCON 2009 (International Scientific Society for Construction with Waste) June 3, 2009 – Lyon.
[9.] D.A. Pereira et al.
"Mechanical behaviour of Portland cement mortars with incorporation of Al-containing salt slags"Cement and Concrete Research 30 (2000), pp.1131-1138.
[10.] Valeria Corinaldesiet al.
"Paper Mill Sludge Ash as Supplementary Cementitious Material", Coventry University and The University of Wisconsin Milwaukee Centre for By- products Utilization, Second International Conference on Sustainable Construction Materials and Technologies, June 28-30, 2010, Ancona, Italy, main Proceeding, ISBN 978-1-4507-1490-7.
[11.] C.M.A. Fonteset al.
"Potentiality of Sewage Sludge Ash as Mineral Additive in Cement Mortar and High Performance Concrete", Use of Recycled Materials in Buildings and Structures, November 2004, Barcelona, pp. 1-10.
[12.] B. Liu et al.
"Strength Development in Autoclaved Alumino-silicate Rich Industrial Wastecement Systems Containing Reactive Magnesia", J. Aust. Ceram. Soc. 43 [1] (2007) 82-87.
[13.] HanifiBinici et al.
"A Study on Cement Mortar Incorporating Plain Portland Cement (PPC), Ground Granulated Blast- Furnace Slag (GGBFS) and Basaltic Pumice", Indian Journal of Engineering & Material Sciences, Vol. 12, June 2005, pp. 214-220.
[14.] Suryavanshi K.E., A.M.Patil,. Dhake R. B.,
"XRD and SEM Characterization of Chemically" Deposited PbxCd1-xS Thin Films. J.Applicable Chem. 2015, 4 (4): 1227-1236.
[15.] Balasubramanian et al.
"Strength and microstructure of mortar with sand substitutes" Gradevinar Journal, Croatia, Vol. 68 (2016), No. 1, pp. 29-37.
[16.] Ozlem Celik, Erdem Damci, Sabriye Piskin. J. ,
"Characterization of Fly ash and its effect on the compressive strength properties of Portland cement." Engg Mat. Science, Vol. 15, 2008, pp.433-440.
[17.] Jiang Jian-guo, Xu Xin, Wang Jun, Yang Shi- jian, Zhang Yan,
"Investigation of basic properties of fly ash from urban waste incinerators in China.", Journal of Environmental Sciences, Vol. 19, 2007, pp.458–463.
[18] Khouryet al.
"Use of XRD patterns to evaluate compressive strength of stabilized aggregates", JCPDS - International Centre for Diffraction Data 2004, Advances in X-ray Analysis, Volume 47, International Centre for Diffraction Data.

REFERENCES (Codes of Practices)

A. BIS 8112 (2005) Specification for 53 grade Ordinary Portland Cement, Manak Bhavan New Delhi.
B. ASTM (C) 150-86, Standard Specification for Portland cement, Annual Book of ASTM Standards, 1988, (Vol. 4.01-Cements, Lime, Gypsum), Easton, USA.

C. BIS 383 (1970), Specification for Coarse and Fine Aggregate from natural Sources for Concrete (Second Revision), Bureau of Indian Standards, New Delhi, India.

D. BIS 2116 (1980) Specification for Sand for masonry mortars (1st revision), Manak Bhavan New Delhi, India.

E. BIS-3812 (Part-1):2003 Pulverized fuel ash-specification for use as pozzolana in cement, cement mortar and concrete, Bureau of Indian Standards, New Delhi, India.

F. EN 1015-10 (1999) European Code for Methods of test for mortar for masonry - Determination of dry bulk density of hardened mortar.

G. ASTM C-780 Standard Test Method for Preconstruction and Construction Evaluation of Mortars for Plain and Reinforced Unit Masonry.

H. BIS-3812 (Part-1):2003 Pulverized fuel ash-specification for use as pozzolana in cement, cement mortar and concrete, Bureau of Indian Standards, New Delhi, India.