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Determination of Optimum Percentage of Metakaolin by Compressive Strength and XRD Analysis.

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Abstarct

The use of pozzolanic materials in concrete as partial replacement of cement is gaining wide acceptance in the construction industry. Metakaolin (MK) and fly ash are the pozzolanas, which conform the requirements of construction industry and largely available in India. In present study, the results of X-ray diffraction (XRD) analysis of concrete containing metakaolin and fly ash were analysed. Four samples K0, K7, K8 and K9 containing 0%, 7%, 8% and 9% MK (by weight of cement) respectively were analysed with XRD at the sample age of 1 day.

Keywords: *Pozzolanic materials, concrete, metakaolin, fly ash, X-ray diffraction*

1. Introduction

Concrete is probably the most extensively used construction material in the world. However, environmental concerns both in terms of damage caused by the extraction of raw material and CO_2 emission during cement manufacture have brought pressures to reduce cement consumption by the use of supplementary materials called pozzolanas[1]. Typically pozzolanas are used as cement replacements rather than cement additions. Adding pozzolans to an existing concrete improve the workability and strength of concrete[2]. Replacing some of the cement with pozzolans preserves the mix proportions. Of all the pozzolanas, metakaolin is most reactive[3]. Metakaolin is refined kaolin clay that is fired (calcined) under carefully controlled conditions to create an amorphous aluminosilicate that is reactive in concrete. Like other pozzolans (fly ash and silica fume are two common pozzolans), metakaolin reacts with the calcium hydroxide (lime) byproducts produced during cement hydration. The use of metakaolin results in considerable enhancement in strength, particularly at the early stages of curing along with the strength at later age[4].

Fly ash produces more cementitious paste as it has a lower unit weight. The greater the percentage of fly ash in the paste, the better lubricated the aggregates are and the better concrete flows. Fly ash reduces the amount of water needed to produce a given slump[5].

When X-rays interact with a crystalline substance (phase), one gets a diffraction pattern. The X-ray diffraction pattern of a pure substance is, therefore, like a fingerprint of the substance. The powder diffraction method is thus ideally suited for characterization and identification of polycrystalline phases[6]. Today about 50,000 inorganic and 25,000 organic single components, crystalline phases and diffraction patterns have been collected and stored on magnetic or optical media as standards[7]. The main use of powder diffraction is to identify components in a sample by a search/match procedure. Furthermore, the areas under the peak are related to the amount of each phase present in the sample. X- ray diffraction is now a common technique for the study of crystal structures and atomic spacing.

2. Materials and methods

2.1 Cement

Cement is a fine, grey powder. It is a fine powder produced by grinding Portland cement clinker (more than 90%), a limited amount of calcium sulphate (which controls the set time) and up to 5% minor constituents. It is mixed with water and materials such as coarse aggregates and fine aggregates to make concrete. The cement and water form a paste that binds the other materials together as the concrete hardens. It is a material with adhesive and cohesive properties which is capable of bonding mineral fragments into a compact-solid. The ordinary Portland cement (OPC) is the most important type of cement. The OPC is classified into three grades (33, 43 and 53) depending upon the compressive strength of cement at 28 days. Ambuja 43 grade OPC was used in this study. It was fresh and free from any lumps and the specific gravity of cement was 3.17.

2.2 Coarse aggregates

Materials which are large to be retained on 4.75 mm IS sieve and contain only that much of fine material as is permitted by the specifications are termed as coarse aggregates. The graded coarse aggregate is described by its nominal size i.e. 40 mm, 20 mm, 16 mm and 10 mm. Since the aggregates are formed due to natural disintegration of rocks or by the artificial crushing of rocks or gravel, they derive many of their properties from the parent rocks. These properties are chemical and mineral composition, specific gravity, hardness, strength, pore structure and colour. Some other properties of the aggregates not possessed by the parent rocks are particle shape and size, surface texture, absorption, etc. All these properties may have considerable effect on the quality of concrete. Crushed stone aggregate (locally available) of nominal size 20 mm and 10 mm in the proportion of 50:50 were used throughout the experimental study. The aggregates were washed to remove dust and dirt and are dried to surface dry condition.

2.3 Fine aggregate

It is aggregates most of which passes through a 4.75 mm IS sieve. Sand is generally considered to have a lower size limit of about 0.07 mm. Material between 0.06 mm and 0.002 mm is classified as silt, and still smaller particles are called clay. The fine aggregate may be one of the following types:-

(i) Natural sand
(ii) Crushed stone sand
(iii)Crushed gravel sand

According to size, the fine aggregate may be described as coarse, medium and fine sands. Depending upon the particle size distribution is: 383-1970 has divided the fine aggregate into four grading zones. The grading zones become finer from grading zone I to grading zone IV. The sand confirming to zone II is used in this study and its specific gravity was 2.59.

2.4 Water

Fresh potable, which is free from concentration of acid and organic substances was used for mixing of concrete.

2.5 Metakaolin

Metakaolin (MK) is a pozzolanic material. It is a dehydroxylated form of the clay mineral kaolinite. It is obtained by calcination of kaolinitic clay at a temperature between 500ºC and 800ºC. Between 100 and 200ºC, clay minerals lose most of their adsorbed water. Between 500 and 800ºC kaolinite becomes calcined by losing water through dehydroxilization. The raw material input in the manufacture of metakaolin ($Al_2Si_2O_7$) is kaolin clay.

Kaolin is a fine, white, clay mineral that has been traditionally used in the manufacture of porcelain. Kaolinite is the mineralogical term that is applicable to kaolin clays. The dehydroxilization of kaolin to metakaolin is an endothermic process due to the large amount of energy required to remove the chemically bonded hydroxyl ions. Above this temperature range, kaolinite becomes metakaolin, with a two dimensional order in crystal structure.

Like other pozzolans (such as fly ash and silica fume), metakaolin reacts with the calcium hydroxide (lime) by-products produced during cement hydration. Calcium hydroxide accounts for up to 25% of the hydrated Portland cement, and calcium hydroxide does not contribute to the concrete's strength or durability. Metakaolin combines with the calcium hydroxide to produce additional cementing compounds, the material responsible for holding concrete together. Stronger concrete are formed with less calcium hydroxide and more cementing compounds. Metakaolin, because it is very fine and highly reactive, gives fresh concrete a creamy, non-sticky texture that makes finishing easier.

2.6 Fly ash

The fly ash, also known as pulverised fuel ash, is produced from burning pulverized coal in electric power generating plants. During combustion, mineral impurities in the coal (clay, feldspar, quartz, and shale) fuse in suspension and float out of the combustion chamber along with exhaust gases. As the fused material rises, it cools and solidifies into spherical glassy particles called fly ash. It is a fine grained powdery particulate material that is collected from the exhaust gases by electrostatic precipitators or bag filters. Depending upon the collection system, varying from mechanical to electrical precipitators or bag houses and fabric filters, approximately 85–99% of the ash from the flue gases in retrieved in the form of fly ash. Fly ash accounts for 75–85% of the total coal ash, and the remainder is collected as bottom ash or boiler slag.

2.7 Superplasticizer

Superplasticizers, also known as high range water reducers, are chemicals used as admixtures. These polymers are used as dispersants to avoid particle aggregation, and to improve the flow characteristics of suspensions such as in concrete applications. Their addition to concrete or mortar allows the reduction of the water to cement ratio, not affecting the workability of the mixture, and enables the production of self-consolidating concrete and high performance concrete. This effect drastically improves the performance of the hardening fresh paste. Indeed the strength of concrete increase whenever the amount of water used for the mix decreases. The percentage of superplasricizer was kept constant in this study.

2.8 Mix design

Concrete mix design (M30) was designed by IS code method using is: 10262-1989.

2.9 Casting of cubes

The 150 mm X 150mm X 150mm size concrete cubes were used as test specimens to determine the compressive strength. Three cubes for each mix were casted. The compressive strength testing of cubes was carried out on a Universal Testing Machine of capacity 2000 kN. The test was performed at 1 day

2.10 XRD

In this study powder method of XRD was used, which is easier to interpret and is capable of high accuracy, especially for determining the spacing of atoms in a solid. Detail of samples used in XRD analysis is given in Table 1. The XRD analysis was conducted on the samples at the age of 1 day. Rigaku Miniflex Table Top spectrometer with Cu-Kα line of wavelength ƛ=1.5418Å at the scanning rate of 2^0/min and varied from 10^0 to 80^0.

3 Results and discussion.

Results of compressive strength of cubes KO, K7, K8 and K9 are given in Table 1.

Table 1: XRD sample detail

S. No.	Sample	MK	Fly ash	Mean compressive strength at 1 day (MPa)
1	K0	0%	10%	5.27
2	K7	7%	10%	8.73
3	K8	8%	10%	12.80
4	K9	9%	10%	11.24

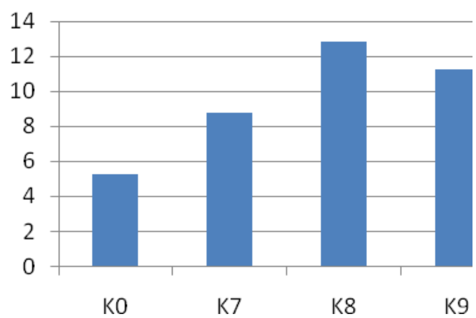

Fig. 1 Compressive strength of samples.

From the compressive strength test it was observed that compressive strength is increased at 0%, 7% and 8% of metakaolin and decreased at 9%. Optimum strength gain is at 8% of matakaolin. By comparing the d-spacing peaks of samples with standard reference pattern or ICDD database. It was also observed that the phase CaO Al_2 SiO_2 H_2O is increasing in sample K0, K7 and K8 which results in increase in compressive strength.

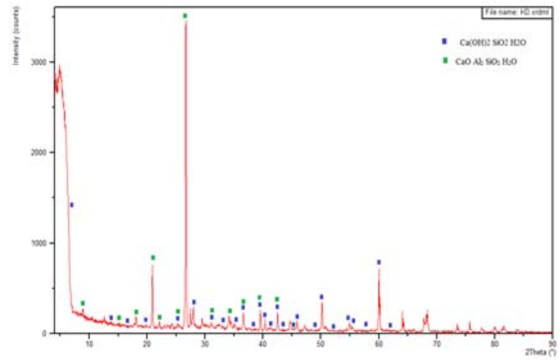

Fig. 2 XRD pattern of K0 (MK 0% and Fly ash 10%)

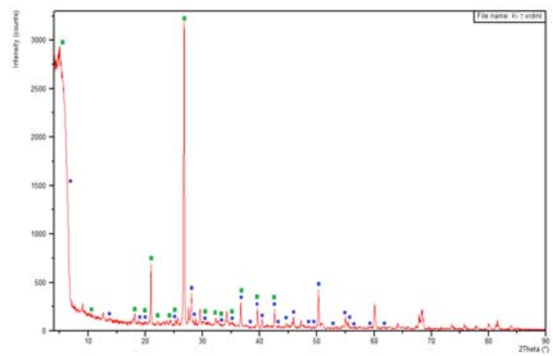

Fig. 3 XRD pattern of K7 (MK 7% and Fly ash 10%)

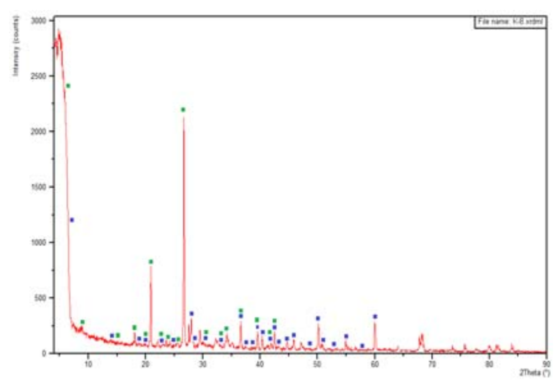

Fig. 4 XRD pattern of K8 (MK 8% and Fly ash 10%)

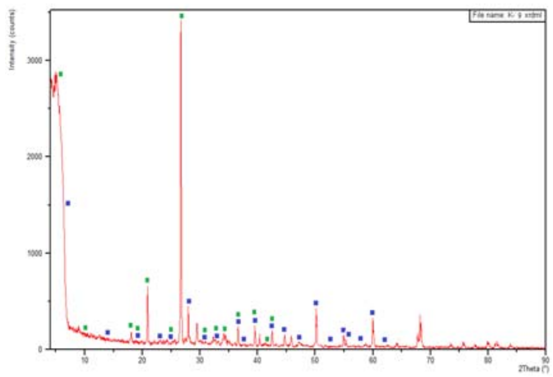

Fig. 5 XRD pattern of K9 (MK 0% and Fly ash 10%)

The powder samples of cement, fly ash, metakaolin, K0, K7, K8 and K9 were analysed by XRD analysis, indexing of cement was done and the peaks were marked on the basis of ICDD database. The XRD pattern of samples K0, K7, K8 and K9 are given in figures 2, 3, 4 and 5 respectively.

The XRD pattern indicates that in all samples the phase of $Ca(OH)_2$ SiO_2 H_2O with a mixed phase of CaO Al_2 SiO_2 H_2O were present. With the increase in the percentage of metakaolin, the phase CaO Al_2 SiO_2 H_2O is increased but in sample K9 phase CaO Al_2 SiO_2 H_2O is decreased at 9% of metakaolin. Results of compressive strength of samples are shown in table I.
Here we mentioned earlier the phase CaO Al_2 SiO_2 H_2O decresed at maximum percentage of metakaolin and as compared with compressive strength of samples, the compressive strength of sample K9 is also decreased. Maximum strength gain is at 8% of metakaolin.

4 Conclusions

From the compressive strength test it was observed that compressive strength is increased at 0%, 7% and 8% of metakaolin and decreased at 9%. Optimum strength gain is at 8% of matakaolin. By comparing the d-spacing peaks of samples with standard reference pattern or ICDD database. It was also observed that the phase CaO Al_2 SiO_2 H_2O is increasing in sample K0, K7 and K8 which results in increase in compressive strength. Results of XRD are supplementing with the results obtained from compressive strength test. From the foregoing study optimum percentage of MK recommended to be used for concrete is 8%.

5 References

1. E Worrell, L Price, N Martin, C Hendriks & L O Meida, "Carbon dioxide emissions from the global cement industry.", Annu. Rev. Energy Environ, 26, 2001, 303–29.
2. A Naceri, H M Chikouche & P Grosseau, "Physico-Chemical Characteristics of Cement Manufactured with Artificial Pozzolan (Waste Brick)", World Academy of Science, Engineering and Technology, 52, 2009, 41-43.
3. U Krajei, I Janotka, I Kraus & P Jamnicky, "Burnt kaolin sand as pozzolanic material for cement hydration", Ceremic silikaty, 51, 2007, 217-224.
4. B B Sabir, S Wild, J Bai, "Metakaolin and calcined clays as pozzolans for concrete: a review", Cement & Concrete Comp, 23, 2001, 441-54.
5. R N Ojha, "Use of fly ash and condensed silica fumes in making concrete", IE(I) Journal CV, 77, 1996, 170-173.
6. C R Ward & D French, "Relation between coal and fly ash mineralogy, based on quantitative X- ray diffraction methods, World of coal ash conference, Lexington, Kentucky, USA, 2005.
7. L L Yang, Synthesis and optical properties of ZnO nanostructure, Thesis, Linkoping university, Sweden, 2008.
8. A Torre & M Aranda, "Accuracy in Rietveld quantitative phase analysis of Portland cements", J. Appl. Cryst., 36,

2003, 1169-1176.

9. K L Scrivener, T Fullmann, E Gallucci, G Walenta & E Bermejo, "Quantitative study of Portland cement hydration by X-ray diffraction/Rietveld analysis and independent methods" Cement and Concrete Res., 34, 2004, 1541-1547.

10. A Palomoa, M W Grutzeck & M T Blancoa, " Alkali- activated fly ashes- A cement for the future" Cement and Concrete Res., 29, 1999, 1323-1329.