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Posted Date: 3 May 2023

doi: 10.20944/preprints202305.0155.v1

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Structural Evolution and Compressive Strength at 3, 7, 14 and 28 Days of Mixtures of Mortars Substituting Portland Cement for 0, 10 and 15% Fly Ash

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Abstract: Samples of mortar mixtures were prepared substituting the cement Portland (CPC-30R) by 0 (standard), 10 and 15% of fly ash and the structural evolution and compressive strength at 3, 7, 14 and 28 days were determined. The results for standard mortar samples showed the mineralogical species portlandite, calcite, ettringite, iron oxide, silicon oxide and sillimanite. Magnetite was identified in the mixtures of mortars with portland cement substitution by 10% and 15% fly ash. The peaks corresponding to the portlandite, and ettringite showed an increase in their intensities with increasing curing time attributed to the consolidation of mineral species. The SEM technique results showed that the mortar samples without fly ash addition contained mainly portlandite and ettringite while the samples with cement substitution by 10 and 15% of fly ash at 28 days further contained particles of fly ash coated with portlandite and ettringite, particles with a smooth surface and particles of fly ash with signs of attack on its surface. An increase in c was observed when the age of the mortar and the substitution of Portland cement by fly ash were increased from 3 to 28 days and from 0 to 15% respectively. The maximum value of c was registered for the mortar sample with Portland cement substitution by 15% fly ash at 28 days of curing with 17.38 Mpa.

Keywords: fly ash; mortar; compressive strength; Portland cement; structural evolution; compressive strength

1. Introduction

One of the main objectives in the manufacture of concrete and mortar in recent years is to reduce the amount of cement used during their manufacture [1]. In this way, in addition to stone aggregates, residues from pyrometallurgical processes are also used as raw materials for the formation of concrete [2,3]. Fly ash is a solid waste that is obtained by electrostatic precipitation or by mechanical collection of dust that accompanies the combustion gases of the burners of thermoelectric plants fed by pulverized coals [4]. The Fly ash has been studied as an additive or substitute for portland cement in concrete in order to improve its compressive strength thanks to its pozzolanic activity which is atribuited to the presence of SiO_2 and Al_2O_3, mineral species that react with calcium hydroxide during the cement hydration process to form calcium silicate hydrate (CSH) and calcium aluminate hydrate (CAH), and that in turn provide a denser matrix, greater resistance and reduction of concrete porosity [5–7]. The strength of mortar depends on the cohesion of the cement paste on its adhesion to the aggregate particle, and to a certain extent on the strength of aggregate itself [8]. Based on the above, research on the development of strength in mixtures of mortar with fly ash substituting Portland cement have been carried out. Chindaprasirt and Rukzon [12] study the strength, porosity and corrosion resistance of mortars made with ternary blends of ordinary Portland cement, ground rice husk ash and classified fly ash and they found that the use of ternary blend produced mortars with improved strengths at the low replacement level with rice husk ash and fly ash and at the later age in comparison to that of ordinary Portland cement mortar. Supit et al. [10], evaluaron el efecto effect of ultrafine fly ash (UFFA) on compressive strength development of mortars containing high volume class fly ash as partial replacement of cement. The study reveals that the cement mortars with 8% UFFA of cement replacement exhibited higher compressive strength at 7 and 28 days than control mortars. Chindaprasirt et al.[11], reviewed the influence of fineness of fly ash on water demand and some of the properties of hardened mortar. They found that the use of fly ashes resulted in significant improvement in drying shrinkage with the coarse fly ash showing the least improvement owing primarily to the high water to binder ratio of the mix. Cheerarot and Jaturapitakkul [12] studied the potential of using disposed fly ashes and replaced the Portland cement by fly ashes at the rate of 10%, 20%, and 30% by weight of cementitious material and they found that the compressive strengths of the fly ash mortars at the age of 7 days were higher than 75% of the standard mortar and increased to be higher than 100% after 60 days. Rukzon and Chindaprasirt [13] studied the strength and chloride resistance of mortars made with ternary blends of ordinary Portland cement, ground palm oil fuel ash, and 0-40wt% classified fly ash and they determined the use of ternary blended cements produces good strength mortars. Fu et al. [14] studied the influence of the contents of the clinker, activators and fly ash on the properties of blended cement with high fly ash content and found that the main hydration product of the fly ash blended cement was CSH gel, ettringite and a small amount of $Ca(OH)_2$. It has been investigated [15] that by using a high percentage of cement substitution by fly ash, the compressive strength tends to decrease due to the fact that there is a saturation of the mixture due to these residues. A method for measuring the consumption of $Ca(OH)_2$, main reactant of pozzolanic reactions, is by XRD analysis, is relatively accurate in evaluating the reactivity of fly ash [15] this method requires a minimum of 28 days for sufficient occurrence of the fly ash pozzolanic reaction. In this article a detailed study of the structural evolution and mechanical performance of mortars with Portland cement substitution by 10 and 15% fly ash is carried out. The study will consist of analyzing the structural evolution of the mineralogical phases formed by means of X-ray diffraction and Scanning Electron Microscopy (MEB) stopping the hydration process of the material at 3, 7, 14 and 28 days and the subsequent evaluation of the mechanical resistance to compression.

2. Materials and Methods

Composite Portland cement (CPC-30R), sand with an average particle size of 4 mm, fly ash and water were used as source materials. Initially, Portland cement and fly ash were structurally characterized by X-ray diffraction. (diffractometer Equinox 2000 Cu Ka) and morphologically using Scanning Electron Microscopy (JEOL JSM-IT300). Additionally, the particle size distribution was determined (Beckman and Coulter LS13320). The quantities of source materials for each mortar sample are shown in Table 1. The amount of fly ash used was 0, 10 and 15 % and it was calculated with respect to 786 g of Portland cement. The mixture of Portland cement and fly ash was kept in a constant ratio of 1:2.75 and 1:0.64 with respect to sand and water, respectively. At the same time, a standard mortar sample (sample 0) was prepared without the addition of fly ash.

Mortar samples were manufactured in bronze molds of cubic geometry of 50 mm per side with a base with smooth and rigid walls, which were previously covered with a thin layer of lubricating oil to prevent sticking. For the preparation of the mortars, the sand and 50% of the water were mixed, later Portland cement was added, fly ash and lastly the remaining 50% water and this was stirred until a homogeneous mixture was obtained.

Table 1. Quantities of raw materials used for each manufactured mortar sample

Sample	Substitution (%)	Portland cement CPC-30R. (g)	Fly ash (g)	Sand (g)	Water (ml)
0	0	786	0	2160)	500
1	10	707	78.6	2160	500
2	15	668	117.9	2160	500

The mixture was placed in the mold filling it halfway and it was compacted with a piston 32 times in a period of 10 seconds. This compaction was carried out in four cycles of eight consecutive blows distributed uniformly on the surface of the mortar, and each cycle was perpendicular to the previous one. Immediately afterwards the second half of the mortar was added and a second compaction was carried out. The excess mortar on the surface of the mold was spread and leveled with a screed applying zigzag movements with a 15° inclination, as stated in the standard [16]. The samples remained in the molds at room temperature for 24 hours, later they were removed from the mold and immersed in tanks with clean water at room temperature where they remained 3, 7, 14 and 28 days until the moment of the compressive strength test. The tank water was renewed every 72 hours. The measurement of the mechanical resistance to compression (c) of the mortar samples was carried out according to the standard NMX-C-486-ONNCCE [17] using a Controls Pilot equipment, model 50 - C43C04, with a load capacity of 150 kN and a load application speed of 2.55 (kg/cm^2)/s. The structural evolution of the mortars was evaluated and monitored using X-ray diffraction and SEM-EDS from the remains of the samples of the compression test [18,19].

3. Results and discussion

3.1. Characterization of source materials

3.1.1. Fly ash

The X-ray diffraction diffractogram of fly ash is shown in Figure 1. A combination of the mineralogical species quartz (SiO_2) (JCPDS 120708), calcium and iron oxide ($Ca_{0.15}Fe_{2.85}O_4$) (JCPDS 460291), hematite (Fe_2O_3) (JCPDS 160653), and alumina Al_2O_3 (JCPDS 350121) was observed being the first predominant. The mineralogical composition identified is similar to the one reported by Gomes and François [20] in an X-ray diffraction characterization study of mullite in aluminosilicate fly ash. Figure 2 a) shows the backscattered electron (BSE) image of the fly ash, it can be observed that the particles composed mainly by O, Fe, Si and Al (Figure 2 b)) are preferentially spherical with an average size of 13.07 microns (Figure 2 c)). This composition is consistent with that reported by Kutchko and Kim [21], who determined that the surface and internal structure of the majority of fly ash particles are comprised mainly of amorphous aluminum-silicate spheres and a smaller amount of iron-rich spheres. It has been reported that the particle size of the fly ash can have a positive impact in the compressive strength of concrete, since it reduces the porosity of the material and he fineness of fly ash, not the chemical composition, is the major factor affecting the strength activity index of fly-ash cement mortar [22,23].

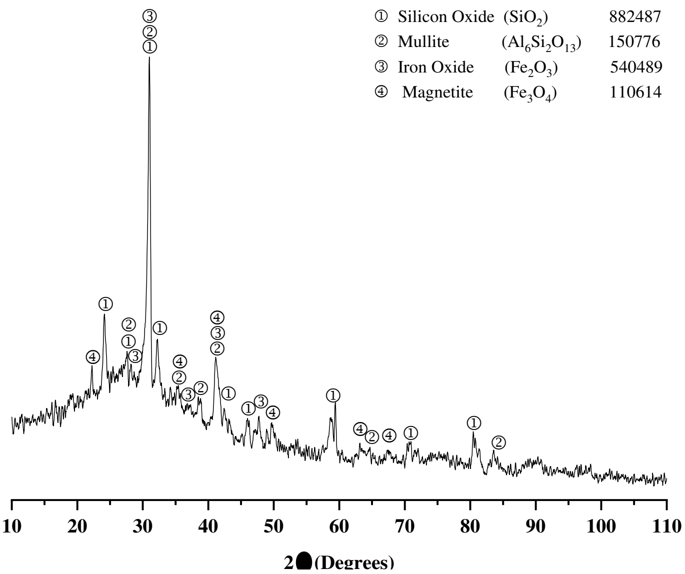

Figure 1. X-ray diffraction diffractogram for fly ash powders.

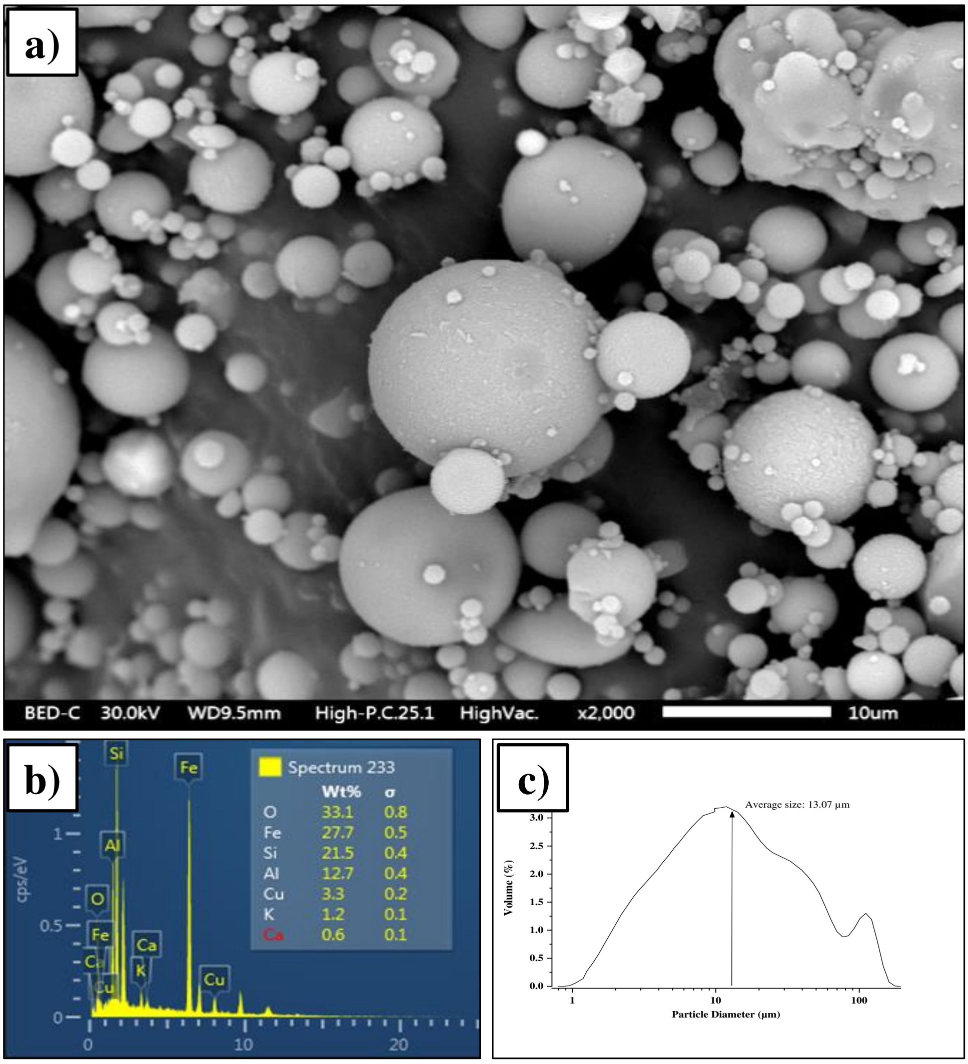

Figure 2. a) Backscattered electron (BSE) image of typical fly ash spheres. b) Elemental spectrum (EDS), c) Particle size distribution.

3.1.2. Portland cement CPC-30R

Figure 3 shows the X-ray diffraction diffractogram of Portland CPC-30R cement. The mineralogical phases identified were: Alite (Ca_3O_5Si) (JCPDS 961540705), Calcite ($CaCO_3$) (JCPDS 969007690), Brownmillerite ($AlCa_2FeO_5$) (JCPDS 961008725) and Tricalcium aluminate ($Al_2Ca_3O_6$) (JCPDS 969014360), being the majority the first. These mineral species coincide with those reported by Young and Yang [24] in studies carried out on the characterization of Portland cement.

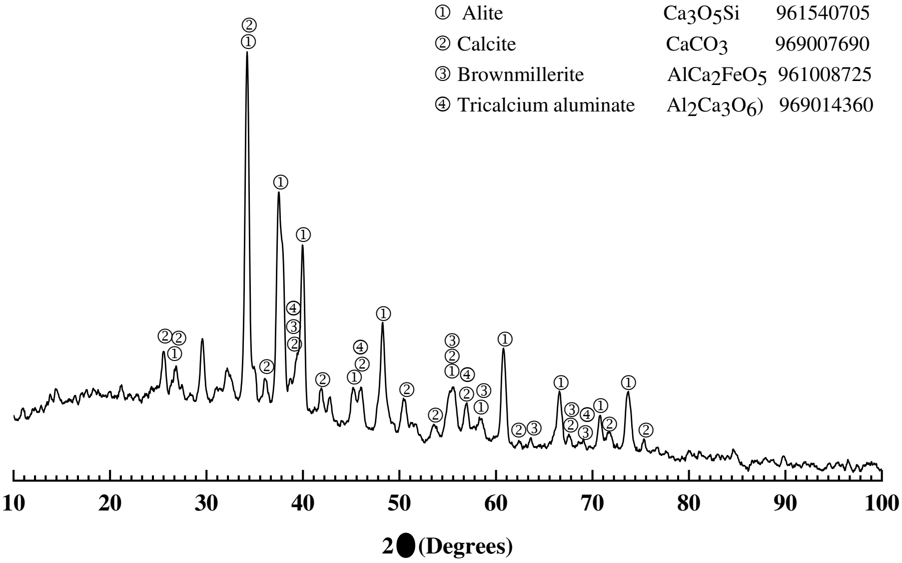

Figure 3. X-ray diffraction diffractogram for portland cement CPC-30R.

Figure 4 a) shows the Backscattered electron (BSE) image of cemento Portland CPC-30R. It was possible to identify that the material is composed mainly of, O, Ca, Si, C, Mg and Al (Figure 4 b)). The morphology is characterized by large crystalline particles of undefined angular shapes with an average size of 5.67 m ((Figure 4 c)) on a visible amorphous material adhered to the surface of the large particles.

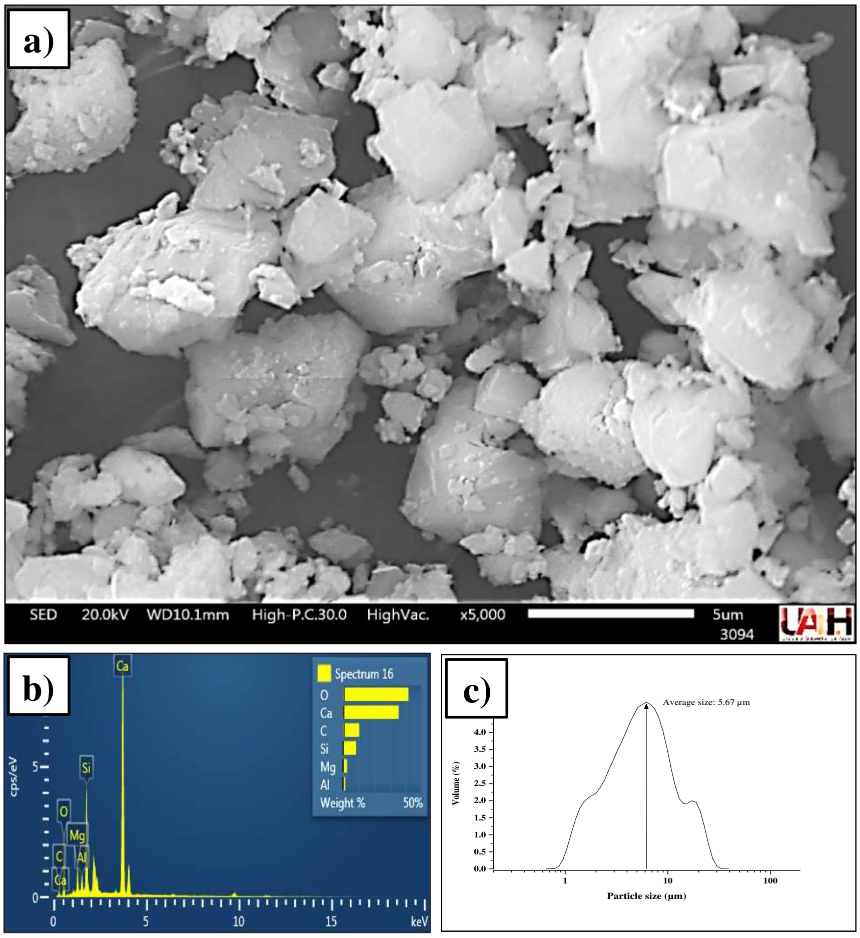

Figure 4. a)Backscattered electron (BSE) image of portland cement CPC-30R, b) Elemental spectrum (EDS), c) Particle size distribution.

3.1.3. Sand

Figure 5 shows the X-ray diffraction diffractogram for the sand used in the manufacture of mortars. The identified phase was calcite ($CaCO_3$) (JCPDS 057690). The presence of this mineralogical specie gives the particles a morphology of flat angular surfaces with a brittle type of fracture (Figure 6a). The average particle size of the particles was 4mm, and the dispersive energy analysis (EDS) indicated a composition consisting of Si, C y Al mostly (Figure 6b)). These results are consistent with the results obtained by X-ray diffraction.

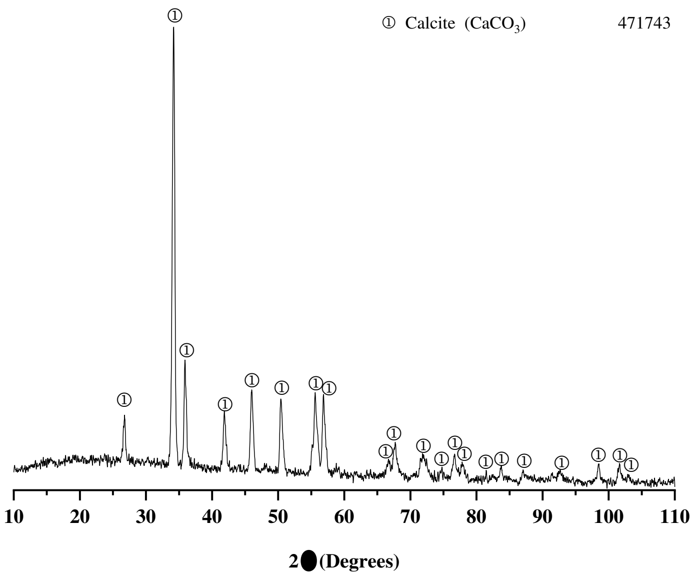

Figure 5. X-ray diffraction diffractogram for sand.

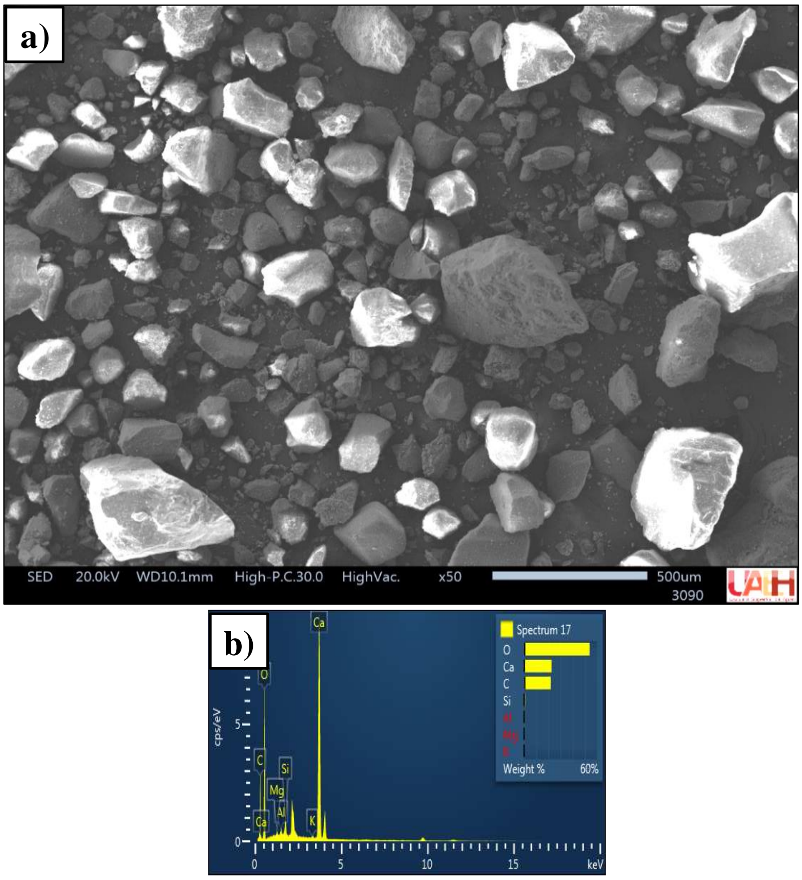

Figure 6. a) Backscattered electron (BSE) image of sand, b) Elemental spectrum (EDS).

3.1.4. X-ray diffraction of mortar samples

Cement hydration depends on reactions between cement minerals and water, as well as the presence of gypsum. The hydration products are hydrated calcium silicates, hydrated calcium hydroxide and calcium sulfoaluminate [7,8]. In Figure 7, the X-ray diffraction diffractograms for the standard mortars are shown (no added fly ash), at 3, 7, 14 and 28 days of curing time. The mineralogical species identified were: portlandite ($CaOH_2O$) (JCPDS 020969), Calcite ($CaCO_3$) (JCPDS 471743), ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12}$) (JCPDS 371476), Iron Oxide (Fe_2O_3) (JCPDS 540489), Silicon Oxide (SiO_2) (JCPDS 882487) and sillimanite (Al_2SiO_4) (JCPDS 831562). These results are characteristic of a typical mineralogical composition of portland cement during the hydration process [25]. Portlandite (calcium hydroxide) is the mineral phase responsible for maintaining the pH of the mixture at high values and to keep the reinforced mixtures protected against electrochemical corrosion. It is the first mineral to decompose at high temperaturas (600°C) which can be reduced with the addition of pozzolans such as fly ash [26]. Ettringite phase (calcium trisulphoaluminate) It is the mineral species that gives cement greater cohesion and is generally produced at late ages (greater than 28 days) by the reaction between gypsum and water [26]. It can be seen in Figure 7, in the spectrum corresponding to 28 days of curing time, an increase in the intensity of the peaks corresponding to this phase located in 2 $\approx$ 27, 44, 48 and 71°, which is indicative of the consolidation of the ettringite phase with increasing curing time. In Figures 8 and 9, X-ray diffraction diffractograms for the mortar mixes with Portland cement replaced by 10% fly ash are shown (Figure 7) and with substitution of portland cement by 15% fly ash (Figure 8) at 3, 7, 14 and 28 days cure time. Again, the mineral species identified were mainly the characteristics of the cement during the hydration process: portlandite ($CaOH_2O$) (JCPDS 020969), Calcite ($CaCO_3$) (JCPDS 471743), ettringite ($Ca_6Al_2(SO4)_3(OH)_{12}$) (JCPDS 371476), Iron Oxide (Fe_2O_3) (JCPDS 540489), Silicon Oxide (SiO_2) (JCPDS 882487), sillimanite (Al_2SiO_4) (JCPDS 831562) and additionally, the Magnetite specie was identified (Fe_3O_4) (JCPDS 110614). It can be noted that the addition of fly ash as a replacement for portland cement in mortar mixes does not drastically influence in the modification of the composition of the mineral species formed, since, with the exception of the magnetite phase (Fe_3O_4) (JCPDS 110614) identified in these samples, the mineral species are the same as those identified in the mortar mixtures without replacing cement with fly ash. Magnetite phase identification (Fe_3O_4) is attributed to the presence of fly ash in 10 and 15% (Figures 8 and 9 respectively), according to the results shown in Figure 1. Has been reported [5,7] that fly ash is a material whose physical and chemical characteristics allow it to develop the pozzolanic function in the mortar and generate a dense microstructure with a discontinuous pore network that makes it difficult for chlorides to pass through the material. It is also noted in Figures 8 and 9 that the peaks corresponding to the main hydration products: (portlandite ($CaOH_2O$) (JCPDS 020969)), identified in the positions 2 $\approx$ 21, 39, 55 y 59°, and ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12}$) identified in the positions 2 $\approx$ 27, 44, 48 y 71° exhibit an increase in their intensities with the increase in the curing time of the mortar mixtures from 3 to 28 days which is attributed to an increase in the consolidation of mineral species.

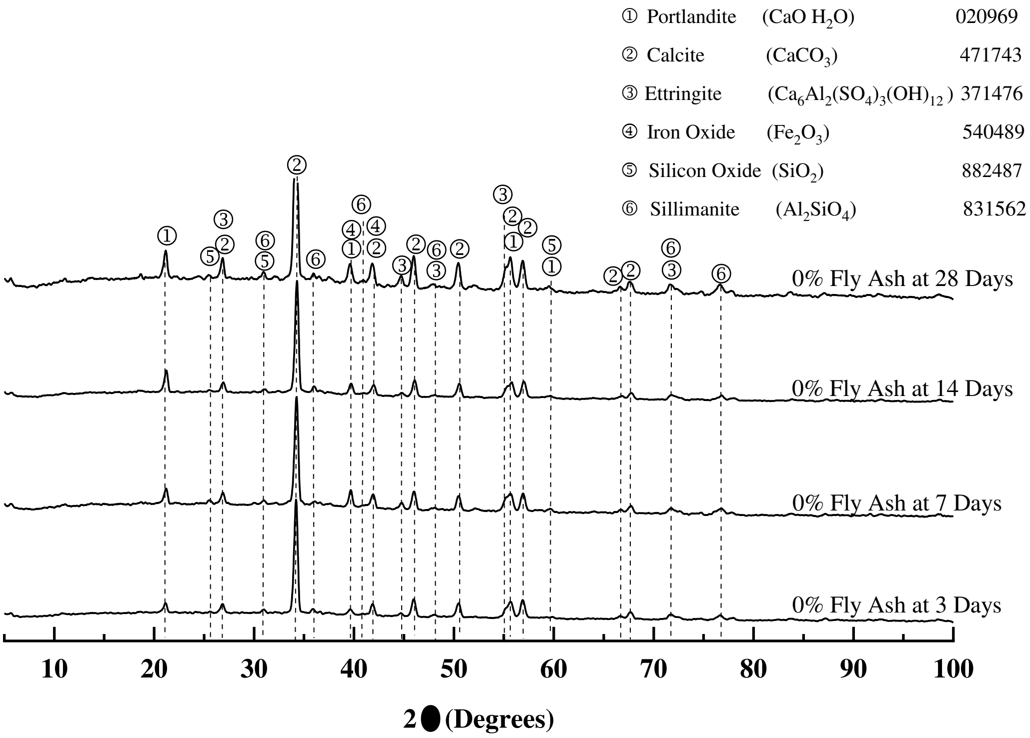

Figure 7. X-ray diffraction diffractograms for standard mortar mixes (without Portland cement substitution) at 3, 7, 14, days of curing time.

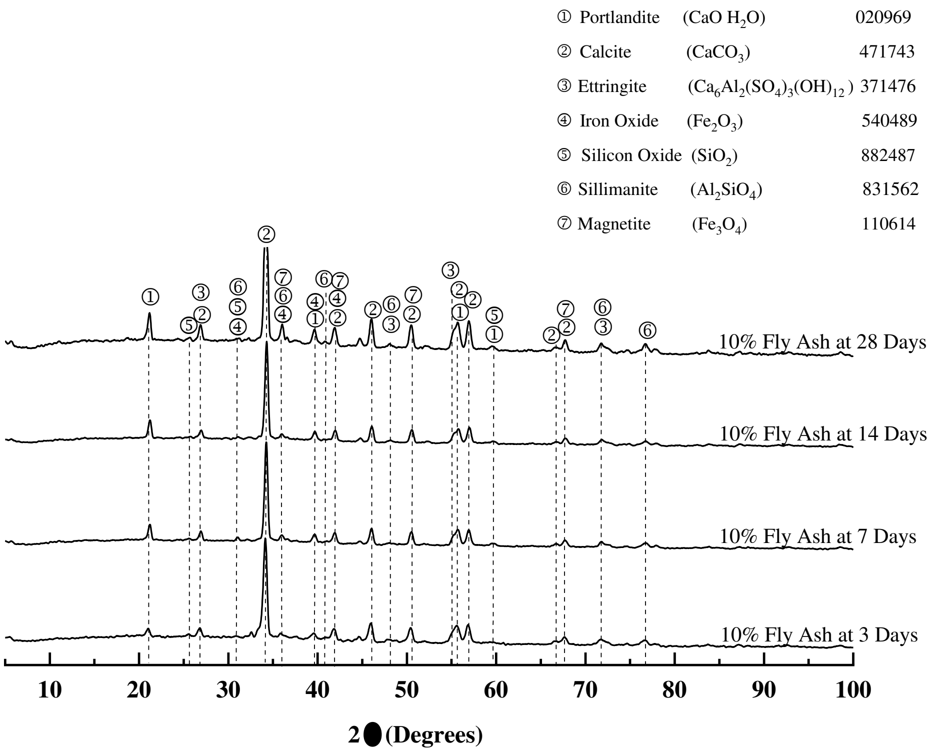

Figure 8. X-ray diffraction diffractograms for the mortar mixtures (with Portland cement substitution by 10% fly ash) at 3, 7, 14 and 28 days of curing time.

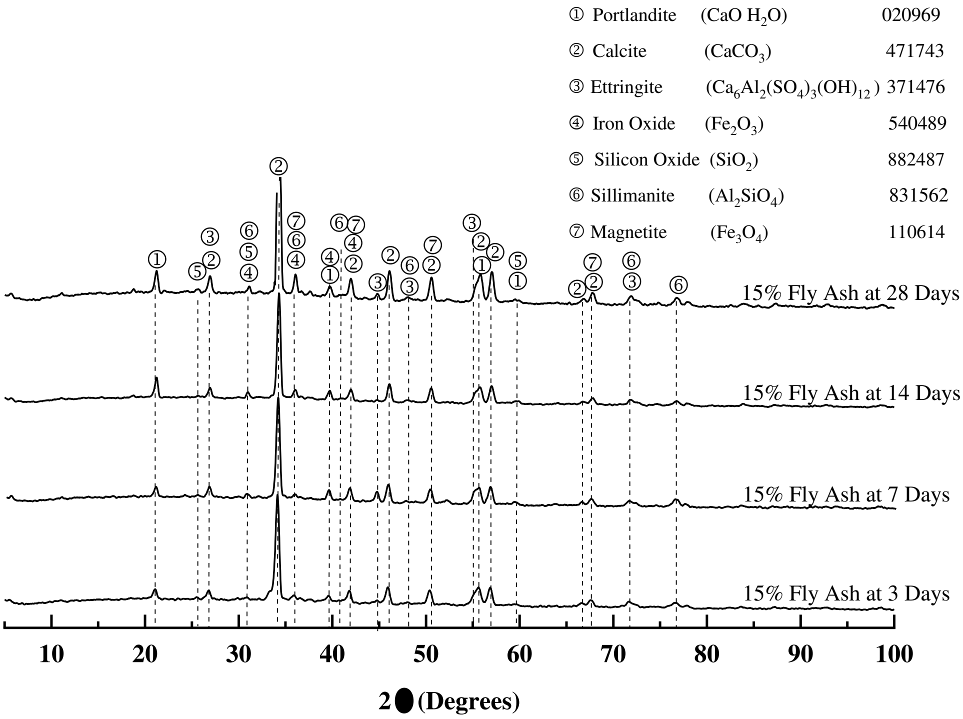

Figure 9. X-ray diffraction diffractograms for the mortar mixtures (with Portland cement substitution by 15% fly ash) at 3, 7, 14 and 28 days of curing time.

3.1.5. Morphology of mortars

Figure 10 shows the detailed images obtained by scanning electron microscopy for the mortar samples without substituting (standard) (a), b)), 10% (b), c)) and 15% (e), f)) of fly ash to 28 days of curing time. In the mortar sample without replacement of cement by fly ash (Figure 8 a), b)) the following mineral species were identified: Portlandite, which is physically arranged in the form of thin hexagonal sheets or platelets and which at 28 days of curing did not show important morphological changes with respect to the portlandites identified in the samples at 3, 7 and 14 days of curing time. Ettringite (ettringite positive), characterized by elongated shapes (elongated rods) in fibrous habits forming oriented formation structures around the aggregate particles. It has been reported [27] that this type of oriented growth habit of ettringite generates an expansion effect and depends on the curing conditions of the mixtures. As mentioned in the previous x-ray diffraction results (Figure 7–9), this species provides the mixtures with an increase in the cohesion of the same. On the other hand, the ettringite identified within the fissures (Figure 10 b)) or pores of the material generates expansion and consolidates at late ages of curing of the mixtures (28 days) [27].

Figures 10 c), d), and 8 e), f) show the detailed images obtained by scanning electron microscopy for the mortar samples substituting by 10% and 15% of fly ash at 28 days of curing time respectively. In these samples, in addition to the hydration product mineral species (portlandite and ettringite) identified in the sample without replacement of cement by fly ash (Figure 10 a), b)) in addition, fly ash particles were observed in three different forms: particles coated with hydration products portlandite y ettringite (Figure 11 a)), particles with a smooth and apparently smooth surface (Figure 11 b)) and fly ash particles that show signs of attack on their surface (Figure 11 c)). It has been reported [28], in an investigation on the effect of fly ash on microstructure of blended cement paste, that at 28 days of curing the ash particles present these characteristics.

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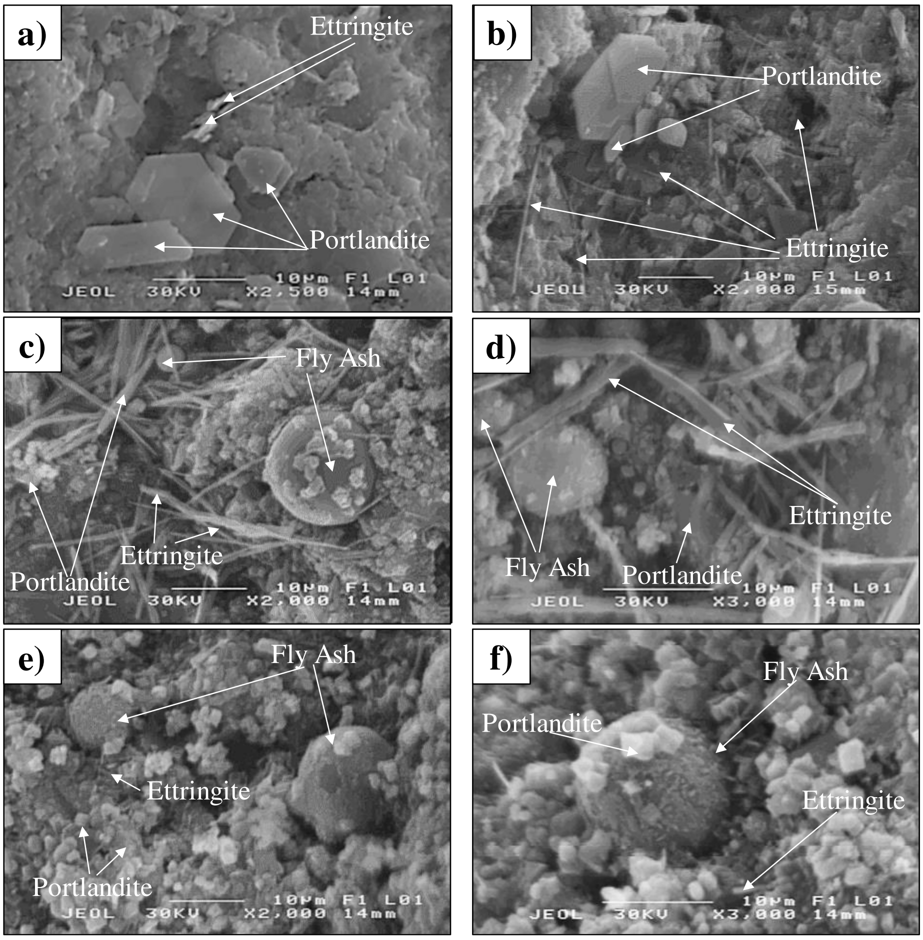

Figure 10. SEM micrograph detail of: a), b) samples of mortars substituting Portland cement for 0 % fly ash. b), c) samples of mortars substituting Portland cement for 10 % fly ash. d), e) samples of mortars substituting Portland cement for 15 % fly ash.

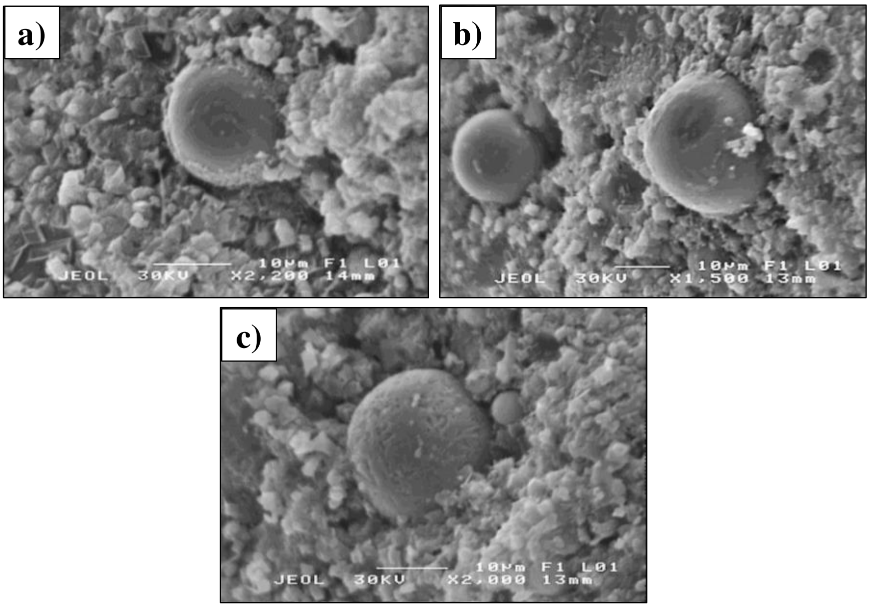

Figure 11. a) Particles coated with hydration products, b) Smooth-surfaced particles, c) Particles with evidence of attack on their surface.

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3.1.6. Compressive strength of mortars.

The mechanical resistance to simple compression of the mortar (c) defined as the ability to support a load per unit area, and is expressed in terms of stress, usually in kg/cm^2, MPa, or pounds per square inch (psi). The results of c obtained for the samples of mortars without substitution of portland cement by fly ash and with substitution of portland cement by 10 and 15% fly ash at 3, 7, 14 and 28 days of age after its manufacture are shown in Figure 12. In general, an increase was observed in c when the age of the mortar and the substitution of portland cement by fly ash were increased from 3 to 28 days and from 0 to 15% respectively, obtaining the maximum values at 28 days. It was found that c for the samples of mortars with 15% substitution of cement by fly ash and for the standard sample at 3 days of age were 12.06 MPa and 13.63 MPa respectively, observing a difference of 1.57 MPa, however, at 28 days of age the difference decreased to 0.07 MPa with 17.45 and 17.38 MPa respectively. Regarding the samples with 10% fly ash, the values of c were 12.57 Mpa at 3 days of age and 17 Mpa at 28 days of age, observing a difference of 1.06 Mpa and 0.38 Mpa with respect to the standard sample at 3 and 28 days of age. It is generally accepted that the pozzolanic reaction in the fly ash/cement systems is important at the ages after 28 days. [29–31]. It has been reported that the reaction between the fly ash and the CH forms calcium silicate hydrates (C–S–H) which have lower calcium-to-silicate ratios (C/S) [32]. The increase in c of the mortars evaluated in this work could be attributed to the improvement in the bond between the hydrated cement matrix and the sand. This is due to the conversion of calcium hydroxide, which tends to form on the surface of aggregate particles to calcium silicate hydrate.

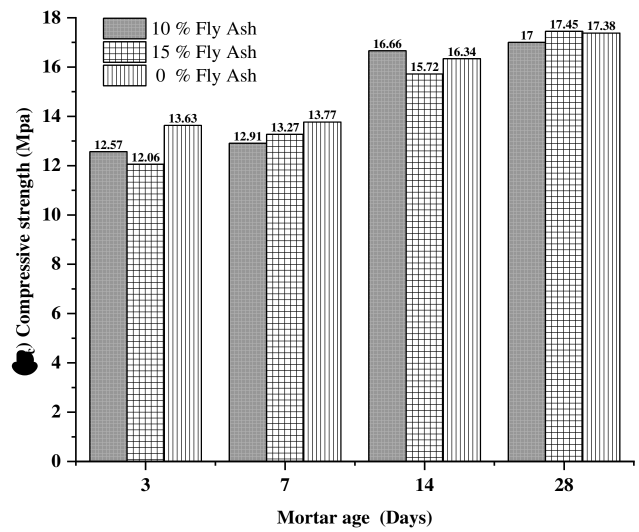

Figure 12. Compressive strength (c) of samples of mortars with 0, 10 and 15% Fly Ash at 3, 7, 14, and 28 days.

4. Conclusions

In this article the structural evolution and compressive strength at 3, 7, 14 and 28 days of curing time of mortars substituting portland cement for 0, 10 and 15% fly ash was analyzed. The mineralogical species identified for the standard mortar mixtures (sample 0% fly ash), at 3, 7, 14 and 28 days of curing time were: Portlandite ($CaOH_2O$), Calcite ($CaCO_3$), Ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12}$), Iron Oxide ($Fe_2O_3$), Silicon Oxide ($SiO_2$) and Sillimanite ($Al_2SiO_4$). These species were also identified in the mortar mixtures with portland cement substitution by 10% and 15% fly ash at 3, 7, 14 and 28 days of curing time and additionally the species Magnetite was identified (Fe_3O_4). The peaks corresponding to the main hydration products: Portlandite ($CaOH_2O$), and ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12}$) showed an increase in their intensities with the increase in the curing time of the mortar mixtures from 3 to 28 days. attributed to an increase in the consolidation of mineral species. The addition of fly ash as a replacement for Portland cement did not drastically influence the modification of the composition of the mineral species formed. The images obtained by SEM technique showed that the samples of mortars without addition of fly ash contained mainly portlandite and ettringite (ettringite positive). In the samples with cement substitution by 10 and 15% of fly ash at 28 days of curing time in addition to the mineral species portlandite and ettringite were also observed particles of fly ash coated with hydration products (portlandite and ettringite), particles with a smooth surface and particles of fly ash with signs of attack on their surface were observed. An increase in the value of c was observed when the age of the mortar and the substitution of Portland cement by fly ash were increased from 3 to 28 days and from 0 to 15%, respectively. The maximum value of c was 17.38 Mpa and it was registered for the mortar mixtures with Portland cement substitution by 15% fly ash at 28 days of curing time.

Author Contributions: Conceptualization, H. Garcia-Ortiz and M. Pérez-Labra, M. Reyes-Pérez; methodology, H. Garcia-Ortiz and J. C. Juarez-Tapia; formal analysis, M. Pérez-Labra; investigation, H. Garcia-Ortiz, F.R. Barrientos-Hernandez, A.M. Teja-Ruiz; resources, J. C. Juarez-Tapia; writing—original draft preparation, M. Pérez-Labra, José A. Romero-Serrano; writing—review and editing, M. Pérez-Labra, José A. Romero-Serrano; visualization, M. Pérez-Labra; supervision, M. Pérez-Labra, J. C. Juarez-Tapia; project administration, M. Pérez-Labra, J. C. Juarez-Tapia; funding acquisition, M. Pérez-Labra, J. C. Juarez-Tapia.

Funding: This research received no external funding.

Acknowledgments: The authors is grateful to PRODEP and CONACyT-México for the financial support.

Conflicts of Interest: The authors declare no conflict of interest.

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