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Evaluation of sustainable high-volume fly ash concretes

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A B S T R A C T

This article presents experimental research work oriented toward developing practical design tools for industrial application, and illustrates the potential benefits of the synergistic effect of an ASTM C 618 Class F fly ash (FA) and a high-range polycarboxylate superplasticizer (SP) in the production of conventional concrete. The different concretes considered in this study were produced with mass substitutions of cement by FA between 15% and 75%, and a target slump of 200 mm ± 20 mm. The total water content was minimized through the use of an optimum SP dosage that resulted in water reductions of 18%, 15% and 11% respectively for the reference mixtures of $w/b = 0.5$, $w/b = 0.55$, and $w/b = 0.6$, which leads to the same percentage reductions of cement. Heat release and heat flow were analyzed through isothermal and semi-adiabatic calorimetry, illustrating that heat release per unit mass of cement is independent of w/b, contrasting with the time of setting results that vary by several hours between the three different w/b ratios. The paper highlights the beneficial effect of the SP in terms of cement reduction and slump retention. Correlations between the FA substitution and slump loss, setting times, compressive strength and static modulus of elasticity (E) were established and they represent very useful tools for the practical applications of the results. Compressive strength developments up to an age of 56 d are also reported, as well as correlations between the modulus of rupture and compressive strength or splitting tensile strength at an age of 28 d.

1. Introduction

Currently, one of the greatest challenges facing the concrete industry is to focus its objectives towards the achievement of sustainable development [1]. In this regard, many steps can be improved in the concrete production process, and one of the first steps is to optimize the mixture proportions by the adoption of technological developments that have not been exploited as they could be in countries with emerging economies such as México. Two technological developments that took place within the last century, and that can improve the ability of the material to maintain ecological processes in the future, are the incorporation of fly ash (FA) into the concrete, and the use of a high-range superplasticizer (SP). There are several publications that explain the technical details for the adoption of these technological developments [2–6]; however, the process of adoption has been slow in some parts of the world.

A significant number of companies from the ready mix concrete industry in México are aware of the benefits of using FA, SP and low water/binder ratios (w/b), but the combination of these three ingredients is rarely found in practice because such concretes are commercialized as special concretes that are not demanded by the potential clients due to their cost. From statistics provided by the National Cement Association of México [7], the production of cement in 2008 was 37.1×10^6 metric tons and the consumption was 35.1×10^6 metric tons; the ready mix concrete industry consumed 19% of this yearly consumption. The other cement consumers were: government (11%), precast concrete and masonry industry (7%), formal contractors (15%) and self construction (48%). The 81% integrated by the last four segments represents the potential users for the exploitation of the research in the present study.

By itself, the use of FA or SP in conventional concrete improves the behavior of concrete in both its fresh and hardened states, but when they are used together the results in terms of mechanical properties, durability and sustainability can be significantly improved [8–14]. The majority of the referenced works deals with superplasticized FA concrete with w/b ratios below 0.4. Conversely, the w/b for this project was fixed between 0.5 and 0.6, a range that is more familiar for the potential users of the results. This work considered two main aspects of sustainability: the use of a SP to reduce total water content requirements and consequently reduce the cement content for a target slump of 200 mm ± 20 mm, and the effect of incorporating a wide range of FA substitutions on various fresh and hardened state properties of concrete.

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In concrete construction, thermal stresses and strains within the concrete will be highly influenced both by the heat transfer conditions between the curing concrete and its environment and by the concrete mixture proportions; in this scenario, one key concrete material property is the energy generated within the concrete element due to the exothermic cement hydration and pozzolanic reactions [15]. Because the *w/b* directly controls the volume of water available for hydration and pozzolanic activity per unit volume of binder and also establishes the initial interparticle spacing between binder particles, it can influence semi-adiabatic temperature rise [16].

The results of this project will give valuable information for the practical application of these concretes in a wide range of projects through useful and friendly tools for many concrete construction segments of the concrete industry in developing countries worldwide.

2. Materials and methods

2.1. Materials

The following materials were used to produce the different concretes considered in the experimental program.

2.1.1. Cement

Blended (with limestone powder) hydraulic cement (designated as CPC 30R by the Mexican standard NMX-C-414-ONNCCE-2004 [17]) that meets ASTM C 595-08 specification for a Type IP (MS) cement [18]. The physical properties and chemical composition are listed in Table 1. The measured particle size distribution is provided in Fig. 1.

2.1.2. Fly ash (FA)

Class F fly ash according to ASTM C 618-08 [18] with an average particle diameter of 80 μm. The fly ash was obtained from the José Lopez Portillo carboelectric plant located in the state of Coahuila in the Northeast of México, which has a monthly average production of 141,000 metric tons, from which only 4700 metric tons are currently used. Physical properties and chemical composition are listed in Table 1 and the measured particle size distribution is given in Fig. 1. It should be noted that the particle sizes of this particular fly ash may improve particle packing by efficiently filling in the particle sizes between those of the cement and the fine aggregate.

Table 1
Physical properties and chemical analysis of cement and FA.

Physical properties		Cement	FA
Fineness – passing 45 μm,%		97.2	Not measured
Blaine, m^2/kg		482	Not measured
Specific gravity		3.13	2.38
Compressive strength of			ASTM C311
50-mm cubes, MPa (psi)	3 d	20.5 (2 973)	21.6 (3 132)
	7 d	24.1 (3 495)	23.7 (3 437)
	28 d	35.6 (5 162)	30.5 (4 423)
Chemical composition,% by mass			
SiO_2		19.6	62.3
Al_2O_3		4.5	22.0
Fe_2O_3		4.0	6.0
CaO		58.8	3.4
MgO		1.6	1.0
SO_3		3.0	0.2
Na_2O		0.4	0.8
K_2O		1.4	1.6
Loss on ignition		6.4	1.6

FA = Fly ash.

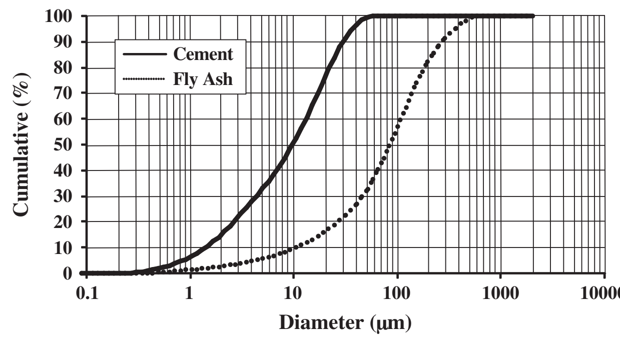

Fig. 1. Measured particle size distributions for the cement and fly ash employed in the current study.

2.1.3. Fine aggregate

Locally available crushed limestone sand that meets the grading requirements of the ASTM C 33-07 specification [18]. It has a specific gravity of 2.65, a fineness modulus of 3.1, and water absorption of 1.25%.

2.1.4. Coarse aggregate

Locally available crushed limestone coarse aggregate with a maximum size of 19 mm that meets the grading requirements of the ASTM C 33-07 # 67 specification [18]. The specific gravity and the water absorption were 2.72% and 0.44%, respectively. For both aggregates, properties were determined according to standard procedures described in the corresponding ASTM test methods.

2.1.5. Water

Potable water that meets the requirements of ASTM C 1602-06 [18] was used to mix the concrete and cure the specimens. Mixtures were prepared at three different *w/b* of: (A) 0.5, (B) 0.55, and (C) 0.60.

2.1.6. Superplasticizer (SP)

A polycarboxylate based high-range water reducer or superplasticizer conforming to ASTM C 494-92 Class A and Class F [18] in aqueous form was used to reduce the paste content and improve workability. The SP has a specific gravity of 1.03 and a total solids content of 22% by mass.

2.2. Test methods

For the concrete mixture production, the general procedure described in ASTM C 192-07 was followed [18]. The proportions were optimized by homogeneity and consistency, and were established for the reference mixtures through trial batches for a target slump of 200 mm ± 20 mm. The following steps describe the procedure that was used to establish the proportions for all the mixtures in a single series:

- The optimization of the SP dosage was established through the saturation point criteria using the Marsh cone test [19,20].
- The initial combination of coarse and fine aggregate was established by the combination that gave the minimum voids content or maximum loose unit weight. At the first trial batch, if necessary, the paste content and aggregates combination were modified to obtain an appropriate homogeneity and the target slump. In order to establish the optimized proportions for the reference mixture, SP was added just before the last mixing period.

Table 2
Mixture proportions for series A, B and C.

Series	FA, (%)	W/(C + FA) (w/b)	Water, (kg/m^3)	Cement, (kg/m^3)	FA, (kg/m^3)	SSD aggregates, (kg/m^3)		SP[a] (L/m^3)
						Coarse	Fine	
A	0	0.50	211	425	–	734	990	2.2
	15			361	64	728	972	
	30			298	128	741	963	
	45			234	191	752	934	
	60			170	255	743	923	
	75			106	319	735	914	
B	0	0.55	222	405	–	774	959	1.3
	15			344	61	772	941	
	30			284	122	771	940	
	45			223	182	770	904	
	60			162	243	767	884	
	75			101	304	770	904	
C	0	0.60	222	370	–	785	973	0.6
	15			315	56	785	928	
	30			259	111	787	921	
	45			204	167	793	917	
	60			148	222	785	883	
	75			93	278	794	886	

FA = Fly ash, C = Cement, SSD = Saturated surface dry.
[a] Superplasticizer.

Table 3
Fresh concrete properties for series A, B, and C.

Series	FA (%)	Unit weight (kg/m^3)	Slump (mm)	Air (%)	Setting times (h:min)	
					Initial	Final
A	0	2350	220	2.5	4:45	6:13
	15	2333	250	1.6	4:53	6:24
	30	2332	250	1.9	5:02	7:08
	45	2308	260	2.0	5:35	7:41
	60	2296	250	2.3	6:30	8:25
	75	2257	230	3.2	7:05	9:30
B	0	2353	220	1.7	5:10	6:18
	15	2320	230	1.8	5:15	6:35
	30	2314	235	1.9	5:30	7:15
	45	2296	245	2.0	6:00	7:50
	60	2271	225	2.2	6:50	9:40
	75	2260	255	1.9	8:10	10:27
C	0	2344	210	2.0	5:15	6:23
	15	2331	235	2.0	5:25	7:00
	30	2316	240	1.8	6:02	8:00
	45	2305	245	1.5	6:30	8:55
	60	2292	245	2.0	8:25	11:32
	75	2288	250	1.5	8:45	12:55

FA = Fly ash.

- Then, the cement was substituted by FA in mass. The mixtures were identified by a three character code in which the first character indicates the series (*w*/*b*) and the second and third characters the FA substitution; A0, B0 and C0 were used to identify the reference mixtures in each series. The resulting difference in absolute volume originating from the differences in specific gravity between the cement and the FA was deducted from the fine aggregate to maintain the design yield. Table 2 presents the mixture proportions for the 18 concrete mixtures studied.

The characterization of all the concrete mixtures in the fresh state consisted of the determination of slump (ASTM C 143-08), unit weight (ASTM C 138-08), air content (ASTM C 231-08b, Class B), times of setting (ASTM C 403-08) and slump loss; this last property was monitored through the procedure described in ASTM C 143-08 [18]. The results for these properties are presented in Table 3. The precisions of the various ASTM test methods are as follows: single laboratory standard deviations of 10 mm for slumps of 160 mm and higher, 10.4 kg/m^3 for unit weight, 0.28% for air content, and 3.5 min and 4.4 min for initial and final setting times, respectively.

Heat of hydration was measured under isothermal conditions (isothermal calorimetry) during the course of 7 d on pre-mixed (as opposed to being mixed in situ in the calorimeter cells) sealed paste samples (cement, FA, water, and SP) with a mass between 4.2 g and 4.6 g [15,16]. For a subset of the eighteen pastes, semi-adiabatic calorimetry was conducted during the course of 2 d, for single specimens with a mass of about 250 g [15,16]. Previous results have indicated a standard deviation of 1.4 °C in the maximum specimen temperature achieved during a 3 d test [15].

Table 4
Compressive strength development on concrete specimens cured under standard conditions for series A, B, and C.

Series (%)	FA	Compressive strength (MPa) Age (d)						
		1	3	7	14	21	28	56
A	0	15.0	26.9	35.1	38.8	41.4	43.5	44.9
	15	15.6	24.8	27.6	29.3	30.5	36.8	40.1
	30	11.7	21.2	24.1	26.2	30.0	32.5	35.5
	45	6.4	14.0	18.6	19.2	23.7	23.8	26.8
	60	4.4	7.2	10.1	11.9	13.8	16.8	17.2
	75	1.1	3.2	4.1	5.4	6.4	7.0	9.9
B	0	19.4	26.8	30.4	30.9	33.7	37.0	38.4
	15	12.5	24.3	27.9	29.8	30.2	32.4	35.7
	30	10.6	18.7	20.7	22.8	24.0	25.4	27.0
	45	5.7	11.4	12.9	18.0	19.0	19.6	21.5
	60	3.8	5.9	7.8	9.5	11.6	13.1	14.0
	75	–	2.6	3.4	4.3	5.5	6.0	7.9
C	0	16.1	22.5	26.3	31.9	29.0	32.1	34.5
	15	8.8	17.4	19.8	24.9	26.9	27.5	29.3
	30	7.5	11.6	16.7	18.5	20.1	21.3	24.5
	45	3.1	6.9	9.1	10.7	12.1	13.4	16.6
	60	–	4.6	6.7	7.9	9.4	9.8	12.6
	75	–	–	2.6	3.8	4.2	5.0	6.7

FA = Fly ash.

The characterization of the concrete properties in its hardened state consisted of the determination of the compressive strength (ASTM C 39-05), the chord static modulus of elasticity (ASTM C 469-02), the modulus of rupture using a simple beam with three point loading (ASTM C 78-08) and the splitting tensile strength (ASTM C 496-04) on standard cured specimens [18]. The precisions of these ASTM test methods are as follows: single laboratory (operator) coefficients of variation of 3.2% for compressive strength, 4.25% for static modulus of elasticity, 5.7% for modulus of rupture, and 5% for splitting tensile strength. Compressive strength results at the ages of (1, 3, 7, 14, 21, 28, and 56) d are presented in Table 4. Fig. 13 presents the results of modulus of rupture and splitting tensile strength and Fig. 14 those for static modulus of elasticity. For compressive and splitting tensile strength determinations, standard cylindrical specimens with a diameter of 100 mm and a length of 200 mm were used, while for static modulus of elasticity standard cylindrical specimens with a diameter of 150 mm and a length of 300 mm were used.

The methodology for the optimization of the concrete mixtures is provided in this study as a systematic procedure that should be equally applicable to other sets of materials.

3. Results and discussion

3.1. Concrete fresh state properties

3.1.1. Slump

At the end of each mixing program, the slump was determined for each mixture and the results of these tests are reported in

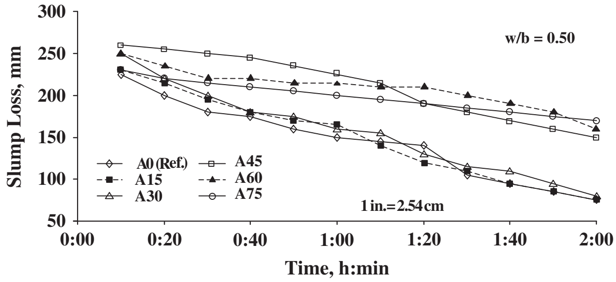

Fig. 2. Slump loss of series A concretes.

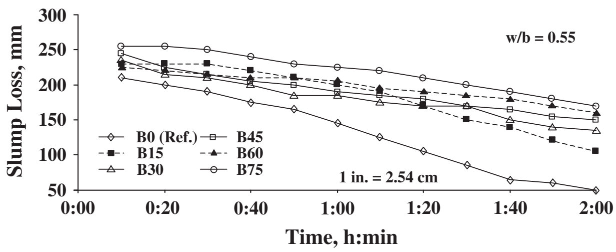

Fig. 3. Slump loss of series B concretes.

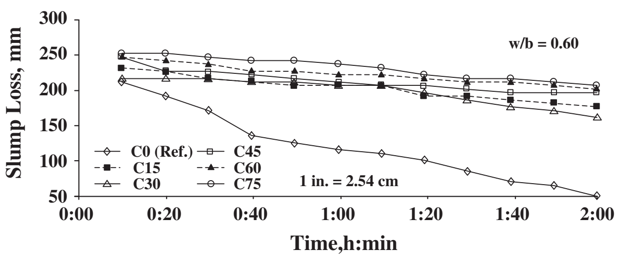

Fig. 4. Slump loss of series C concretes.

Table 3. From the results, it can be observed that for the three series, the increase in FA substitution generally leads to higher slump values in the range of 10–40 mm.

3.1.2. Slump loss

Fig. 2 presents the slump loss for concretes of series A; this property was monitored for 2 h, and during this period, mixtures A0, A15, and A30 presented a similar average slump loss trend of 80 mm per hour. In the same time period, mixtures A45, A60 and A75 presented a similar average slump loss trend of 43 mm per hour. In concretes of series A, FA substitutions of 45%, 60%, and 75% improved the slump loss by 50% compared with mixtures A0, A15, and A30. Figs. 3 and 4 illustrate the slump loss of concretes for series B and C. Reference mixtures in both series presented a slump loss of 80 mm per hour, similar to that of the A reference mixture. Concretes with FA in series B presented slump loss rates between 60 mm per hour and 40 mm per hour and in series C between 40 mm per hour and 23 mm per hour. Figs. 2–4 also illustrate that as the FA substitution increases, the slump loss decreases.

3.1.3. Setting times

It is well known that FA substitutions may produce retardation in the setting times of concretes; as was expected, the higher retardation times were obtained for the concretes with the higher FA substitutions. For this specific FA, we can observe from Table 3 that

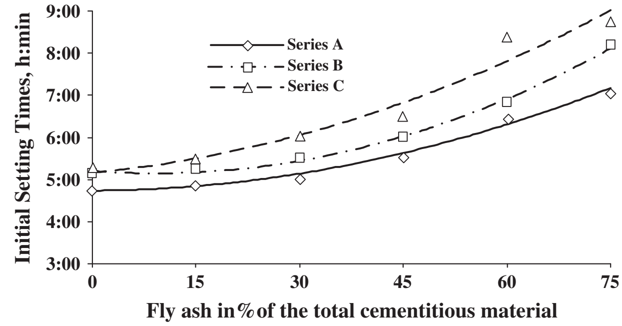

Fig. 5. Initial setting times vs. fly ash content in% of the total cementitious material.

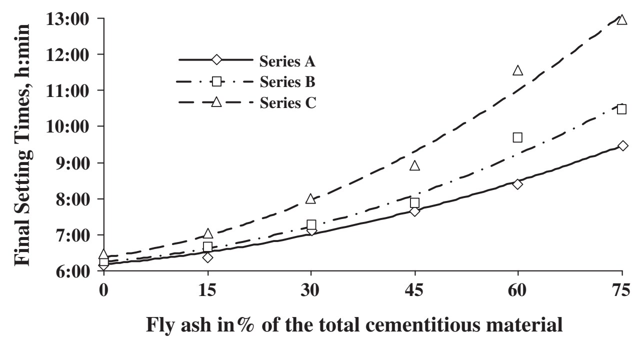

Fig. 6. Final setting times vs. fly ash content in% of the total cementitious material.

the studied concretes presented retardations of the initial setting times of 2:20, 2:05, and 3:30 (h:min) for series A, B, and C respectively, and for final setting times, retardations of 3:17, 4:09, and 6:32 (h:min), for the 75% substitution of cement by fly ash. Figs. 5 and 6 illustrate respectively the influence of the different FA substitutions on the initial and final setting times; these graphs clearly illustrate that as the *w/b* and FA substitution increases, the times of setting increases.

3.1.4. Calorimetry

Isothermal calorimetry results are presented in Fig. 7 and illustrate that during the first 24 h, the reference and fly ash pastes hydrate in a similar fashion, indicating that while setting times are delayed, the fly ash is not retarding the hydration of the cement. It is simply that there is less cement to percolate the solid backbone and cause setting when FA is incorporated into the mixture and/or *w/b* is increased. Bentz et al. have attributed this disparity between calorimetry and setting to fundamental differences between a physical process such as setting and heat release which is purely a quantification of chemical reaction [16].

The heat flow can be further quantified by plotting the cumulative heat release to see how much the fly ash is contributing to the overall reactions; Fig. 8 shows the heat flow development for the three series over the course of the 7 d test on a per gram of cement basis for the three *w/b*, indicating in each case a small contribution of the pozzolanic reactions, but almost no contribution during the first 24 h (where the curves basically overlap one another). These results illustrate that fly ash additions, through their dilution effect, should significantly reduce the peak temperature achieved in concrete structures. Semi-adiabatic calorimetry executed on a subset of the pastes has verified that this is indeed the case (Fig. 9). For large structures, this reduction in peak temperature should significantly reduce the propensity for early-age cracking due to thermal issues.

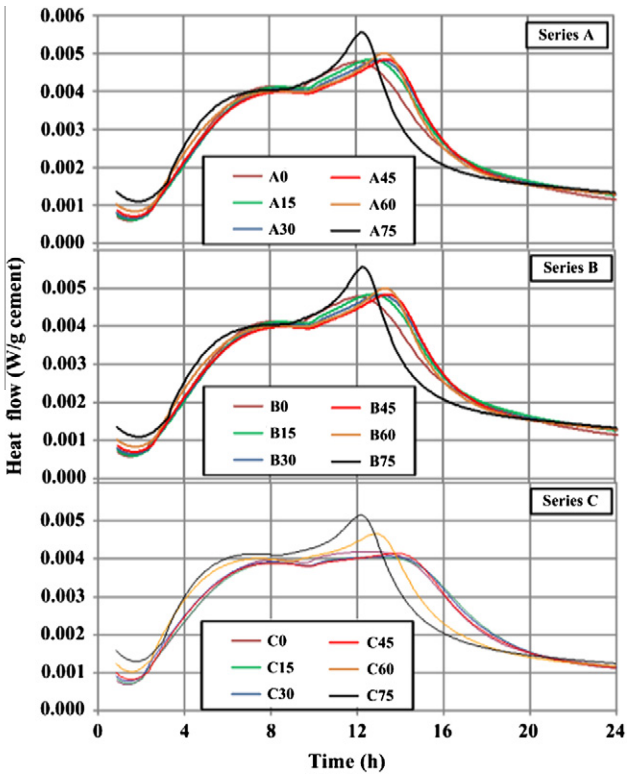

Fig. 7. Early-age heat release rates from isothermal calorimetry for the cement pastes reproduced according to the *w/b* of series A, B and C. Replicates for A0 (not shown) overlap one another and cannot be distinguished as individual curves.

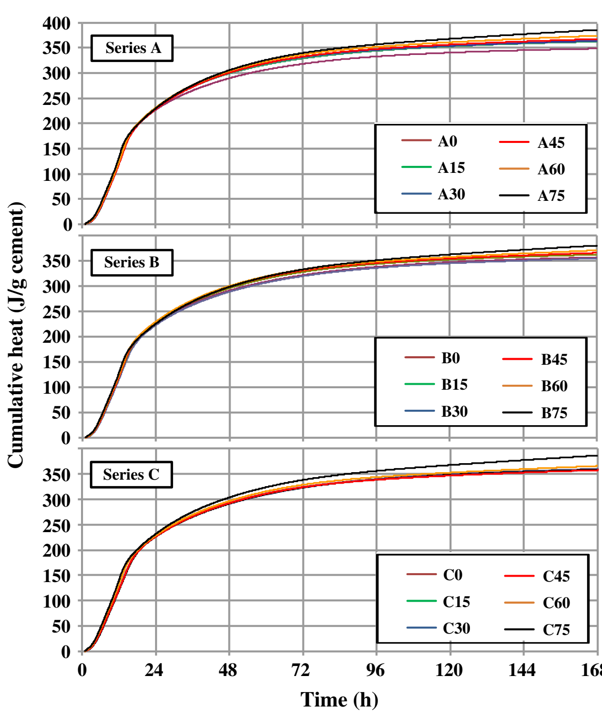

Fig. 8. Cumulative heat release for 7 d from isothermal calorimetry for the cement pastes reproduced according to the *w/b* of series A, B and C.

3.2. Hardened state properties

3.2.1. Compressive strength development

Table 4 presents the compressive strength development for all the concretes considered in the study; at ages up to 3 d, some values are not included because the concrete was too weak to be tested at those ages. For ages of (1, 3, 7, 14, 21, 28, and 56) d it can be observed from this table that as the FA content increases the compressive strength decreases, and also as a result of the pozzolanic activity of the FA, that as the age increases, the difference between the compressive strength of the concretes with FA and the reference concretes tends to decrease. In practice, usually the compressive strength of the concrete is specified at the age of 28 d. Figs. 10 and 11 are intended to illustrate the wide range of potential compressive strengths for concretes made with this particular cement and FA, with a wide range of substitutions. At an age of 28 d, Fig. 10 illustrates that for concretes with *w/b* of 0.50, 0.55, and 0.6 and FA substitutions between 0% and 75%, strengths from 45.0 MPa down to 5.0 MPa can be obtained. In this regard, Fig. 11 illustrates that at an age of 56 d the compressive strengths ranged from 47.0 MPa down to 7.0 MPa for the studied concretes.

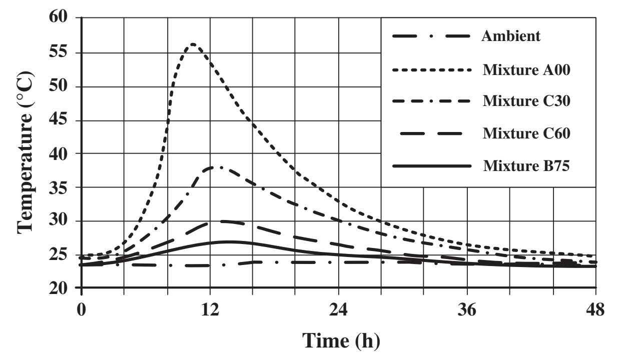

Fig. 9. Semi-adiabatic calorimetry curves for four of the pastes examined in this study.

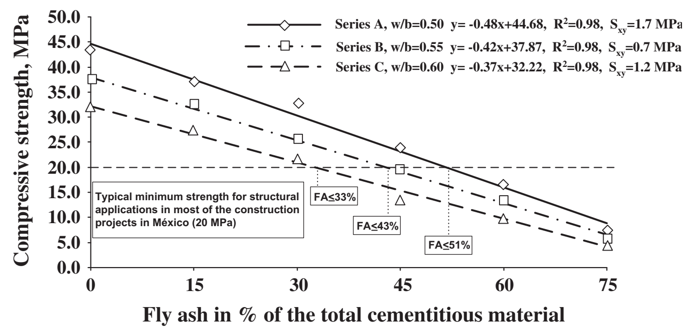

Fig. 10. Compressive strength at 28 d vs. fly ash content in% of the total cementitious material. S_{yx} indicates standard error from linear regression.

In Figs. 10 and 11, one can observe that there was a good linear relationship between strength and FA replacement level for these concretes, with correlation coefficients (R^2) of 0.98 in all cases, and that the slopes of these linear relationships were the same at both ages, being −0.48, −0.42, and −0.37 for series A, B, and C, respectively. Computed standard errors (S_{yx}) were between 0.7 MPa and 1.7 MPa at 28 d and between 0.8 MPa and 2.1 MPa at 56 d.

3.2.2. Modulus of rupture

Fig. 13 presents the results of modulus of rupture at an age of 28 d, measured on standard cured specimens for series A, B and C; these results were used to establish a linear fit between modulus of rupture and compressive strength that had a correlation coefficient (R^2) of 0.93 and a standard error of 0.32 MPa, as shown in Fig. 12. This figure also contains the curve produced by an equa-

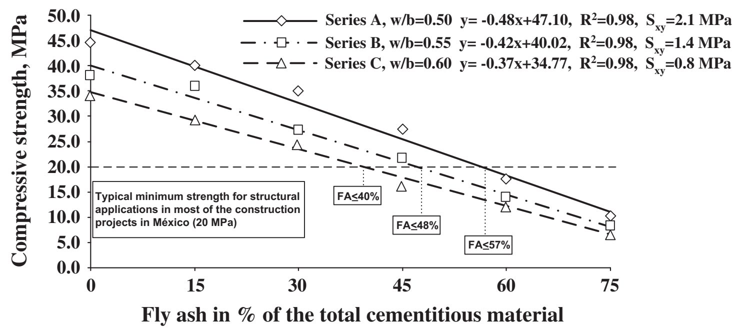

Fig. 11. Compressive strength at 56 d vs. fly ash content in% of the total cementitious material. S_{yx} indicates standard error from linear regression.

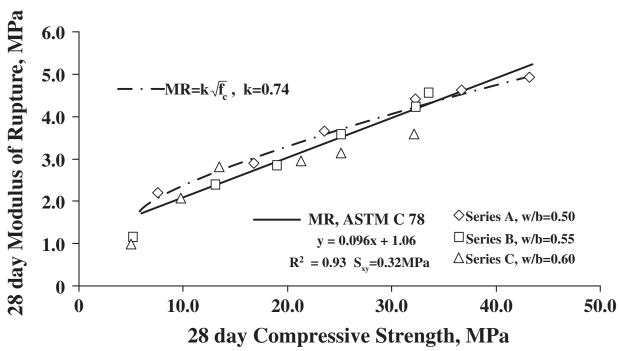

Fig. 12. Modulus of rupture vs. compressive strength at 28 d for series A, B, and C, and curve for the equation MR = $k\ \sqrt{fc}$ with k = 0.74. S_{xy} indicates standard error from linear regression.

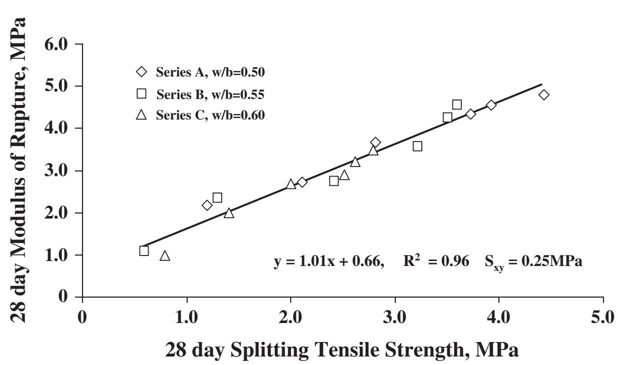

Fig. 13. Splitting tensile strength vs. modulus of rupture at 28 d for series A, B, and C. S_{yx} indicates standard error from linear regression.

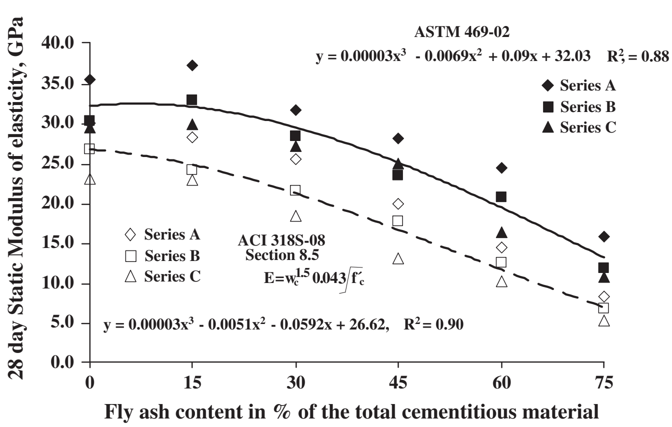

Fig. 14. Static modulus of elasticity (E) at 28 d vs. fly ash content in% of the total cementitious material.

tion that has been previously developed to describe the relationship between flexural strength and compressive strength, namely MR = k (fc)$^{1/2}$ with a value of k = 0.74 recommended in the literature [21,22]. This expression also provides a reasonable fit to the concrete data produced in this study.

3.2.3. Splitting tensile strength

Fig. 13 presents the results of splitting tensile strength at an age of 28 d, measured on standard cured specimens for series A, B and C; these results were used to establish a linear relationship between modulus of rupture and splitting tensile strength that had a correlation coefficient (R^2) of 0.96 and a standard error (S_{yx}) of 0.25 MPa, as shown in Fig. 13.

3.2.4. Modulus of elasticity

Fig. 14 presents the results of static modulus of elasticity at an age of 28 d, measured on standard cured specimens. For series A, B and C, concretes with an FA substitution of 15% produced E values between 1% and 8% higher than the corresponding reference concretes. With the information contained in this table, a cubic regression of the form $y = ax^3 + bx^2 + cx + d$ was established and appears in Fig. 14. This figure presents two curves that illustrate the relations between the modulus of elasticity and the FA contents in percentage of the total cementitious material for the results obtained by the ASTM procedure, and for the corresponding values obtained with the equation provided by ACI 318-08 [23], section 8.5. For FA substitutions between 15% and 75%, E diminished at a rate of 1.0% and 0.7%, respectively for the ASTM procedure and the ACI equation. The ACI equation produces conservative estimates compared with the values obtained through the ASTM procedure. For the whole range of FA substitutions, the differences in modulus of elasticity between the two cubic regressions obtained by the two methods were between 5.4 GPa and 8.5 GPa.

In summary, Figs. 10–14 represent useful tools for practical applications of these concretes in a wide variety of projects, as they can be used to initially select w/b and fly ash substitution at the beginning of mixture optimization for many project specific properties such as: 28 and 56 d compressive strength (Figs. 10 and 11) and the 28 d modulus of rupture (Fig. 12) or the 28 d splitting tensile strength (Fig. 13) in the case of pavement design. In structural analysis, Fig. 14 provides information to evaluate the effect of this fly ash in the determination of the potential structural stiffness. These figures are also useful tools to encourage potential users to apply an improved sustainability approach to their concrete construction practices.

4. Conclusions

For the studied materials and proportions, based on the aforementioned results and discussion, the following conclusions can be drawn:

1. For these particular cement-FA combinations, the observed correlations represent a practical tool to select initial proportions for target properties including compressive strength, modulus of rupture, splitting tensile strength, and static modulus of elasticity.
2. For the range of FA substitutions studied in this work, the slump was increased by 10–40 mm.
3. The incorporation of FA produced an average slump loss reduction of 50% in comparison to the characteristic slump loss rate obtained for all the reference mixtures.
4. As the *w/b* and FA substitution increases, the setting times increase. A maximum retardation in final set of 6:32 (h:min) was obtained for *w/b* of 0.6 and FA substitution of 75%.
5. Fly ash additions do not retard the hydration reactions of the cement but through dilution do significantly reduce the heat release-peak temperature at early ages under semi-adiabatic conditions.
6. FA substitutions of 15% increased the modulus of elasticity by up to 8% in comparison with the reference concrete.
7. For the range of FA substitutions studied in this work, the ACI equation to calculate the modulus of elasticity produces conservative estimates when compared with the values obtained through the ASTM procedure. For the entire set of FA substitutions, the average values obtained by the ACI equation were between 17% and 51% lower than the average values obtained by the ASTM procedure. This reduction increases as the FA substitution rate increases.

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