OPUNTIA FICUS INDICA MUCILAGE (OFIM) CA ADITIV DE ÎMBUNĂTĂȚIRE A PROCESELOR DE ÎNTĂRIRE ALE BETONULUI AUTOCOMPACTANT

OPUNTIA FICUS INDICA MUCILAGE (OFIM) AS INTERNAL CURING ENHANCER IN SELF CONSOLIDATING CONCRETE

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Two viable technologies that may be used to reduce shrinkage at early ages in high-performance concrete, such as self-consolidating concrete, include internal curing and shrinkage- reducing admixtures. By combining an internal source of curing water and an admixture that decreases the surface tension of pore solution, an improved overall shrinkage reduction can be achieved. Based on the evidence resulting from this synergy, this investigation focuses on the effect of Opuntia Ficus Indica Mucilage (OFIM) as a reducer of the pore solution surface tension, to establish its effectiveness in shrinkage mitigation, in comparison with the effect of conventional internal curing with water and that of a commercial shrinkage-reducing admixture. Autogenous strain and drying shrinkage deformations were determined according to ASTM C 1698 and C 157 test methods, respectively. Result show that the incorporation of OFIM does not have a significant effect on mechanical and elastic properties of the concrete, and could lead to substantial benefits in terms of shrinkage mitigation, particularly at early ages.

***Keywords**: self-consolidating concrete, internal curing, autogenous shrinkage, drying shrinkage, shrinkage-reducing admixture, opuntia ficus indica mucilage.*

1. Introduction

Currently, there is no doubt that self-consolidating concrete (SCC) has many advantages over conventional concrete, and has increased interest in research and industrial applications [1]. Due to their high flowability and instability/inhomogeneity of ingredients, these concretes require a tight optimization of their rheological properties in the fresh state to meet the following target characteristics: increase in productivity, ease of placement, improved appearance in hardened stage with high quality aesthetics, labor cost reduction, great flexibility at pouring and pumping, reduced noise pollution on the work site, the elimination of vibration and an increase in the service life of formworks [2]. In these mixtures, the use of high amounts of powder along with a reduced water/cement ratio, increase the concrete's volume instability [3]. These factors increase the risk of shrinkage cracking in self-consolidating concrete and have a direct effect on the durability of the material. The most common solution to reduce volume instability at early ages is to prevent drying by the appropriate handling and curing of concrete from very early ages after casting [4]. Typically, in its formulation, an SCC have a higher powder content and in many cases fly ash is one of the preferred sources of powder that additionally to participate in the system as a cementitious material and filler, it improves rheology and drying shrinkage[5].

Internal curing has proven to be effective in shrinkage reduction. This technology provides a source of water through the incorporation of uniformly distributed reservoirs within the cement-based materials that will satisfy the water demand produced by self-desiccation. Such reservoirs could be provided by water saturated lightweight aggregates [6, 7].

Another alternative for shrinkage reduction is the use of shrinkage-reducing admixtures (SRAs), which are typically organic chemical compounds that substantially reduce the surface tension of the pore solution [8, 9], increase its viscosity [10] and reduce early-age drying rates in cement-based materials [8, 9].

Among the natural substances that can also reduce, surface tension of the pore solutions is Opuntia Ficus Indica Mucilage (OFIM), a material that can absorb water and reduce surface tension in aqueous solutions [11]. Mucilage is a complex carbohydrate that constitutes a hydrocolloid, presenting a thickening power that is still being studied [12-16]. Back in 1998, Chandra et al, indicated that the incorporation of cactus extract could have useful results in Portland cement mortar [17]. As an additive, OFIM has potential uses for several industrial products, including the food industry. Introducing OFIM in industrial applications

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brings additional advantages related to its cultivation like erosion control and partner-economic development of sectors that are traditionally depressed. These characteristics are strong arguments to increase the use of this species [18]. However to date, the practical application of OFIM is mainly based on empirical knowledge, and just recently there have been a few formal experimental works that explored the effect of OFIM in some mechanical and durability properties of concrete [19, 20]

As OFIM solutions increase viscosity and decrease surface tension, it is seen as a potential shrinkage reducer in cement-based materials, and as a surface tension modifier of the solution for internal curing of concretes, because when internal curing is implemented through a saturated-surface-dry lightweight sand, the greater the viscosity of the solution for internal curing the lower the release rate from the aggregates. Analyzing the effect of surface tension in Equation 1, suggested by the Laplace model [5], where (r) is the radius of the pore, we can see that when surface tension (σ) is reduced, suction pressure (s) is also reduced. This is one of the main mechanisms related to shrinkage mitigation in cement-based materials.

$$s = \frac{2\sigma}{r} \qquad \text{Eq. 1}$$

Cracking caused by shrinkage may affect the durability of a concrete structure, and is often a major concern in high performance concrete. In this regard, it is necessary to study the technical feasibility of alternative technologies that may reduce autogenous and drying shrinkage. The aim of this research work was to establish the potential of OFIM extracted by boiling in an aqueous solution and without adding any conserving agents, to reduce shrinkage in self-consolidating concrete. OFIM was added to self-consolidating concrete as an internal curing enhancer and as an admixture. The effectiveness of this product was compared against existing technologies, such as the conventional internal water curing, and a commercial shrinkage-reducing admixture (SRA). For this purpose, an experimental program was designed and oriented to evaluate autogenous and drying shrinkage, as well as to characterize typical fresh and hardened stage properties of concrete.

2. Experimental

2.1. Materials

2.1.1 Cement

Ordinary Portland cement (OPC) that meets the requirements of the Mexican standard NMX-C-414 and the ASTM C-150 for a OPC 40 and for a Type I cement, respectively, was employed. The density of the cement was 3080 kg/m^3 with a Bogue potential mineralogical composition by mass of C_3S= 57%; C_2S= 15%; C_3A= 9% and C_4AF= 6%.

2.1.2. Aggregates

Crushed limestone aggregates from the northeast of Mexico with density and water absorption (by dry mass) of 2670 kg/m^3 and 1.3 %, respectively, for the coarse aggregate, and of 2640 kg/m^3 and 2.3 % for the fine aggregate. The lightweight sand used as a medium to introduce the internal curing solution into the concrete was a pumice sand from the central region of Mexico, with density and water absorption of 1570 kg/m^3 and 31.8 %, respectively.

2.1.3. High-range water reducing admixture (HRWRA)

The HRWRA used as a superplasticizer admixture (SP) was a polycarboxylate-ether based high-range water reducer, manufactured by BASF[1] and commercialized in Mexico as GLENIUM 3030NS, in which 30% by mass of the solution constituted the dispersing active agent.

2.1.4. Shrinkage-reducing admixture (SRA)

BASF[1] MASTERLIFE SRA20 was used; this admixture was diluted in distilled water to produce an aqueous solution of 1:1 (by mass), and introduced to the concrete directly to the mixture or within the saturated lightweight aggregate as an internal curing enhancer.

2.1.5. Opuntia Ficus Indica Mucilage (OFIM)

For the extraction of OFIM, the cladodes were cut into sections of 1 cm wide by 1 cm long, and boiled for five minutes in distilled water (DW) at an average temperature of 94 °C. After the boiling process, when the solution reaches the ambient temperature, the following mass proportions of distilled water-OFIM solution were prepared: 1:1, 1:3.5 and 1:5.

2.2 Methods, Concrete identification and proportions

2.2.1. Surface Tension of OFIM

Twenty-four hours after extraction, surface tension was determined through the ring method in a KSV tensiometer model Sigma 701, viscosity and density of OFIM solutions were determined in a Brookfield viscometer model DV II at 5 rpm with a RV #1 spindle according to ASTM D 2196. The results of these tests are reported in Table 1 and Figure 1. Based on these results, it was decided

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[1] Certain commercial entities, equipment, or materials may be identified in this document to describe an experimental procedure or concept adequately. Such identification is not intended to imply recommendation or endorsement by the Universidad Autónoma de Nuevo León, nor is it intended to imply that the entities, materials, or equipment are necessarily the best available for the purpose.

Table 1

Properties of the distilled water: OFIM solutions.

Parameter	Tap water	SRA–water 1:1	Mucilage 1:1	Mucilage 1:3.5	Mucilage 1:5
Viscosity, cP	3.0	15.0	10.0	5.0	5.0
Surface tension mN/m	68.49	30.60	49.24	47.64	49.95
Density, kg/m^3	994.7	1012.9	999.2	998.6	995.5

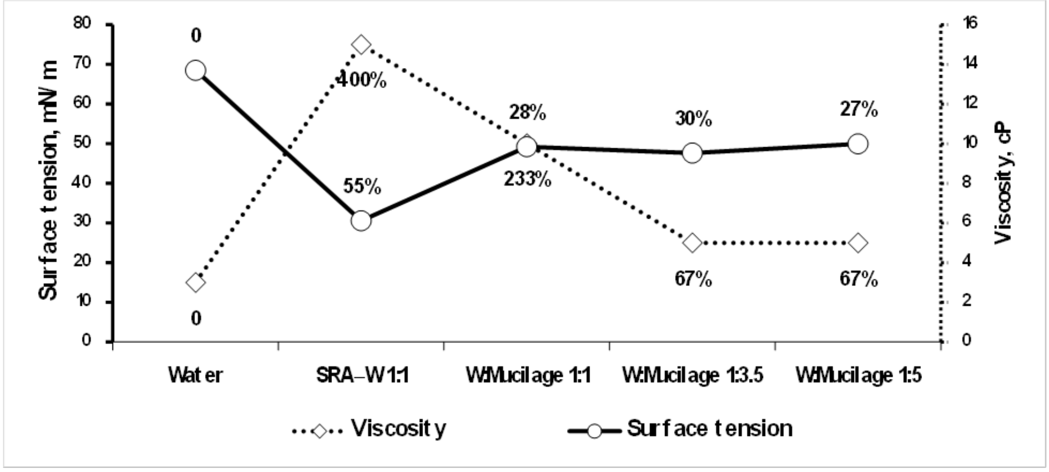


Fig. 1 - Surface tension and viscosity for tap water and DW-SRA, DW-OFIM aqueous solutions.

to use the 1:3.5 solution, since it had the lowest measured surface tension.

Figure 1 clearly exhibit that OFIM reduces Surface tension and increases the viscosity. The same trend but with much higher impact is observed for the DW-SRA solution by increasing 4 times the viscosity and by decreasing 55 % the surface tension.

2.2.2. Optimization of HRWRA dosage

For a fixed water/cementitious material ratio (*w/cm*) of 0.4 by mass, the optimum dosage of HRWRA was determined based on the saturation point criterion through the Marsh cone. The saturation point of the HRWRA was established based on the curve obtained for a paste volume of 750 ml. Although saturation point provides a dosage very close to that needed to obtain the target SCC consistency, to establish the definitive content of HRWRA, it was necessary to carry out trial SCC mixtures to attain the target flow extensibility (700 ± 25 mm) and extended loss of consistency.

2.2.3. Optimization of the granular skeleton

The proportion of fine to coarse aggregate was selected based on the maximum compactness criteria. The selected sand:gravel or fine:coarse proportion was 60:40 by mass because it produces the highest volumetric (unit) mass and the lowest voids volume, an important aspect from an economic point of view and with regard to sustainability, since such a combination will require the lowest volume of cement paste.

2.2.4. Internal curing

To introduce internal curing within the concrete, a lightweight pumice sand with a maximum size of 5 mm was used. Based on previous studies, the pumice was used in substitution of 20% by volume of the normal weight sand, because this substitution does not affect compressive strength and modulus of elasticity [21]. The amount of solution required to theoretically ensure saturation was calculated based on the absorption-desorption capacity of pumice sand over a period of 24 h. For this purpose, 25.6 liters of solution were required for the concrete mixture.

The SRA was used in liquid solution at a proportion of 1:1 by mass. For all internal curing modes, the saturation period of the pumice sand was constant and fixed at 24 h.

2.2.5. Mixtures and proportions

The six studied SCC mixtures are described in Table 2, and the corresponding proportions with the materials in dry condition and without the absorption water are shown in Table 3. All the mixtures in Table 3 have a w/c ratio of 0.4, to maintain this parameter constant in all the mixtures, the amount of SP, SRA-water solution and OFIM-water solution were considered part of the reaction water, so in total all the mixtures had a reaction water content of ≈ 220 l/m^3.

2.2.6.Fresh stage characterization of SCC

Fresh stage characterization of SCC included the properties reported in Table 4. To establish final proportions, a target slump flow of 700 ± 25 mm was fixed. With exception of the J Ring test described in ASTM C-1621-09, the procedures of all the other tests in Table 4 are described in the EFNARC guidelines for self-consolidating concrete [22].

Table 2

Mixture description.

Mix number	Nomenclature	Description
1	R	Reference mixture.
2	IC	Conventional internal water cured mixture
3	ICSR	Internally cured mixture. Pumice saturated with SRA aqueous solution.
4	IC-MSR	Conventional internal water curing and SRA aqueous solution dosed in the mixture.
5	ICNA	Internally cured mixture. Pumice saturated with OFIM aqueous solution.
6	IC-MNA	Conventional internal water curing and OFIM aqueous solution dosed in the mixture.

Table 3

SCC Mixture proportions.

Ingredients	Materials in kg/m^3					
	R	IC	ICSR	IC-MSR	ICNA	IC-MNA
OPC	544	544	544	544	544	544
Water	213	213	213	187	213	187
Limestone sand (dry)	900	720	720	720	720	720
Limestone gravel (dry)	600	600	600	600	600	600
Pumice sand (dry)	---	83.1	83.1	83.1	83.1	83.1
SP	7.36	7.36	7.36	7.36	7.36	7.36
SRA-water solution	---	---	---	25.57	---	---
OFIM-water solution	---	---	---	---	---	25.57

Table 4

Reference values for SCC properties.

SCC parameter	Test method	Target values
Deformability and flow rate (filling ability at non restricted flow)	Slump flow T- 500	700 ± 25 mm 2 to 5 s
Passing ability (Passing ability through narrow openings, confined flow, restricted flow, dynamic stability)	V-funnel (65 x 75 mm opening) L-box, h_1/h_2 J-ring	< 8 s > 0.8 < 15 mm
Filling ability (Filling ability + passing ability + restricted deformability)	L-box, h_1/h_2 J-ring	> 0.8 > 0.8
Static stability (segregation resistance)	Sieving segregation resistance	≤ 15 %

2.2.7.Compressive strength and static modulus of elasticity

To determine compressive strength (ASTM C 39-05) and static modulus of elasticity (ASTM C 469-10), cylindrical specimens of 100 mm in diameter by 200 mm in height were poured without any compaction or vibration. According to ASTM C192-07, all the specimens were standard cured for 7 days under controlled conditions of temperature (T= 23 ± 1.7 °C) and relative humidity (RH= ≥ 95%). After standard curing, all specimens were then kept under controlled laboratory conditions at a relative humidity of 55 ± 5% and a temperature of 23 ± 1.7 °C.

2.2.8. Shrinkage measurements

To evaluate the effect on volume stability, autogenous deformation and drying shrinkage deformations were evaluated through the procedures described in ASTM C-1698-09 and ASTM C-157-08, respectively. Regarding the measurement of autogenous deformation, the tests were carried out using the wet-sieved mortar fraction (ASTM C 172-10) of each mixture of concrete.

3. Results and Discussion

The three modes to administer the internal curing (IC, ICSR and ICNA) that were studied in this work, and the synergy of conventional internal curing with either of the two admixtures studied (IC-MSR and IC-MNA) produced different hydration kinetics and impacts on the results as discussed below.

3.1. Fresh stage

Previous works stated that the presence of hydrophobic and hydrophilic terminations within the chains of polysaccharides and proteins contained in OFIM reduces friction and increases the fluidity in cement-based compounds [17]. When the aqueous solution of OFIM was added directly to the SCC mixture, deformability, flow rate and flowability were increased without disturbing static stability. Results of the different fresh stage properties considered in this work are presented in Table 5.

From the results reported in Table 5, it has been identified that the unique fresh stage parameters that exhibit variations attributed to the implementation of one of the technologies evaluated in this work, were the unit weight and the viscosity/static stability of SCC quantified trough the V-funnel results. Reductions in unit weight of mixtures with IC are attributed to the lower density of the pumice. The increase in V-funnel results from 3.5 to 4.4-5.6 s, exhibit a slightly more viscous cementitious matrix with negligible effect in the static stability of the concrete (absence of segregation).

3.2. Compressive strength

Comparing compressive strength results presented in Figure 2, it can be observed that in comparison to R mixture, the internal curing (IC mixture) presents a similar strength at 7 days and an average increase of 2.5 % at 28 days and 91 days. These results are the average of three individual results that satisfy the acceptable variation range for three tests of 10.6 %, established in the precision and bias section of ASTM C 39.

The ICNA and IC-MNA mixtures presented similar results to those of R mixture at 28 days, but were lower at 91 days by 7.3 % and 4.5 % respectively. In relation to the IC mixture, the results were 9.5 % and 6.8 % lower, respectively. Compared to ICSR mixture, ICNA mixture exhibited a significant strength gain of 23.8 % at 7 days that decreased at 28 days and 91 days, with 1.5 % and 5.5 % lower strength respectively. The IC-MNA mixture exhibited a similar behavior in comparison to IC-MSR mixture, IC-MNA mixture exhibited a strength gain of 5.1 % at 7 days and a decrease of 5.0 % and 7.7 % at 28 days and 91 days, respectively.

In contrast to what it is reported in previous works, OFIM mixtures did not exhibit the expected higher compressive strengths [17]. In presence of SRA, expansive compounds will grow within concrete at early ages [23]. The higher compressive strengths exhibited at 91 days by IC and ICSR mixtures are attributed to an enhanced hydration provided by conventional (IC) and improved (ICSR) internal curing, as well as to the shrinkage reduction.

3.3. Static modulus of elasticity.

Figure 3 presents the results of the static modulus of elasticity at 28 days and 91 days. R mixture presented average E values of 35 GPa and 38 GPa at 28 days and 91 days respectively, which represents a gain of 8 % between these two ages. Each result is an average of two individual tests; the maximum difference found between test specimens of a given mixture was lower than the

Table 5

Properties of fresh mixtures.

Tests	R	IC	ICSR	IC-MSR	ICNA	IC-MNA
Unit weight, kg/m^3	2285	2198	2219	2207	2213	2230
Air content, %	2.5	3	2.5	2.5	2.5	2.0
Slump flow, mm	705	685	695	705	675	715
T500, s	2.0	2.3	2.6	2.5	2.8	2.0
J-ring, mm	13	14	12	14	14	12
V-funnel, s	3.5	4.8	5.5	4.5	5.6	4.4
L-box, H2/H1	0.85	0.87	0.88	0.86	0.85	0.90
Static stability, %	4.8	4.4	4.1	4.7	4.5	5.1

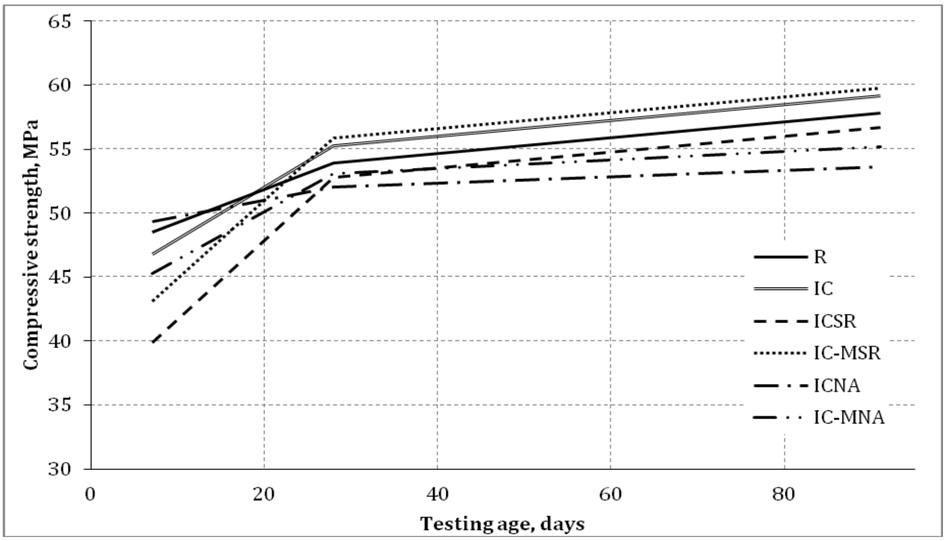
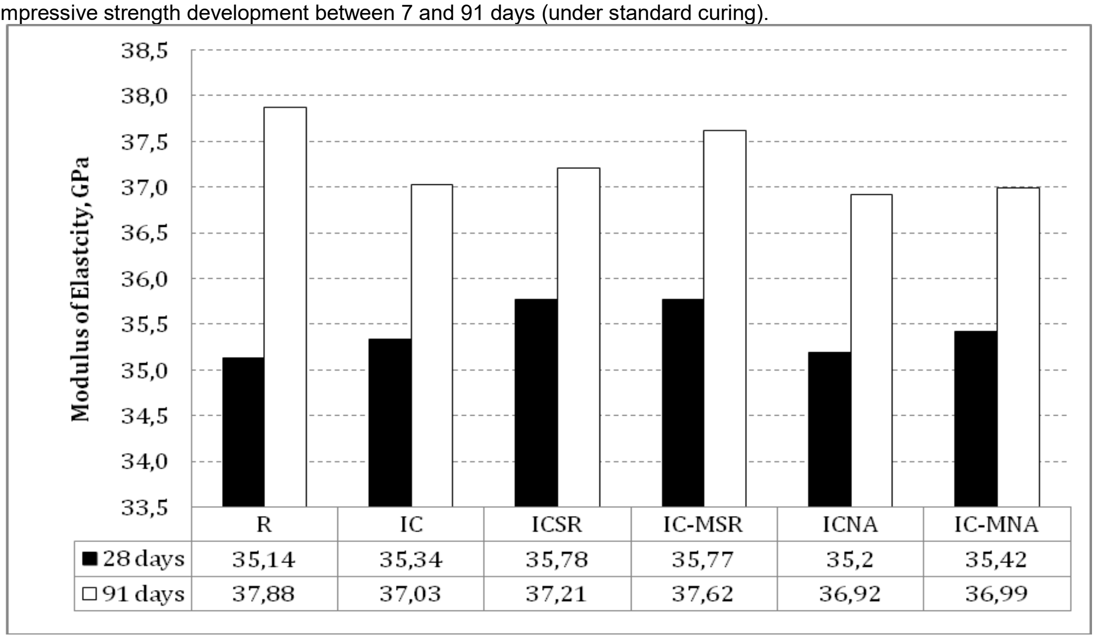


Fig. 2 - Compressive strength development between 7 and 91 days (under standard curing).



	R	IC	ICSR	IC-MSR	ICNA	IC-MNA
■ 28 days	35,14	35,34	35,78	35,77	35,2	35,42
□ 91 days	37,88	37,03	37,21	37,62	36,92	36,99

Fig. 3 - Effect of modified internal curing on static modulus of elasticity.

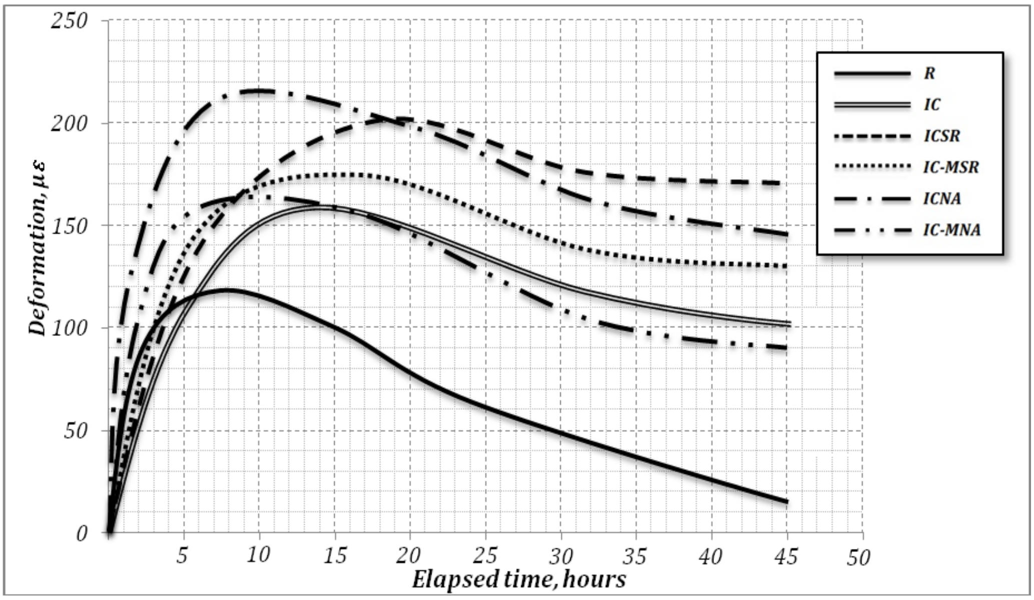


Fig. 4 - Development of autogenous deformation for the first 45 h.

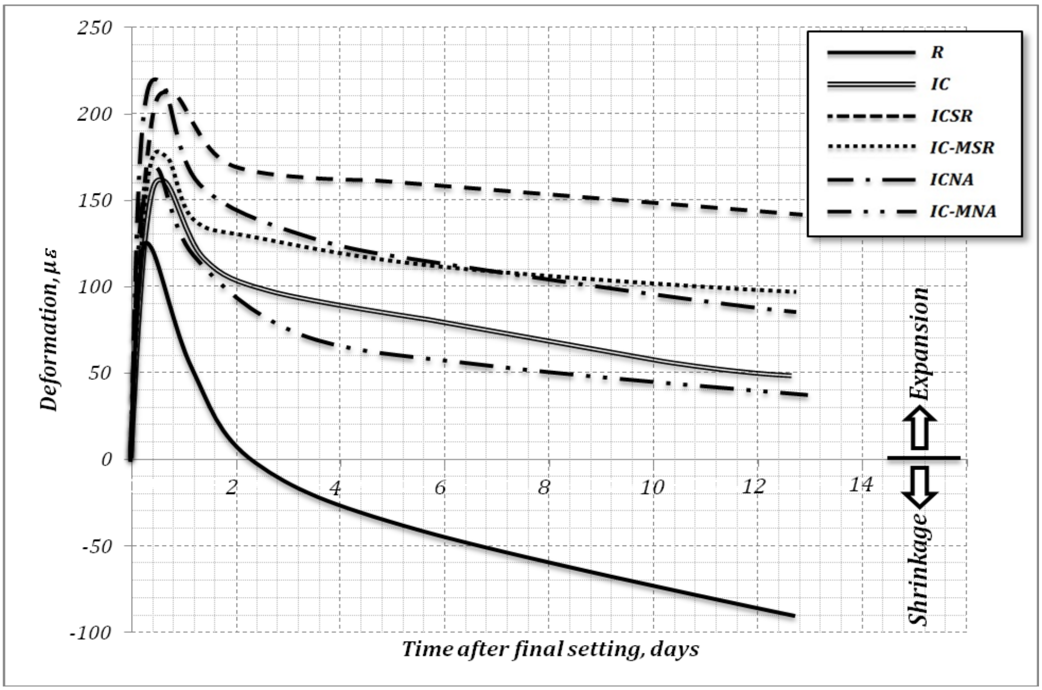


Fig. 5 - Autogenous deformation for a period out to 13 days.

maximum allowable deviation from the average of two cylinders (5 %) established in the precision and bias section of ASTM C-469-02.

In comparison to R mixture, at 28 days, IC, ICSR, IC-MSR, ICNA and ICMNA mixtures presented slightly greater E values, with increments ranging from 0.2 % to 1.8 %. At 91 days, E values were below the reference, with reductions ranging from 0.7 % to 2.5 %.

The higher stiffness exhibited at 91 days by the cementitious matrix in R mixture can be contrasted with the E values of the mixtures with lightweight aggregate (lower stiffness). For practical applications, differences in E values between R mixture and all the other mixtures could be considered negligible at 28 days and 91 days.

3.4. Autogenous deformation

The results of autogenous deformation are presented in Figures 4 and 5, for periods between 0 h to 45 h and between 0 days to 13 days, respectively. All the average values reported in figures 4 and 5 meet the precision and bias requirements of ASTM C-1698-09 that establish a standard deviation of 28 μm/m for mortar specimens with values of *w/cm* between 0.30 and 0.43.

Results in Figure 4 indicate that IC, ICSR, IC-MSR, ICNA and ICMNA mixtures had greater initial expansion at early ages (t < 15 h) than the reference that presented an expansion of 115 μƐ at 7.5 h after mixing; then, this concrete began to shrink, and recovered its original casting volume after 56 h. Afterwards, shrinkage may be considered as critical, because at that age concrete strength is relatively low (< 3.5 MPa) to counteract the potential appearance of cracks induced by the internal stresses caused by shrinkage.

IC, IC-SR and IC-MSR mixtures, presented maximum expansion deformations at 14 h, 19 h and 16 h respectively (Figure 4). Results suggest that both modes of OFIM incorporation (ICNA and IC-MNA) induced a more active and faster hydration reaction, achieving maximum expansions at 9 h after mixing. However, with maximum expansions of 212 μƐ and 200 μƐ for ICNA and ICSR mixtures respectively, values suggest that both OFIM and SRA aqueous solutions for internal curing, induces a more active hydration than that observed for R and IC mixtures. When both admixtures are dosed directly in the mixture (IC-MSR and IC-MNA mixtures) the respective expansions (170 μƐ and 160 μƐ respectively) were like that obtained for the conventional internal water cured mixture (IC = 155 μƐ).

Figure 5 presents autogenous defor-mations up to an age of 13 days. Excluding R mixture, that started to exhibit shrinkage at an age of 2.4 days, IC, ICSR. IC-MSR, ICNA and IC-MNA mixtures retained much of their initial expansion up to an age of 13 days. As a result of the expansion exhibited during the first few hours, Figure 5 shows that at 13 days the mixture with conventional internal water curing (IC = 50 μƐ), as well as the mixtures with the admixture dosed directly in the mixture (IC-MSR = 98 μƐ and IC-MNA = 38 μƐ) presented a reduced autogenous deformation by 1.6 times, 2.0 times and 1.4 times in comparison to the reference mixture (R = -91 μƐ), respectively. For mixtures where only internal curing was used (IC, ICSR = 142 μƐ, and ICNA = 88 μƐ), autogenous shrinkage reductions were 1.5 times, 2.5 times, and 2.0 times less than the shrinkage exhibited by mixture R.

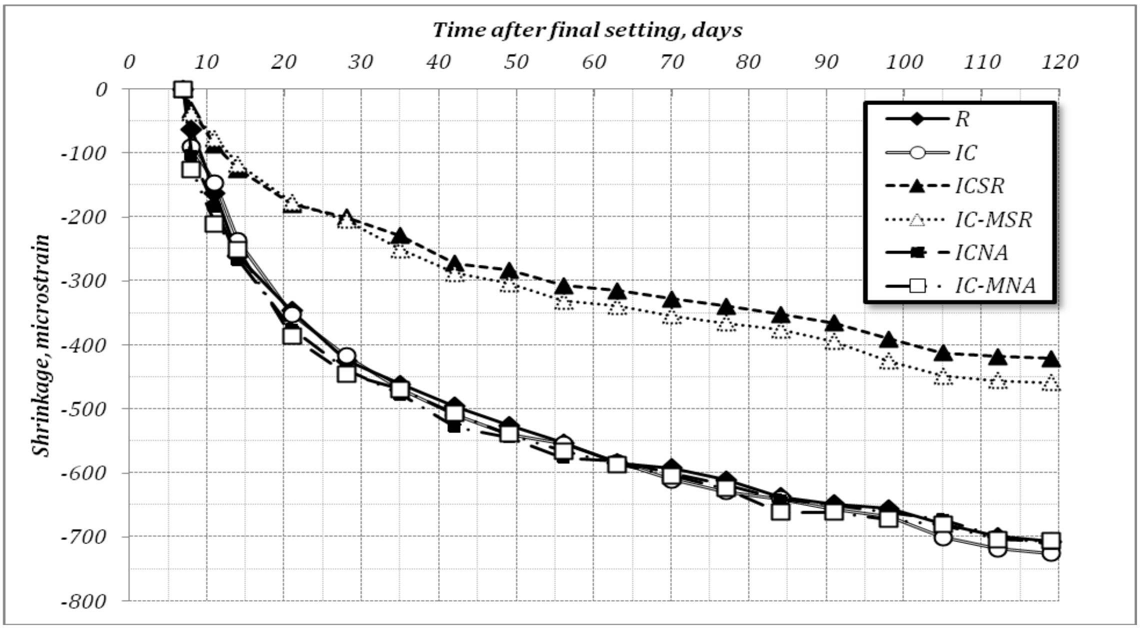


Fig. 6 - Drying shrinkage for ages ranging between 7 d and 120 days.

3.5 Drying shrinkage.

Figure 6 presents the development of drying shrinkage up to an age of three months. Results indicate that both modes of OFIM dosing (IC NA and IC-MNA mixtures) and the conventional internal water curing (IC) did not affect this property, since together with R mixture, they exhibited the greatest drying shrinkage, with maximum values between 706 μƐ and 725 μƐ at an age of 120 days. Individual results reported in Figure 6 are the average of three specimens. For these results, the standard deviation falls within the one required by the precision and bias section of ASTM C-157-08 (0.0048 %) for specimens stored in air.

Mixtures with SRA (ICSR and IC-MSR) substantially reduced drying shrinkage. For an average shrinkage of 712 μƐ for R, IC, ICNA and IC-MNA mixtures, the SRA in ICSR (463 μƐ) and IC-MSR (427 μƐ) mixtures led to drying shrinkage mitigations of 35 % and 40 %, respectively. These results clearly illustrate the benefit resulting from the incorporation of SRA into concrete; this technology exhibited an improved performance to reduce drying shrinkage when SRA in an aqueous solution is incorporated as an internal curing agent through a saturated fine lightweight aggregate.

4. Conclusions

The following conclusions were established for the constants and variables studied in this work:

1. The addition of OFIM induces compressive strength reductions between 2 % and 9 %, with negligible effect on static modulus of elasticity
2. A fine normal weight aggregate substitution of 20 % by volume by fine lightweight pumice aggregate, contributed to negligible reductions of static modulus of elasticity between 0.7 % and 2.5 %.
3. Initial autogenous expansions caused by OFIM substantially reduced autogenous deformations; when OFIM is used a greater reduction in total deformation is produced.
4. The effect of OFIM and conventional internal curing on drying shrinkage was negligible.
5. The effect of SRA on compressive strength and modulus of elasticity was negligible, and drove to significant reductions of autogenous and drying shrinkage deformations.
6. From a practical point of view, the obtained results illustrate that the incorporation of an aqueous solution of OFIM does not produce adverse effects on mechanical and elastic properties of high performance concretes, and induces a substantial reduction in autogenous shrinkage deformation.

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