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Effects of RHA on autogenous shrinkage of Portland cement pastes

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ABSTRACT

In this work, the effects of partial replacements of Portland cement by rice-husk ash (RHA) on the autogenous shrinkage were investigated. Pastes with water/binder ratio 0.30 and substitutions of 5% and 10% cement by RHA were tested. Two RHAs, both amorphous and partially crystalline, were studied. Comparisons between pastes with silica fume, and control pastes (without RHA or silica fume) are presented. Autogenous deformations, internal relative humidity and compressive strength were recorded. The RHA, amorphous or partially crystalline, when used in an appropriate way, reduces autogenous shrinkage.

1. Introduction

Autogenous shrinkage is a phenomenon known from the beginning of the 20th century [1,2], but its practical importance has only been recognized in recent years [3,4]. It is a problem in high-strength (HS) and high-performance concrete (HPC) mixtures [5]. Typical HS/HPC mixtures are characterized by a low water-to-cement ratio, an increased cement content, and the incorporation of mineral admixture additions and superplasticizer. In these concretes, a dense microstructure is formed in few days or less. Theoretically, for w/c below 0.42 [5–7], there is insufficient water in the initial mixture to complete the "potential" hydration; if external or internal supply of water are not available, considerable shrinkage can occur.

Autogenous shrinkage has been linked to lower w/c ratios when cement hydration consumes water from not only the larger pores, but also from the more refined pore structure, thus the concrete may exhibits enough self-desiccation to produce cracks. With the development and use of chemical admixtures such as superplasticizers, it is possible to produce workable mixtures with very low w/c, and the proneness to cracking increased and highlight the need of a correct concrete mixture design.

It is known that mineral admixtures lead to a densification of the concrete internal structure. In this sense, the mechanical characteristics of concrete, durability, and non-autogenous phenomena are modified with the use of mineral admixtures [8]. The improvements produced by the mineral additions can also modify the autogenous phenomena in concrete. Results are found concerning autogenous phenomena in cement-based materials due to the presence of mineral admixtures such as silica fume [3,5,9], fly ash [9–12], metakaolin [13–15], and blast furnace slag [16,17]. Fly ash, silica fume, metakaolin and blast furnace slag have played an important part in the production of HS/HPC [18–21].

The development and use of rice-husk ash (RHA) is not new [22]. RHA is a mineral admixture for concrete [22–23] and a lot of data has been published about its influence on the mechanical properties and durability of concretes. The literature regarding the effect of the RHA on autogenous shrinkage is scarce [24,25] and vague. For this reason, the main objective of this paper is to determine the effects of partial replacements of Portland cement by RHA on the autogenous phenomena, and provide relevant data in autogenous deformation and internal relative humidity. As RHA is not commonly used in the production of HS/HPC, the research done here gives relevant data concerning the autogenous problem and contributes toward that goal [26].

2. Autogenous phenomena

Early-age shrinkage of cementitious matrices is the result of several complex physico-chemical phenomena [27]; it can result in cracks that form in the same manner as at later ages. Even if the early resulting cracks are internal and microscopic, further shrinkage at later ages may merely open the existing cracks and cause problems [28–30].

Early-age shrinkage measurements provide a challenge due to the difficulty in making accurate measurements of the concrete prior to demolding. The shrinkage must be measured immediately after casting in a mold which permits constant reading without disturbing the concrete.

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Early-age shrinkage is associated with autogenous phenomena [16,28–30]:

- *Chemical shrinkage*: absolute volume reduction associated with the hydration reactions in a cementitious material.
- *Autogenous deformation*: the bulk deformation of a closed, isothermal cementitious material system not subjected to external forces.
- *Autogenous relative humidity change*: the change of internal relative humidity in a closed, isothermal, cementitious material system not subjected to external forces.
- *Self-desiccation*: autogenous relative humidity change of a cementitious material system after setting, caused by chemical shrinkage.

Autogenous shrinkage of cement paste and concrete is defined as the macroscopic volume change occurring when there is no moisture exchange between the material and the exterior surrounding environment. It is the result of chemical shrinkage produced by the hydration of cement particles [5,28–30].

Autogenous shrinkage has only recently been documented and accurately measured [28,29]. In the tests the concrete sample is sealed, there is no moisture transfer to the surrounding environment, and can be measured by volumetric or linear measurements. Jensen and Hansen [31], developed a special, corrugated mould system, which combines the advantages of linear and volumetric measurement. Before set, the corrugated mould system transforms the volumetric deformation into a linear deformation, and, after set, a normal, linear deformation is measured. In this way, it is possible to commence linear measurements immediately after casting. In this paper this technique was used to measure autogenous deformation; the description is presented in Section 4. In conventional concretes, autogenous deformation is generally negligible, whereas in HS/HPC it can be considerable [30–35].

3. Rice-husk ash

The rice husk is a ligneous hard layer of the cereal with high silicate content. When subjected to combustion, almost 20% of the husk becomes ash with a porous cellular structure (Fig. 1), a high specific surface (50–100 m^2/g) and high silica content. RHA is a waste product. The chemical composition of RHA depends on burning temperature and time of burning, but the variations in the elements are not significant.

The ash from open-field burning or from non-controlled combustion in industrial furnaces usually contains a higher proportion of non-reactive silica minerals such as cristobalite and tridymite, and it should be ground to very fine particles to develop pozzolanic activity. In addition, highly pozzolanic ash can be produced by means of controlled combustion, when silica is kept in non-crystalline form and cellular structure. Such silica can react when added to cement in the presence of water, with calcium hydroxide, resulting in cementitious compounds [8]. Most researchers confirm the fact that the burning temperature is a critical point in the production of amorphous reactive ash [36].

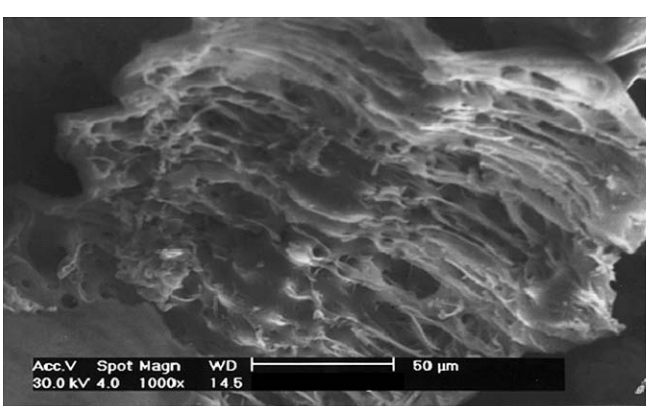

Fig. 1. Scanning electron micrograph of the RHA.

RHA is a mineral admixture for concrete. Mineral admixtures have a favorable influence on the strength and durability of concrete. In the case of RHA, the chemical effect is related to the fact that it is a highly pozzolanic material if its is produced by controlled combustion (reaction of amorphous silica with calcium hydroxide to form C–S–H); the physical effect is linked to particle size (less than 45 μm on average), which operates a refinement on the pore structure, acts as nucleation point for hydration products, and restricts growth of the crystals generated in the hydration process. The increment in the compressive strength of concretes with residual RHA may be better justified by the filler effect (physical); when working with RHA produced by controlled incineration, this increment is mainly due to the pozzolanic effect [37].

A lot of data has been published concerning the influence of RHA on the behaviour of pastes, mortars and concretes. Concretes with substitution of 10% of Portland cement by RHA showed excellent mechanical properties and durability when compared to control concretes [8,36–40]. However, limited data is available concerning the effect of the RHA on the autogenous shrinkage [24,25]; as a matter of fact, none investigates its effect on the autogenous deformation and relative humidity changes.

4. Experimental program

In this paper, in order to investigate the effects of partial replacements of Portland cement by RHA on the autogenous shrinkage, pastes with water/binder ratio of 0.30 and substitutions of 5% and 10% cement by RHA were tested. Two RHAs, both amorphous and partially crystalline, were studied. Comparison between pastes with silica fume and control pastes (without RHA or silica fume) are presented. Autogenous deformations and compressive strength for all mixtures were recorded; also, autogenous relative humidity changes are measured.

4.1. Materials

The following materials were used in the preparation of the paste specimens: a commercially-available ordinary Portland cement CEM I 42.5 R manufactured in Portugal, distilled water and a polymer based high range superplasticizer (PCE). Two sources of ash were considered; a residual RHA (RRHA) from the common rice paddy milling industries in Uruguay and a homogeneous ash produced by controlled incineration from the United States (CRHA). A commercially-available powder silica fume acquired in Portugal was used (SF) for comparison.

The residual RHA used for this work was a processed waste dry-milled for the necessary time to obtain a median particle size of 8 μm, a defined specific surface by nitrogen adsorption [41], and the maximum activity index according to the ASTM C311-98b. The procedure of RRHA optimization is presented [42,43].

Table 1 shows the chemical composition and physical properties of the Portland cement, the two ashes (RRHA and CRHA), and the SF used. Chemical analysis indicates that the two ashes are mainly composed of SiO_2. The median particle size of the two ashes is the same and also is the activity index (activity index = 92).

X-ray diffraction analysis, Fig. 2, indicates that the CRHA can be considered as non-crystalline; but the RRHA showed crystalline materials, which were identified as cristobalite. A rapid analytical

Table 1
Physical properties and chemical analyses of the two RHAs, silica fume and Portland cement used

	Cement	RRHA	CRHA	SF
Physical tests				
Specific gravity	3.15	2.06	2.16	2.12
Fineness	–	–	–	–
-Blaine specific surface, m^2/kg	429.50	–	–	–
-nitrogen adsorption, m^2/kg	–	28800	24300	24540
Chemical analyses, %				
Silicon dioxide (SiO_2)	19.21	87.2	88	91.92
Aluminium oxide (Al_2O_3)	4.64	0.15	–	0.35
Ferric oxide (Fe_2O_3)	3.30	0.16	0.1	0.15
Calcium oxide (CaO)	62.41	0.55	0.8	0.95
Magnesium oxide (MgO)	2.30	0.35	0.2	0.31
Manganese oxide (MnO)	–	–	0.2	–
Sodium oxide (Na_2O)	0.11	1.12	0.7	0.31
Potassium oxide (K_2O)	0,50	3.60	2.2	0.40
Sulphur oxide (SO_3)	3.46	0.32	–	0.26
Loss on ignition	3.33	6.55	8.1	5.56

method to evaluate amorphous silica in the rice husk ashes according to [44] has been used; the percentage of reactive silica contained in the CRHA was 98.5% and in the RRHA was 39.6%.

4.2. Mix design and testing program

The experimental program designed to study the effects of partial replacements of Portland cement by RHA on the autogenous shrinkage focused on the autogenous deformation, autogenous relative humidity changes and compressive strength.

The autogenous deformation was measured by a special measuring technique developed by Jensen and Hansen [31]. Linear measurements of autogenous deformation with CT1 digital dilatometers were used. The dilatometric technique has been tested during years of research work on binder phases of HPC [8,16,31,32,45–47]. A particular characteristic of the measuring technique is the encapsulation of the hardening cement-based material in a specially designed, corrugated polyethylene mould; this effectively prevents moisture loss and ensures insignificant restraint during hardening. The technique used is computer-controlled and permits measurements immediately after casting; but its use is limited to aggregates with maximum size of 2.5 mm. Since concrete has more aggregate than mortar, its autogenous shrinkage is lower; due to the same reason it is that mortar has less autogenous shrinkage than paste [28]. The use of paste and mortar mixtures would have more autogenous shrinkage than the concrete mixtures, and for this reason the differences between samples are expected to be higher, which allow better evaluation of the effect of the mineral admixtures.

A total of seven paste mixtures were made; for each RHA and SF two paste mixtures were made, and one paste without RHA or SF for comparison (control paste). The water/binder was kept constant and equal to 0.30, and substitutions of 5% and 10% cement were tested. Using percentages between 5% and 10% imply that the strengths of RHA and SF mixtures should be similar of the control pastes [8,48]. The replacement of cement by RHA and SF was made by mass. A constant consistency was achieved by adopting a 200 ± 20 mm flow diameter, only due to variation on superplasticizer dosage.

Table 2
Superplasticizer, flow diameter and setting time of the pastes

Paste	Superplasticizer (%)	Flow diameter (mm)	Setting time (min)	
			Initial	Final
Control	0.95	206	410	490
5% RRHA	0.925	220	284	359
10% RRHA	0.83	217	255	305
5% CRHA	1	213	316	373
10% CRHA	1.6	205	413	495
5% SF	1.37	217	315	376
10% SF	1.37	189	302	354

The pastes were mixed in a semi-automatic standard paste mixer (EN 196-1) for a total of 3 min. The flow test was performed immediately after the mixing period. No external forces or vibration was applied in the test. The values of flow diameter obtained and superplasticizer used are presented in Table 2. The superplasticizer percentages was used in relation to the weight of binder (cement + RHA or SF). In all the replacement levels, the CRHA and SF pastes required more superplasticizer, compared with the control paste, to obtain the desired flow diameter.

After the slump test, and for each paste, two specimens were cast for autogenous deformation, two specimens for autogenous relative humidity changes, 15 specimens for compressive strength, and the mould for setting time was filled.

4.3. Experimental details

According to the technique and equipment to measure autogenous deformation [31], two sealed corrugated tubes were carefully filled with fresh paste and sealed with two plastic end caps. The corrugated mould of 440 mm ∅28.5 mm specially designed to minimize restraint on the paste (made of 0.5 mm low density polyethylene plastic, LDPE) transforms volumetric deformations into linear deformations when the paste is in a fluid state, due to a greater stiffness in the radial than in the longitudinal direction of the mold.

Each specimen was placed in a dilatometer computer-controlled, and linear measurements every 10 min were recorded in a room with controlled temperature at 20 ± 1 °C (continuously monitored). The duration of the autogenous deformation test was 28 days.

For each cement paste, three simultaneous tests are carried out to determine the average setting time. Setting time was

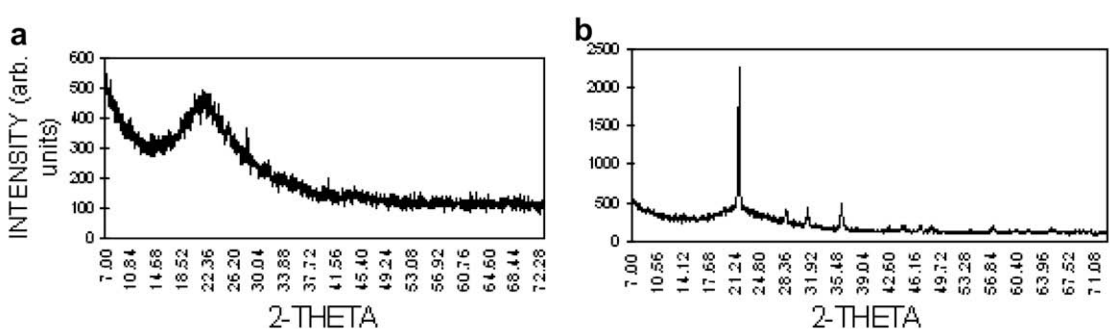

Fig. 2. X-ray diffraction of the RHA used. (a) CRHA; (b) RRHA.

determined by Vicat needle penetration (EN 196-3) for determination of "time zero", the starting time of the relevant autogenous deformations [49,50].

At early age thermal deformation occur simultaneously with autogenous shrinkage; for this reason a thermocouple was placed at the centre of each specimen (parallel specimen). Assuming a constant coefficient of thermal expansion $\alpha_{avg} = 18.0 \times 10^{-6}\ K^{-1}$ [43], the autogenous deformation at early age was corrected by a term $\alpha_{avg}\ \Delta T$ [16,47] where ΔT is the change of the temperature between the dilatometer and the test specimens. The temperature of the dilatometer bench and those of the specimens were controlled to approximately 0.1 °C during the early age (1–2 days). In all cases measurements of autogenous deformation and internal temperature were carried out simultaneously.

The internal relative humidity was measured on two independent sealed paste specimens [51], for each mixture and for a period of at least 6 days. For this determination the fresh paste was placed in two plastic tubes, with diameter 32 mm not much bigger than the RH sensors (12 mm) and minimum length 10 times the diameter of the sensors. After filling the plastic tubes two rubber plugs were placed immediately between the RH sensors and the tubes. Measurements of RH every 10 min were recorded in a room with a continuously monitored controlled temperature at 20 ± 1 °C. The RH sensors were calibrated before and after every experiment. According to the calibration, the maximum measurement error of the RH sensors was ±1%.

For compressive strength fifteen 50 mm cubes were cast into steel molds and demolded after 1 day. Curing of the paste specimens was conducted under sealed conditions at 20 °C for 7 days and at 50 °C after the first week, in order to accelerate the hydration rate. The compressive strength tests were carried out at the age of 1, 7 and 28 days.

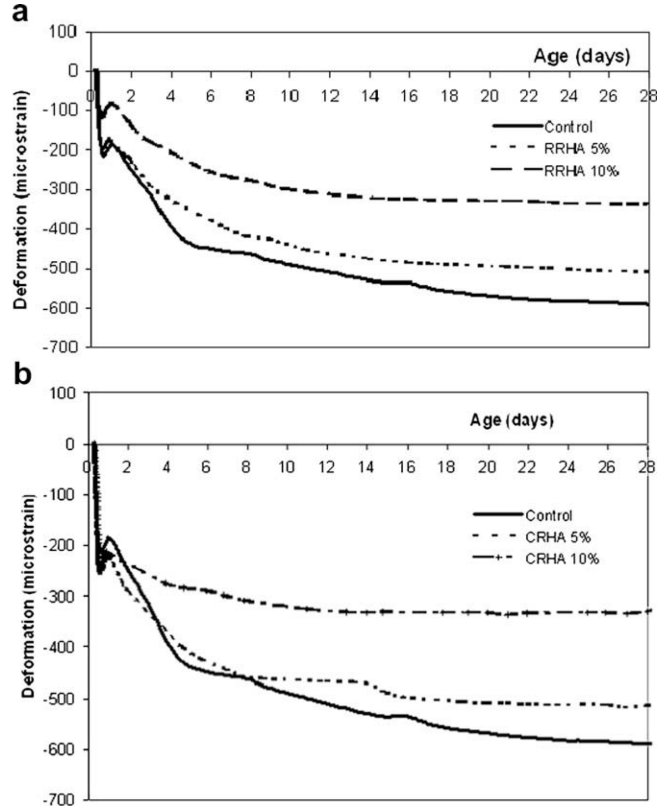

Fig. 3. Autogenous deformation vs age of Portland and RHA cement pastes.

5. Results and discussion

The measured autogenous deformation for the mixtures is provided in Figs. 3 and 4. Deformations were recorded for 28 days. The deformations were zeroed at the moment of final setting time, according Table 2. The inflections of the curves that occurred between 1 and 2 days may be due to the following effects; reabsorption of detrimental bleeding water accumulated at very early age between paste and the corrugated tube; ettingite needles growth; crystals of calcium hydroxide growth; topochemical reaction of C3S. However, the origin of this swelling was not investigated.

As shown in Fig. 3, RHA could decrease the autogenous deformation. With no RHA, a considerable autogenous deformation, approximately 600 μm/m, is developed during 4 weeks of sealed hardening. Replacement of 5% and 10% of Portland cement by RHA lead to a successive reduction of this autogenous deformation. At 10% RHA could decrease the autogenous deformation after 4 weeks by 250 μm/m.

RRHA provides a positive effect on the autogenous deformation, since early ages (1–2 days) which is attributed to the filler effect, when compared with the hydration effect of an equal dosage of Portland cement. The humidity measurements, Fig. 5, confirm a higher humidity of paste with RRHA. The cement substitution by CRHA at early age increases the autogenous deformation due to the pozzolanic effect, since chemical shrinkage of the silica reaction is much higher than Portland cement reaction; but with time, probably due to its cellular structure the autogenous deformation decreases in comparison with that of the paste without RHA (control). In the long time the behavior of the pastes with CRHA was similar to those of RRHA pastes. The lower strength of mixtures with CRHA is an indication of higher porous structure which may lead to lower capillary stresses in these pastes. The humidity values of the CRHA paste are higher than RRHA paste, which may also result of changes on porous structure. In fact, assuming that only Portland cement consumes water during hydration, the higher relative humidity of CRHA, when compared with RRHA, may be explained by the existence of larger pores in the CRHA paste.

As shown in Fig. 4, cement substitution by 5% SF increases the autogenous deformation. The increase of autogenous deformation due to SF, when used in addition to the cement (not replacement), is presented in the literature [6,16]. This fact leads to greater chemical shrinkage of the silica fume pastes [6,16,3] and to microcracking of the cement paste restrained by the aggregates [16,52].

Replacement of 10% of Portland cement by SF also increases the autogenous deformation at early ages; however, at 70 h the influence of the lower dosage of Portland cement equals the pozzolanic effect of the SF, and the autogenous shrinkage is comparable with the control paste; later, the autogenous deformation is similar to those of 5% RHA pastes.

The development of RH with hydration time is provided in Fig. 5. This figure shows that the initial RH is similar in the four pastes, about 97–98%. In the first hours, the humidity increasing period is due to the disequilibrium between the humidity of the sensor and the humidity of the paste, and does not correspond to valid measurements.

Initial RH values around 97–98% were measured on a number of Portland cement pastes [6,16].

As reaction of RHA and SF might not consume water a less RH-drop is expected on mixtures with partial substitution of Portland cement. In fact, it was found that the pozzolanic reaction of SF and calcium hydroxide only slightly affects the RH [6,16,47,53,54]. The behavior of RHA is analogous to that of SF since the ongoing pozzolanic reaction will result in a progressive RH decrease due to the refinement of the porosity and water absorption on the CSH gel formed. The reactive silica contained in the RRHA

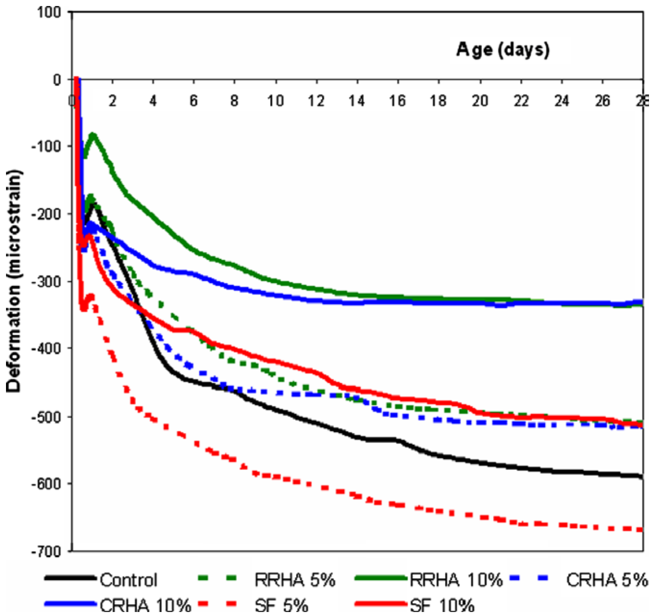

Fig. 4. Autogenous deformation vs age of all pastes.

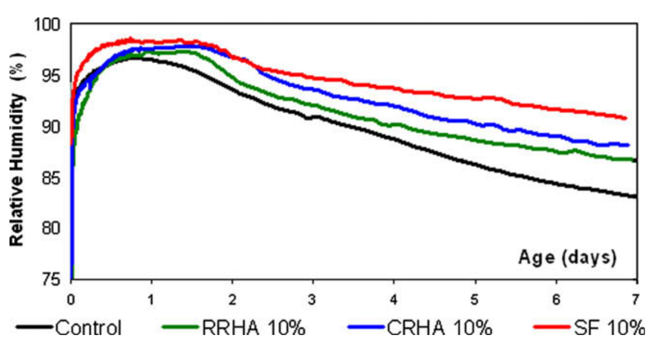

Fig. 5. Internal RH vs age for four cement pastes.

is approximately 40% and the effects of the pozzolanic reaction are lower than those obtained in the CRHA and SF pastes.

The internal relative humidity of the control paste decreases and stabilizes at approximately 83% RH within one week of hardening. The values obtained for the control and SF pastes, 83% and 90%, respectively, are in reasonable accordance with the literature [46,47,53,54]; the RHA pastes values are 87% and 88%, respectively for the RRHA and the CRHA.

Fig. 5 indicates a marked influence of the silica-content on the reached level of equilibrium relative humidities (see Table 1). The higher RH of pastes with mineral admixtures, when compared with control mixture, is explained by the lower water consumption during hydration.

The autogenous shrinkage results indicate that the use of RHA (RRHA and CRHA) has a positive effect, which may be due to its porous cellular structure, Fig. 1. The RHA probably acts as an internal reservoir, providing a source of curing water to the paste volume in its vicinity. The control mixture has the same amount of internal water, but mixtures with RHA may provide more homogenous distribution of large pores, that feed the capillary pores. Assuming that RHA provides a homogenous distribution of large pores, a decrease of capillary tension may be also expected, according to the Kelvin–Laplace equation.

Table 3 shows the compressive strength at the age of 1, 7 and 28 days on 50 mm cubes under sealed conditions. As expected, all the

Table 3
Compressive strength (MPa) of the pastes

fc (MPa)	1day	7 days	28 days
Control	52.5	88.9	100.5
RRHA 5%	41.1	80.8	100.5
RRHA 10%	48.3	86.7	105.4
CRHA 5%	37.6	70.6	85.9
CRHA 10%	39.7	83.3	97.4
SF 5%	43.1	83.1	95.3
SF10%	43.8	87.9	100.3

pastes show an increase in strength with age. From the results it arises that the compressive strength of RRHA pastes exhibit an excellent performance at ages of 7 days and over.

It is known that CRHA provides a positive effect on the compressive strength at long term [37,42]; the results of this study show that the behavior of pastes with RRHA is not inferior. No significant changes in long term compressive strength were observed by RRHA or SF incorporation.

6. Conclusion

The results show that RHA has considerable influence on autogenous deformation and on the autogenous relative humidity change of hardening Portland cement paste with low W/B. RHA markedly decreases the autogenous shrinkage as well as produces an autogenous relative humidity change.

An increase in the RHA content decreases the autogenous deformation. The experiments performed indicate that RHA and SF dosage have a dominating influence.

For autogenous shrinkage the efficacy of the use of RRHA and RHA produced by controlled incineration (CRHA) is confirmed through a performance evaluation in the pastes studied and the discussion presented. The positive effect of RHA on autogenous shrinkage is attributed to its porous cellular structure, which affects the distribution of water during hydration.

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