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Compressive strength and shrinkage of mortar containing various amounts of mineral additions

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ABSTRACT

Three mineral additions largely used in cementitious materials were tested in order to follow the shrinkage behaviour for 1 year of observation when they substitute a part of cement. The tests were carried out on standardized mortars specimen where cement was replaced by 5%, 15% and 25% of limestone, 10%, 20%, 30% of natural pozzolan and 10%, 30% and 50% of slag. The substitution of cement by 10%, 20% and 30% of limestone powder, natural pozzolan and slag respectively involves an optimal improvement of compressive strength of mortar. The separate quantification of the autogeneous and drying shrinkage development shows the effective contribution of each addition on microstructure modification and of the additional hydrates production. The microstructure was improved in the presence of limestone and of a moderate rate of slag, whereas it remains normal with natural pozzolan. The replacement rate of an active addition lower than 10% led to an additional hydrates production. This overproduction which accompanies the autogeneous shrinkage is more pronounced when cement is largely replaced by limestone. The evolutions of strength and shrinkage of mortars follow the same tendency from where it is easier to find a linear relationship giving the shrinkage deformation according to the compressive strength.

1. Introduction

The shrinkage of the concrete can be defined as the deformation of concrete element free of any external mechanical solicitation in a constant thermodynamic environment. Thus, one can distinguish between the shrinkage in a dry condition and the swelling in a saturated environment. The prediction of this deformation is of a very great importance for the study of the durability and the aptitude for long-term functioning of concrete structures. Indeed, it can be at the origin of cracking, prestress losses, stress redistribution, and even more rarely, ruin of the structure [1]. This dimensional variation depends on several parameters such as the concrete composition, the quality of its components, the elements size as well as the curing conditions. Also, the shrinkage of the concrete is largely influenced by mineral additions where they substitute a part of ordinary cement. Moreover, its development is similar to that of compressive strength from where it will be more easily deduced as a function of strength.

In general, the incorporation of mineral additions in the concrete composition involves a demand for additional water, a more important volume of paste and an additional formation of CSH products, which generates a more important shrinkage. In the same way, the presence of the mineral additions is accompanied by a modification of the microstructure and an increase of the fine pore percentage as in the case of silica fume [2], which does not strongly involve an important shrinkage.

In a synthesis study, Mehta [3] shows that shrinkage of pozzolanic cement is generally higher than that of ordinary cement. This additional shrinkage is due to the CSH content which would be relatively higher in the case of pozzolanic cement. The paste with the silica fume has a finer pore structure and consequently, retains more condensate water at the capillary level with given relative moisture. For the relative humidity located under the capillary level, weight losses and shrinkage are limited by the quantity of CSH; which is larger in silica fume paste [2,4]. Katri et al. [5] examined the shrinkage of a concrete with an W/C ratio equal to 0.35, they found that the substitution of 10% of cement by silica fume increases the shrinkage at early age and reduces it at long-term. This effect is largely reduced when silica fume is replaced with slag cement where the calcium hydroxide is less available. In the same way, Jianyong and Yan [6] observed, on three concretes with an equal ratio W/C of 0.26 and made with ordinary cement, 30% of slag and the third of 10% of silica fume and 30% of slag, that the drying shrinkage was practically identical at early age. After 28 days the values started to vary, and at 180 days the shrinkage reached 220, 96 and 127 μm respectively. This reduction is due to the great number of hydrates formed where mineral additions

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replace ordinary cement which makes the paste more rigid and less deformable.

ACI Committee 232 [7] reported that the shrinkage of fly ash cement increases slightly if the W/C ratio remains constant. This variation remains weak for a replacement rate less than 20%. Even, Mehta [4] confirmed that no difference in shrinkage was observed on concretes containing less than 25% of fly ash. On the other side, where 50% of cement was replaced by slag the shrinkage was largely higher. The experimental results taken by Chen and Chan [8] on mortars containing 35% and 68% of slag present a higher shrinkage than that of ordinary cement, causing tension stresses and surface cracking what explains the strength falls with a strong presence of slag. As for the silica fume, the results of shrinkage are less influenced by a replacement rate lower than 10%, and the difference in shrinkage becomes more important beyond 25% and even more when the concrete is exposed to drying condition [4].

Atis et al. [9] studied the shrinkage evolution of mortars specimen having a W/C ratio equal to 0.4. They concluded that the shrinkage of a mortar containing 10%, 20% and 30% of fly ash was reduced by 25%, 37% and 43% respectively after 5 months of observation. This is certainly due to the high concentration of free lime and MgO in these ashes. Chindaprasirt et al. [10] tested the effect of the fineness of fly ash on the drying shrinkage strain. The results confirm that all the mortars with fly ash present a less important shrinkage whatever the fineness is. This is due to the water demand which is reduced with the presence of these ashes.

On concrete specimen preserved 7 days in water, Ravindrarajah and Tam [11] observed that the shrinkage increased by 23% and 38% when cement was substituted by 20% and 40% of fly ash for a concrete strength of 25 MPa class and by 10% and 32% for a concrete strength of 35 MPa class. When the curing time passed from 7 to 28 days the shrinkage was much more reduced particularly for higher rates. They showed a reduction of 2%, 15% and 33% for replacement rates of 0%, 20% and 40% respectively. This shrinkage reduction with the increase of the cure was associated with the low porosity resulting from the slowness of the pozzolanic reaction.

The objective of this work is to evaluate the compressive strength and the shrinkage of mortars when mineral additions replace a part of cement. Cement was replaced by several replacement ratios of three mineral additions and the strain was measured on free and sealed samples. The determination of shrinkage deformation enables to identify the contribution of each addition in the hydration kinetic and the pore quality of cement. The objective is also to find a suitable procedure to obtain the optimal performances of these materials and to be able to predict their shrinkage according to the compressive strength results.

2. Experimental program

To better develop the mineral addition used in cement factory, it will be proceeded to the quantification of their effects on compressive strength and shrinkage deformation. The mortars were prepared with standardized sand with cement–sand–water mass proportions of 1:3:0.47. A part of cement has been substituted by mass levels of: 5%, 15% and 25% for the limestone powder; 10%, 20% and 30% for the natural pozzolan, and 10%, 30% and 50% for the slag. The composition and the characteristics of the used materials are given in Table 1. A superplasticizer based on melamine resin was used (1% of the mass of the binder) in order to obtain an acceptable consistency.

Compressive strength of mortar specimens were measured by casting 40 mm side cubes. After 1 day, specimens were removed from their moulds and stored in moist rooms until testing. Compressive strength tests were made at various ages of 1, 3, 7, 28 and 90 days. For shrinkage strain measurement, the mixtures were cast in prismatic moulds $40 \times 40 \times 160$ mm and covered with a moist tissue to prevent any evaporation at early age. At 1 day of age, the mortars are unmolded and prepared for shrinkage measurements. They were provided with an adhesive band at both ends to avoid the edge effects desiccation. For the autogenous shrinkage measurement the mortar samples were completely covered with a thin layer of bitumen surrounded by an adhesive band to ensure no exchange water with the external environment. During shrinkage measurement, samples were placed in a frame for strain shrinkage measurement equipped with a micrometer precision comparator. For each mixture, the measured values of shrinkage were done for two samples and average values were reported and recalculated for 1 m long. It is seen that the error variation for all measurements is acceptable and does not exceed 5%. Fig. 1 shows the position of samples in the frame measurement.

3. Results analysis

3.1. Compressive strength

The compressive strength was measured from the average of three cube samples of 40 mm sides in which the standard deviation was less than 8%. The obtained results are presented in Fig. 2. Compressive strengths of all mortars progress with the age according to the nature and the quantity of the mineral additions used. Generally, a strength reduction is noted at early age in case of inert mineral additions because of the dilution effect. After the first week, the conditions of activity of all additions are met, and the release of their reactions makes the microstructure more resistant.

Fig. 2 shows the beneficial effect of limestone powder on the strength at early age especially for low replacement rates. The replacement rate of 5% accelerates the hydration of cement and gives strengths higher than those of ordinary cement. The replacement rate of 25% proves to be ineffective because the strengths are stabilized after only 2 weeks. The use of this replacement rate can be justified only by special economic or ecological considerations. The effect of the replacement rate of 5% and 15% of limestone powder proves to be beneficial and the two mortars reach compressive strengths similar to those of ordinary cement before 28 days of age. After 28 days only the 5% replacement rate gives strength comparable to that of mortar without mineral additions.

The mortar with natural pozzolan has compressive strength at early age close to that of ordinary cement except for the high replacement rate. For replacement rates of 10% and 20%, the compressive strengths are slightly lower than those of ordinary cement and they are higher after 2 weeks. For high replacement rates, the strengths at early age are relatively low because of the dilution effect and the absence of pozzolanic reaction. On Fig. 2, it is shown that the replacement rates of 10%, 20% and 30% give compressive strength higher or equal to that of mortar without mineral addition at later age. The incorporation of 10% and 20% of natural pozzolan gives compressive strengths close to those of ordinary cement and no strength reduction was observed at later age, thus testifying the great activity of this material.

The slag mortar strengths are lower than those of ordinary cement at early age whatever the proportion of the slag used. This is explained by the fact that the slag reactivity requires a very basic solution to start, leading to a slow development of the compressive strength. After 2 weeks of hydration, the mortar strength containing 10% of slag reaches that of ordinary cement, where the dilution effect is compensated by the hydraulic effect of this slag. For 30% replacement rate, the hydraulic effect is significant

Table 1
Characteristics of used materials.

Chemical composition %	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	CL	Na_2O	K_2O	PAF	SSB (m^2/kg)	Glass content
CEM I	20.58	4.90	4.70	62.8	0.53	2.28	2.17			1.00	310	–
Slag	42.2	5.85	1.9	42.2	4.72	1.54		0.12	0.43	0.8	320	80%
Natural pozzolan	46.4	17.5	10.5	10.5	3.8	0.4		3.4	1.5	4.31	320	15%
Limestone powder	2.5	0.6	0.9	52.6	0.5			0.02	0.05	41.9	340	–

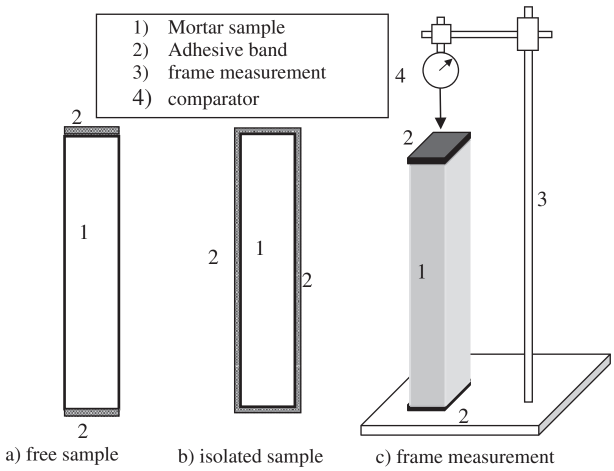

Fig. 1. Frame to measure shrinkage deformation.

and compressive strengths of the slag mortars are largely improved. On the other hand for high replacement rates of 50%, the development of compressive strengths requires 28 days of curing to reach those of the mortar without slag.

3.2. Total shrinkage

Figs. 3–5 show that the total shrinkage evolves very quickly for all the mixes because of their small size which makes the desiccation more favourable. At early age the shrinkage is almost independent of the composition of the mortar, the effect of the mineral additions appears only after the first week with a slight superiority for ordinary cement. At long-term, the presence of the mineral additions decreases the final shrinkage according to the quality and the quantity of these additions.

The total shrinkage of the limestone samples represented on Fig. 3 shows an important strain exceeding that of ordinary cement. After the seventh day, the shrinkage of the ordinary mortar evolves more rapidly and is distinguished from those of limestone until 6 months of age. At long-term, limestone does not have a significant effect on the total shrinkage where its values meet with those of ordinary cement, around 1250 µm. On Fig. 4, the shrinkage of mortars with natural pozzolan is identical to ordinary cement because of the great porosity generated by pozzolanic cements at early age. After the fifth day, pozzolan generates a less significant shrinkage than that of ordinary cement after the release of its reactivity. After the tenth day, the 10% pozzolan mortar starts to show a very important shrinkage and exceeds that of ordinary cement. At 1 year of observation, the shrinkage of the 20% natural pozzolan sample reaches that without any mineral additions. On the other hand, the shrinkage of 30% natural pozzolan mortar remains slightly lower. On Fig. 5, the slag cement decreases the shrinkage such as after 1 year of observation 1250, 1163, 1140 and 1030 µm are obtained for the mortars with ordinary cement and those containing 10%, 30% and 50% of slag respectively. This reduction of shrinkage is much more marked after the first week because of the filler effect by the slag and the quality of the long-term pores of this mortar.

3.3. Autogenous shrinkage

The autogenous shrinkage is the strain of the isolated mortars without water exchange with the external environment. These values give evidence of hydration progress and the self desiccation due to water consumption by the chemical process of the hydrates formation. Fig. 6 illustrates the results of the autogenous shrinkage according to the replacement rate of limestone powder measured at various ages. With its filler effect and its creation of other nucleation sites, the formation of hydrates is accelerated causing more shrinkage during the first week. At long-term, the shrinkage of 5% limestone mortars reaches that of ordinary cement; on the other hand those with 15% and 25% of limestone powder show a slight superiority. The curves evolution of autogenous shrinkage of natural pozzolan mortars represented on Fig. 7 shows an activity

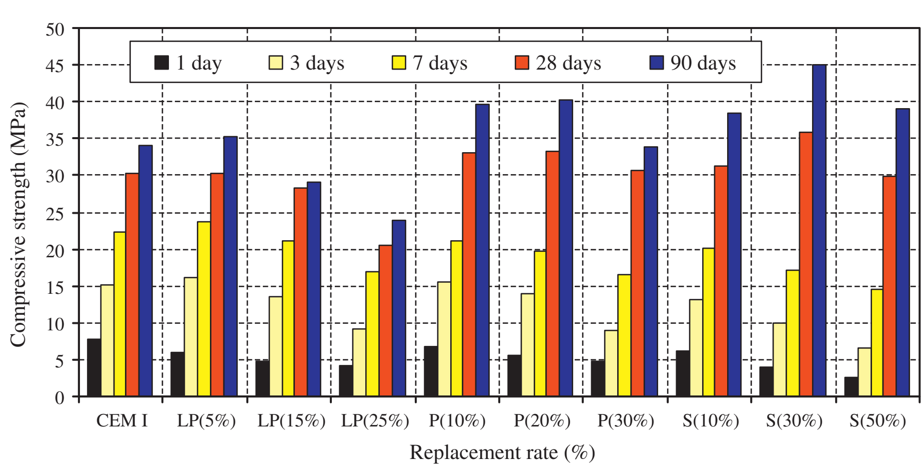

Fig. 2. Evolution compressive strength of mortars with various amounts of mineral additions (LP: limestone powder, P: natural pozzolan, S: slag).

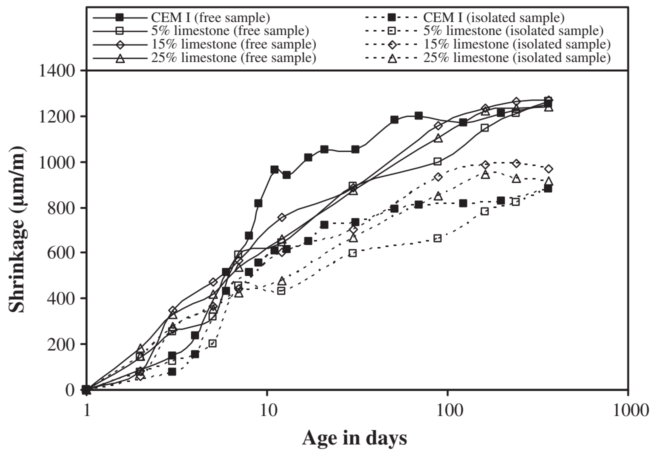

Fig. 3. Evolution of shrinkage for free and isolated samples of mortars with limestone powder.

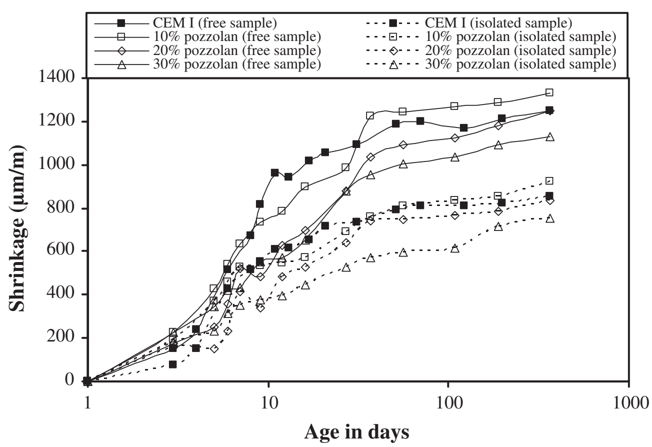

Fig. 4. Evolution of shrinkage for free and isolated samples of mortars with natural pozzolan.

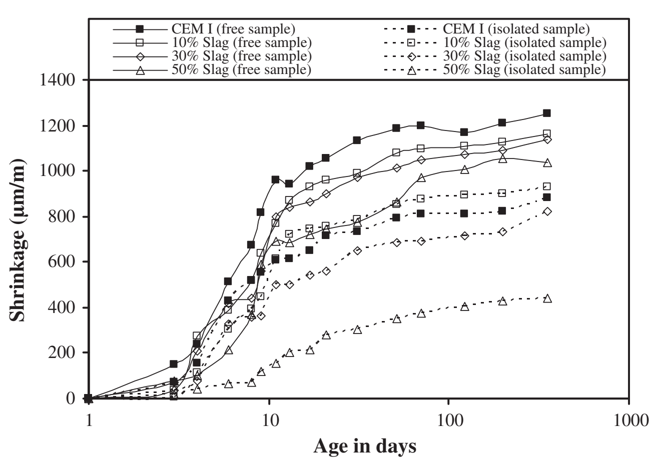

Fig. 5. Evolution of shrinkage for free and isolated samples of mortars with slag.

of pozzolan at early age due to important water consumption by the hydration. This result is in conformity with the results of setting time where a high rate of this mineral addition generates an acceleration of the setting [12]. After 7 days, the autogenous shrinkage of natural pozzolan mortars is reduced and becomes lower than that of ordinary cement. This confirms the weak

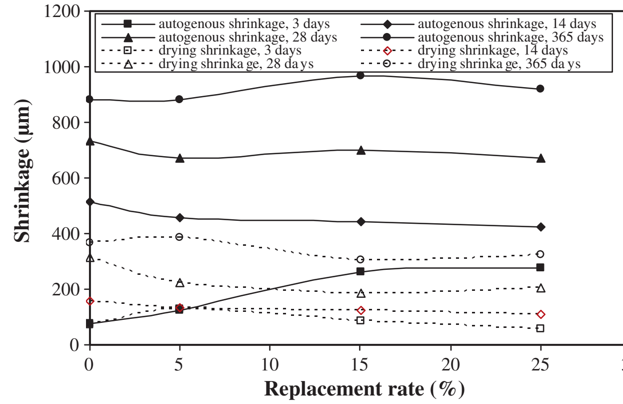

Fig. 6. Autogenous and drying shrinkage of mortars according to the replacement rate of limestone powder.

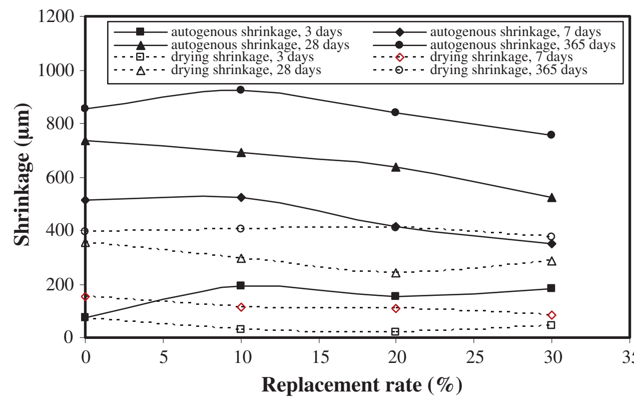

Fig. 7. Autogenous and drying shrinkage of mortars according to the replacement rate of natural pozzolan.

pozzolanic reactivity of this addition led by low glass content [13]. At 1 year of age, the autogenous shrinkage increases when 10% of cement is replaced by natural pozzolan which testifies a great production of hydrates. The effect of the slag on the hydration is illustrated on Fig. 8 where the evolution of the autogenous shrinkage shows a passive effect of the slag at 1 day, especially before the age of 7 days. After 10 days, the replacement rate becomes more marked where the slag starts reacting because of its hydraulic reactivity released after the saturation of the interstitial solution by the lime. The 10% slag mortar already generates a higher autogenous shrinkage, leading to a stronger formation of hydrates.

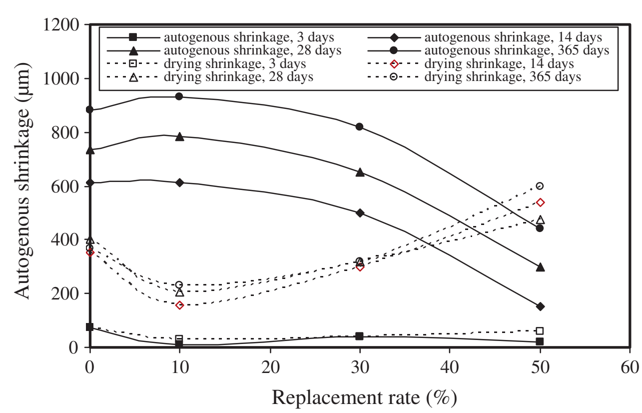

Fig. 8. Autogenous and drying shrinkage of mortars according to the replacement rate of slag.

On the other hand, that of 50% generates an autogenous shrinkage largely lower testifying an excessive replacement rate leading to few hydration products.

3.4. Drying shrinkage

The drying shrinkage represents the deformation of the mortar samples in the presence of water exchange with the external environment. These values represent the combination between the desiccation and the carbonation which testifies the porosity of the microstructure and its ability to support drying. It is measured as the difference of the shrinkages between free and sealed samples. Figs. 6–8 illustrate the results for the various mineral additions and the replacement rates used. The drying shrinkage of the limestone mortar specimen represented on Fig. 6 shows a positive effect at one age for replacement rates lower than 15%. After the first week, limestone mortar paste is less porous with more refined pores thus preventing the desiccation. At 1 year of observation, the recorded shrinkages are of 367, 384, 305 and 325 µm for ordinary mortars and mortars containing 5%, 15% and 25% of limestone powder respectively. The drying shrinkage of mortar with natural pozzolan is represented on Fig. 7 which shows an early activity of this addition characterized by a clear reduction of the shrinkage at 1 day. This reduction is more obvious after the first week when the pozzolanic reaction starts to take effect. This is due to the quality of the microstructure and the pores refinement which prevent water evaporation. At 1 year of observation, the drying shrinkage is almost identical independently to the replacement rate used. As shown in Fig. 8, the slag contributes by its fineness and its hydraulic reaction to reduce the drying for moderate replacement rates of 10% and 30%. At early age, the slag cement mortars develop an identical shrinkage to that of ordinary mortars until the seventh day where the addition of 50% of slag appears by a stronger drying. At later age, it can be concluded that a moderate use of the slag reduces the drying shrinkage by the improvement of the microstructure quality. The drying shrinkage represented in Fig. 8 shows a quality of the microstructure of cement containing 30% of slag identical to that of ordinary cement and much better for lower replacement rates.

3.5. Relationship between compressive strength and shrinkage

By comparing the development of compressive strength and the shrinkage deformations, it appears possible to predict the shrinkage according to the acquired compressive strength. Fig. 9, clearly illustrates this linear relationship for the various deformations of shrinkage where the coefficient of correlation is rather acceptable. This coefficient is higher for total shrinkage where the water exchange is allowed and better characterises the shrinkage deformation. It is established that this correlation is more adequate for ordinary cement and in the presence of natural pozzolan while it is weaker for limestone powder and the slag.

$$\varepsilon_{\text{shrinkage}} = A * S_{\text{strength}} + B$$

where A represents the kinetic coefficient according to the development of compressive strength and B the initial shrinkage when strength starts to develop, ε and S are the shrinkage and the compressive strength measured at the same age respectively. This relation will be more adequate if the measurement of shrinkage would have started from the setting time as reported in previous research [14,15] instead of 1 day as it is presented in this work.

According to the results illustrated in Fig. 9, it appears clearly that the correlation of the total shrinkage with the compressive strength is well accepted where the coefficient of correlation is higher. By representing the shrinkage strain with compressive strength for each type of concrete in Fig. 10–12, it will be noted that this correlation is not single and depends largely on the type and the replacement rate of the mineral additions. From these results, it will be shown that as long as the mortar does not reach the strength of 20 MPa the presence of mineral additions increases the total shrinkage. Once this strength is exceeded the mortars containing mineral additions generate a less significant total shrinkage. This result remains a simple proposal which needs further experimental results to confirm its conformity.

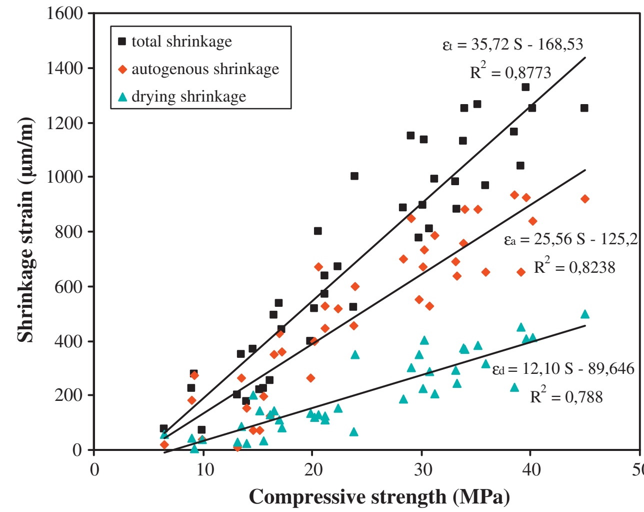

Fig. 9. Relationship between compressive strength and shrinkage. (ε_t: total shrinkage, ε_a: autogenous shrinkage, ε_d: drying shrinkage, S: compressive strength, R^2: coefficient of correlation).

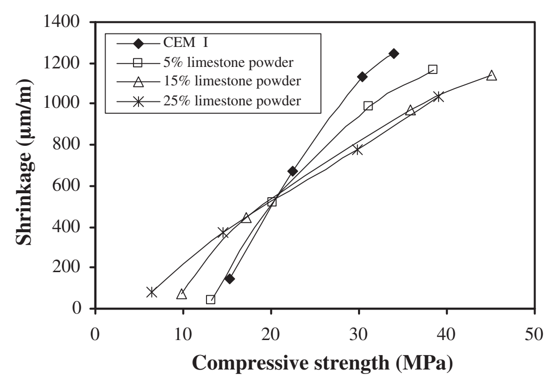

Fig. 10. Variation of shrinkage according to compressive strength for several replacement rate of limestone powder.

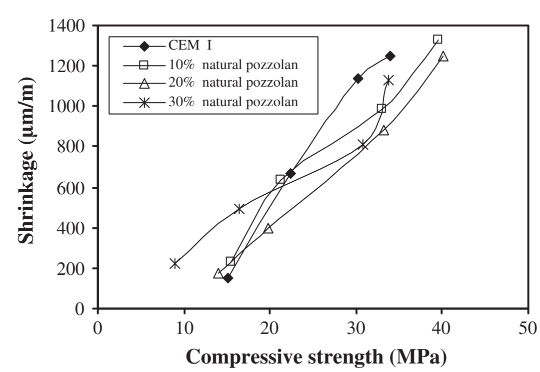

Fig. 11. Variation of shrinkage according to compressive strength for several replacement rate of natural pozzolan.

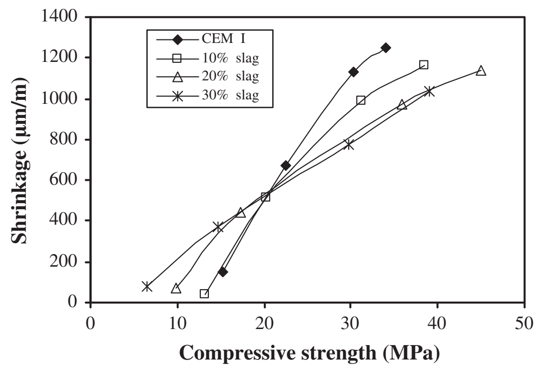

Fig. 12. Variation of shrinkage according to compressive strength for several replacement rate of slag.

4. Discussion

The fine mineral additions can substantially promote hydration of cement and increase the amount of the hydrates in cement paste which offers hardened cement a stronger structure and higher resistance to deformation. As well, blended cement made with mineral additions may fill small pores and reduces the shrinkage as reported by several researchers [16,17].

It is shown from Figs. 6–8 that autogenous shrinkage at 3 days increases according to the replacement rate of natural pozzolan and limestone powder whereas it is lower when slag is used. The presence of limestone powder and natural pozzolan causes an acceleration of the autogenous shrinkage and the hydration process since the very early age. This effect is generally explained by supplementary surface activity provided by the mineral addition particles, creating nucleation sites for hydrates as reported by others researchers [18,19]. At this age, autogenous shrinkage for mortars with slag remains lower than that of ordinary cement for all replacement rates. This proves that slag cement is less reactive than ordinary cement which is in conformity with Cassagnabère et al. [20] results where 18% replacement rate of slag involved 19% decrease in strength and 33% of the amount of hydrated phase. Also, the results obtained by Bougara et al. [21] on the same slag indicate that after 7 days of hydration only 8% of slag had reacted in slag cement systems, however after 28 days of hydration the percentage of slag that had reacted was 21%.

Natural pozzolan is the deposits of a volcanic ash characterized by its high pozzolanic reactivity where its contribution to the development of compressive strength is clearly shown in Fig. 3. For replacement rates of natural pozzolan up to 20%, the compressive strengths are lower than those of ordinary cement mortars at early age and they are higher beyond 28 days. This is in conformity with other results [22,23] where pozzolanic additions decreased the early mortar and concrete strengths but resulted strengths were comparable after 28 or 90 days. The finely divided silica present in natural pozzolan can combine with calcium hydroxide in the presence of water to form stable compounds like calcium silicates, which have cementitious properties. Such pozzolanic action of natural pozzolan contributes to the enhancement of strength mortars [13,24] and the densification of the hardened cement paste since it is deposited in the pores which offer high durability [23]. This action of the natural pozzolan has a great effect on the mortar shrinking since the autogenous shrinkage testifies to the advance in the hydration and the drying shrinkage to the quality of the microstructure [24]. This confirms the results of the shrinkage presented in Fig. 7 at early age where the values of the autogenous shrinkage increase and those of the drying shrinkage decrease with the increase in the replacement rates. The shrinkage results reported here is in agreement with the sorptivity coefficient results reported by Ghrici et al. [23] in which a mortar containing 30% of the same natural pozzolan was 34% lower than the corresponding OPC mortar where the cement past was filled and the capillarity pores are reduced by the formation of secondary CSH gel.

It is to be noted that the autogenous shrinkage of slag mortar improves with the age for low replacement rate of 10% as it is shown in Fig. 8. The autogenous shrinkage represents only 10% of that of OPC at 3 days, at 7 days they are identical, but at 28 days and 1 year that of slag is 7% and 6% higher respectively. This result is in agreement with those reported by Lee et al. [14] where slag concrete had higher autogenous shrinkage than the OPC concrete. Also, Lim and Wee [25] observed that there was a maximum autogenous shrinkage with respect to the replacement rate of slag. The higher autogenous shrinkage of mortar containing 10% of slag beyond 28 days can be attributed to the greater chemical shrinkage and high capillary pressure resulting by its higher hydraulic activity at this age which led to more hydrates products. Thus, the greater autogenous shrinkage of slag mortar led to low drying shrinkage for a low replacement rate as shown in Fig. 8. The autogenous shrinkage increases when the replacement rate of slag is small. However, when the replacement rate reaches 50%, the autogenous shrinkage becomes remarkably small; this result is in agreement with those reported by other researchers [26]. It was reported [26] that the autogenous shrinkage increased when the replacement rate of fly ash was 25%, however when the replacement rate reached 50%, the autogenous shrinkage had become remarkably small. Moreover, the use of suitable replacement rate of slag makes a cement paste have finer pore structures, which contribute to increase autogenous shrinkage and to decrease drying shrinkage.

It is well established by the users of the cement that various properties of the cement are predicted according to the compressive strength. The shrinkage deformations which include the quality of the microstructure and the advance of the cement hydration can be predicted as a function of compressive strength. Similar propositions were presented in several works in which Haque [27] presented a linear relationship between the strength of a concrete and the 24 h water penetration of the skin concrete. As well a significant positive correlation was established by Li et al. [28] between the percentage of 5–50 nm diameter pores and the natural logarithm of autogenous shrinkage. It seems that the value of 20 MPa represents a threshold of deformability beyond which composed cement will be less deformable.

5. Conclusion

The presence of mineral additions at suitable replacement rates makes an enhancement of compressive strength and a decrease of drying shrinkage, which is the result of their contribution to the production of other hydrates and to the improvement of the microstructure quality.

- The blended cement containing less than 15% of limestone powder seem to be beneficial and has compressive strengths similar to those of ordinary cement before 28 days of age. Up to 20% replacement rate of natural pozzolan, the compressive strengths are slightly lower than those of ordinary cement mortars at early age and they are higher after 2 weeks of cure. After 2 weeks of hydration, the mortar strength containing 10% of slag reaches that of ordinary cement. On the other hand for high replacement rates of 50%, the development of compressive strengths requires 28 days cure to reach those of the mortar without slag.

- Limestone powder generates a similar total shrinkage to that of ordinary cement. Besides, the slag makes a decrease of final shrinkage proportionally to the replacement rates. On the other hand, natural pozzolan makes an increase of shrinkage with 10% of replacement rate and beyond this rate, shrinkage is reduced.
- At early age, the substitution of cement with limestone powder or natural pozzolan increases the autogenous shrinkage by generating an acceleration of the cement hydration. The autogenous shrinkage increases by 10%, 8% and 6% when the cement is replaced by 15%, 10% and 10% of limestone powder, natural pozzolan and slag respectively.
- Before the first week, the drying shrinkage of limestone powder and natural pozzolan mortars is reduced. At 1 year of observation, the drying shrinkage is almost identical independently to the replacement rate used but a moderate use of the slag reduces the drying shrinkage at later age.
- Limestone powder did not affect the total shrinkage, however as the proportion of limestone powder increases the autogenous shrinkage of the mortar increases and its drying shrinkage decreases for high replacement rate.

A linear relationship is established between compressive strength and shrinkage, this correlation is more significant for the total shrinkage and it will be more adequate if the shrinkage is measured starting from the setting time. It is shown that there is a strength value of 20 MPa from which the shrinkage behaviour between blended cement and ordinary cement changes.

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