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THE EFFECT OF LIMESTONE FINENESS ON TERNARY CEMENT FRESH-STATE AND EARLY-AGE PROPERTIES

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Abstract: During cement production, a significant amount of CO_2 is released into the atmosphere, it is estimated that the production of each ton of clinker free about a ton of second carbon oxide. The use of additions as constituents of cement reduces the amount of clinker, where $C0_2$ emissions are reduced.
The combination of two or three additions with Portland cement can develop new types of binders (ternary or quaternary cement) with improved physical and mechanical properties compared to Portland cement alone. The objective of this work involves the study of the effects of the fineness of limestone on the physical and mechanical properties of ternary cements containing pozzolan and limestone with specific area of 3500, 5500 and 11000 cm^2/g, respectively. The amount of clinker is fixed at 65% , that of limestone is varied from 10 to 35% by weight of cement, the remain is constituted of pozzolanic addition.
The results showed that increasing the surface area of limestone could be with a favorable effect on the physical properties in particular the setting time and the shrinkage; further to good strength, mainly at early ages. The higher dosages of pozzolan reaching 25 % gave better mechanical performances among all other mixtures.
It can be concluded that the use of combined mineral additions, limestone and pozzolan could be beneficial to formulate ternary cements with improved physical and mechanical properties for mortars based on such binders.

Keywords: *Addition, limestone, pozzolan, fineness, shrinkage, setting time, mechanical performance.*

INTRODUCTION

For environmental reasons and the shortage marked in the manufacturing of ordinary portland cement (OPC) and further to lower its cost, researchers have developed a binder in which are embedded natural resources such as limestone (Torres C et al.,

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2017), naturel resources like pozzolan. Algerian cement companies incorporate into the production process these compounds to produce such type of cement with pozzolan and limestone as additions in the cement manufactured (Belagraa L and Bouzid A., 2016; Deboucha W. et al., 2012; 2015). It has been shown in several studies that the incorporation of additions mentioned above has several environmental and economical advantages, and sometimes even improving the mechanical properties of mortars containing these cements (Cassagnabère F et al, 2011; Imbabi M. S et al, 2012).

It can be concluded that the reuse of mineral additions as a substitution to normal cement is a promising venture and is being considered as a research area of great interest. As this valuation of industrial wastes contributes to the limiting of the dust and CO_2 emissions in the atmosphere. So, such benefit of reuse of these additions has a positive impact on the technical, economical and environmental levels (Men G et al., 2003; Menéndez G et al., 2003; Caid R et al., 2010; Torres C et al., 2017).

The beneficial effect of the limestone fillers on the speed of hydration of the Portland cement at the early age was already highlighted by previous studies (Lothenbach B et al., 2008; Bouasker M et al., 2008; Mounanga P et al., 2010). This improves the hydrates formation within matrices during the hydration process and also constitutes sites of additional nucleation for the development of such substances necessary for acquiring resistance of mortar cement based materials (Schindler A.K., 2005; Khalifa N. E. H et al., 2013). The incorporation of pozzolan in cement matrix is reported to improve the rheological properties of mortars mainly the workability, water retention and good particle homogeneity, beside the reduction of bleeding phenomenon at fresh state (Macleod., 2005). A significant reduction of shrinkage and limiting cracking is resulted by pozzolan addition for mortars based on ternary cement (Baron et al., 1997; Macleod, 2005). Also, the added pozzolan leads to better internal cohesion hence, higher compactness of the cement paste resulting in an improved mechanical strengths and the durability of mortars in general at hard state (Baron et al., 1997; Macleod., 2005).

This research work studies the effect of adding limestone and pozzolan on the physical properties and mechanical strengths of cements paste and mortars. In addition, the setting time and shrinkage have been determined for this compound cement. For this we compared the behavior mixes containing different combinations of additions (10 to 25 % limestone), (10 to 25 % pozzolan). The maximum amount of combined additions is 35 % with fixed specific area of 3500cm²/g for pozzolan. When, the limestone addition having a variable fineness of 3500, 5500 and11000 cm²/g, successively.

The prepared specimens of mortars based on these additions (limestone and pozzolan) were compared to reference control mix with 100 % normal cement at fresh and hardened state. The flexural and compressive strengths were assessed at 2, 7 and 28 days.

The study here in focusing on the reuse of mineral additives (limestone and pozzolan) as a substitution to normal cement is a promising venture and is being considered as a research area of great interest. As this valuation of industrial wastes leads to reducing the clinker hence, contributes to the limiting of the dust and CO_2 emissions in the atmosphere. So, such benefit of reutilization of these additions has a positive impact on the technical, economical and environmental levels.

EXPERIMENTAL PROGRAM

MATERIALS AND TEST PROCEDURES

The clinker used was delivered by a local company, with a specific area of 3500 cm²/g and a Bulk density of 3.2. In this study an inert natural limestone addition was used.The pozzolan is a byproduct of iron ore processing obtained from plant for steel manufacturing. The pozzolan composition can vary within wide limits, depending on the nature of the ore. The pozzolan used in this study is a waste of the steel local company. The gypsum used in this study is a natural addition delivered from a nearby deposit quarry. The sand used in the mortars is a normalized sand, according to the European standard (EN 933-1., 2000; EN 933-2., 1999). The water supplied by the network of public service in the civil engineering department laboratory for mixtures preparation was utilized. The chemical and mineralogical composition of clinker and additions are given in table 1

Tab. 1. The chemical and mineralogical composition of materials

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	L.O.I	Σ
Clinker	21.42	4.58	4.96	63.73	1.43	0.72	0.24	/	2.94	100
Gypsum	3.8	0.5	0.1	22.5	0.58	32.84	/	/	39.09	99.41
Limestone	11.06	1.65	0.58	47.85	0.73	0 .24	0.079	0.053	37.54	99.78
Pozzolan	45.59	17.81	9.04	10 .71	3.48	0.21	5.12	1.04	6 .96	99.96

The mineralogical composition of clinker			
C_3S	C_2S	C_3A	C_4AF
58.11	17.03	3.75	15.09

MIX DESIGN

To identify the benefits from the substitution of ternary additions in cements, we opted for making mixes of clinker, limestone and pozzolan. In the mix limestone with three specific areas 3500, 5500 and 11000 cm²/g, clinker and pozzolan with specific area fixed at 3500 cm ²/g were used. The sets of mortar obtained are related respectively to the proportions of the additions used in each mix. The mortar specimens adopted the following proportions 1/4 cement, 3/4 sand and water cement ratio w/c equals to 0.5. The cement paste was prepared at a normal consistency, according to the European standard (EN 196-3., 2003) having a water cement ratio about 0,26. For the characterization of the mechanical resistance of cubes of cement pastes and the shrinkage of mortars, prismatic specimens of dimensions (40 x 40 x 160) mm^3 were used. After twenty four hours, specimens were demolded and cured in the water until the time of testing for compressive strength. Shrinkage specimens were air dried for 28 days till the due age of the test. The different proportions for the mix series are given in table 2.

Tab. 2. The cement pastes and Mortars mixtures combinations for the testing program

Mixtures			M 1		M 2		M 3	
Proportions , %			P	L	P	L	P	L
			25%	10%	17,5%	17,5%	10%	25%
Mix \ Specific Area	Pozzolan cm² /g	Limestone cm² /g	1		2		3	
Group 01	3500	3500*	M_1G_1		M_2G_1		M_3G_1	
Group 02	3500	5500**	M_1G_2		M_2G_2		M_3G_2	
Group 03	3500	11000***	M_1G_3		M_2G_3		M_3G_3	

§ M_iG_j§ refers to Mix i (i=1,.,3) and group j (j=1, 2, 3).

*Group01 refers to the mixes having a specific area of limestone L1 of 3500 cm² /g.

** Group02 refers to the mixes having a specific area of limestone L2 of 5500 cm² /g.

***Group03 refers to the mixes having a specific area of limestone L3 equal to 11000 cm² /g.

TESTING

The physical properties of the mortars were performed mainly to assess the setting time and consistency as it is necessary to know the beginning and end of setting of cement pastes studied. Thus, the time available for proper placement of mortars or concretes which will then be made based on this type of cement is assessed. In addition, tests to evaluate the mechanical responses of the behavior of the different mixtures for flexural and compressive strengths were undertaken. The strength tests were performed according to European standards (NA EN 12390-3, 2009; NA EN 12390-6 2003). The shrinkage test has for objective to define the linear unidirectional variation of mortar specimen length of the three combinations of hydraulic binders obtained. Before each set of measurement taken, the instrument is calibrated with a metal rod of 160 mm in length and whose ends reproduce the face of the sides of the specimen.

RESULTS AND DISCUSSION

CONSISTENCY

The normal consistency is obtained for a W/C at values of 0.24, 0.29 and 0.34 for mixes of reference cements OPC, Limestone and Pozzolan, respectively. For the mixtures studied, it is noted that the higher the percentage of the addition, the higher the amount of water is needed to have a normal consistency as presented in table 3 and figure 1. For the set of cement paste specimens with higher fineness 11000 g/cm^2; the recorded consistency ratio showed an important increase for all mixtures in the whole groups at values ranging from of 0.25 to 0.34 compared to the reference one (0.24).

It can be noticed that a higher demand of water is needed, when pozzolan addition is present in the mixtures. The ratio of water to cement reaches the value of 0.34 for the M_1 mixtures in all mortars and independently of fineness variations. This evidence could be justified by the nature of addition, its chemical composition as well as its porous microstructure (Chaib O et al., 2015). However, for the limestone the need for mixing water is less important namely with a fineness superior to that of clinker. One can note that the minimum value for reference cement is equal to 0.24.

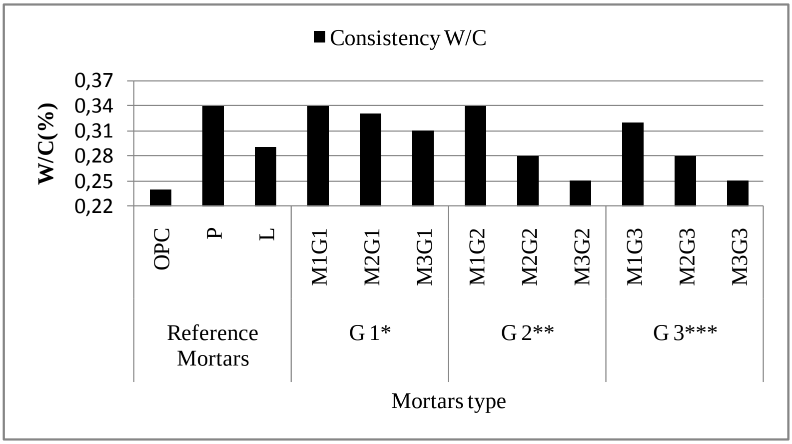

Fig. 1. The change in Consistency of studied mixtures

SETTING TIME

The results of setting time tests for the different mixes are shown in table 3.

The comparison of variation in the setting time of three sets of cement is presented in figure 2. It can be noted that the setting time decreases with higher dosages of pozzolan amounts; with additions at dosages of (35 % Pozzolan +65% Clinker) at fixed fineness of 3500 cm²/g; which can be explained by the activity of this addition type. For the whole mixtures based on natural additions (M1, M2, M3) and varied fineness, it can be observed that both initial and final setting time for control mix (OPC) remains higher than that of all additive mortars.

Tab. 3. The change in the setting time as a function of the addition content

		Reference mortars			G 1*			G 2**			G 3***		
		OPC	P	L	M1G1	M2G1	M3G1	M1G2	M2G2	M3G2	M1G3	M2G3	M3G3
Consistency	W/C	0,24	0,34	0,29	0,34	0,33	0,31	0,34	0,28	0,25	0,32	0,28	0,25
Setting time	Initial min	170	85	190	50	55	70	50	90	100	90	90	105
	Final min	200	160	240	150	160	165	160	175	190	180	180	220

G1* Refers to the limestone addition in group 1 with the fineness of 3500 cm^2/g.
G2** Refers to the limestone addition in group 2 with the fineness of 5500 cm^2/g.
G3*** Refers to the limestone addition in group 3 with the fineness of 11000 cm^2/g.

However, the increase in surface area from 3500, 5500 to 11000 cm²/g, influences the setting time for all mortars with ternary cement resulting in a longer initial and final registered setting time. This can be observed for the whole mortars in the three groups. The initial setting time showed lower values for all mortars with additives compared to the reference mix based on OPC cement.

The final setting time seems to be similar at values of three hours for both materials control one and with addition. Thus, the development of hydration process and mechanical strength is more acquired with this advantageous combination of pozzolan and limestone for ternary cement binders.

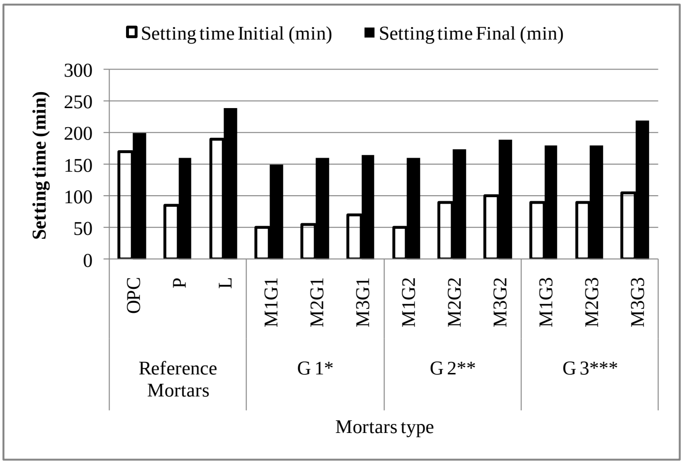

Fig. 2. The change in the setting time of studied mixtures

The setting time for mixtures containing great amount of pozzolan appeared to be affected with the addition blend type. A shorter initial and final setting time was recorded in regards to the pozzolan activity degree for such cement incorporating this addition (Ezziane K et al ., 2010).

DENSITY

The table 4 presents the results of the bulk density for the studied mixtures at 02, 07 and 28 days. Also, figure 3 illustrates these obtained values.

Tab. 4. The results of the bulk density variation for the different mixtures at 02, 07 and 28 days

		Reference mortars			G 1*			G 2**			G 3***		
Density variation %	Age days	OPC	P	L	M1G1	M2G1	M3G1	M1G2	M2G2	M3G2	M1G3	M2G3	M3G3
	2	7,15	7,54	7,88	7,68	8,82	8,52	7,84	9,4	8,51	7,15	8,14	7,86
	7	8,04	8	8,1	8,05	9,38	9,09	8,58	9,98	8,51	7,87	8,33	8,17
	28	8,17	8,13	9,02	8,05	9,38	9,09	8,58	9,98	8,7	7,87	8,33	9,17

It can be noticed that there is a slight increase of the density, for the mixtures containing great amount of both of additions at equal percentages of 17.5 %. This can be explained by the effect of the type of this addition with higher content and greater fineness. This fact could be due to the additive playing the role of filling voids (fillers role); resulting in a more compactness obtained for cement matrices of such mortars.

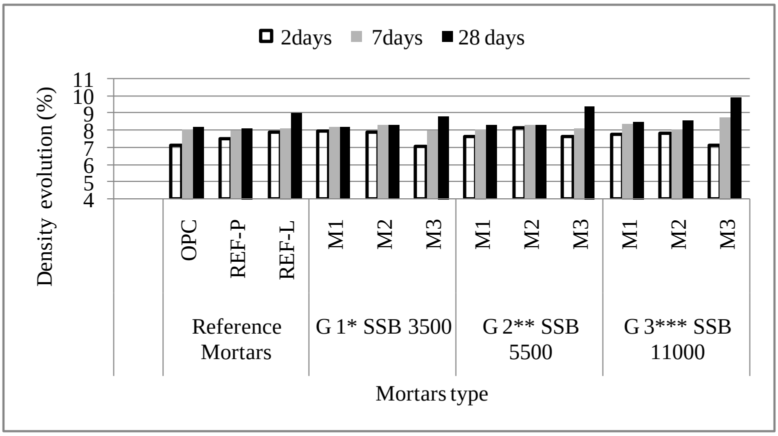

Fig. 3. Variation of the bulk density (%) for the different mixtures at 02, 07 and 28 days

SHRINKAGE TEST

The results obtained are presented in table 5 and plotted in figure 4, these are in a good agreement with European standard (NF EN 196-3., 2003), which specifies that the cements shrinkage should be around a 800 (μ/m) for the CEM II grade 32.5 and 1000 (μ/m) for the grade.

Tab. 5. The change in the shrinkage of the mixtures studied.

Shrinkage, μ/m		Reference mortars			G 1*			G 2**			G 3***		
Cure mode	(Air dried)	OPC	P	L	M1G1	M2G1	M3G1	M1G2	M2G2	M3G2	M1G3	M2G3	M3G3
		555	447	400	320	280	240	216	165	130	184	119	90

At the age of 28 days, the obtained results for shrinkage correlate very well with those for densities evolution increase namely, the mortars with great amount of limestone at higher fineness (M_3G_2 and M_3G_3).)

These results are in good accordance with previous research that reported the limiting of shrinkage due to limestone added for mortars formulation (Guemmadi1 Z et al., 2010).

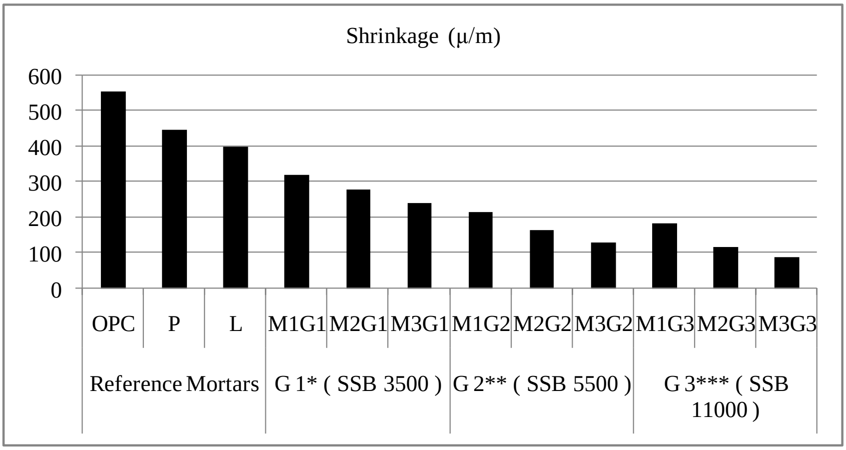

Fig. 4. Shrinkage for the different mixtures

Based on the obtained results, it can be noticed a limiting shrinkage; which is based on the hardening age at 28 days. This reduction could be explained by the creation of a fine network of capillary pores within the cement hydrated pastes. When, these capillaries are saturated with water, its consumption for hydration process in cement causes drying; this results in the formation of meniscuses which produce attractive forces causing the shrinkage (Doug Hooton R et al., 2002).

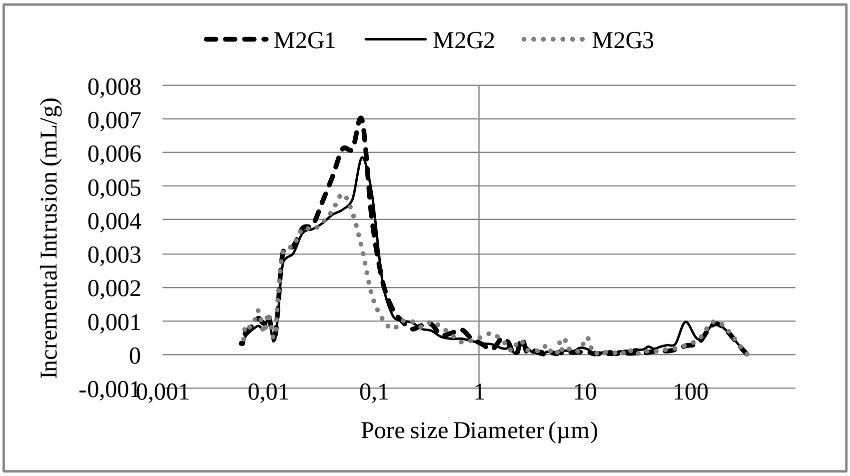

Fig. 5. Pore size distributions of combined mortars studied (M2)

The shrinkage of reference mortars is more important for binary cement compared to other mixtures based on ternary cements at various fineness of limestone particularly, for group 2 and 3 at the fineness of 5500 and 11000 cm^2 /g, respectively. This could be explained by the fact that the filling role of the limestone addition prohibiting water evaporation where, the voids are occupied with fine grains of such addition.

The increase of the shrinkage also depends on the change in fineness of grinding. It is mainly due to the presence of a high capillary porosity. This causes the formation of a large amount of meniscuses and the kinetic of hydration becomes very fast within the hydrated cement paste (increased tensile forces that is developed within the capillaries). The minimum values for shrinkage are recorded for mixtures containing more limestone additions especially, for those with a specific surface area exceeding 5500 cm^2/g (Guemmadi1 Z et al., 2010).

For a better understanding of the fineness effect of limestone addition on the relationship among physical properties mainly, the porosity and the shrinkage, the microporosimetry tests (MIP) was carried out. The mixture at equal ratio of additions (M2) at varied fineness of limestone was chosen for the undertaken tests.

In general, the increase of the pores is due to the fact that the porosity of the mineral addition mainly the pozzolan is higher than that of clinker. The pore size distribution of mortars with pozzolan and limestone combination blends at the same amount (17. 5 % P+17.5 % L) evidently, shows the effect of the limestone addition and its fineness reaching 11000 cm^2 /g on the pores size; which are reduced with greater content of limestone as illustrated in figure 5. This fact is revealed by previous study to enhance hydrate chemical reactions of cement (Bellifa S et al., 2014).

COMPRESSIVE AND FLEXURAL STRENGTHS

The comparison of the development in for the mechanical strengths of three sets of mortars at 2, 7 and 28 days are shown in figures 6 and 7.

Tab. 6. The Compressive and flexural strength results for the different Mixtures

Compressive strength		Reference mortars			G 1*			G 2**			G 3***		
R_c (MPa)	Age (days)	OPC	P	L	M1G1	M2G1	M3G1	M1G2	M2G2	M3G2	M1G3	M2G3	M3G3
	2	17,2	8,5	10,5	7,5	6,8	10,2	7,6	7,8	8,9	9,8	10,8	11,5
	7	30	25,4	18,7	25,6	19,9	24,5	25,1	22,1	23,9	25,5	24,7	23,8
	28	52,6	38,8	30,1	40,7	35,4	36	43,5	36,7	35,4	52,3	38,7	39,4
Flexural strength		Reference mortars			G 1*			G 2**			G 3***		
R_f (MPa)	Age (days)	OPC	P	L	M1G1	M2G1	M3G1	M1G2	M2G2	M3G2	M1G3	M2G3	M3G3
	2	2,1	2,5	2,1	2,4	2,1	1,9	2,1	1,9	1,8	2,9	2,7	2,6
	7	5	4,9	3,9	4,8	3,7	5,1	5,1	4,8	5,4	4,4	4,7	5,6
	28	8,9	7,8	6,8	6,7	6,7	6,9	6,4	6,6	7,1	7,4	8,2	9,1

G1* Refers to the limestone addition fineness of 3500 cm^2/g.

G2** Refers to the limestone addition fineness of 5500 cm^2/g.

G3*** Refers to the limestone addition fineness of 11000 cm^2/g.

The benefic side on strength improvement development in regards to physical limestone fillers contribution effect at higher fineness reaching 11000 cm^2/g could be noted for the choice of ternary cement compositions with additions which is favored by such utilization in mortars formulation.

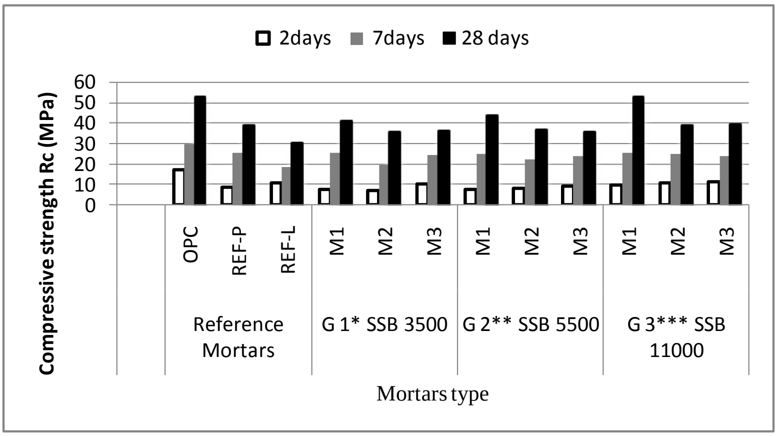

Fig. 6. The variation of compressive strengths for different mixtures at 02, 07 and 28 days

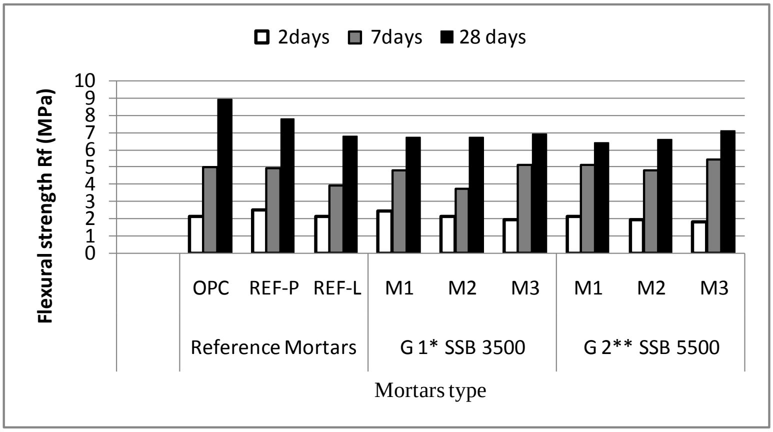

Fig. 7. The variation of flexural strengths for different mixtures at 02, 07 and 28 days

The comparison of the mechanical strengths development of three sets of mortars at 2, 7 and 28 days are shown in figures 6 and 7. In regards of the addition type effect, the results obtained show that at early age all the values of the compressive strength of whole mixes with addition and the control one is lower compared to the mix containing 25 % of limestone. This decrease is mainly attributed to the slow activity of added pozzolan at young age and the quick response of added limestone at the short term. This fact could be explained by the physico-chemical effect of such combination with 10 % pozzolan and 25 % limestone that enhances development of strength and accelerates the formation of hydrated products. This is in good agreement with results obtained by other researchers (Doug Hooton R et al., 2002).

According to figure 6 and table 6, it can be noticed that the composition of the mixtures set (M_1) for all groups containing 25% pozzolan gave compressive strengths comparables to the reference mortar (OPC). This fact showed the influence of the addition on its reactivity and then on the mechanical strength of the mortars at later ages. Beside the contribution related to the surface area of the limestone at higher fineness of around 11000 cm²/g. These values of compressive strengths were 523, 387 and 394 bars at 28 days of age for the three compositions in group 3, respectively.

The maximum value of the compressive strength is recorded for the mixture M1G3 which contains 25 % pozzolan and 10% limestone (SSB = 11000 cm2 / g), it is comparable to that of the reference mortar (OPC) at 28 days. As well, the based ternary cement mixtures (M2) [17.5 % P, 17.5 % L) gave acceptable mechanical performance higher than 325 bars; when compared to reference binary mixes mainly with limestone alone.

The results of such combination for ternary cement contribute to the limiting of pores and make the cement matrix more compact hence, the improvement of compressive strength.

Regarding the evolution of the strength as a function of the fineness and the additions contents at 28 days (Fig 6), it can be noticed that the strength value of the mixtures in group 3 had a significant increase compared overall the other mixes with additions.

Further, It can be noticed from figure 7, that the flexural strength follows the same development trend as for compression at 28 days of age. These values of flexural strength for ternary cement mortars are almost similar to reference one (OPC).

This finding about the positive effect of limestone addition in ternary cements on the porosity and hence the mechanical responses of the present research studied mortars are in accordance with previous research works on the subject (Guemmadi1 Z et al., 2010; Bellifa S et al., 2014).

CONCLUSION

The results obtained in this study lead to the following conclusions to be drawn;

Regarding the consistency of ternary cements incorporating pozzolan, the water demand is higher for such mixtures compared to the reference one.

The setting time is much influenced by the type of additions which is appeared to be advantageous for the decrease of both initial and final setting time of ternary blended cements (limestone and pozzolan). This reflects the activity of pozzolan accelerating hydrates formation process.

The results obtained for the density indicated similar values compared to reference mortar. The effect of addition type is not much significant for pozzolan. However, likely limestone and its higher fineness lead to a slight amelioration of the mortars densities with additive compositions.

The decrease of shrinkage based on the age of hardening and assessed at 28 days is mainly depending on the degree of fineness of limestone addition used. This could be advantageous for novel combined compositions for ternary cement based on (limestone and pozzolan).

At early age, all values of the compressive strength for all the mixtures remain lower compared to the mix containing great amount of limestone at a dosage of 25 %. The influence of the surface area on the reactivity of limestone addition and consequently on the mechanical strength performance is appreciated.

The percentage of 17.5 % for the additives (pozzolan and limestone) gave acceptable results for the compressive and flexural strength for all fineness of limestone used so far. Thus, the optimal dosage around 35 % could be suggested for ternary cement. The higher dosages of pozzolan reaching 25 % gave better mechanical performances.

Finally, It can be concluded that the use of mineral additions, limestone and pozzolan could be beneficial to formulate ternary cements. Thus, an alternative environmental friendly cement binder could be used matching with a sustainable development objectives.

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