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Research Paper

Effect of limestone fines on the mechanical properties and durability of mortar made with crushed sand

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ARTICLE INFO	ABSTRACT					
Article history :						
Received : 17 December 2019	This study investigate the influence of limestone fines (LF) on the mechanical properties,					
Revised : 28 May 2020	hydration heat and durability (acid and sulfate environments) of mortars made with					
Accepted : 31 May 2020	crushed sand (CS) and different rate replacement of limestone fines of the CS (0%, 5%, 10%, 15%) using three types of cement, that is, ordinary Portland cement, limestone cement and pozzolanic cement. The obtained results show that the limestone fines increase					
	the compressive strength and generate more hydration heat for all the used cements.					
Keywords:	Mortars made with blended cements, limestone or pozzolan, with 5% and 10% limestone					
Cement	fines, have low expansion when imerged in 5% Na2SO4 solution. In addition, when immerged in acids environment, the mortars with limestone fines exhibit negative effect					
Crushed sand	at long-term durability. However, the mortar manufactured with ordinary Portland cement					
Fines	containing 15% of limestone fines will be strongly affected. It was found that no benefit can be gained from eliminating limestone fines fraction from the CS.					

1 Introduction

Mortar

In order to build all kinds of structures, a consumption of a huge quantity of sand is needed for making concretes. At the present time, the most commonly used sand is the one coming up from rivers. Though, as it contributes to a lack of natural sand, an excessive use of alluvial sand has been banned for several technical and environmental reasons. It is worth to note that the extensive use of this kind of sand has led naturally to the depletion of the resources and has affected seriously the environment. Thus, it is imperative to reduce their consumption as well as to find out other alternative sources. Crushed sand is a frequent alternative to the river sand. However, crushed sand has to satisfy some quality criteria and be available in sufficient quantities with low cost, in order to be practical alternative in manufacturing concrete [1].

* *Corresponding author. Tel.:* +213 696460548. E-mail address: hadjmeziane@yahoo.fr Indeed, the use of crushed sand in concrete is required to obtain higher mechanical strengths [2, 3]. However, this operation has the drawback of requiring an amount of water higher than it is for rolled aggregates. This is due essentially to the reduced compactness associated to their shape, angularity and roughness [4].

Previous studies have found that up to 15% of limestone fines in CS do not affect strength performances of concrete [5]. Also, it has been established that a range of 12-18% of fines could be allowed in sand without harmful effects on the physical and mechanical properties of mortar and concrete [6-8]. Due to the grinding process, crushed sand differs from natural sand in particle size, shape and texture; and generally, includes between 10% to 20% of fines (the particles passing through 80 µm sieve).

In recent years, several researches were focused on the effect of different grades of crushed limestone sand on the properties of the concrete. Their results established that 10% to 15% of the lime scale fines might be replaced in crushed sand without negative effects on the physical and mechanical properties of the concrete [9].

Limestone fines content in CS plays a fundamental role in improving the compressive strength of the concrete. It was found that the addition of up to 15% of limestone fines as a partial replacement for fine aggregates improves the compressive strength of the concrete [9, 10]. Celik and Marar have studied the influence of various proportions of the dust on the workability and mechanical strengths of concrete. They concluded that incorporating an amount of 10% of the dust content in the mix has enhanced the compressive and the tensile strength of the concrete. But, exceeding this proportion, a reduction of the compressive and tensile strength was observed [11]. Also, the substitution of 10% of cement with quarry limestone powder leads an improve of the compressive strength of the cement pastes, which can be considered as a positive parameter in the use of quarry limestone powder in self-compacting paste [12].

It the other hand, the durability properties of the mortar and the concrete are improved when using CS in the same manner as it is for natural sand. The resistance of concrete made with limestone fines against sulfate and acid attack depends on the replacement level. The results found by Pipilikaki et al. [13] indicate that limestone cement mortars is strongly affected in sulfate environment and display the first macroscopic cracks after immersion in a 5% Na₂SO₄ solution for a period of 6 months. Besides, it was stated that sewer pipes made with limestone aggregates has a lifetime much longer than those made with siliceous aggregates [14].

Cement hydration products in the concrete a reaction with acid and its neutralization effect is found to be very limited. Tough, in concrete made with limestone aggregates, the reaction of the calcium carbonate with acid likely to neutralize the acid effect in local zones surrounded by the concrete. The local acid concentration and the rate of acid attack on concrete could be significantly reduced by the neutralization effect [14]. Moreover, mortars made with Portland limestone cement containing 20% of limestone exhibits a reduction of the carbonation depth and the total porosity of the mortar [15]. The incorporation of silica fume to limestone cement enhanced significantly its sulfate resistance; yet the absorption of sulfate and the formation of sulfate minerals in the cement paste were reduced [16, 17].

The main purpose of this study is to investigate the effects of limestone fines contained in the CS on the mechanical's properties, hydration heat and durability of mortars. These properties are studied with three types of cements. The experimental tests are conducted on mortar specimens made with various replacements of limestone fines of the CS.

2 Materials and methods

2.1 Materials

Three types of binders were used, namely an ordinary Portland cement C1 (CEM I 52.5), limestone cement C2 (CEM II/A-L 42.5) containing 10% of limestone and pozzolanic cement C3 (CEM II/A-P 42.5) containing 15% of natural pozzolan. The cements C1 and C3 are produced by the cement factory of Zahana, the cement C2 is supplied by the cement factory of Chlef. The replacement rates of mineral additions investigated for the binders C2 and C3 are those commonly used in by the cement factories. The CS used provided from Oued Fodda quarries displays a continuous size distribution of the particles with a maximum diameter of 4 mm, the percentage of grains less than 80 μ m (i.e. LF) is about 15%; the fineness modulus is 3.05, as shown in Fig. 1. The mineralogical composition of CS is schematized on Fig. 2. Chemical compositions and physical properties of the materials used are given in Table 1.



Fig. 1 - Particle size distribution of CS.



Fig. 2 - X-ray diffraction of limestone sand.

Table 1 - Properties of materials used.								
Cement C1	Cement C2	Cement C3						

	Cement C1	Cement C2	Cement C3	LF	
Chemical properties (%)					
SiO ₂	22.53	21.23	22.96	2.5	
Al ₂ O ₃	4.26	6.08	6.28	0.6	
Fe ₂ O ₃	5.38	4.67	3.62	0.9	
CaO	59.68	61.25	58.76	52.6	
MgO	1.28	1.61	1.13	0.5	
SO ₃	2.3	2.16	1.47	/	
K ₂ O	0.51	0.93	0.84	0.05	
Na ₂ O	0.14	0.22	0.17	0.02	
Free CaO	1.05	0.35	0.70	/	
Loss on ignition	1.6	2.34	5.07	41.9	
Mineralogical properties (%)					
C ₃ S	52.81	41.8	62.3	/	
C ₂ S	21.91	33.3	15.2	/	
C ₃ A	2.02	5.1	3.8	/	
C ₄ AF	16.36	10.7	8.8	/	
Physical properties					
Specific density (g/cm ³)	3.1	3.1	3.1	2.7	
Blaine fineness (cm ² /g)	3500	3700	3300	/	

Limestone fines used as partial replacement of sand are provided by sieving the CS below 80 μ m. In the mortars prepared, limestone fines are replaced by weight in CS at rates of 0%, 5%, 10% and 15%. The mortars were prepared with cement, crushed sand and water mass at proportions of 1:3:0.5. Table 2 specifies the contents of the mortars mix tested.

	Percentage of limestone fines							
	0%	5%	10%	15%				
Cement (g)	450	450	450	450				
Crushed sand (g)	1350.0	1282.5	1215.0	1147.5				
Limestone fines (g)	0	67.5	135.0	202.5				
Water (g)	225	225	225	225				

2.2 Mechanical strength tests

For each mix, three mortars sample of $40 \times 40 \times 160$ mm were made for testing the compressive strength. The samples were cast in steel moulds and stored in a moist room at 20°C for 24h. Demoulding process took place at one day after casting and specimens were kept in lime-saturated water at 20 °C until the testing day. This alkaline environment is considered as non aggressive, because it is rich in calcium resulted from the dissolution of lime in water, so the hydration take place in a normal manner, away from any leaching that may be due to acidic environments. Compressive tests are determined at the age of 2, 7, 28 and 90 days in accordance with EN 196-1 [18].

2.3 Hydration heat test

Hydration heat measurements were carried out according to NF 15-436 [19]. The method uses the Langavant Calorimeter as illustrated in Fig. 3. This semi-adiabatic method consists of calculating the heat released during cement hydration by use of a thermally isolated bottle. Since the exterior conditions were very important, the test was performed in a curing room at $20\pm2^{\circ}$ C. This temperature is the numerical difference between the temperature of the tested mortar and that of the inert mortar with over than three months of age. This temperature was used to evaluate the hydration heat generated by the test sample. Since the evolution heat was observed to be very low at later ages, the measurement tests are taken for 5 days. The variation of the temperature in the mortar versus time was measured immediately instantly after mixing. Also, for each mixture, the heat flow was measured along with the temperature of the mortar. The hydration heat of the mortar being the sum of the quantity of the accumulated heat in the calorimeter and the heat dissipates in the environment.



Fig. 3 - Test setup for the hydration heat test.

2.4 Durability test

For the durability tests, mortar specimens were prepared with the similar mixture proportions as in 2.3, and stored in water at 20 ± 2 °C for 28 days, before being immersed in several aggressive environments. The aggressive environments are: Hydrochloric acid (HCl) and sulfuric acid (H₂SO₄) with a purity of 38% and 98% respectively; sodium sulfate (Na₂SO₄) and magnesium sulfate (MgSO₄) having a purity of 99.5 and 99% respectively.

Sulfate attack tests were performed on mortars specimens immersed in 5% Na₂SO₄ solution and 5% MgSO₄ solution at laboratory temperature (20 ± 2 °C). The sulfate solution was changed every 15 days. The mortar samples were stored in the former environments for a period of one year. The sulfate attack was estimated throughout the measurement of the expansion on three mortar samples of $40 \times 40 \times 160$ mm in accordance to ASTM C-1012 [20].

Three cube mortar specimens of 40×40 mm were immersed in 1% hydrochloric acid and 3% sulfuric acid for each mixture. The attacked mortar samples were cleaned up using demonized water. The acid attack resistance was assessed through the measurement of the weight loss of the samples over 6 month's period according to ASTM C-267 [21].

3 Results and discussion

3.1 Compressive strength

The compressive strength, as a function of percentage of limestone fines contained in the CS with different types of cement is shown in Fig. 4.



Fig. 4 - Compressive strength mortar for different rates of limestone fines.

From this Figure, it can be noted that the compressive strength increases with age of mortars with and without fines. This is due to the growth of the hydration of cement. For mortars made with cement C1, a significant improvement of the compressive strength is noted compared to those of the control mortar at 2 days, as shown in Fig. 4(a). This increase is about 42%, 45% and 57% for the mortar containing 5%, 10% and 15% of limestone fines respectively. However, at 90 days, the improvement decreases to 3%, 10% and 23% for the mortar with 5%, 10% and 15% of limestone fines respectively. At early age, the fines act as filler allowing the filling of the gaps between the grains, accelerating cement hydration, leading to an increase of their strength [5, 22, 23]. At long-term, the effect of the fines becomes insignificant, which is probably due, to the dilution effect of limestone fines [20, 24-26].

Fig. 4(b) shows the development of the compressive strength of the mortars cement C2 for different fines content in the CS. The effect of the fine contents is more significant beyond 7 days and reached for 10% of fines an improvement in compressive strength of 25%, 42%, 25% and 18% at 2, 7, 28 and 90 days respectively. It seems that the cement C2 rich in limestone, does not allow more than 10% of the limestone fines in the CS.

It can be suggested that the sand incorporating high rate of fines are not recommended with this type of cement. It would be useful to reduce the content of the fines in their original quarries or to proceed to a washing of the sand before using it.

The results of the compressive strength of the pozzolanic mortars cement C3 are illustrated in Fig. 4(c). The presence of the fines in the CS with the natural pozzolan leads to the enhancement of the compressive strength around 30% at 28 days. At 90 days, this improvement is reduced at 10% for all the rates used. These fine powders contributed positively along with the presence of the pozzolan to activate the cement grains and to fill the granular distribution of the mixture. This has resulted in an increase of the compressive strength with incorporated limestone fines [27].

Table 3 shows the ratio between the compressive strength of the mortars for different rates of fines in the CS and the compressive strength of the mortars without fines. The compressive strength increases along with the rate increase of limestone fines used. It can be said that, at early age, the development of the strength is more significant than that at long-term.

		0%	5%	10%	15%
	2j	1	1.42	1.45	1.57
Comont C1	7j	1	1.25	1.27	1.35
Cement CI	28j	1	1.12	1.23	1.37
	90j	1	1.03	1.10	1.16
	2j	1	1.35	1.27	1.26
Comont C2	7j	1	1.31	1.25	1.20
Cement C2	28j	1	1.15	1.25	1.17
	90j	1	1.09	1.18	1.12
	2j	1	1.27	1.34	1.33
Comont C2	7j	1	1.09	1.21	1.28
Cement C5	28j	1	1.07	1.30	1.31
	90j	1	1.11	1.15	1.08

Table 3 - Ratio's values of the compressive strength of the mortars

Regarding the effect of each type of cement on the compressive strength of the mortars with and without limestone fines, it appears that the behavior of the cement C1 is more contributing to strength increasing than the other cements. Introducing the limestone fines in the mixture made with cement C1 is probably the reason for this positive effect. While incorporating limestone fines into the mixtures made with the other cements (C2 and C3) has less pronounced effect than that those made with cement C1. It is thought that the two former mixtures contain already a mineral addition.

3.2 Hydration heat

The hydration process characterizes the performance of the cement, in which it represents the mass conversion process between the different phases in the cement paste. It is possible to estimate the hydration kinetic of a mortar using the heat registered by a calorimeter. Fig. 5(a) shows the evolution of the total heat hydration in J/g of the mortar of cement C1 up to 5 days period. The limestone fines have a positive effect on the hydration particularly beyond 5% of replacement level. After 12 hours of hydration, mortar with 15% of limestone fines provides an increase of 66% of hydration heat, and then the heat curves of all the mortars become similar after 120 hours. It is noted that the maximum quantity of the heat released by the mortars made with cement C1 is approximately 200 J/g after 120 hours of hydration.

Fig. 5(b) shows the behavior of the mortars made with cement C2 under the heat effect. The heat hydration released by this mixture increases as the limestone fines content rises in the mixture. A content of 5% of fines in the mixture allows an increase almost of 25% of heat. This increase rate decreases with the evolution of the cement hydration. This may be explained by addition of the limestone fines to the fillers which do exist in the cement C2 and consequently contribute to the creation of new nucleation sites. This promotes the hydration of cement grains and generates additional heat [28].

The effect of limestone fines on the heat hydration of mixture made with cement C3 cementitious is shown in Fig. 5(c). The efficiency of the fines on the hydration of the pozzolanic cement C3 is insignificant for rate less than 10%, since the mortars release an amount of heat smaller than that released by the mixture with cement C2. It can be seen that introducing 15% of limestone fines in the mixture can cause a clear acceleration of the hydration, reflected by a release of 218 J/g of heat. This change in the amount of heat hydration may be attributed somewhat to the late synergistic effect in high fine contents. This is caused by the pozzolanic effect of the cement conditioned by the saturation of the interstitial solution of lime which governs the engagement of this reaction.

It is also worth to note that the highest heat hydration, around 250 J/g for 15% replacement rate, is released from the mixture made with cement C2, due to the presence of limestone in this cement.



Fig. 5 - Variation of heat hydration of mortar with rates of limestone fines of CS.

Table 4 presents the result of the final hydration heat, rate of hydration and time of peak for all mortars mixtures versus replacement rates of limestone fines. For all types of cement, mixture with 15% of fines indicates the highest peak of hydration rate as shown in Fig. 6. The increase in the intensity of the heat flow is more than 61%, 97% and 41% for cement C1, C2 and C3 respectively compared to that of mortar without limestone fines. The appearance of the peak is more noticeable for mixture with limestone cement C2, characterized by an accelerated hydration, as shown in Fig. 6(b). The peak hydration heat of the mixture made with cement C2 occurs well before the other blended cements, which indicates the most active mixture. It has been noticed during the testing procedure that all the mixtures approximately stop to release heat after 60 hours of hydration, while continuing the release of low heat until the end of the test, as shown in Fig. 6.

	Cement C1			Cement C2			Cement C3					
% des fines	0%	5%	10%	15%	0%	5%	10%	15%	0%	5%	10%	15%
Final hydration heat (J/g)	199.2	203.0	207.3	209.9	205.4	226.2	239.0	249.7	192.6	203.3	206.2	218.6
Peak of hydration rate (J/g/h)	8.28	7.30	10.32	13.38	9.53	13.26	12.11	18.80	8.94	6.28	9.31	12.6
Time of peak (h)	12	11.5	11.5	10.5	10	10	9.5	8.5	11.5	14	12	9.5

 Table 4 - Hydration heat results for all mortars mixtures.



Fig. 6 - Variation of heat flow of mortar with rates of limestone fines.

4 Durability

4.1 Sulfate attack

Fig. 7 shows the expansion of the mortar with different replacement rates of limestone fines immersed in 5% Na₂SO₄ solution. The cement mortars exhibit rapid expansion, which become more stable after 6 months period of immersion.

It can be noted in Fig. 7(a) that an excess amount of limestone fines in the mortar promotes the expansion in contrary to mixtures containing low proportions of limestone fines. Mortars containing 5 or 10% limestone fines preserve their sizes and have low expansion up to a period of three months of immersion. After this period, their expansions increase rapidly and approach to the expansion of the mixture without limestone fines. After one year of immersion, the replacement rate of 15% of limestone fines gives an increase of 10% of expansion. However, the replacement rate of 5% and 10% reduce the expansion about 10%.

When the mortar is made with cement C2, it was observed that limestone fines have a considerable influence on the observed expansion, as shown in Fig. 7(b). After one year of immersion, mortars containing 5%, 10% and 15% of limestone fines exhibit a decrease in the expansion about 10%, 30% and 70% respectively compared to that of mortar without limestone fines. These limestone fines combined with the particles cement may act as barrier for sulfates penetration.

The positive effect of the presence of limestone fines on the blended cement C3 starts to be apparent after three months of immersion in the sulfate solution, where amount of expansion is twice at 10% and 15% of limestone fines used as shown in Fig. 7(c). After one year, mortars with different replacement rates of limestone fines have almost similar expansion, about 40% of reduction is observed.

Regarding the expansion, the effect of introducing limestone fines is noticeable for all mixtures except the mixture made with cement C1 including 15%.



Fig. 7 - Evolution of expansion of mortars immersed in 5% Na₂SO₄ solution.

The results of the sulfate attack with the $MgSO_4$ solution are illustrated in Fig. 8. In which, the mortar with cement C1 is very influenced by the presence of fines in the CS. The effect of the limestone fines is favorable for reducing expansion in the mortars up to three months period. Within one year of immersion in the solution, the rate of 15% of limestone fines leads to an excessive expansion, exceeding 45% with the regard to the mortars without fines (Fig. 8(a)).

These findings are similar for mortar manufactured with cement C2, where the increase in the expansion in one-year period reached 50% for 15% of limestone fines, as illustrated in Fig. 8(b). The obtained results have good agreement with those obtained by Pipilikaki et al. [13].

When the cement is made with natural pozzolan (cement C3), the expansion of the mortars immersed in the $MgSO_4$ solution is reduced with the increase of the limestone fines, as shown in Fig. 8(c). For a short period of immersion, it has been remarked that the expansion is not influenced by the replacement rate of limestone fines. The combination of limestone fines and the natural pozzolan particles leads to a dense and less porous paste, which prevent the sulfate penetration into the mortar.



Fig. 8 - Evolution of expansion of mortars immersed in 5% MgSO4 solution.

4.2 Acid attack

Fig. 9 shows the weight loss of the mortars specimens immersed in hydrochloric acid solution (1% HCl). A decrease of the mortars weight is observed as the increase of the immersed time. During the first three months of immersion, it is noted that the effect of the limestone fines on the mortars weight loss is not significant for the three types of cement. Also, it can be seen that the limestone fines contribute to the increase of the weight loss of mortars regardless the nature of the cement used [11].

All mortars including less limestone fines are more efficient and less degradable in the acidic environment [29].

Fig. 10 illustrates the weight loss of the mortar specimens immersed in sulfuric acid solution (3% H₂SO₄). When mortars are immerged in the sulfuric acid solution, the effect of the presence of the fines is very significant at long-term especially for cement C1. The weight loss reaches 10%, 12%, 14% and 15% for mortars made with C1 cement containing 0%, 5%, 10% and 15% of limestone fines respectively.

When using cement C2 and C3, no clear effect of limestone fines in the sand is noticed before 4 months of immersion. However, after this period, the positive effect of the fines is more pronounced. Similar results were found by Chang et al. [14]. When comparing the effect of the two acidic environments, it appears that the results of the weight loss in mortars doesn't exceed 15% for the sulfuric acid, in contrary, those of the mortars stored in the hydrochloric acid indicate values more than 16%.



Fig. 9 - Evolution of weight loss of mortars immersed in 1% HCl.



Fig. 10 - Evolution of weight loss mortars immersed in 3% H₂SO₄

5 Conclusions

This paper presents the results of an experimental study carried out in order to assess the suitability of crushed sand mixed with different limestone fines rates to make a mortar with three types of cement. Based on the obtained experimental results, the following conclusions can be drawn:

According to the strength results, the use of CS with limestone fines to make mortar is acceptable with cement without mineral addition up to 15% of fines. In the contrary, sands incorporating high rate of fines exceeding 10% are not recommended when using cements blended with limestone or pozzolan.

The amount of the limestone fines in CS has a positive effect on the hydration heat of the mixtures particularly beyond 5% of replacement level. The total heat of hydration of cement blended with limestone increases with the proportion of fines and the greatest heat hydration is released, around 250 J/g for 15% replacement rate. Otherwise, the mixture of blended with pozzolan becomes sensitive only in the case of sands mixed with 15% of limestone fines.

The peak of hydration heat flow of the mixture blended with limestone fines occurs sooner than that of the others, indicating which is the most activated mixture. For all types of cement, mixture with 15% of limestone fines content indicates the highest peak of hydration rate.

With the respect to the expansion effect due to 5% Na₂SO₄ solution, the presence of limestone fines in CS is noticeable for all the mixtures except the mixture made with Portland cement and including 15% replacement rate.

In 5% MgSO₄ solution, the limestone fines in the CS can positively contribute to the reduction of the expansion of the mortars made with the blended cements and with less effect than the ordinary Portland cement.

The influence of limestone fines on the mortars weight loss in the hydrochloric solution (1% HCl) is very considerable regardless the type of the considered cement. Mortar made with CS including lower limestone fines contents is more efficient and less degradable in an acidic environment.

The positive effect of limestone fines in CS on the weight loss of the mortars made with the blended cements is well observed after 4 months of immersion in a sulfuric acid solution $(3\% H_2SO_4)$.

Finally, the analysis of the obtained results shows that the mixture most recommended for use in construction is C2 cement incorporating 10% of limestone fines due to its best performances.

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