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OPTIMAL CRITERIA OF ALGERIAN BLENDED CEMENT USING LIMESTONE FINES

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Abstract. The effect of substitution of Portland cement by limestone up to 40% as well as its fineness on the physico-mechanical properties of fresh and hardened cement pastes is studied. The binder was prepared by substitution of cement by limestone filler. Fillers were chosen of various particle sizes and with percentages from 5 to 40. Test results revealed that the replacement of Portland cement by the finest filler of limestone slightly decreases the consistency and the setting times (initial and final). The total porosity decreases and accordingly the compressive strength is improved with the content and fines of limestone. Although limestone has a little accelerating effect on the hydration process of Portland cement, but acts only as a filler reducing the porosity due to its compact structure, in which the compressive strength of the hardened cement paste is enhanced. The XRD and DTA analyses of samples cured up to 28 days showed that this amelioration is due to formation of new hydrated compounds. It is concluded that an addition of finely ground limestone filler only up to 15% gives a better strength.

Keywords: limestone fillers, cement paste, fineness, compressive strength, X-ray.

1. Introduction

In many developing countries, crushed limestone is the source of aggregates that can be used for concrete industry. The production of building materials is associated with limestone fillers which have a direct impact on the environment. In order to minimize this impact and reduce the quantities of this waste, it is desirable to make use of such limestone fines in the cement for reducing its production cost.

In Algeria, crushed limestone is the main source of aggregates used in concrete. However, such production is associated with high percentages of fines that makes these aggregates not acceptable in the concrete design mix. As a result, over 20% of such products could be not used and become hazardous to the environment.

This study aims at attempting to make use of these limestone fines in the production of blend cement. An experimental investigation is carried out to examine the impact of adding these fines to the physical-mechanical properties of the cement paste such as the water consistency, setting time, total porosity and compressive strength.

The use of Portland cement containing limestone is a common practice in European countries. The recent European Standard EN 197 identifies two types of Portland limestone cements (PLC): Type II/A-L containing 6–20% and Type II/B-L containing 21–35%. In addition, the inclusion of 5% of filler material that can be calcareous is accepted in all cements. During the 1990s, Latin American countries also moved in this direction and the use of limestone filler in Portland cements (PCs) was standardized. After many years of discussion, in 2004 the ASTM C150 standard specification for Portland cement was modified to allow the incorporation of up to a 5% mass fraction of limestone in ordinary Portland cements (ASTM Annual Book of Standards 2004). An extensive survey of the literature conducted by the Portland Cement Association (Hawkins *et al.* 2003) concluded that "in general, the use of up to 5% limestone does not affect the performance of Portland cement. This type of cement is formulated to achieve certain goals in technical, economic, and ecological fields. Among the technical benefits, there are the increase of early strength, the control of bleeding in concrete with low cement content, and the low sensibility to the lack of curing (Moir, Kelham 1997). The economic benefits are related to development similar to that of Portland cement at low production and investment costs per ton (Baron, Douvre 1987). The ecological advantages are the possibility to obtain cement with a significant reduction of CO_2 and NO_2 emissions per ton of cement manufactured, the conservation of fossil fuels and mineral resources.

The performance of limestone filler addition to Portland cement has been widely studied in cement pastes, mortars, and concretes (Baron, Douvre 1987; Tsivilis *et al.* 2002; Taoufik *et al.* 2008; Bentz 2006). In general, limestone fillers are incorporated for completing the granulometric distribution of cement decreasing the water

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demand, to enhance its granular packing factor and to block up capillary pores. Moreover, filler particles improve the hydration rate of cement compounds and consequently increases the strength at early ages (Bachiorrini 1985; European Committee for Standardization... 2000). From a chemical point of view, limestone filler does not have pozzolanic properties, but it reacts with the alumina phases of cement to form an AFm phase (calcium monocarboaluminate hydrate) with significant changes in the strength of blended cement (Guemmadi, Houari 2002; Bachiorrini 1985; Bonavetti 1976).

The main effects of limestone filler are of physical nature. It causes a better packing of cement granular skeleton and a large dispersion of cement grains (Helmuth 1980; El-Alfi *et al.* 2004). Furthermore, limestone filler acts as the crystallization nucleus for the precipitation of cement hydration (European Committee for Standardization... 2000; Gutteridge, Dalziel 1990). These simultaneous effects produce an acceleration of the cement grains hydration. In addition, the amount of limestone increase the heat of hydration and the free lime contents enhance slightly. On the other hand, the total porosity decreases and the compressive strength enhances at early age (Neville 1996). In general, research studies on limestone cement are focused in 3 areas: the first one is the effect of limestone on the cement performance (Guemmadi, Houari 2002; Escadeillas 1988; Soroka, Stern 1976); the second one dealt with the participation of limestone in the hydration reactions of clinker (Moir, Kelham 1997; Baron, Douvre 1987; Guemmadi, Houari 2002 and Vuk *et al.* 2001), while the third one is the production process and specifically the intergrading of clinker and limestone (Negro 1974; Gutteridge, Dalziel 1990 and Ingram, Daugherty 1992). These studies examined the limestone fines as fillers or inert material and did not go beyond the chemical reaction with the main chemical C_3A of cement. It is thus essential to study the behaviour of the limestone fillers considering their quantities and fineness on the cementing matrices in general and the influence on hydraulic cement pastes in particular.

2. Experimental program

A total of 25 mixes (Table 1) were designed to study all parameters involved, namely, the percentage of the fines, the median diameter of fines and the water to binder ratio, in addition. The reference mix was made without fines.

Table 1. Composition of blended cements

Paste	Content (g)				
	CEM I	Fillers			
		LF5	LF10	LF15	LF29
REF MC	100	0			
MCF5P05	95	5			
MCF5P10	90	10			
MCF5P15	85	15			
MCF5P20	80	20			
MCF5P30	70	30			
MCF5P40	60	40			
MCF10P05	95		5		
MCF10P10	90		10		
MCF10P15	85		15		
MCF10P20	80		20		
MCF10P30	70		30		
MCF10P40	60		40		
MCF15P05	95			5	
MCF15P10	90			10	
MCF15P15	85			15	
MCF15P20	80			20	
MCF15P30	70			30	
MCF15P40	60			40	
MCF29P05	95				5
MCF29P10	90				10
MCF29P15	85				15
MCF29P20	80				20
MCF29P30	70				30
MCF29P40	60				40

The addition of fines was taken with percentages ranging from 5 to 40%, also the median particle size of fines was ranging from 5 to 40 µm. The fines were designated by LF5, LF10, LF15 and LF29 respectively with the fillers diameter of 5 µm, 10 µm, 15 µm and 29 µm. Water to binder ratios was 0.24.

2.1. Materials used

Cement. The cement used is Algerian CPA-CEMI cement, (CEM I 42.5 RR according to EN 197/1) (European Committee for Standardization… 2000). From the clinker chemical and mineralogical characteristics, the laser gradation analysis reveals a distribution of the various particle-size ranges of grains between 1 and 100 µm. The mineralogical compositions (Bogue) of Portland cement was C_3S = 61.3%, C_2S = 15.9%, C_3A = 8% and C_4AF = 9.6%. The X-ray diffractogram of anhydrous cement is shown in Fig. 1.

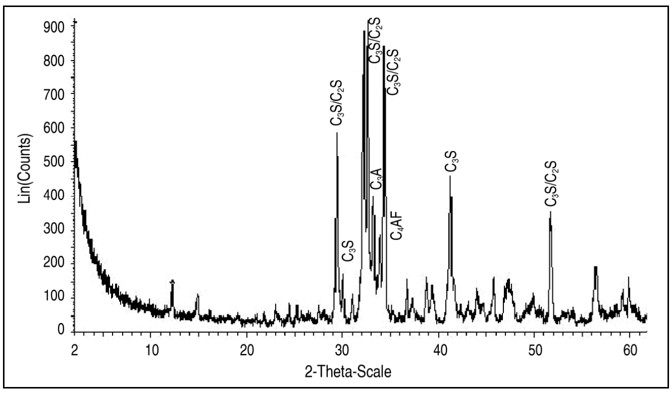

Fig. 1. X-rays diffractogram of anhydrous cement CEM I

Fillers. The fillers were limestone fines and provided by a local company in the eastern part of the country. Their content of $CaCO_3$ is about 98%, as the XRD analysis shows in Fig. 2. The fillers used are designated as F5, F10, F15, F29, where the subscript denotes the median diameter of the fines and their Blaine specific surface of 5400, 4500, 3500 and 2640 cm^2/g, respectively.

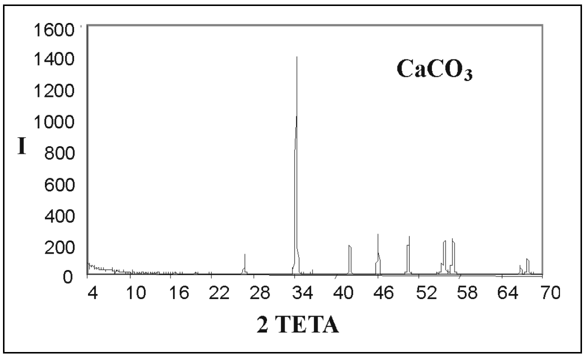

Fig. 2. X-rays diffractogram of filler

Portland cement (MC) and limestone filler (LF) were used in this investigation. The chemical composition obtained by X-rays fluorescence spectrometry, and the physical characteristics of these materials are reported in Table 2.

Table 2. Chemical composition and physical characteristics of material

	CEM I	Limestone filler			
		F5	F10	F15	F29
Chemical composition (%)					
SiO_2	20.01	0.58	0.58	0.58	0.58
Fe_2O_3	2.97	0.02	0.02	0.02	0.02
Al_2O_3	4.65	0.06	0.06	0.06	0.06
CaO	64.01	55.85	55.85	55.85	55.85
MgO	0.62	0.06	0.06	0.06	0.06
SO_3	2.15	0.07	0.07	0.07	0.07
K_2O	0.02	0.25	0.25	0.25	0.25
Na_2O	0.24	0.31	0.31	0.31	0.31
LI *	4.34	43.58	43.58	43.58	43.58
Physical characteristics					
SG**	3.2	2.7	2.7	2.7	2.7
SSB m/kg***	290	540	450	350	264
Retained on sieve (%)					
75 µm (#200)	1.8	7.5	6.5	3.2	1.6
45 µm (#325)	13.8	30.5	24.3	15.5	12.3

* Loss by ignition, ** Specific gravity, ***Fineness Blaine

2.2. Preparation of specimens and testing

The water of consistency was directly carried out by Vicat apparatus (Hewlett Lea's 1997; European Committee for Standardization... 2000). The cement pastes were mixed with the predetermined water of consistency, cast in prismatic moulds of 40x40x160 mm^3, cubic moulds of internal dimensions 25×25×25 mm^3 and then cured for 24 h. At this age, specimens were demolded and immersed in lime saturated water at 20±1 °C until test time of 2, 7, 28 and 90 days. Compressive strength was determined according to ISO 679 standard, on cubic pieces resulting from previous flexural strength test. Data represent the average values obtained from 3 flexural strength tests and 6 compressive strength tests. After mechanical tests, the mechanism of hydration was studied by the XRD (X-ray diffraction) and DTA (Differential thermal analysis). XRD analysis was performed using a Philips X-Ray Diffractometer Mod. P W 1390, with Ni-filtered, Cu–Kα radiation. DTA analysis was carried using NETZSCH Geratobau Selb, Bestell-Nr. 348472c at heating rate 10 °C/min up to 1000 °C.

3. Results and discussion

The results of consistency for the various cement pastes are plotted in Fig. 3. Generally, as the amount of limestone increases, the water of consistency decreases slightly that up to 15%. The finely ground limestone (F5 and F10) have a positive effect on the consistency, they play the role of plasticizer. Beyond this percentage these fines have a thickening effect. The coarse particles (F15 and F29) have a weak effect with means on consistency.

The influence of substitution rates of the finely ground limestone F5 and F10 are weak to mean son the initial and final of setting times in the interval of 0% to 20% (Figs 4 and 5). The setting time accelerates, i.e. more rapidly. This is essentially due to the presence of the predominant phase C_2A. Beyond this value, the coarse particles (F15 and F29) the time of initial and final of setting rise from 20 to 90 min, so fillers play a retarding role on the setting time.

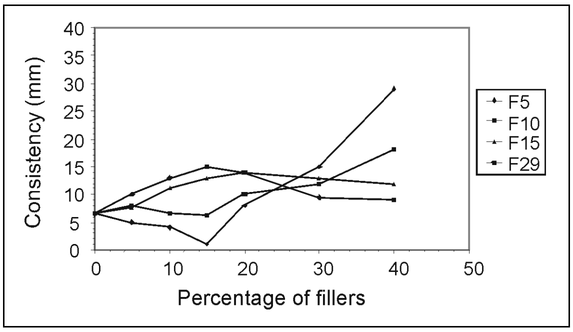

Fig. 3. Consistencies of the cement paste as function of percentage of substitution

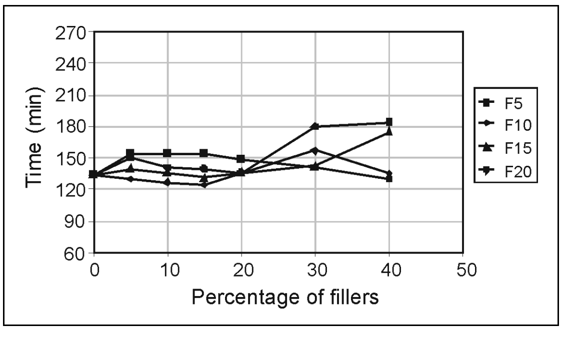

Fig. 4. Initial setting time

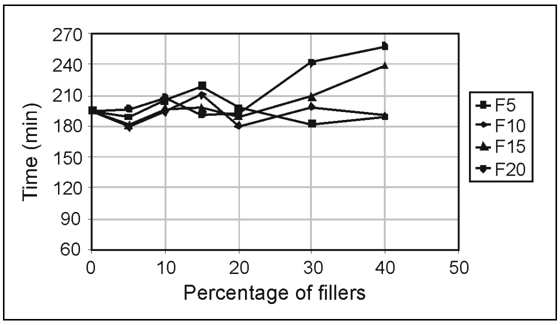

Fig. 5. Final setting time

The compressive strength values of the hardened cement pastes are reported in Table 3. The results indicate that the compressive strength of all hardened cement pastes increases continuously with time. This is mainly due to the continual formation of hydration products which always deposit inside the available pore structure leading to a decrease in the total porosity.

Therefore, the specific volume and bulk density must increase resulting in a clear improvement of compressive strength (Negro 1974). Moreover, the cement pastes containing limestone filler give a higher initial strength than those of the cement especially with finely ground limestone F5 (5400 cm^2/g); this behaviour increases with the amount of limestone filler but only up to 15%. This is essentially due to the acceleration effect of limestone filler related to the formation of calcium carboaluminate hydrate, which may be contributed to the overall increase in the rate of hydration. Also, the increased binding capacity of carboaluminate is likely due to its compact structure (Baron, Douvre 1987). Furthermore, the consumption of calcite in the formation of carboaluminate hydrates, the accelerating influence on the hydration of CA, the changes in the calcium aluminates hydrates between limestone filler and the cement constituents, in addition to the fineness of limestone are the different factors specific to the reactivity of limestone filler (Guemmadi, Houari 2002; Tsivilis *et al.* 2002). The incorporation of fines having a high specific area such as the finely ground limestone F5 and F10 (Fig. 6) considerably improves the compressive strengths especially for values of substitution of about 15%. Beyond this value, the resistance decreases by about 45% for the coarse particles F29 (2640 cm^2/g) to 40% of substitution.

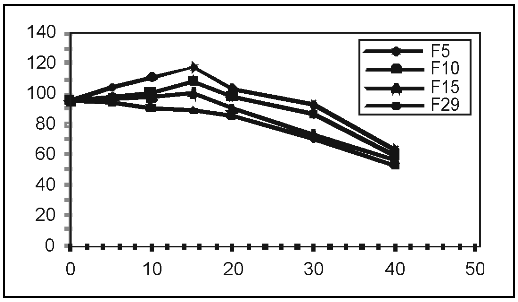

Fig. 6. Influence of filler concentration on 28-day compressive strengths

Table 3. Compressive and tensile strength of cement

	Compressive strength (MPa)				Tensile strength (MPa)			
Mixture	2d	7d	28d	90d	2d	7d	28d	90d
REF MC	65.5	80.0	95.0	105.0	4.2	5.2	6.3	6.8
MCF5P05	70.0	86.0	105.0	112.0	4.9	5.8	6.9	7.2
MCF5P10	72.0	88.0	110.5	118.0	5.1	6.2	7.2	7.8
MCF5P15	76.4	93.5	118.0	125.0	6.3	7.8	8.1	8.3
MCF5P20	68.0	85.0	103.5	110.5	4.6	5.4	6.5	7.0
MCF5P30	56.5	77.8	92.5	101.5	3.9	4.7	5.8	6.0
MCF5P40	48.3	58.0	62.5	67.7	3.6	4.0	4.9	5.2
MCF10P05	66.8	82.7	98.5	108.0	4.5	5.4	6.5	7.0
MCF10P10	67.8	83.4	100.2	110.5	4.8	5.7	6.7	7.1
MCF10P15	72.4	88.5	108.2	114.0	5.1	6.5	7.0	7.4
MCF10P20	66.0	81.8	97.8	105.8	4.3	5.0	5.8	6.8
MCF10P30	58.5	72.3	86.7	94.5	3.8	4.5	5.2	5.8
MCF10P40	45.6	52.9	59.2	64.5	3.2	3.7	3.9	4.4
MCF15P05	66.2	82.0	96.5	106.3	4.1	4.8	5.2	5.5
MCF15P10	67.9	85.4	98.3	107.9	3.8	4.2	4.7	5.1
MCF15P15	69.8	87.5	100.8	108.9	4.2	5.1	5.8	6.1
MCF15P20	62.3	78.5	90.2	98.5	4.0	4.9	5.5	5.8
MCF15P30	52.1	68.2	73.5	84.2	3.7	4.0	4.8	5.0
MCF15P40	42.5	50.3	56.8	61.2	3.1	3.7	4.4	4.7
MCF29P05	64.5	78.7	93.8	103.5	3.5	4.2	4.9	5.0
MCF29P10	63.2	76.0	90.5	101.0	3.1	3.8	4.2	4.8
MCF29P15	62.2	74.5	89.5	100.8	2.9	3.5	4.6	4.7
MCF29P20	60.0	72.8	85.3	93.2	2.8	3.4	3.9	4.2
MCF29P30	50.5	65.8	70.3	80.4	2.7	3.0	3.5	4.0
MCF29P40	40.5	48.2	52.8	55.7	2.5	2.9	3.1	3.8

The finest fillers (F5) improve a better resistance in compression. Choosing this type of fillers with an optimal value of 15% is particularly well highlighted.

The results of tensile strength of the hardened cement paste and reference mixes are also given in Table 3. As expected from their higher compressive strength, the hardened cement paste mixes MCF5P15 showed significantly higher tensile strengths than did the corresponding reference MC. Different mixes compared at the same time, reveal a little effect on tensile strengths. The evaluation of tensile strength shows a similar trend to compressive strength.

For the reference mix (100% cement), a very tightened porosity was noted in a marked principal peak, between 10 and 100 μm this peak is reduced by the incorporation of the very fine fillers F5 and F10 at a rate of 15% (Fig. 7).

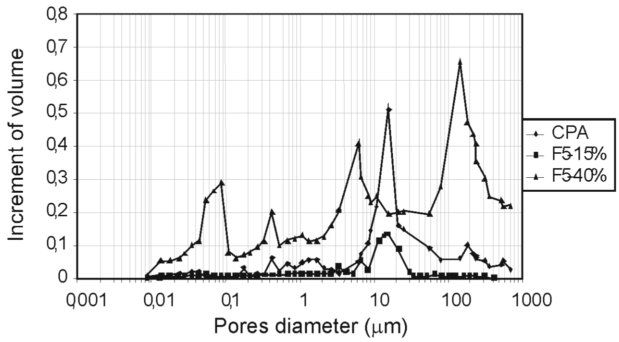

Fig. 7. Distribution of the pores according to the fine rate (F5 fillers; W/B=0,24)

So the hardened cement paste with 15% F5 filler seems as dense and homogeneous material, which explains the improvement of mechanical performance. The cement paste is more compact in case of the finest filler at 15%, but for the addition of F5 fillers to 40%; several families of pores were observed which point that this paste is more porous and have a porosity coarser than that observed in the paste with 15% (Guemmadi, Houari 2002; Escadeillas 1988).

Fig. 8 represents the XRD patterns of hydrated specimens of the cement with various proportions of limestone up to 28 days. It is clear that new peaks representing the formation of monocarboaluminates which appears after 7 days in the paste with 15% of F5 filler content is very important, compared to the paste with F29 filler. At a higher addition of 15% limestone, the carboaluminate hydrate phase is clearly detected which the content of limestone followed by a gradual disappearance of C3AH6. The limestone fillers are thus active chemically and their activity influences the mechanical resistance of the binder (Guemmadi, Houari 2002; Escadeillas 1988; Hewlett Lea's 1997; Voglis *et al.* 2005).

Fig. 9 shows the DTA thermograms of the same hydrated specimens. The endothermic peaks at the temperature range (100–135 °C) are due to the decomposition of the intermediate met stable products CAH_{10} and C4AH3, while those at temperature ranges 250-280, 405-435 and 698-767 °C are mainly due to the decomposition of stable hydrates C_3AH_6, and C_3A. $CaCO_3$. H_{12}, respectively. The newly formed hydration product tends to deposit into the pore structure of cement paste resulting in a strength gain. Therefore, the addition of limestone filler modifies the deterious effect of this conversion (Tsivilis *et al.* 2002;

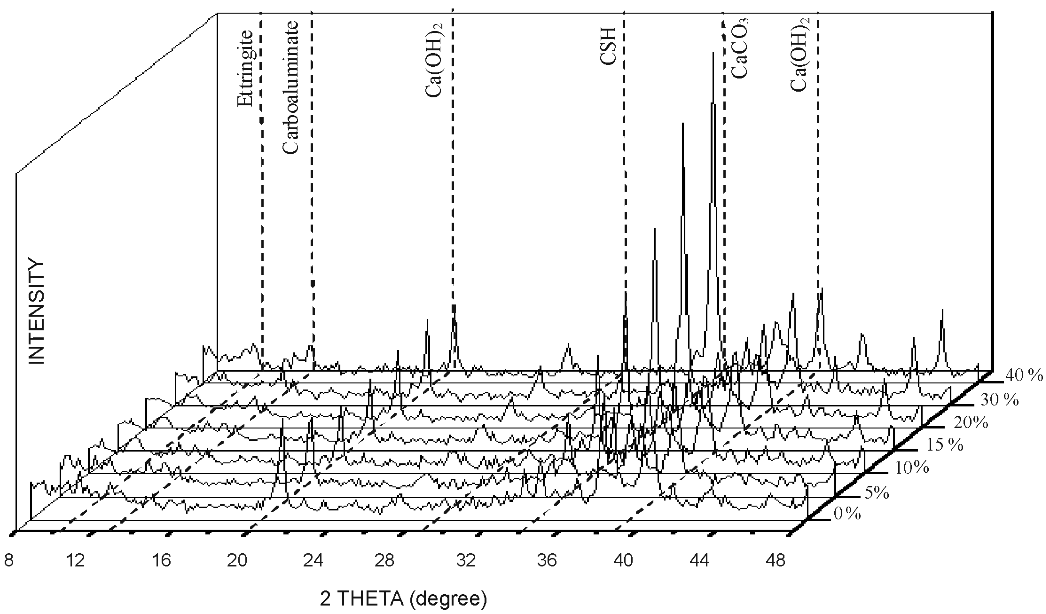

Fig. 8. X-rays diffraction of hardened cement pastes containing 5400 cm^2/g limestone filler

Negro 1974; Ingram, Daugherty 1992). The XRD patterns as well as DTA thermograms show a corresponding increase in the amount and intensity of carboaluminate. Hence, the results obtained are in a good agreement with those of XRD and DTA analyses.

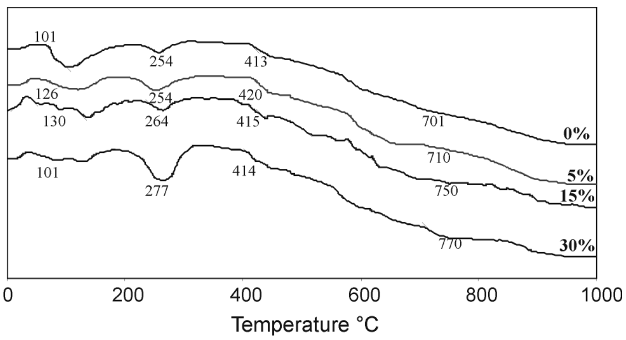

Fig. 9. DTA thermograms of cement pastes containing the percentage of fillers

4. Conclusions

In Algeria, limestone dust, which is produced in quarries operate disposal and environmental problems. There is a current interest, however, in the use of limestone as an additive to Portland cements. The addition of a small quantity up to 10% by weight of unspecified filler such as limestone has been a common practice in many European countries, such as France, which has permitted such an addition since 1979. The consumption of calcite, the formation of carbo-aluminates, the accelerating effect on the hydration of C_3A, C_3S, the change in the CSH and formation of transition zone between the filler and cement paste, are all facts specific of the reactivity of limestone fillers which are mainly conditioned by the fineness.

The various results obtained show well that the limestone fillers can play some roles:

- ✓ A filling role by off setting the cement lack of fine elements in the grading curve.
- ✓ A rheological role by their dispersing capacity on the grains of cement which results in a reduction in the water content at a constant hardness.
- ✓ A chemical and physical role by formation of carboaluminates, crystal nuclei (with acceleration of the hydration).
- ✓ The variation of smoothness of the fillers practically does not have a significant influence on the demand for water.
- ✓ The influence of the fillers is marked favourably when their content is lower than 25%.
- ✓ Porosity of the pastes decreases with the addition of the limestone fillers up to 15%.

The optimal fillers content that allows the obtaining of highest resistance is 15%. The addition of 15% of limestone to cement paste determines good mechanical properties as well as the lowest porosity in all periods.

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ALŽYRIETIŠKAS MIŠRUSIS CEMENTAS SU MALTU KALKAKMENIU

Z. Guemmadi, M. Resheidat, H. Houari, B. Toumi

S a n t r a u k a

Tirtos šviežios ir sukietėjusios cementinės tešlos, kurioje iki 40 % cemento pakeista įvairaus smulkumo maltu kalkakmeniu, savybės. Rišiklis buvo paruoštas dalį cemento pakeitus maltu kalkakmenio užpildu. Užpildo dalelės buvo įvairaus dydžio, o jų kiekis buvo keičiamas nuo 5 % iki 40 %. Tyrimai parodė, kad priedas leidžia sumažinti vandens kiekį, reikalingą tos pačios konsistencijos mišiniui gauti, taip pat cemento rišimosi pradžiai ir pabaigai paankstinti. Sumažėja cementinio akmens suminis poringumas ir atitinkamai padidėja stipris gniuždant cementinio akmens, kuriame yra kalkakmenio priedų. Nors kalkakmenio priedas nedaug pagreitina portlandcemenčio hidratacijos procesą, tačiau veikia kaip užpildas, sutankinantis struktūrą, dėl to labai padidėja sukietėjusio cementinio akmens stipris gniuždant. Bandinių, išlaikytų 28 dienas, rentgenostruktūrinė ir diferencinė terminė analizė parodė, kad pagerėjimas yra dėl susidariusių naujadarų. Apibendrinant galima teigti, kad 15 % malto kalkakmenio priedas turi didžiausią įtaką stiprumo rezultatams.

Reikšminiai žodžiai: kalkakmenio užpildai, cemento tešla, smulkumas, stipris gniuždant.

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