Global Advanced Research Journal of Engineering, Technology and Innovation (ISSN: 2315-5124) Vol. 5(3) pp. 040-044, July, 2016
Available online http://garj.org/garjeti/index.htm

Full Length Research Paper

Quality Control of Cement for Works in Water at High Tenor Sulphate-es

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Accepted 23 May 2016

In order to control the quality of cement for watery works at HTS (High Tenor in Sulphate), four samples of cement have been manufactured from a clinker-gypsum mixture ground into powder at intervals of time (30, 35, 45 and 55 minutes). Several analyses have been carried out afterwards on these samples: specific surface area, start making time and compressive strength after 28 days. The results of these analyses showed that the two cements with start making times of 165 and 114 minutes have respectively the specifics areas of 3130 cm^2/g and 3917 cm^2/g, these materials develop the best compressive strengths (54.40 MPa and 61.00 MPa respectively) in accordance with the norm XP P 15-319 of cement: [35 < 53.40 < 55] MPa and [45 < 61.00 < 65] MPa. These two cements are the finest and most reactive. Cement with start making time of 165 minutes is close to CPA ES 45 whereas cement with start making time of 114 minutes is rather close to CPA ES 55.

Keywords: Control quality, High tenor, sulphate, clinker, gypsum, cement standard.

INTRODUCTION

Cement is a hydraulic binder, that is a finely ground inorganic material which is wasted with water. This form a paste that becomes harden as the reaction goes on and during the dehydration process also. After curing it retains their strength and stability even under water (Papadakis and Vernuat, 1969). Thus it may agglomerate a number of inert materials (gravel, sand) to form mortar and concrete which have many applications like in buildings, bridges, dams, etc ... (Horkoss et al., 2011; Standing Committee of the Standardization of Building Materials, 1994).

The alteration of the concrete varies; they reach either the cement matrix or frames and sometimes both. Most of the concrete properties are related to the nature of the cement and its composition: rheology, mechanical strength, chemical resistance, durability, appearance ... where the interest to know the characteristics of its components (Achour, 2007).

Table 1. Clinker Certificate of Analysis

Certificate of chemical analysis							potential composition				Sadran index
SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	LOI	C_3S	C_2S	C_3A	C_4AF	
1.45	4.10	4.90	64.00	2.70	0.80	0.25	56.07	19.22	2.58	14.90	17.72

LOI = Loss on Ignition; C_3S = tricalcium silicate; C_2S = silicate bicalcium; C_3A = tricalcium aluminate; C_4AF = alumino-ferrite tetra calcium

Table 2. Composition of clinker + gypsum mixture

CPA ES LAB composition	Clinker	Gypsum	Total grinding
weight (g)	4800	200	5000
weight percent	96	4	100

Table 3. Summary of the BSS values and initial setting time

Cement samples	Grinding time (min)	% Clinker	% Gypsum	BSS (cm^2/g)	Setting time (min)
CIM 30	30	96	4	2977	170
CIM 35	35	96	4	3130	165
CIM 45	45	96	4	3778	120
CIM 55	55	96	4	3917	114

BSS: Blaine specific surface

Table 4. Summary of LOI, tenor on sulfate (SO_3), residues on the sieve of 100 µm for the four cement samples

Cement samples	%LOI	%SO_3 tenor	%Residue on the sieve 100 µm
CIM 30	1.65	2.69	3.6
CIM 35	1.92	2.67	2.9
CIM 45	1.97	3.11	2.5
CIM 55	1.98	3.44	2.3

Thus, a properly formulated concrete may suffer degradation from an attack by acidic agents or saline contained in the water with which it is in contact. Sulphates can react with the cement causing expansion and destruction. Some sources or sea waters contain enough sulphate to attack the concrete (Horkoss et al., 2016; Dupain et al., 2000).

Among the components of the cement without additions (tricalcium silicate or Alite (C_3S), dicalcium silicate or Belite (C_2S), tricalcium aluminate (C_3A), alumino ferrite tetra calcium (C_4AF) and calcium silicate or gypsum dihydrated ($CaSO_4.2H_2O$)), the most vulnerable component of the cement is tricalcium aluminate (C_3A.). Cement resistant to sulphate will have a low amount of C_3A (Cimbeton, 2005 and 2006; Horkoss et al., 2011). It is therefore necessary to know the quality of different types of cements suitable for various industries.

In order to develop appropriate cements in needs and to satisfy user demand, a study on the quality control of cement for works in water at high tenor sulphate was presented in this work, the influence that high tenor sulphate content can have on the quality of cement manufactured in the Lafarge cement plants in Cameroon in accordance with the XP P 15-319 standard was also presented (Afnor, 1995).

MATERIALS AND METHODS

Sampling Condition and mixture grinding (clinker + gypsum)

This study was made by using some technical analyses used by the laboratories of Lafarge Cement of Cameroon (CIMENCAM). This cement plant was a grinding plant;

Table 5. Values of resistance to wet compression at 28 days (**Rc_{28d}**) of the four cement samples

Cement samples	BSS	Rc_{28d} (MPa)
CIM 30	2977	50.20
CIM 35	3130	53.40
CIM 45	3778	59.30
CIM 55	3917	61.00

BSS: Blaine specific surface area; Rc28d: compressive strength at 28 days

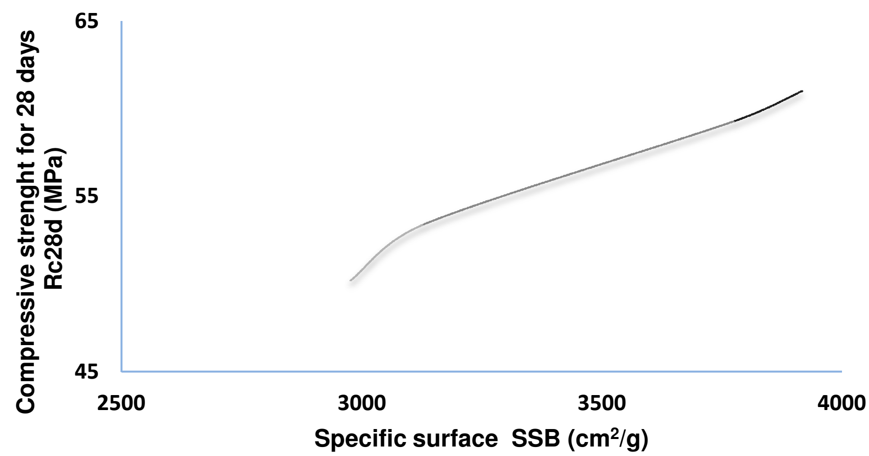

Figure 1. Evolution of resistance depending on the specific surface area

clinker and gypsum were imported and were the main raw material components used.

To prepare cement without addition for work in water at high tenor sulphate (CPA ES LAB), a clinker ship ALTHEA from Alexandra (Egypt), whose Sadran index 17.72 in line with the standard XP P 15-319 was used. The composition of the clinker is given in Table 1.

For sampling, about 25 kg of clinker in bulk were taken by ship and submitted to the quartering to be the average sample vessel. This means sample and gypsum were separately crushed and sieved using a 315 µm mesh sieve.

Both sieves were subsequently mixed to form the total sample to be ground. The composition is given in Table 2.

The crushing operation was made in the laboratory, staggered in time, and four cement samples labeled CIM 30, CIM 35, CIM 45 and CIM 55 according respectively to different grinding times 30, 35, 45 and 55 minutes were obtained. The samples were analyzed immediately to avoid any influence of storage and moisture.

Laboratory analysis

Several physical-mechanical analyses were conducted on the four previous samples of cement prepared in the laboratory. These tests were made on the anhydrous cement on hardening cement paste and mortar. On the anhydrous cement, measuring the fineness of grind was made in accordance with the recommendations of EN 196-6 standard. The tests were conducted on hardening cement pastes to monitor their standard consistency in accordance with EN 196-3. Measurement testing of normal mortar compressive strengths were carried out according to the EN 196-1standard.

The loss on ignition (LOI) with calcination (decarbonization) was performed to determine the percentage of volatiles cement (carbon dioxide, water) according to EN 196-2 standard. The latter (EN 196-2) was also followed for the gravimetric determination of sulphate (SO_3 rate), in order to regularize the cement.

RESULTS AND DISCUSSION

The experimental results of the Blaine specific surface (BSS) and the initial setting time are reported in Table 3.

These results show that the surface area increases with the cement grinding time. This is due to the fact that more the mixture is ground, the more the particles decrease in sizes. The specific surface area becomes large and cement is fine, this fineness is an important feature because during the mixing, more cement surface is in contact with water and the greater the hydration will be rapid and complete.

The introduction of water to anhydrous cement grains, followed by kneading leads to the immediate formation of hydrate crystals in the periphery of the cement grain. The hydration occurs, but more slowly when there is enough gypsum (not combined in C_3S). Once the gypsum is consumed, hydration accelerates. Hydrated bridges begin to connect the cement grains together; this is the beginning of initial setting (Cimbeton, 2006). Unlike the specific surface area, the time of initial setting decreases with the cement grinding time, and depends on several parameters. It varies in particular according to the chemical composition and fineness of grind of the studied cement. Thus, the larger the specific surface of the cement, the greater the reactivity is high, which causes a decrease of setting time (Horkoss et al., 2011).

The different results obtained for the determination of the percentage of the loss on ignition (LOI), the tenor on sulfate (SO_3) and the residues on the sieve of 100 µm for the four samples of cement are shown in Table 4.

Table 4 shows that the four cement samples can be used for work in water at high tenor sulphate (HTS). For each cement sample, the percentage of loss on ignition (LOI) is less than 3% and sulphate contents (%SO_3) less than 3.5%, respecting therefore the XP P 15-319 standard.

The values of resistance to wet compression at 28 days of the four cement samples are summarized in Table 5.

Table 5 shows that the resistance increases with the specific surface area. It is thus appeared that the specific surface area plays a significant role in the resistance: the more the cement is small, the more the concrete is resistant. Figure 1 represents the evolution of compressive strength for 28 days according to the specific surface area.

Figure 1 shows that the compressive strength at 28 days increases with the specific surface area. This increase could scale up to 65 MPa, value on which an early stage is observed. This is due to the fact that 65 MPa is the last strength class of the CPA ES.

According to the results and the specifications of the XP P 15-319 standard, the cement samples label CIM 30 and CIM 35 approach a CPA ES 45; their resistances are in the range of [35-55] MPa. The cement samples CIM 45 and CIM 55 are rather closer to a CPA ES 55 because their resistances meet the range of [45-65] MPa.

CONCLUSION

This study shows that the quality of cement depends on the nature and quality of raw materials used, the respect of the procedure of preparation and the mastering of analyses methods. The clinker and gypsum which are raw materials must have certain levels in accordance with the standard ($C_3A \leq 5\%$ for clinker; $SO_3 \leq 3.5\%$ if $C_3A \leq 3\%$). This is due to the fact that hydrated aluminate cements are attacked by sulphate water, it is the same for sulphate ions from the gypsum; therefore causes swelling of the concrete when they are in excess.

According to the XP P 15-319 standard of cement and results obtained from the four cement samples at different grinding times, cement samples CIM 30 and CIM 35 approach a CPA ES 45 while samples CIM 45 and CIM 55 are rather closer to a CPA ES 55. In addition, cement samples CIM 35 and CIM 55 have the best quality because they have the highest resistance in their class (53.40 MPa and 61 00 MPa) in accordance with the standard (XP P 15-319). A mixture (clinker + gypsum), ground at different times provides different CPA ES classes, thus reducing the grain size gives finer cement and more resistant to sulphate ions.

To avoid attacks by waters at high tenor sulphate, the constitution of concrete and the nature of the cement used (CPA ES 45, ES 55 CPA) are important. Taking into consideration that the resistance is directly proportional to compactness, the concrete with the highest strength is also the most resistant to aggressive agents, thus more sustainable otherwise concrete is more durable as its porosity is low.

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