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REACTIVITY OF GRANULATED BLAST FURNACE SLAG

ABSTRACT

The Algerian iron and steel complex of El Hadjar, near the city of Annaba, produces a granulated blast furnace slag (GBFS) mainly used by the local cement factories as an addition to clinker of up to 30% as maximum content, for manufacturing a compound CEM II type cement (Algerian Standard).

With the aim, on the one hand, of limiting the use of high clinker content in Algerian cement plants and replacing it with other constituents such as granulated blast furnace slag and, on the other hand, to use this slag as an addition to local concrete, we have attempted to characterize it according to its degree of reactivity.

The use of chemical activity indexes and caustic soda tests lead to disappointing results that are contradicted by the results obtained on mortar within the scope of the standard determination of a hydraulic efficient index. The results obtained indicated that the classification of El Hadjar slag strongly depends on its degree of fineness; it also has weak short-term activity, denoting a slower kinetics of reaction compared to classical slag. These results permit us to recommend a minimal Blaine of 3500 cm²/g for this slag.

A more intensive use of this slag in cement and concrete is undoubtedly possible, but this must be demonstrated by conducting tests on mortar and validating by a study on concrete.

1. INTRODUCTION

The iron and steel complex of El Hadjar is the largest integrated iron and steel plant in the east region of Algeria. It is located 12 km south of the city of Annaba. It has a steel making capacity of 2 millions tones a year. It also has its own captive iron ore mines and dedicated port facilities, which are connected by rail to the plant.

As is well known, cement, which accounts for about 20% of the volume of concrete, is its most expensive constituent. The huge quantities of granulated blast furnace slag produced by this plant have mainly been used by the local cement factories to develop a CEM II type cement (Composed Portland Cement).

A research project was carried out by the Laboratory of Civil Engineering at Annaba University, Algeria, in collaboration with the

Laboratory of Materials and Durability of Constructions of Toulouse, France, to valorize this mineral waste in construction materials.

The main objectives are aimed at developing the use of such slag in cement manufactured in the east of Algeria. In other words, we want to develop CEM II, and even CEM III type cements, and to study the feasibility of using this granulated slag as an addition to the concrete produced from local materials. This is slightly practiced not only in Algeria but also in France.

Granulated blast furnace (GBFS) slag has been used for many years as a supplementary cementitious material in Portland cement concrete, either as a mineral admixture or as a component of blended cement. The addition of slag to the clinker or to the concrete permits the obtaining of a concrete which can set and harden with a lower release of heat than standard concrete and behave better in an aggres-

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KEY WORDS

- Granulated slag,
- reactivity,
- activity indexes.

sive environment, such as in a saline or sulfate attack. These two types of attack need to be studied and guidelines need to be developed for using the slag in various conditions of exposure in Algeria.

Initially, the work focused on studying the slag of El-Hadjar by characterizing it according to its chemical and physical properties and reactivity aspects, and identifying the parameters which influence this reactivity. Various methods which characterize the slag's reactivity were reported in this study as background for the main objective, which was aimed at finding the degree of hydraulic reactivity of El-Hadjar slag as well as quantifying the influence of the fineness of the grinding on this reactivity.

2. REVIEW OF THE EVALUATION METHODS OF THE HYDRAULIC REACTIVITY OF THE GRANULATED SLAG

Several evaluative methods exist, but only the method which uses the measurement of the mechanical strength of granulated slag on mortar has been standardized [NF EN 15167-1, (2000)].

2.1 Chemical indexes

The chemical indexes are calculated from the chemical composition of the slag and are supposed to be connected to its hydraulic activity. Many indexes, as shown in Table 1, have been proposed by several authors [DEMOULIAN E., et al.(1980), SMOLCYCK H.G., (1980), ALEXANDRE J. and SEBELAU J.L. (1986)], but they often contradict each other and are not always relevant, particularly compared to our aim, which is to predict the reactivity of slag from its chemical composition. DEMOULIAN et al. [DEMOULIAN E., et al, (1980)], reviewed and tested many indexes by looking for those which gave the best correlations with the compressive strengths.

They noted that the indexes I_8 , I_9 , I_6 , I_{10} , and I_1 have, in descending order, the strongest coefficient (r) of correlation: where r varies from 0.91 to 0.85 for two days of compressive strength, from 0.82 to 0.78 for those of 7 days, and from 0.84 to 0.76 for 28 days. These indexes were used to characterize the granulated slag of El-Hadjar.

2.2 Sodium hydroxide test

This method, which is not standardized, consists of preparing 4 x 4 x 16 cm prismatic samples from standard mortar [NF EN 196-1, (2006)], the water being replaced by 200 gr of concentrated solution of NaOH, which was diluted with water to form one liter of diluted solution, and the binding part being made up of ground granulated slag. The solution / slag ratio is equal to 0.5. All the test samples are demoulded and tested in compression with 6 and 24 hours of hardening. The compressive strength values must be in a range between 7 and 8 MPa after 6 hours of hardening and between 12 and 15 MPa after 24 hours.

2.3 Determination of the hydraulic efficiency index h

The hydraulic capacity is conventionally estimated using the hydraulic efficiency index h [French standard NF P 18 – 506 (1992)]. This index is defined as a ratio, at a given age, of the compressive strength of a mortar in which the binding material is composed of 50 % slag and 50 % Portland cement, which were obtained at the same age as the reference mortar samples made from the same cement. The reference cement has a content of C_3A ranging between 6 and 10 % and a maximal SO_3 content of 3 % as prescribed by the standard [NF P 18–506 (1992)]. According to the numerical value of h , the slag can be classified according to NF P-18 506 into three classes (h_1 , h_2 , h_3) with increasing reactivity as shown in Table 2.

Tab. 1 Chemical indexes used to estimate the reactivity of GBFS.

Formulas	
$I_1 = \frac{CaO}{SiO_2}, I_2 = \frac{CaO}{Al_2O_3}, I_3 = \frac{SiO_2}{Al_2O_3}$	$I_9 = 20 + CaO + Al_2O_3 + 0.5 MgO - 2SiO_2$
$I_4 = \frac{CaO + MgO}{SiO_2}, I_5 = \frac{CaO + MgO}{SiO_2 + Al_2O_3}$	$I_{10} = \frac{CaO + MgO + 0.66Al_2O_3}{SiO_2 + 0.33Al_2O_3}$
$I_6 = \frac{CaO + 0.56Al_2O_3 + 1.4MgO}{SiO_2}$	$I_{11} = \frac{CaO + Al_2O_3 + 0.5MgO + CaS}{SiO_2 + MnO}$
$I_7 = \frac{CaO + 1.4Al_2O_3 + MgO}{SiO_2 + 0.33Al_2O_3}$	$I_{12} = \frac{CaO + 0.33Al_2O_3 + MgO}{SiO_2 + 0.66Al_2O_3}$
$I_8 = \frac{CaO + Al_2O_3 + MgO}{SiO_2}$	$I_{13} = \frac{CaO + Al_2O_3 + 10}{SiO_2 + 10}$

Tab. 2 Classification of the hydraulic efficiency index according to the mechanical test results.

index		28 days
h_1	–	> 0.60
h_2	> 0.60	> 0.75
h_3	> 0.70	> 0.85

3. EXPERIMENTAL

3.1 Characterization of Materials

3.1.1 The granulated slag of El-Hadjar

The granulated slag of El-Hadjar is a by-product of the steel complex located near the city of Annaba. It is formed from the non-ferrous part of the ore, coke ash and flux. The iron ore comes from two deposits (BOUKHADRA and EL OUEZNA), situated in the south east of Algeria.

The granulated slag of El-Hadjar is presented in the form of spherical grains. Its particle sizes range from 0 to 5 mm. It has a clear yellow color and a porous structure. Its bulk density (r_{app}) is 1000 kg/m³, whereas its real density (r_{abs}) is 2800 kg/m³. The chemical composition of the slag resulting from the control and the follow-up of manufacturing carried out by the company is given in Table 3. The last line in the table corresponds to the average composition of the granulated slag of another slag produced as given by DEMOULIAN [DEMOULIAN E., et al, (1980)].

Tab. 3 Chemical composition of the granulated blast furnace slag of El Hadjar.

Year	Chemical composition (%)								
	SiO ₂	CaO	MgO	Al ₂ O ₃	FeO	S	MnO	K ₂ O	TiO ₂
1987	39.2	39.5	9.5	8.8	0.8	1.10	–	–	–
1995	39.3	39.4	6.0	8.2	0.7	0.07	2.4	–	–
2000	40.1	42.2	4.7	6.0	2.0	0.15	2.6	1.2	1.2
[1]	33.5	42.2	6.0	13.3	1.2	0.94	0.6	0.7	0.6

According to the European standard NF EN 197-1 [NF EN 197-1, (2001)], the granulated blast furnace must contain at least two thirds by mass of CaO, MgO and SiO₂; the mass ratio (CaO + MgO)/(SiO₂) must be higher than 1. These ratios are always verified. As shown in Table 3, we note that the slag used has a high percentage of silica and a small percentage of alumina compared to the average chemical composition of the slag recommended by DEMOULIAN [DEMOULIAN E., et al, (1980)].

According to the French standard NF P 18-506 [NF P 18-506 (1992)], concerning the use of slag as an addition to concrete, the nature of the slag is evaluated by the modulus $I = C.A$, which is the product of the percentage of C (CaO) and A (Al₂O₃). The found values range between 253 and 345, which classify this slag in the category 1 ($C.A < 425$).

An X-ray diffraction analysis of the blast furnace slag was carried out on a Siemens D 5000 diffractometer type, using a cobalt anticathode (Co Ka, $\lambda = 1.789 \text{ \AA}$); the scan is taken between 10 and 70° (2 θ) at increments of 0.02° and a count of 12 seconds. These angles were selected because an important reflection for most minerals and other relevant impurities lies in the region. The count time was selected as 12 seconds to enable the analysis to take place over a reasonable period. Figure 1 shows a diffractogram of the slag, which is typical of an essentially vitreous material. In fact, the vitreous slag present in X-ray diffraction patterns contains one or more broad and diffuse halos: these halos are the images of a local disorder, which existed in the liquid and was solidified by hardening. In addition to the vitreous fraction, the slag presents small quantities of crystallized minerals, probably calcite (CaCO₃, $d = 3.035 \text{ \AA}$), metallic iron (Fe, $d = 2.021 \text{ \AA}$), traces of Gehlenite and/or Akermanite. Table 4 shows the mineral formed in this slag when the material is slowly cooled [MALEK A, (1998)].

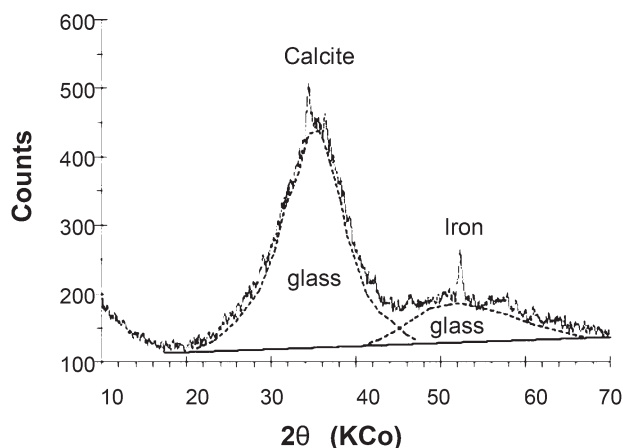


Fig 1 X-ray diffraction diagram of granulated furnace slag of El Hadjar.

Tab. 4 Mineralogical composition of slowly cooled GBFS of El-Hadjar which was analyzed by [MALEK A, (1998)].

N°	Minerals	Designation	Chemical formulas
1	Gehlenite	C ₂ AS	2 CaO Al ₂ O ₃ SiO ₂
2	Akermanite	C ₂ MS ₂	2 CaO MgO SiO ₂
3	Merwinite	C ₃ MS ₂	3 CaO MgO 2 SiO ₂

It is generally recognized that the reactivity of GSBF has been primarily governed by three material properties, i.e., chemical composition, degree of vitrification or glass content, and fineness when ground. The glass content of a slag is influenced by how rapidly it is cooled from the molten state. Rapidly cooled molten slag solidifies as a supercooled liquid or glass. This glassy material, when finely ground and in the presence of a suitable activator, will hydrate to form a stable solid product similar to the hydration products of Portland cement [REGOURD M.; et al., (1980)]. To be used in a cement factory, the granulated blast furnace slag must contain at least 2/3 of the slag and must be in a vitreous state, according to the French standard NF EN 197-1 [NF EN 197-1, (2001)]. According to an X-ray diffraction diagram, we have aimed at evaluating the percentage of the vitreous phase.

The method used consists of withdrawing 100% of the sum of the contents of the crystallized phase (CaCO_3 and Fe). The quantitative and chemical compositions of the crystallized phases compounds are given by XRD analysis [KLUG H.P. and ALEXANDER L.E. (1974), CYR M., et al., (1998)]. The iron quantity is calculated from the chemical analysis of the slag presented in Table 3 by using equation 1.

$$\chi_{\text{Fe}} = \frac{M_{\text{Fe}}}{M_{\text{FeO(mol)}}} \cdot C_{\text{FeO}} \cdot 100\% = 1,6\% \quad (1)$$

Where

- χ_{Fe} : Metal quantity of iron in the sample (%).
- C_{FeO} : Quantity of iron oxide in the granulated slag (Table 3: 2.0%)
- M_{Fe} : Atomic mass of iron (55.8 g/mol)
- M_{FeO} : Molecular mass of Fe O (71.8 g/mol)

• The calcite quantity is calculated according to the XRD results using the quantitative analysis as shown in equation 2.

$$\chi_{\text{Calcite}} = \frac{I_{\text{Calcite}} \cdot \mu_{\text{slag}}}{I_{\text{pure calcite}} \cdot \mu_{\text{pure calcite}}} \cdot 100\% = 0.7\% \quad (2)$$

Tab. 5 Chemical composition of the clinker (%).

Oxydes	CaO (free)	Al ₂ O ₃	Fe ₂ O ₃	SiO ₂	MgO	K ₂ O	Na ₂ O	Cl	SiO ₂ insoluble	Loss of ignition	SO ₃
Content (%)	65.7	5.2	2.7	21.7	0.7	0.4	0.7	0.01	0.3	0.3	0.6

Tab. 6 Mineralogical composition estimated in the clinker.

Minerals	Designation	Chemical Formula	Calculated content
1 Tricalcium Silicate	C ₃ S	3 CaO SiO ₂	58.2
2 Dicalcium Silicate	C ₂ S	2 CaO SiO ₂	18.5
3 Tricalcium Aluminate	C ₃ A	3 CaO Al ₂ O ₃	9.3
4 Alumno-Ferrite Phase	C ₄ AF	4 CaO Al ₂ O ₃ Fe ₂ O ₃	8.2

Where

- χ_{Calcite} : Calcite quantity in the tested sample (%)
- I_{Calcite} : intensity of the calcite line (3.03Å) in the slag diffractogram ($I_{\text{Calcite}} = 3$ counts)
- $I_{\text{pure calcite}}$: intensity of the pure calcite line (3.03Å) of a standard sample ($I_{\text{pure calcite}} = 427$ counts).
- m_{Slag} : mass absorption coefficient of the granulated slag ($m_{\text{slag}} = 118 \text{ cm}^2/\text{g}$)
- $m_{\text{pure calcite}}$: mass absorption coefficient of the pure calcite ($m_{\text{pure calcite}} = 113.4 \text{ cm}^2/\text{g}$)

• The quantity of the vitreous phase in the blast furnace slag may then be estimated at approximately 97% by considering the traces of Gehlenite and/or Akermanite).

3.1.2 Cement

The cement used is an ordinary Portland cement (CPA-CEM I) in conformity with the Algerian standard NA 442 [NA 442, (1994)]. It is the only OPC type cement produced by the cement plant of Hadjr Essoud without any additions. It has an apparent bulk density (ρ_{app}) of 1108 kg/m³ and a real density (ρ_{abs}) of 3150 kg/m³. According to the Algerian standard [NA 442, (1994)], the minimal compressive strength guaranteed at 28 days is 35 MPa. Table 5 gives the chemical composition of the clinker, which is the principal component of cement, whereas Table 6 gives its mineralogical composition calculated according to the corrected formulas by BOGUE [BARON J. and OLLIVIER J.P. (1997)].

3.1.3 Sand

The sand used in this study to prepare the mortar samples was siliceous; it is clean and fine sand. This natural sand, which was taken from a river, has a fine granulometry of 0 / 2.5 mm, and a fineness modulus of $M_f = 2.04$, an apparent bulk density ρ_{app} of 1450 kg/m³, and a real density ρ_{abs} of 2700 kg/m³.

Tab. 7 Calculated values of the different chemical indexes.

Indexes	Calculated values		Optimal values found in the literature
	(1)	(2)	
$I_8 = \frac{\text{CaO} + \text{Al}_2\text{O}_3 + \text{MgO}}{\text{SiO}_2}$	1.31	1.84	> 1 [14]
$I_9 = 20 + \text{CaO} + \text{Al}_2\text{O}_3 + 0.5 \text{MgO} - 2\text{SiO}_2$	-9.8	11.57	$12 < I < 20$ [13]
$I_6 = \frac{\text{CaO} + 0.56\text{Al}_2\text{O}_3 + 1.4\text{MgO}}{\text{SiO}_2 + \text{Al}_2\text{O}_3}$	1.12	1.54	> 1.65 [4]
$I_{10} = \frac{\text{CaO} + \text{MgO} + 0.66\text{Al}_2\text{O}_3}{\text{SiO}_2 + 0.33\text{Al}_2\text{O}_3}$	1.23	1.82	-
$I_1 = \frac{\text{CaO}}{\text{SiO}_2}$	1.04	1.26	Max 1.4 [14]

(1) According to the chemical composition of the El Hadjar Slag for 2000. (2) According to the chemical composition as given by DEMOULIAN [1].

3.2 Experimental testing of the hydraulic activity of slag

The tests described in paragraphs § 2.2 and 2.3 were carried out on this slag in order:

- To determine its degree of hydraulic reactivity
- To quantify the influence of the grinding fineness on its reactivity.

4. RESULTS AND DISCUSSION

4.1 Results

Table 7 gives the chemical indices I_8 , I_9 , I_6 , I_{10} and I_1 , for the slag of El Hadjar in comparison with the indexes obtained from the average chemical composition given by DEMOULIAN [DEMOULIAN E., et al (1980)]. This table also gives the optimal values of these indexes, as specified in the literature from various sources. Tables 8 and 9 present the results of a caustic soda test for the three grinding finenesses of slag (2850, 3570 and 4620 cm²/g). Lastly, Table 10 gives the hydraulic efficiency indices h of the ground

slag with four of Blaine's fineness tests (2480, 2900, 3470 and 4150 cm²/g).

Tab. 8 Caustic soda test: Results of the compressive tests of the activated slag mortar.

N°	Specific surface area of Blaine (cm ² / g)					
	2850		3570		4620	
	Time of hardening (hours)					
	6	24	6	24	6	24
	Compressive strength, s _c (MPa)					
1	1.03	3.75	3.06	3.12	3.13	4.50
2	1.25	3.37	2.5	3.12	3.13	2.87
3	1.25	3.37	2.44	3.75	3.25	3.88
4	1.03	3.37	2.38	4.00	3.06	4.62
5	1.15	3.15	2.00	3.88	3.25	4.62
6	1.15	3.15	—	4.00	—	4.88
σ _{average values}	1.14	3.36	2.47	3.64	3.16	4.50

Tab. 9 Influence of the fineness of the grinding slag on the K ratio ($\sigma_{\text{average}}/\sigma_{\text{min}}$).

Fineness	2850 cm ² /g		3570 cm ² /g		4620 cm ² /g	
Age	6 h	24 h	6 h	24 h	6 h	24 h
$K = \sigma_{\text{average}}/\sigma_{\text{min}}$	0.14	0.22	0.31	0.24	0.40	0.30

σ_{average} : Average values of the compressive strength of the slag-reference cement mortar cubes at the designed ages (Table 8)

σ_{min} : minimum values of the compressive strength of the reference mortar cubes at the designed ages (6h:8MPa, 24h:15 MPa)

Tab. 10 Hydraulic efficiency index h .

	Age of samples , days							
	7				28			
	Slag fineness, cm²/g							
	2480	2900	3470	4150	2480	2900	3470	4150
$\sigma_{(CEM II)}$, MPa CEM II = OPC + 50 % of slag	15.0	18	20.5	21.5	31.5	36	39	44.5
$\sigma_{(OPC)}$, MPa	36.25				48			
$h = \sigma_{CEM II} / \sigma_{OPC}$	0.42	0.50	0.57	0.60	0.66	0.75	0.81	0.93

4.2 Discussion

The results obtained show that the degree of the El Hadjar slag vitrification is satisfactory. The chemical composition of the slag also falls under the limits recommended by the standard [NF EN 197-1, (2001)]. In Table 3, the results also show that the slag of the El Hadjar plant is essentially composed of limestone ($CaO = 42.2\%$), silica ($SiO_2 = 40.1\%$), Alumina ($Al_2O_3 = 6\%$) and magnesium oxide ($MgO = 4.7\%$). Therefore, it can be said that the CaO contents are close to the SiO_2 content, whereas for the majority of slags, the CaO content of about 5 to 10 % is generally higher than that of the SiO_2 . It can also be noted that the chemical composition of the El Hadjar slag varied a little between 1987 and 2000. In this period, there was little modification in the manufacturing process as well as in the type and origin of the ore.

An evaluation of the hydraulic activity of the granulated slag of El Hadjar by the indexes is more complex, especially in the absence

of an Algerian standard. Therefore, we have tried to use the indexes which can give the best correlations with the mechanical strengths [DEMOULIAN E, et al (1980)].

The chemical composition given in Table 3 (year 2000) led to the calculation of indexes (Table 7), 2, 4 out of which classify the slag of El Hadjar as being a very low hydraulic activity. However, this result should be balanced because the same calculation starting from the average composition given by DEMOULIAN led to hardly better values. Be may it is necessary to ask a question about the relevance of these indexes or more surely about the limiting values reported in the literature?

The tests on the slag mortar activated by soda (Table 9) confirm this bad classification (Table 8), the achieved values being from 2 to 7 times weaker than the necessary minima. From the results, it can be observed that an increase in the grinding fineness of 62% activates the initial reaction of 180% (the compressive strength at 6 h is almost tripled), but only influences to a very little extent its total capacity of reaction (the increase at 24 hours is only 34%), which remains at a very low level (only 30%) compared to the level fixed by the test.

On the other hand, the compressive resistance (Table 10) obtained from the cement made of 50% OPC and 50% ground slag (equivalent to CEM II) is satisfactory as well at 7 days and at 28 days, mainly for the slag most finely ground at 4150 cm²/g. The classification of the slag according to Table 2 must take into account its fineness and hardening age (Figure 2):

- At 7 days of hardening, the ground slags with 2480, 2900 and 3470 cm²/g are displaced in accordance with the indices h_2 and h_3 , because the indexes are too weak (0.42 to 0.57) ; the finest slag with 4150 cm²/g of fineness is classified in the 2nd category ($h = 0.60$).

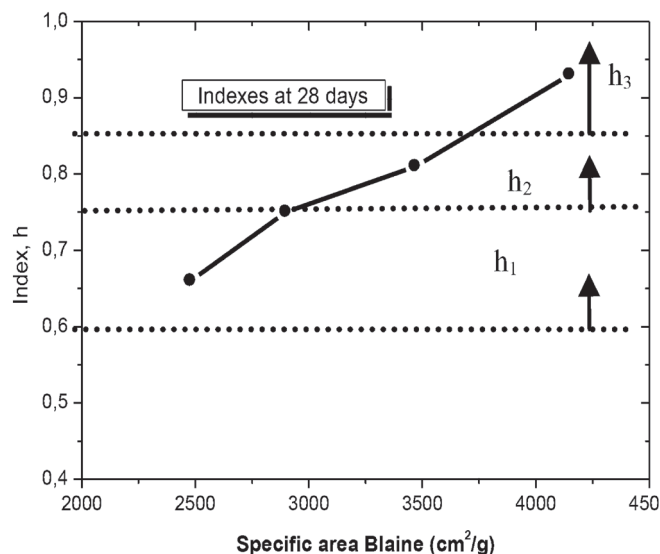
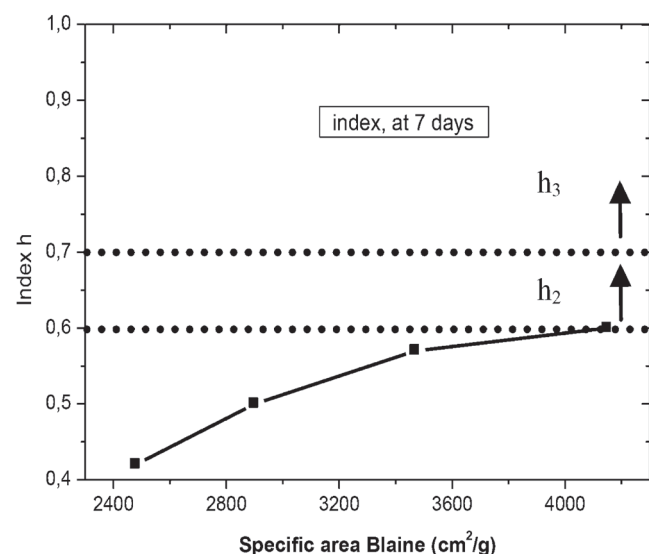


Fig. 2 Evolution of the index h at 7 and 28 days as a function of the fineness of the granulated slag of El Hadjar.

- At 28 days of hardening all the slags are classified; in category 1 for that of 2480 cm²/g, in category 2 for those with a fineness of 2900 and 3470 cm²/g, and in category 3 for the finest, with 4150 cm²/g, due to its index of $h = 0.93$.

It is noted that the fineness of the ground slag is a strong influence on its reactivity and that the slag of El Hadjar has a rather low short-term reactivity, but is more interesting at 28 days, thus showing kinetics of reaction slower than for a standard granulated slag.

5. CONCLUSION

Based on the results of this experimental study, the following conclusions can be drawn:

1. The slag of El Hadjar is vitreous to approximately 97%, which is an indication of good reactivity but as it is also in a high range of glass content, the El Hadjar slag could be classified as a material having moderate reactivity.
2. Its chemical composition shows a slightly higher quantity of SiO₂ and CaO but a lesser quantity of Al₂O₃ and MgO than the mean of the granulated slag.
3. Several activity indexes have been calculated from the chemical composition of the El Hadjar slag. Among the calculated indexes for the slag used, two of them, i.e., I_1 and I_8 , for which we have found optimal values in the literature, give satisfactory values. Two other indexes, I_9 and I_{60} , are outside of the recommended values, but the same calculations carried out on an average

slag gives a value close to the recommended one of (I_9) and an outside limit for the other (I_{60}).

4. The caustic soda test seems to be necessary to determine the activity indexes but is not sufficient. Therefore, other methods were recommended by ALEXANDRE [ALEXANDRE J. and SEBELAU J.L. (1986)] and DEMOULIAN [DEMOULIAN E., et al (1980)] to predict the hydraulic activity of the slag.
5. The results obtained from the activity index tests allow us to classify the El Hadjar slag in the categories h_2 and h_3 . This classification, which is satisfactory, strongly depends on the fineness of the slag. A slag fineness of more than 3500 cm²/g is recommended before adding it to the clinker.
6. The slag used in this study also had weak short-term activity, denoting a slower kinetics of reaction compared to the classical slag cited in many references [DEMOULIAN E., et al, (1980), SMOLCYCK H.G., (1980), ALEXANDRE J. and SEBELAU J.L. (1986)].
7. The results obtained on the mortars thus partly contradict the results of the various calculated indexes. They have been confirmed and refined, in particular by a study of a mortar, in order to examine the influence of the chemical and mechanical properties on the fineness and slag percentage [BEHIM, M (2005)].

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