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Treatment of phosphogypsum and mechanical characterization of solid blocks

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Abstract: *Phosphogypsum is a solid waste generated in phosphoric acid production. Several researchers had studied the use of phosphogypsum in various fields: plaster manufacture, agriculture, cement manufacturing, roadway structures, embankments, solid bricks, clay bricks, raw non-fired bricks blocks, hollow blocks … This study aims to determine the percentages of materials (crushed sand, tuf, cement, lime) that neutralized phosphogypsum and to specify the blocks having the best properties in terms of ultrasonic velocity and compressive strength. The obtained results showed that the treatment of phosphogypsum by cement or lime and cement has absorbed huge quantities of phosphogypsum. The blocks, constituted by 92% of phosphogypsum, 5% of cement, 3% of lime and 8% of water, have the best mechanical properties with minimum compaction energy.*
Keywords: *Compressive strength, pH, Phosphogypsum, Solid blocks, Ultrasonic velocity*

I. INTRODUCTION

The management of the industrial phosphogypsum (PG) is an environmental problem which concern several countries such as: Florida, India, Jordan, Turkey, Morocco, Senegal, etc [1]. It is stored into piles in the proximity of factories or is discharged directly into the sea. It is responsible of the adverse effects on the environment with all its components (sea, water table, soil). Therefore, it is necessary to find other treatment processes in order to reduce the stored quantities and to protect the fauna and flora.

Several researchers have studied the use of PG in various fields (in cement, in embankments, in roadway structures, in bricks, in blocks etc). The PG was used in the manufacture of cement by substitution of the gypsum (cement ultimax). It showed good performances but the used quantity is low [2]. Moussa had studied the possibility of the use of the PG in the embankments. This study showed a behavior to the compaction not similar to that of a soil. Furthermore, the fill showed also a continuous settlement because of the PG solubility [3]. Sfar et al. have explored the PG for a road use. The study proposed the following formulation: 46.5% of PG, 46.5% of sand and 7% of cement. But this study recommended the Tunisian south region, where the rainfall is low [4]. Belaiba et al. studied the use of PG in solid raw bricks. In this study, the following formulation has been considered: PG: 40–45%, Sand: same as PG, Cement: the complementary (10–15%). This application gave encouraging results but the product needed high compaction energy to obtain the bricks [5]. Finally, Ajam and al. have concluded that bricks, containing 30% of PG, are consistent with the standard requirements. But the quantities used are limited [6].

To overcome the failures of these applications, another construction technique is developed. It is consisted in the manufacture of blocks with stabilized and hardened PG by using aggregates additions (crushed sand and tuf) and/or hydraulic binder (cement and lime). It is served to use the maximum amounts of PG with less compaction energy.

In this article, at first, the materials are characterized (absolute density, bulk density, fineness modulus, fragmentation dynamics, $CaCO_3$ and pH). The second part deals with the valorization of PG in blocks. In particular, the study is focused on the neutralization of PG and mechanical characterization of solid blocks.

II. EXPERIMENTAL PROGRAMME

1. Materials

The materials used for blocks were phosphogypsum (PG), crushed sand (CS), tuf (T), cement (High Sulfate Resistance) (C), lime (L) and tap water (w_i) (Photo 1).

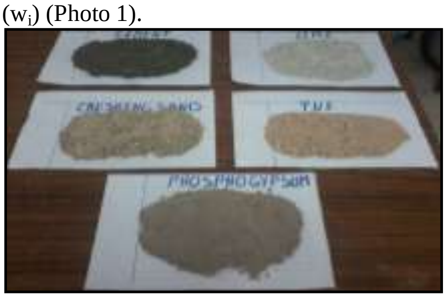

Photo 1. Materials used in the manufacture of blocks

The properties of the materials are given in Table 1. The absolute density of PG is usually close to the value given by the natural gypsum (2320kg/m^3) [6]. The CS and T, having an absolute density between 2000 and 3000kg/m^3, are common materials [7]. The fineness modulus of CS is lower than that of T. Thus, the fine fraction in the T is larger which causes the increase of water dosage [8]. The dynamic fragmentation of T is greater than that of CS. Therefore, the CS is more resistant [9]. The percentage of calcium carbonate ($CaCO_3$) of T, determined by testing the calcimetry [10], is greater than that of CS. Therefore, the addition of T decreases the acidity of PG as the CS. The pH of other materials is basic.

Table 1. Properties of the materials

	PG	CS	T	C	L
Absolute density (kg/m^3)	2430	2720	2140	3080	-
Bulk density (kg/m^3)	850	1800	1481	1200	1465
Fineness modulus	-	3.16	3.38	-	-
Dynamic fragmentation (%)	-	21	41	-	-
$CaCO_3$ (%)	-	58	77	-	-
pH	2.1	8	8.6	11.5	12.6

2. Mix proportions

The mix proportions of blocks are given in Table 2. The water contents added are 2, 4, 6, 8 and 10%.

Table 2. Proportions of the materials

Series	Mix-design	Materials (%)				
		PG	C	CS	L	T
A	**M_0**	92	8	-	-	-
B	**M_1**	45	5	50	-	-
C	**M_2**	92	5	-	3	-
D	**M_3**	56	4	-	-	40

3. Process operations

3.1. Mixing of materials

The mixing procedure is described as follows: First the components were placed in the tank with sand at the bottom then water was added. Mixing for 1 min 30 s at low speed was achieved later. The sides of the mold were scraped with the blade of the mixer before mixing again for 1 min 30 s at high speed [11] (Photo 2).

Photo 2. Mortar mixer

3.2. Preparation of blocks

The blocks are constructed using a semi automatic press (Photo 3) with a static compaction 10MPa. Theirs dimensions are 200 x 100 x 50mm. The manufacturing process is made according to the following steps: place the mixture into the mold, apply pressure by the descent of the press part to obtain the desired pressure, install the piece releases automatically and kick block manually by the creek.

Photo 3. Semi automatic press

3.3. Method of storage and testing of blocks

The blocks are stored in the air (at ambient temperature). They are tested after 28 days. The mechanical tests carried out are as follows: density (D), ultrasonic velocity (V) [12] and compressive strength (R_c) [13].

III. RESULTS AND DISCUSSION

1. Neutralization of phosphogypsum

The neutralization of PG is obtained by adding aggregates (CS, T) having a coarser particle size than the PG or by adding hydraulic binders (C, L). The test consists on putting ten grams of materials in 100ml of distilled water and shakes the mixture for 15 minutes with a magnetic bar.

Fig 1 presents that the addition of CS or T causes an increase in pH without reaching the neutralization. The CS has its greatest efficacity at dosages greater than or equal to 30% but from doses greater than or equal to 10% T is more efficient. The greater efficiency of CS is obtained especially at moderate doses (>10%) [14].
The T reduces the acidity of PG remarkably compared to the CS. This is due to the intervention of two factors: the presence of 77% $CaCO_3$ in the T while the CS contains only 58% and the portion of the fines in the T is larger compared to the CS (MF_T = 3.38 and MF_{CS} = 3.16). The fineness of the sand affects the kinetic of dissolution of carbonate [14].

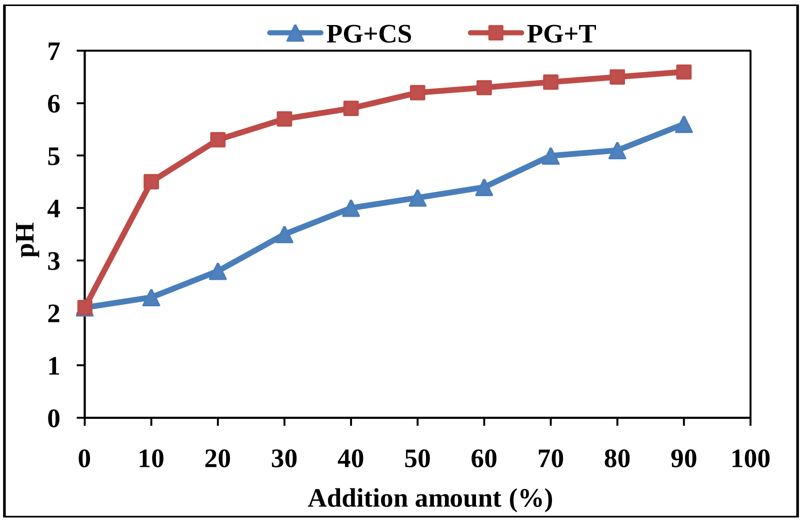

Figure 1. The pH of PG treated by CS or T

Fig 2 shows that the neutrality is achieved with 45% PG, 50% CS and 5% C. While the treatment of PG by the C and T provides neutrality with 56% PG, 40% T and 4% C.

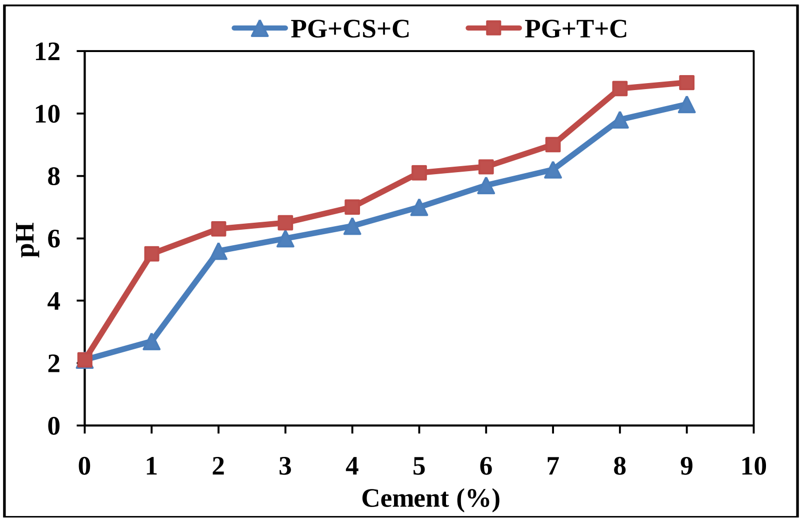

Figure 2. The pH of PG treated by CS+C or T+C

Fig 3 illustrates that the neutrality is reached with percentages of 92% PG and 8% C. The pH increases when 3% of C were added and can reach 8. Also, it presents that the addition of 3% of L permits to have a greater pH values and to achieve the neutralization with percentages of 92% PG and 5% C. The addition of L allows to exceed the neutrality [14].

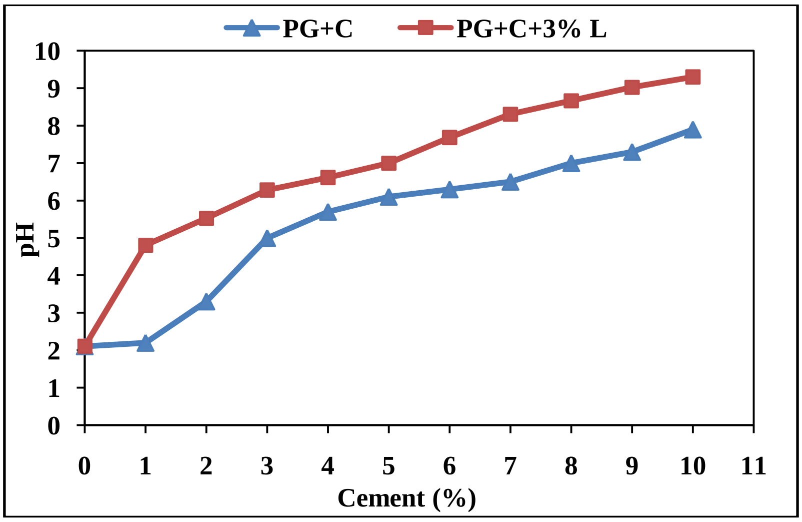

Figure 3. The pH of PG treated by C or C+3% L

2. Visual description of the solid blocks

Photos 4 and 6 show that white layers are spread totally on the sides of the series A and C while those of series D is partially extended (photo 7). The deposit is probably due to the dissolution of PG. The blocks of series B have developed layers of powder on their surfaces which have the same color as the PG (Photo 5).

Photo 4. Blocks of series A (w_i= 10%)

Photo 5. Blocks of series B (w_i= 10%)

Photo 6. Blocks of series C (w_i= 10%)

Photo 7. Blocks of series D (w_i= 10%)

3. Mechanical characterization of solid blocks

3.1. Density

Fig 4 shows that the density increases slightly in function of time. Densities of series D are less important than those series B. They extend respectively from 1.59 to 1.63 and from 1.93 to 1.96. The densities of series A and C are almost comparable. They varied respectively from 1.35 to 1.42 and from 1.37 to 1.44.
They are classified in the following order: series B > series D > series C > series A. Blocks produced have densities greater than 1. Therefore, they are dense (high density class) according to the standards [15].

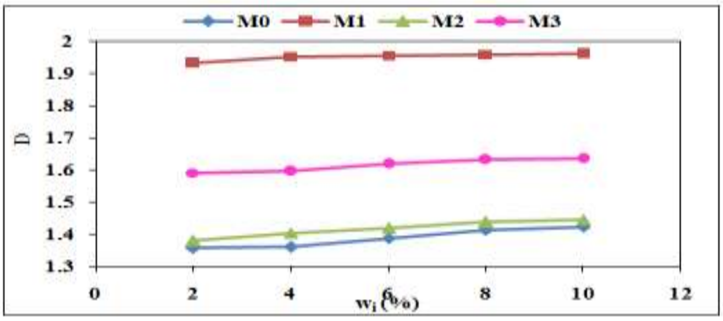

Figure 4. Density of solid blocks cured in air

3.2. Ultrasonic velocity

Fig 5 illustrates that the increase of the velocities of series B is more significant than those of series D. They extend respectively from 810 to 1950m/s and from 770 to 1450m/s. The velocities of series C are more important than those of series A. They vary respectively from 1090 to 1920m/s and from 1100 to 1740m/s. With 10% water content, the velocities of series B and C decrease respectively from 2020 to 1950m/s and from 1950 to 1920m/s. But the velocities of series D and A increase respectively from 1410 to 1450m/s and from 1720 to 1740m/s. The velocities of series B and C are higher than the others. Therefore, their particles are well distributed. They have more homogeneous structure [16].

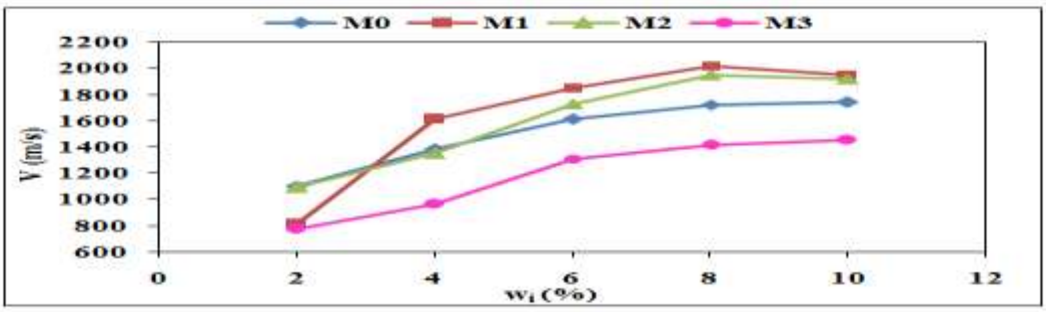

Figure 5. Ultrasonic velocity of solid blocks cured in air

3.3. Compressive strength

Fig 6 shows that the compressive strengths of the series D are smaller than those of series B. In fact, the dynamic fragmentation of T (40%) is greater than that of the CS (20%). Therefore, the addition of the CS causes a significant strength gain than that of T. With water content greater than 8%, the resistance of the series D increases slightly (from 5.347 to

5.647MPa), while that of series B decreases (from 8.267 to 8.014MPa). Indeed, the portion of fine existing in the T is more important than in the CS which requires an additional amount of water. The resistances of the series C evolve significantly more than the series A. The strength gain is explained by the addition of L (3%). The formation of stable compounds of C and L further improves the development of the mechanical strength. With 10% water content, the resistance of the series C decreases (7.934MPa) while the series A increases (7.473MPa). They have respectively 5 and 8% of C. Therefore, the hydration reaction of C requires more water to it unfolds correctly and easily. The resistances of series B are more important than those of others for 4 and 6% water content. However, with 8% water content, the resistances obtained are almost comparable to the series B and C (8.3 MPa). Series B and C are substantially similar in terms of velocity and compressive strength with 8% water content. Therefore, they are more homogeneous and more resistant. As the formulation of the series C absorbs the maximum of PG (92%), it can be selected. The strengths of the total solid blocks were higher than 2.3MPa which is the minimum strength indicated by the standards [15].

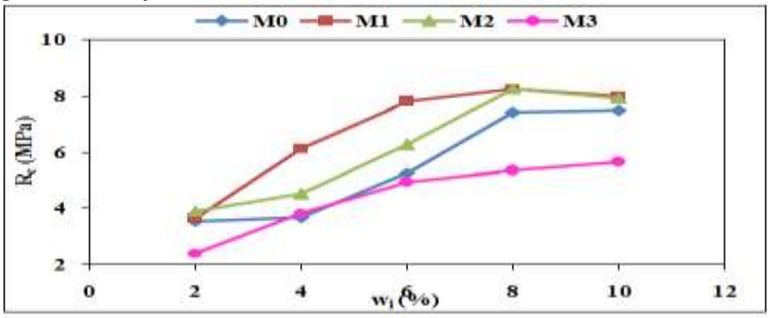

Figure 6. Compressive strength of solid blocks cured in air

IV. CONCLUSION

Based on the experimental investigation reported in the article, the following conclusions are drawn:

- The addition of CS or T causes an increase in pH without neutralization.
- The T reduces the acidity of PG remarkably compared to the CS.
- The treatment of PG by the C and CS provides neutrality with percentages 45% PG, 50% CS and 5% C.
- The treatment of PG by the C and T provides neutrality with the percentages of 56% PG, 40% T and 4% C.
- The addition of T and C allows for a greater amount of PG in comparison with the addition of CS and C.
- The treatment of PG by the C provides neutrality with percentages 92% PG and 8% C.
- The addition of 3% L is used to provide a higher pH values and to achieve neutralization with percentages of 92% PG and 5% C.
- The treatment of PG by C or L and C absorbs huge quantities of PG (92%).
- Blocks, constituted by 92% PG + 5% C + 3% L + 8% w, have the best mechanical properties in terms of ultrasonic velocity and compressive strength with minimum compaction energy (10MPa).

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