UN STUDIU REFERITOR LA INFLUENȚA PRESIUNII DE COMPACTARE ASUPRA PROPRIETĂȚILOR CĂRĂMIZILOR PE BAZĂ DE FOSFOGHIPS
A STUDY OF COMPACTION PRESSURE INFLUENCE ON PROPERTIES OF PHOSPHOGYPSUM- BASED BRICKS

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A feasibility study was undertaken on the production of phosphogypsum-crushing sand based bricks to build houses economically by utilizing of industrial wastes. All full bricks were made on a bench model, semiautomatic press having a capacity of 25 tons, as shown in appendix, to produce bricks of 51 mm×95 mm×203 mm in size under a static compaction of 15, 20 and 27MPa. The compressive strength, flexural strength water absorption, density and speed of sound for these bricks were investigated. It was observed that these bricks have sufficient mechanical strengths for their use in low cost housing development. Tests were also conducted to study the pressure influence on bricks properties. The results suggest that compressive and flexural strength increase with pressure. This increase is more important for pressure increase from 20 to 27 MPa. The concentrations of the selected metal species, i.e., Cd, Cu, Zn, Ni, Pb, and Cr, for all mixes-design, were well below the regulatory limits. Thus, this result indicates that phosphogypsum (PG) amended bricks specimens and can be considered as non-hazardous material.

***Keywords**: Phosphogypsum, full bricks, mechanical strengths, compaction pressure*

1. Introduction

There is a general exodus of rural population to the cities with the rapid industrialization in developing countries. Buildings for housing and industry, mass transit for moving people and goods, and facilities for handling water and sewage will require large amounts of construction materials. Enhanced construction activities, shortage of conventional building materials and abundantly available industrial wastes have promoted the development of new building materials.

Phosphogypsum (PG) is an important by-product of phosphoric acid and fertilizer industries. It consists of $CaSO_4$.$2H_2O$ and contains some impurities such as phosphate, fluoride, organic matter and alkalis. In Tunisia, for several years, a set of phosphoric acid production factories have produced PG in large quantities (approximately 10 million tons per year [1]. Currently, the PG is stored into piles in the vicinity of the factory, by dry or wet process. The storage of PG causes the pollution of the water table and the soils by acid and heavy metals infiltrations.

Its valorization leads to environmental protection and to minimization of the storage costs. Several researchers had studied the use of PG in various fields. The PG was treated to be used in the plaster manufacture. It has been found that the PG is suitable for making good quality plaster showing similar proprieties to plaster from natural gypsum [2-4]. This field is advantageous for the countries which do not have natural quarries of gypsum such as Japan and India. It has been sought also to use phosphogypsum in agriculture [5]. It is as effective as the crushed natural gypsum. However, the quantities used are limited and moreover the health standards became increasingly restrictive. But the most interesting use of the PG is for the cement manufacturing: either by natural gypsum substitution (about 5%), which will play the role of a set retarder [6-7], or to reduce the clinkerization temperature [8]. The PG was also used in soil stabilization [9]. Finally phosphogypsum has been studied for be used in hollow blocks [10] and light bricks [11].

In Tunisia, the PG was studied for be used in similar fields. The most successful application so far is in the manufacture of cement by substitution of the gypsum. It shown good performances but the used quantity is low [12,13]. In the paper [14] was studied the possibility of the PG use in the embankments like a fill. This study showed a behavior to the compaction not similar to that of a soil. Further more, the filling shown also a continuous settlement because of the PG solubility. Moreover, Ambroise and Pera [15] have studied the Gabes and Skhira PG for to be used as a hydraulic binder. Finally, Sfar[16] has explored the PG for a road use. The study proposed the following composition: 46.5% PG, 46.5% sand and 7% cement. But this study was conducted in the

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Tunisian south region, where the rainfall is low.

In this paper compressive strength, flexural strength water absorption, density and speed of sound for phosphogypsum- crushing sand-lime-cement bricks were investigated. The influence of pressure on these characteristics was studied.

2. Experimental program

2.1. Materials

2.1.1. Characteristics of the studied phosphogypsum

The characteristics of a PG depend on its composition and on its manufacturing process and control. Samples were obtained from the PG piles in the vicinity of the factory of Sfax (Center-East of Tunisia).

Figure 1 presents the granulometric curve of the studied PG. The granular distribution of PG was obtained by laser diffraction (laser granulometry). This figure shows that the studied PG appears like fine sand, of a granulometry lower than 250 µm. The granular distribution of PG depends on the quality and the reaction time of the phosphate attack. The test allowed calculating the Coefficient of uniformity (Cu) and the Coefficient of conformity (Cc).

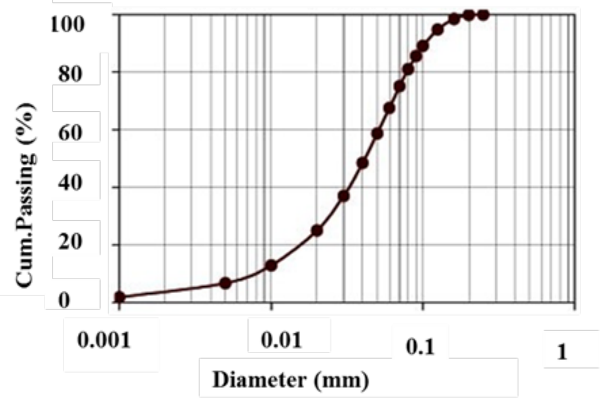

Fig. 1 - Granulometric curve of the studied Tunisian PG.

The Cc (resp. Cu) was evaluated at 8 (resp. 3.125). Figure 1 shows also a suitable uniform granulometry, consequently, it gives an optimum compactness which influences the strength, mainly in compression. The PG is considered to have a fine granulometry which is similarly to those of sand. The Tunisian PG presents 80% [17,18].

The real density of the grains was determined by the pycnometer method, by using water as work liquid. The studied PG showed a density of about 2300 kg/m3. This value is similary to that for the natural gypsum (2320 kg/m3). This value is also closely to that indicated for the Brazilian PG (2308 kg/m3) [19]. But this density is lower than the one of the Turkish PG (2890 kg/m3) [20].

The chemical analyses, considered most relevant to the prospect use in commercial bricks making, were undertaken on the PG. The results of these analyses are presented in Table 1. The PG is primarily (about 77%) made up of calcium sulfate ($CaSO_4$). The remaining components are present in low percentage. It should be noted that the pH of PG samples has been found to be around 2.9, indicating a high acidity of the PG.

2.1.2. Characteristics of the crushing sand

Figure 2 present the granulometric curve of the crushing sand. The granular distribution of crushing sand was obtained by dry sieving. Figure 2 shows also a suitable uniform granulometry, which gives an optimum compactness which influences the strength, mainly in compression.

The real density of the grains was determined by the pycnometer method. The studied crushing sand presented a density of about 2740 kg/m3. This value is higher than the one of phosphogypsum.

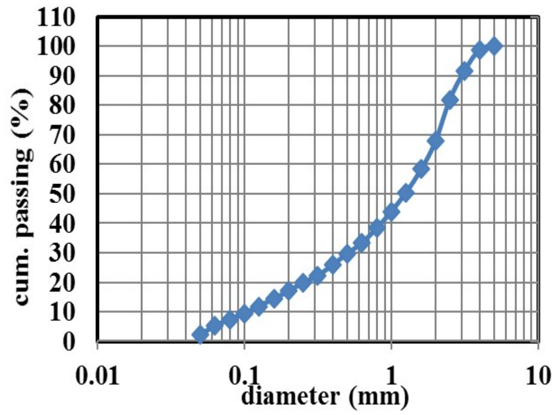

Fig. 2 - Granulometric curve of the crushing sand.

The content of $CaCO_3$ in crushing sand determined by calcimetry was 63.63 %.

2.1.3. Characteristics of hydraulic lime

Hydraulic lime was obtained by calcinations of calcareous rock containing a percentage of clay greater than 5% (generally 15-20%). it contains 6.5 to 7% at least (and often much more) clay. They are therefore made of mixtures of limestone and marls, or clay limestone. The firing is carried out at

Table1

Chemical composition of phosphogypsum

Components (%)								
CaO	SO_3	P_2O_5	F	SiO_2	Fe_2O_3	Al_2O_3	MgO	Ignition loss at 1000°C
32.8	44.4	1.69	0.55	1.37	0.03	0.11	0.07	18.98

Table 2

Mix proportions of PG-CS-C-HL full bricks

Mix indicative	Constituant materials (weight %)			
	Phosphogypsum	Crushing Sand (CS)	Portland cement	Lime
M-1	60	33	5	2
M-2	60	30.5	7.5	2
M-3	60	28	10	2
M-4	70	23	5	2
M-5	70	20.5	7.5	2
M-6	70	18	10	2
M-7	80	13	5	2
M-8	80	10.5	7.5	2
M-9	80	8	10	2

about 1000 ° C., which causes some reactions between the oxides CaO and SiO2 and Al_2O_3. The clusters are formed from the main products of CaO and minerals clay reactions. What distinguish the hydraulic lime from cement are the absence of C3S and the presence of a significant amount of free lime.

The hydraulic lime presented a density of 2900 kg/m^3 and a bulk density of 800g/l.

2.1.4. Cement

The used cement was of type HRS 42.5. Portland cement with high sulphate resistance conforming to NT 47.01 and NT 47.26, belonging to Resistance Class 42.5 and presenting ordinary short-term resistance.

2.2. Use of Phosphogypsum in the solid bricks preparation

2.2.1. Mixes compositions

Shrinkage cracking and spalling are major weakness for phosphogypsum-based bricks. Shrinkage cracking can be minimized by keeping the water content of binder as low as possible. Hence, in the present study, a low slump mix was considered to limit the shrinkage. The water content of Phosphogypsum-crushing sand-limemixtures was fixed at 5 %. Bricks produced with more than 5 % showed cracks after fabrication due to excessive water content.

The mixes compositions of phosphogypsum, crushing sand (CS), lime and Portland cement (PC) for bricks are given in Table 2.

2.2.2. Manufacturing process

2.2.2.1. Mixing of raw materials

A mixer was used for materials mixing. The dry phosphogypsum was sieved through 1 mm sieve. The weighed quantities of phosphogypsum, crushing sand, lime and cement were first thoroughly mixed in dry state for a period of 10 minutes for obtain uniform mixes. The required water was then gradually added and the mixing continued for another 5 min.

2.2.2.2 .Preparation of bricks

All full bricks were made on a bench model, semiautomatic press having a capacity of 25 tons, as shown in appendix, to produce bricks of 51 mm ×95 mm×203 mm in size under a static compaction of 15, 20 and 27MPa.

2.3. Testing of bricks

All bricks were cured into the free air for a period of 28 days. Compressive strength, density, water absorption, saturation coefficient and leaching data of bricks from different mixes are shown in the Table 2.

Compressive strength of the samples was determined according to ASTM C67 [17]. Test bricks consisting of a halfs brick with full height and width. Flexural strength test on the bricks were performed according to ASTM C 67[17]. Test specimens were taken as full-size brick samples.

Rate of water absorption is an important property of brick because it affects mortar and grout bond. If the rate of absorption is too high, brick will absorb moisture from the mortar or grout at a rapid rate, thus impairing the strength development and extend of bend.

Bricks contain pores; some may be “through” pores, others are “cul-de-sac” or even sealed and inaccessible. The “through” pores allow air to escape in the 24 hours absorption test and permit free passage of water. However, others in a simple immersion test or vacuum test do not allow the passage of water, hence the requirement for five hours boiling test. The water absorption is the amount of water which is taken up to fill these pores in a brick by displacing the air. The saturation coefficient is the ratio of cold water absorption for 24 hours and maximum absorption in boiling water.

Water absorption and saturation coefficient were determined according to ASTM C67 (Ahmadi BH. 1989). The test specimens were consisted of halfs brick. Five specimens were tested.

In order to determine the density of each brick, dry bricks were weighed accurately and their volumes were measured.

The leaching tests has been performed according to the French norm NF EN 12457-3 [19], at ambient temperature (20 ± 5°C). The specimens (three different samples for each mix-design) were crushed and an amount of 0.175 kg was taken to be analyzed. The distilled water was added to obtain a liquid/solid ratio of 10.

Table 3

Dry densities (kg/m^3) of PG- CS-HL-PC full bricks versus compaction pressure

Mix indicative	Dry densities (kg/m^3) , for compaction presures		
	15MPa	20MPa	27MPa
M1	1446	1535	1621.5
M2	1523	1564.5	1636
M3	1564	1594	1662
M4	1556	1570	1630
M5	1594.5	1606	1660
M6	1613.5	1642.5	1690.5
M7	1619	1633	1759
M8	1636	1670	1796
M9	1700	1751.5	1832

Table 4

Cold water absorption after 24 h of CS-HL-C-PG full bricks

Mix indicative	Cold water absorption after 24 hr (%) for 15MPa	Cold water absorption after 24 hr (%) for 20 MPa	Cold water absorption after 24 hr (%) for 27 MPa
M1	27	24.65	13.38
M2	26.3	23.78	11.32
M3	25.1	22.02	10.36
M4	26.6	23.3	12.1
M5	25.5	22.07	10
M6	24	21.46	9.1
M7	25.3	21.52	9.1
M8	24	20.7	8.7
M9	23	19	7.5

Table 5

Water absorption after curing for 24 hrs in cold water + 5hr in boiling water (%) of PG-CS-HL-PC full bricks

Mix indicative	Water absorption after curing for 24 hrs in cold water +5hrs boiling (%) (15MPa)	Water absorption after curing for 24 hrs in cold water +5hrs boiling (%) (20MPa)	Water absorption after curing for 24 hrs in cold water +5hrs boiling (%) (27MPa)
M1	28.4	26.23	19
M2	27.97	25.85	17.5
M3	26.98	24.2	16.3
M4	28.29	25.33	18.02
M5	27.41	24.26	16.2
M6	26.37	23.85	15.4
M7	27.2	23.65	17.1
M8	26.66	23	15.1
M9	25.84	21.6	14.2

3. Test results and discussions

3.1. Dry density

The densities of PG-CS-HL-PC full bricks are shown in Table 3. These densities range from 1446 to 1832kg/m^3. Therefore the PG-CS-HL-PC full bricks have high densities.

The results show also increase of the densities with the compaction pressure increase. This increase is more significant from 20 to 27 MPa. The maximum of compressive strengths is reached for 27 MPa.

3.2. Water absorption and saturation coefficient of studied bricks

3.2.1. The cold water absorption after 24-h

The water absorption is a principal factor for the durability of the product and its behavior to natural environment. Table 4 presents the results of the water absorption for CS-HL-C-PG full bricks. It can observe that the water absorption of bricks decreases with the increase of pressure. Also, it was observed that the values obtained were favorable when compared with those of clay bricks (0 to 30%) [19].

3.2.2. Water absorption after curing for 24 hours in cold water +5hours in boiling water

The water absorption after curing for 24 hours in cold water + 5hours in boiling water is shown in Table 5. The fact that water absorption of bricks increased after 5hours of boiling was noticed. This is due to the fact that the 24 hours submergence alone will not fill all the pores spaces in a brick, so more will become filled during the boiling stage.

Besides, the results show that the water absorption for 5-h boiling of M9 bricks composition compacted at pressures of 20 MPa and 27 MPa

Table 6

Saturation coefficient of PG-CS-HL-PC full bricks compacted at different pressures

Mix indicative	Saturation coefficient (15MPa)	Saturation coefficient (20MPa)	Saturation coefficient (27MPa)
M1	0.95	0.94	0.83
M2	0.94	0.92	0.8
M3	0.93	0.91	0.77
M4	0.94	0.92	0.81
M5	0.93	0.91	0.77
M6	0.91	0.9	0.75
M7	0.93	0.91	0.78
M8	0.91	0.9	0.75
M9	0.89	0.88	0.73

and also, for all bricks compositions compacted at 27 MPa pressure is lesser than the maximum limit for clay building bricks set by ASTM (22%) (ASTM international: C62-08, 2008). Water absorption of bricks manufactured with 15 and 20 MPa exceeds this limit.

3.2.3. Saturation coefficient

The saturation coefficient of the same bricks is shown in Table 6. It can observe that the saturation coefficient of bricks decreased with the increase of pressure. Only the saturation coefficients of M9 bricks manufactured at 20 MPa and for all bricks compositions compacted at 27 MPa are lesser than the maximum limit for clay building bricks set by ASTM (0.88) (ASTM international: C62-08, 2008). Saturation coefficient of other bricks manufactured at 15 and 20 MPa exceeds this limit.

3.3. Compressive strength of studied bricks

The results, obtained as an average of measurements performed on three specimens, are shown in Figures 3-5. The BS6073-Part 1 [20] requires minimum compressive strengths of 7.0 MPa after 28-days curing period for load-bearing concrete masonry units. All mixtures investigated in this study satisfied the standard for compressive strength.

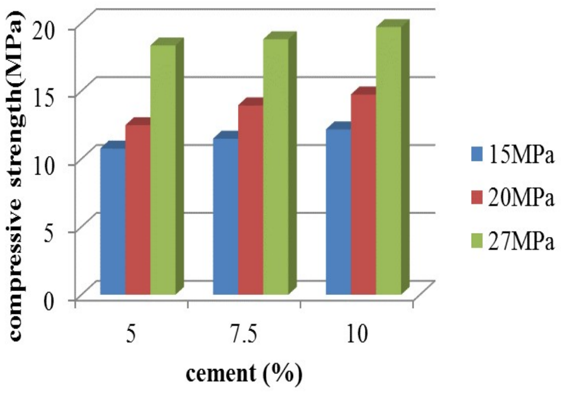

Fig.3 - Compressive strengths of PG-CS-HL-PC full bricks versus cement content and compaction pressure (phosphogypsum=60%).

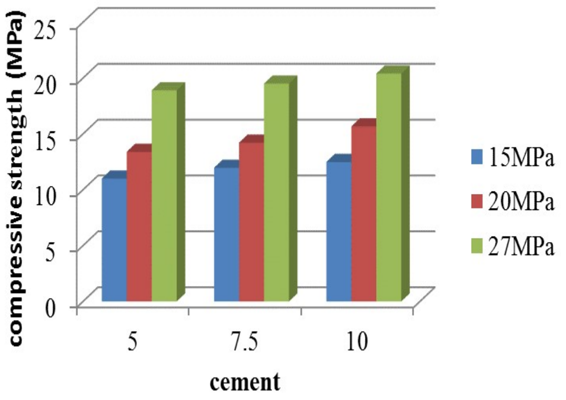

Fig.4 - Compressive strengths of PG-CS-HL-PC full bricks versus cement content and compaction pressure (phosphogypsum=70%).

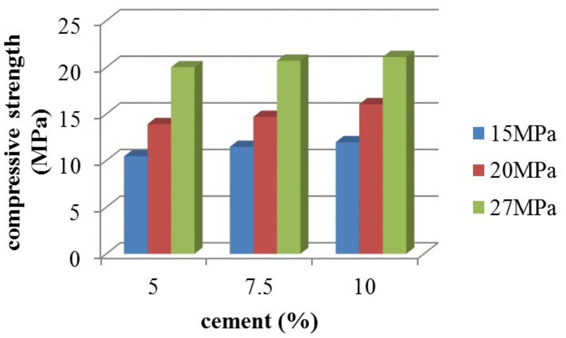

Fig.5 - Compressive strengths of PG-CS-HL-PC full bricks versus cement content and compaction pressure (phosphogypsum=80%).

For all the studied cases, the strength of the 90 days cured full bricks was higher than 10.3 MPa, which is the minimum strength indicated by the standards [21]. Cement as a source of reactive silicates and aluminates, by reactions with water, gives silicate and aluminate hydrates, which are responsible for the development of mechanical strengths.

The Figures 3-5 show also increase of the strengths with the compaction pressure increase. This increase is more significant from 20 to 27 MPa, the maximum of compressive strengths being reached for 27 MPa. The increase of cement content determines also the increase of compressive strengths.

These results are in good correlation with the water absorption test outcome that has been indicated that the increase of pressure produces a decrease of water absorption as result of internal pore size decrease.

3.4. Flexural strength of studied bricks

The results, obtained as an average of measurements performed on three specimens, are shown in Figures. 6-8. The code ASTM does not specify a requirement for flexural strength. However the values obtained were favorable when compared with the minimum flexural strength required by BS6073-Part 1[20] (0.65 MPa) after 28-days, curing period which was surpassed by all the samples. These figures show also the increase of the flexural strengths with the compaction pressure increase. This increase is more significant for pressure increase from 20 to 27 MPa. The maximum of flexural strength is reached for 27 MPa.

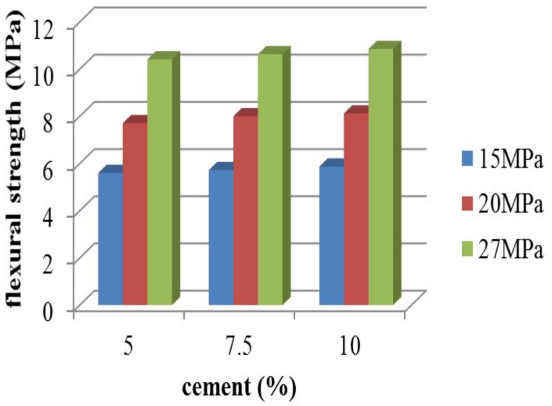

Fig.6 - Flexural strength of PG-CS-HL-PC full bricks versus cement content and compaction pressure (phosphogypsum=60%) .

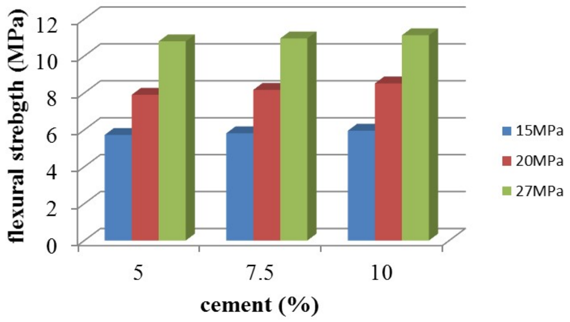

Fig.7 - Flexural strength of PG-CS-HL-PC full bricks versus cement content and compaction pressure (phosphogypsum=70%) .

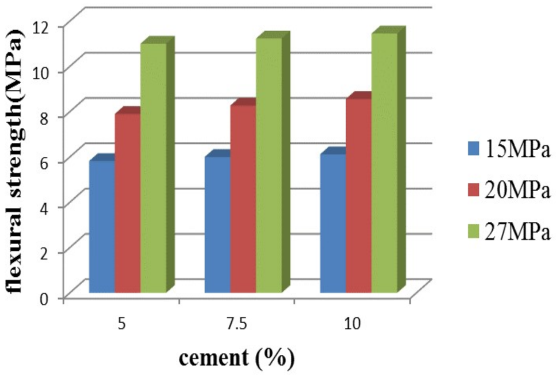

Fig.8 - Flexural strength of PG-CS-HL-PC full bricks versus cement content and compaction pressure (phosphogypsum=80%) .

Table 7

Leaching test results, in mg/kg for L/S = 10 l/kg (pressure 15MPa)

Elements	**M1**	**M2**	**M3**	**M4**	**M5**	**M6**	**M7**	**M8**	**M9**	**Limit values acceptable as inert**	**Limit values acceptable as non-hazardous**
Cr	0.08	0.077	0.76	0.07	0.068	0.07	0.058	0.059	0.053	4	50
Ni	0.078	0.073	0.063	0.069	0.064	0.056	0.064	0.06	0.058	0.4	10
Zn	0.058	0.056	0.054	0.054	0.052	0.05	0.05	0.048	0.046	4	50
Pb	0.1	0.095	0.09	0.095	0.093	0.09	0.092	0.09	0.088	0.5	10
Cu	0.12	0.098	0.096	0.099	0.096	0.094	0.09	0.094	0.09	2	50
Cd	0.07	0.067	0.062	0.06	0.04	0.035	0.045	0.031	0.03	0.04	1

Table 8

Leaching test results, in mg/kg for L/S = 10 (pressure 20MPa)

Elements	M1	M2	M3	M4	M5	M6	M7	M8	M9	Limit values acceptable as inert	Limit values acceptable as non-hazardous
Cr	0.058	0.055	0.14	0.048	0.045	0.037	0.035	0.026	0.02	4	50
Ni	0.055	0.05	0.04	0.046	0.041	0.033	0.041	0.039	0.035	0.4	10
Zn	0.036	0.034	0.032	0.032	0.03	0.029	0.028	0.026	0.024	4	50
Pb	0.085	0.08	0.078	0.081	0.079	0.077	0.078	0.077	0.075	0.5	10
Cu	0.095	0.085	0.083	0.08	0.082	0.079	0.078	0.076	0.069	2	50
Cd	0.05	0.047	0.042	0.04	0.02	0.01	0.025	0.01	0.01	0.04	1

Table 9

Leaching test results, in mg/kg for L/S = 10 (pressure 27MPa)

Elements	M1	M2	M3	M4	M5	M6	M7	M8	M9	Limit values acceptable as inert	Limit values acceptable as non-hazardous
Cr	0.048	0.045	0.04	0.038	0.035	0.027	0.025	N	N	4	50
Ni	0.045	0.04	0.032	0.035	0.03	N	0.03	N	N	0.4	10
Zn	N	N	N	N	N	N	N	N	N	4	50
Pb	0.074	0.065	0.043	0.064	0.055	0.036	0.055	0.037	N	0.5	10
Cu	0.09	0.055	0.03	0.08	0.045	0.02	0.06	0.035	0.02	2	50
Cd	0.04	0.02	0.01	0.025	0.01	N	N	N	N	0.04	1

3.5. Leaching behaviour

The average values of the leaching test are presented in Tables 7, 8 and 9, for the samples compacted at different pressures. The concentrations of the selected metal species, i.e., Cd, Cu, Zn, Ni, Pb, and Cr, for all mixes, were well below the regulatory limits. Thus, these results indicate that PG amended brick specimens can be considered as non hazardous materials.

4. Conclusions

Based on the experimental investigations data reported in this paper, following conclusions can be drawn:

(1) The PG-CS-HL-PC bricks have enough high strength and have potential as a replacement for load-bearing concrete masonry units.

(2) The increase of the compaction pressure resulted in increase of the mechanical strengths of bricks.

(3) The increase of compressive and flexural strengths is more significant for increase of pressure from 20 to 27 MPa

(4) The diminish of water absorption and saturation coefficient is more pronounced too for increase of pressure from 20 to 27 MPa.

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MANIFESTĂRI ȘTIINȚIFICE / SCIENTIFIC EVENTS

1st Global GypSupply Conference & Exhibition on gypsum supply trends and technology

13 - 14 March 2018, Brussels, Belgium

Global GypSupply Conference will look at the different supply sources of gypsum worldwide, including natural gypsum, synthetic gypsum and recycled gypsum. Supplies of FGD gypsum will drop dramatically in the coming years as coal-fired power stations are progressively closed, and environmental and other regulations are curtailing the supply of some sources of natural gypsum. Recycling is becoming a potentially significant new source of raw materials. Given these changes in the markets, gypsum users (wallboard and plaster manufacturers, cement producers and agricultural users) are likely to seek new supplies and new suppliers of their crucial raw materials.

Themes and topics:

- Gypsum mining
- Gypsum recycling
- Synthetic Gypsum
- Gypsum shipping and trade
- Gypsum markets

Contact: http://www.globalgypsum.com/conferences/global-gypsupply/introduction