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T. Tahri[1],
orcid.org/0000-0002-3953-9914,
N. Bezzi[1],
orcid.org/0000-0002-5343-5089,
A. Bouzenzana[2],
orcid.org/0000-0003-0440-9471,
K. M. Benghadab[3],
orcid.org/0000-0002-3423-4273,
A. Benselhoub[4],
orcid.org/0000-0001-5891-2860

1 – Laboratory of Materials Technology and Process Engineering (LTMGP), University of Bejaia, 06000, Bejaia, Algeria, e-mail: tahri2008@yahoo.fr
2 – Mining Laboratory, Mining Institute, Larbi Tebessi University, Tebessa, Algeria
3 – Laboratory of Metallurgy and Material Sciences, Badji Mokhtar University, Annaba, Algeria
4 – Environment, Modeling and Climate Change Division, Environmental Research Center (C.R.E), Annaba, Algeria

USE OF NATURAL PHOSPHATE WASTES IN THE MANUFACTURE OF CONSTRUCTION BRICKS

Purpose. Valorization of phosphate waste and diversification of the range of bricks by improving their mechanical properties. In this context, our work focused on the use of phosphate wastes from Djebel Onk (Tebessa) combined with clays from the Sidi Aich region (Bejaia) to manufacture a new variety of brick. The principle consists in varying the percentage of these wastes and the firing temperature.

Methodology. After mechanical preparation in the laboratory, which consists of crushing, grinding and sieving operations, the two materials of "phosphate and clays" are characterized by several analysis techniques (XRD, IR and XRF). The brick manufacturing tests from these two materials were carried out for different weight percentages and for different firing temperatures.

Findings. The combination of these two materials constitutes a new approach to the recovery of natural phosphates, which has a very high waste rate. Tests of making bricks from these two materials have yielded promising results. From a mechanical point of view, the best flexural strengths obtained at a temperature of 900 °C vary from 3 to 5 MPa depending on the type of phosphate/clay mixture. The same goes for the compressive strength, which varies from 15 to 27 MPa depending on the type of mixture as well. These results conform to ASTM-C674, 1999 construction standards.

Originality. The use of natural phosphate wastes from Djebel Onk in various proportions has resulted in a quality of brick that meets the international standards.

Practical value. The mechanical properties of the brick made from these two materials, such as resistance to bending and compression constitutes a real significant recovery for the phosphate wastes from Djebel Onk.

Keywords: *phosphate wastes, clay, manufacturing, bricks*

Introduction. The major current problems, from an economic and environmental point of view, is the treatment of large volumes of mining waste produced continuously by industrial and mining activities as well as the excessive consumption of non-renewable natural resources in the field of construction such as clay.

Clay is a raw material used in the manufacture of construction bricks and is of growing interest for its various industrial applications, which are constantly diversifying. Among the clay-based building materials, there is a brick that is made at an appropriate temperature called "the sintering temperature". At this temperature, the clay particles begin to melt and agglomerate to form a mass with a stony character. After firing, the brick retains certain porosity, which gives it specific properties and distinguishes it from other construction materials [1].

Several research works have been carried out: on different qualities and sources of industrial waste used in the manufacture of fired bricks such as waste from the wood, paper mill, tobacco, sugar cane and biomass industries are increasingly more used [2], also recycling different types of waste into eco-friendly fired clay bricks [3]. Other studies propose the production of bricks based on industrial waste by passing through a series of firing, cementing and geopolymerization [4]. To develop clay brick as a sustainable construction material, the use of agricultural and industrial waste is a practical solution [5]. A classification of waste according to its properties and its origin was carried out according to the European waste catalog EWC (European waste catalogue, 2002) and according to its role as an alternative material for the manufacture of fired bricks [6]. Wastes classified in the EWC 01 code are wastes resulting from the exploration, extraction or physical and chemical treatment of minerals; they are the most used in the manufacture of fired bricks.

We are interested in this study, in the manufacture of a brick using the clays of Oued Remila (Bejaia) on the integration of the wastes. It is in particular, a question of associating with these clays and the wastes of natural phosphates of Djebel Onk whose qualities are considerable. They are characterized by mixed grains of dolomite-phosphate cemented by a carbonate cement and clay [7].

Many industries produce waste from industrial waste, which is considered not only as waste but rather as a by-product that can be a means of increasing the life of clays or increasing the profitability of production.

The main objective of this work is to beneficiate further the phosphate ore; to diversify the range of brick manufacturing and contribute to the protection of the phosphate-processing unit from drunkenness.

The study consists of two materials, phosphate wastes and clay; bricks are made with different percentages and different temperatures.

Materials and methods. ***Characterisation studies.*** The samples of phosphate wastes larger than 1mm for the three sublayers of Bled El Hadba and the sample of clay taken from the stock of clays from the Oud Remila brickyard are subjected to mechanical preparation in the laboratory to reduce the dimensions of the grains, on the one hand, and to obtain representative samples, on the other hand. These are homogenization, crushing, quartering and grinding.

The different analysis techniques used are X-ray diffraction (XRD), X-ray fluorescence spectrometry (XRF) and Infrared spectrometry (IR).

The phosphate wastes and the clay are crushed in a jaw crusher, the aim of which is to obtain a maximum diameter of 1 mm (i.e. the opening is 1 mm), and then dried in a furnace at 105 °C for 24 hours until weight stabilized. The humidity ratio measured on clay is about 9 %. The two samples will then undergo grinding to dimensions of less than 100 μm in a FRISCH RM200 grinder. The grinding conditions used are grinding time 10 to 15 minutes and speed of rotation 200 rev/minute.

The mixtures of the two samples are then prepared according to the desired substitution rates. These last ones change according to the percentages of phosphate waste (Table 1).

Prior to starting to prepare samples, the prerequisite is to manufacture the mold to be used. For this, we used a mechanical manufacturing machine at the level of the Technological Hall of the University of Bejaia. This machine is of parallelepipedal shape and has dimensions of $4 \times 4 \times 16$ cm^3. The raw materials are mixed with a sufficient quantity of water (20 %) in a mixer then deposited in a mold, pressed with a hydraulic press to evacuate the air and water, dried in the open air at 100 °C for two days. They are then fired in a furnace at a well-defined temperature and firing time.

The firing temperature is gradually increased for 5 hours. This test was carried out for firing temperatures of 850, 900 and 950 °C to determine the important indices of brick quality. Then a gradual cooling is performed for 5 hours to a room temperature and then exposed to the open air [8].

Experimental procedure. Using phosphate waste outside the mine site is one of the methods that could reduce its volume. The idea is to find ways to recover these wastes that can consume these large quantities. The manufacture of fired bricks based on phosphate wastes presents a specific example of industrial symbiosis.

Results and discussion. ***Chemical analysis and X-ray Diffraction analysis.*** The mineralogical study on the phosphate ore, performed by X-ray diffraction (XRD) focused on the two crushed samples < 100 μm.

The obtained results of the phosphate waste sample are reported in Fig. 1, *a*. They revealed the presence of the following:

- dolomite and calcite as main minerals;
- quartz and fluorpatite in low quantities.

Phosphate wastes from Bled El Hadba are rich in calcite but low in Fluoroapatite and quartz [9].

The obtained XRD results of the clay sample are reported in Fig. 1, *b*. They have highlighted the presence of the following minerals:

- illite, muscovite and calcite as main minerals;
- quartz and dolomite in small quantities.

The Oued Remila clays have a poly-mineral composition.

IR infrared spectroscopy analysis. Analyses by infrared spectroscopy were carried out for the two ground samples < 100 μm of phosphate wastes and clay. The obtained results are illustrated in Tables 2, 3 and Fig. 2. They highlighted several main bands, in particular those attributed to calcite carbonates with a deformation vibration of Si—O—Al for phosphate wastes and for clay.

Chemical compositions. The mechanical preparation of the two samples (phosphate wastes and clay) was performed at the LTMGP laboratory, University of Bejaia.

The chemical composition of these two samples was determined by XRF, at the laboratory of Farhat Abes University, Setif. The obtained results showed the existence of the following elements: Ca, Si, Al, Fe, P, Mg, K, S, Na, Ti, F, Sr, Mn, and Cl as presented in Table 4 and Fig. 6. From these results, the clay is rich in silicon (34.9 %), aluminum (12.2 %), iron (11.6 %) and potassium (4.23 %) unlike phosphate wastes which record: 4.82 % silicon, 1.35 % aluminum, 1.18 % iron and 0.51 % potassium. It should be noted that phosphate wastes are characterized by high contents of: calcium (71.4 %), phosphorus (9.32 %) and magnesium (5.90 %) compared to clay: calcium (30.6 %), phosphorus (0.18 %) and magnesium (2.21 %).

Silicon (Si) plays a role as a filler material and is used in the manufacture of fired bricks to reduce plasticity and increase its hardness.

Alumina (Al) makes it possible to improve the plasticity of the mixtures and to obtain pressed products with significant reliefs.

Calcium (Ca), potassium (K), sodium (Na) and magnesium (Mg) are also considered fluxing elements and tend to combine with silicates during cooking [10]. The rest of the chemical elements are presented at similar levels.

Manufacture of fired bricks. In this study, we used phosphate wastes to manufacture fired bricks. The principle is to formulate bricks by substituting the natural clay of Oued Remila with phosphate wastes in different proportions, ranging from 0 to 30 % phosphate waste.

Analysis of fired bricks by XRF. The obtained results of the chemical analysis of the bricks, by XRF at a temperature of 850 °C, are shown in Table 5 and Fig. 4. These results show that the contents of silicon (34.3 %), aluminum (11.9 %) and iron (11 %) obtained for MX1 are higher compared to the other MX2 bricks (Si: 30.1 %, Al: 10.6 % and Fe: 10.1 %), MX4 (Si: 23.6 % Al: 8.53 % and Fe: 7.8 %) and MX3 (Si: 26.3 %, Al: 9.47 % and Fe: 8.87 %). On the other hand, the contents of calcium (31.7 %) and phosphorus (0.189 %) recorded by the MX1 brick are lower than those for the other MX2 bricks (Ca: 36.3 % and P: 1.84 %), MX3 (Ca: 39.9 % and P: 3.16 %) and MX4 (Ca: 43.7 % and P: 4.34 %). On the other hand, the contents of the other elements are very close for four other types of bricks.

The obtained results by XRF at a temperature of 900 °C are illustrated in Table 6 and Fig. 5. It appears from these results that the contents of the elements of silicon (33.4 %), aluminum (11.7 %) and iron (11.2 %) for MX1 are higher compared to those of three other MX2 bricks (Si: 29.7 %, Al: 10.4 % and Fe: 10.2 %), MX3 (Si: 26.4 %, Al: 9.11 % and Fe: 8.81 %) and MX4 (Si: 23.2 % Al: 8.29 % and Fe: 7.84 %). On the other hand, the calcium content (32.2 %) recorded by the MX1 brick is lower compared to the MX4 (Ca: 44.3 %), MX3 (Ca: 39.9 %) and MX2 (Ca: 36.4 %).

For the other elements, the contents are very close for four other types of bricks manufactured.

The obtained results by XRF at a temperature of 950 °C are presented in Table 7 and Fig. 6.

These results show that the contents of silicon (33.9 %), aluminum (11.9 %) and iron (11 %) elements for MX1 are high compared to the other three MX2 bricks (Si: 29.8 %, Al: 10.6 % and Fe: 10 %), MX3 (Si: 26.4 %, Al: 9.47 % and Fe: 8.96 %) and MX4 (Si: 23.4 % Al: 8.54 % and Fe: 7.82 %). However, the calcium content (Ca: 32.2 %) recorded by the MX1 brick is lower than that for the other MX2 bricks (Ca: 36.4 %), MX3 (Ca: 40.3 %)

Table 1

Percentage of phosphate wastes and clay

Sample	% Clay	% Phosphate wastes
Mixture 1 (**MX1**)	100	0
Mixture 2 (**MX2**)	90	10
Mixture 3 (**MX3**)	80	20
Mixture 4 (**MX4**)	70	30

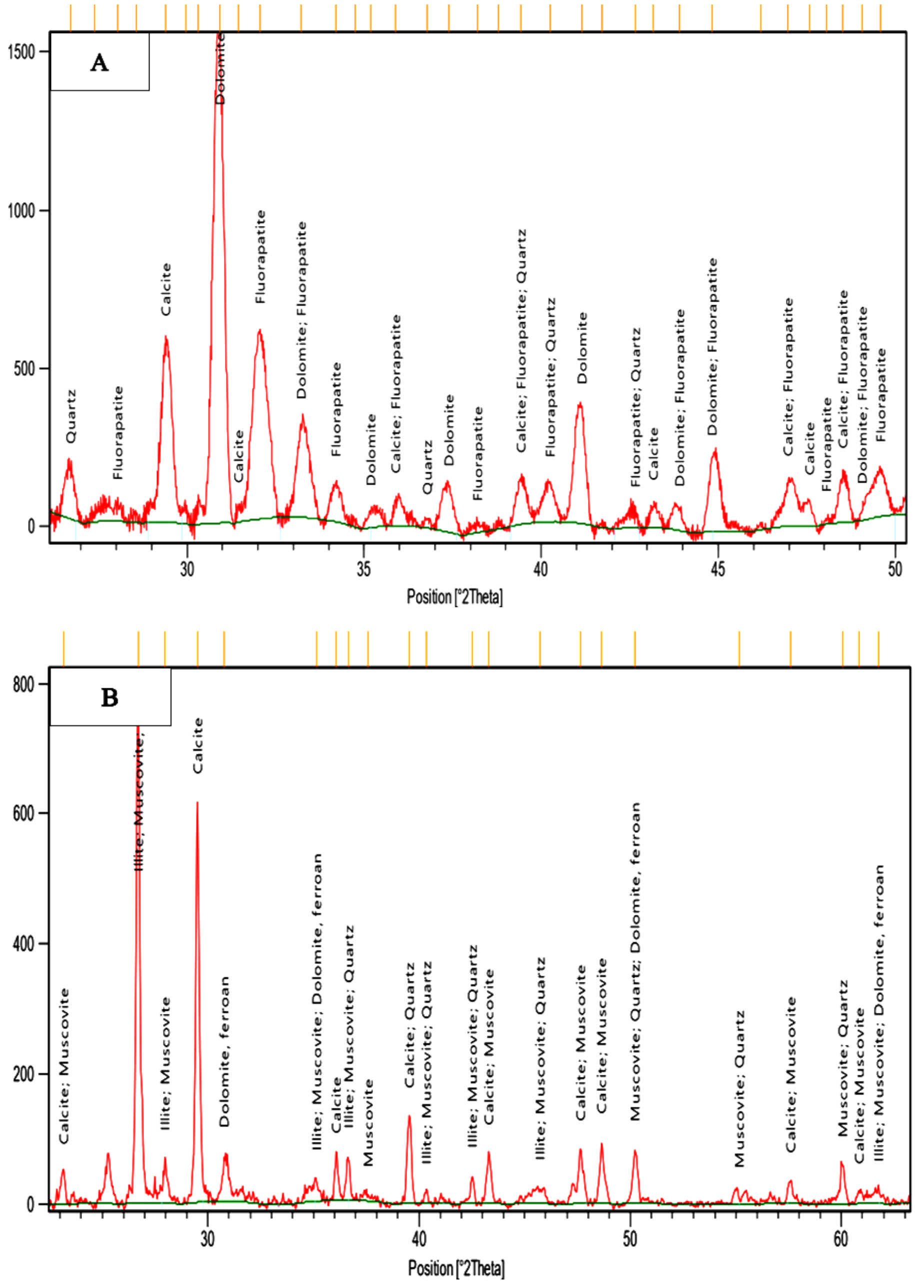

Fig. 1. X-ray diffractograms:
A — Phosphate wastes; B — Clay

Table 2

Identification of IR bands for the clay sample

Positions in cm^{-1}	Band intensities	Band identification
586.36	Strong	Deformation vibration of Si—O—Al
873.75	Weak	Asymmetric vibration Si—O—Al
1047.35	Very strong	Calcite $CaCO_3$
1436.97	Very strong	Carbonate CO_3^{-2}
2524.82	Weak	P—H bond
3425.58	Strong	Hydroxyls OH

Table 3

Identification of IR bands for phosphate wastes

Positions in cm^{-1}	Band intensities	Band identification
588.29	Strong	Deformation vibration of Si—O—Al
856.39	Weak	Asymmetric vibration Si—O—Al
1053.13	Very strong	Calcite $CaCO_3$
1429.25	Strong	Carbonate CO_3^{-2}
1579.70	Average	Aromatic C=C

and MX4 (Ca: 44.3 %). The rest of the elements presented very rocky grades for the other four types of bricks manufactured.

Bending and compressive strength tests for bricks. Mechanical resistance is one of the quality indicators required by construction standards. The mechanical characterization includes uniaxial compression and bending tests on the manufactured specimens and allows the evaluation of the suitability of a material for use in the field of construction.

We carried out the physical tests on different samples of bricks using the CMA compression device, Ko3313 at the laboratory of the Ain Kbira cement plant. It has the following characteristics:

C1: compressive strength test number 1.

C2: compressive strength test number 2.

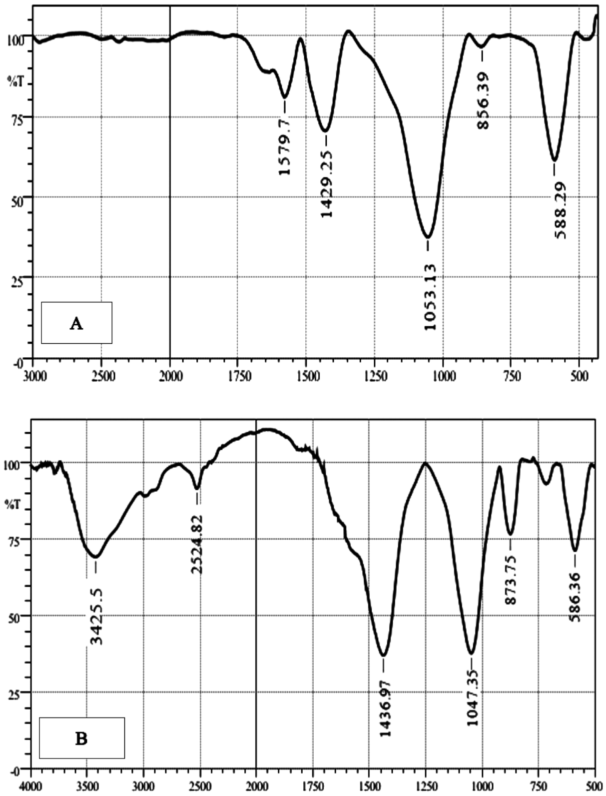

Fig. 2. Infrared spectrum of the sample:
A – Phosphate wastes; B – Clay

CM: average of the two compressions 1 and 2.
F: resistance to bending.

The obtained results of the flexural and compressive strength of the bricks at 850 °C are shown in Table 8 and Fig. 7. These results show that the flexural strength of the bricks for the 100 % clay mixture (MX1) 4.43 MPa is greater compared to that of the other MX2 mixtures: 3.62 MPa; MX3: 3.07 MPa and MX4: 2.89 MPa. Also, the compressive strength of MX1 (23.89 MPa) is higher than those obtained for the three mixtures MX2 (20.25 MPa), MX3 (17.12 MPa) and MX4 (16.03 MPa).

The obtained results of the flexural and compressive strength of the bricks at 900 °C are given in Table 9 and Fig. 8. It appears from these results that the flexural strength of 100 % clay bricks (MX1) 5.03 MPa is greater compared to those of

Table 4

Chemical composition of the used clay and of phosphate wastes

Elements	Contents in %	
	Clay	Phosphate waste
Ca	30.6	71.4
Si	34.9	4.82
Al	12.2	1.35
Fe	11.6	1.18
P	0.18	9.32
Mg	2.21	5.9
K	4.23	0.51
S	0.66	1.55
Na	1.33	0.66
Ti	1.22	0.14
F	–	2.42
Sr	0.2	0.5
Mn	0.1	0.04
Cl	0.41	0.1

Table 5

Chemical composition of bricks at 850 °C

Elements	Contents in (%)			
	MX1	MX2	MX3	MX4
Ca	31.7	36.3	39.9	43.7
Si	34.3	30.1	26.3	23.5
Al	11.9	10.6	9.47	8.53
Fe	11	10.1	8.78	7.8
P	0.19	1.84	3.16	4.34
Mg	2.66	3.19	3.89	4.29
K	3.93	3.48	3.03	2.65
S	1.19	1.29	1.4	1.47
Na	1.38	1.37	1.38	1.24
Ti	1.17	1.07	1.17	0.9
F	–	–	0.9	0.9
Sr	0.19	0.24	0.26	0.28
Mn	0.09	0.08	0.09	0.08
Cl	0.15	0.22	0.18	0.29

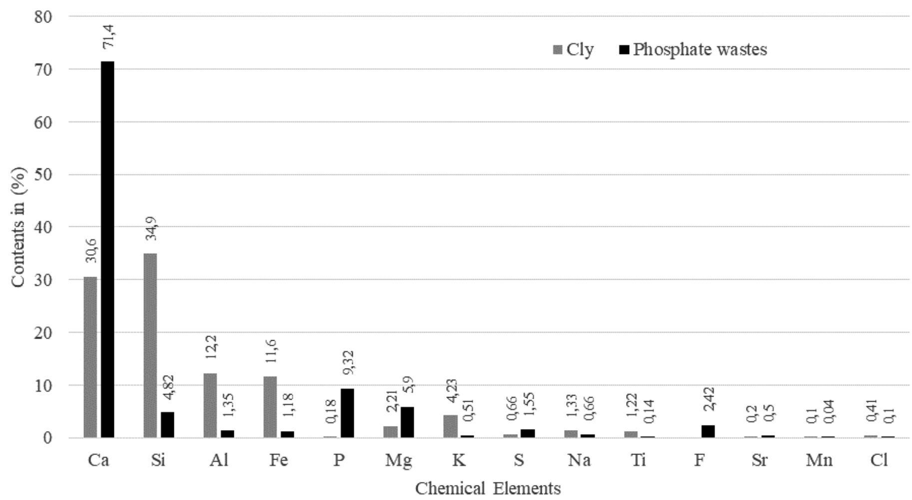

Fig. 3. Chemical composition of the used clay and of the phosphate wastes

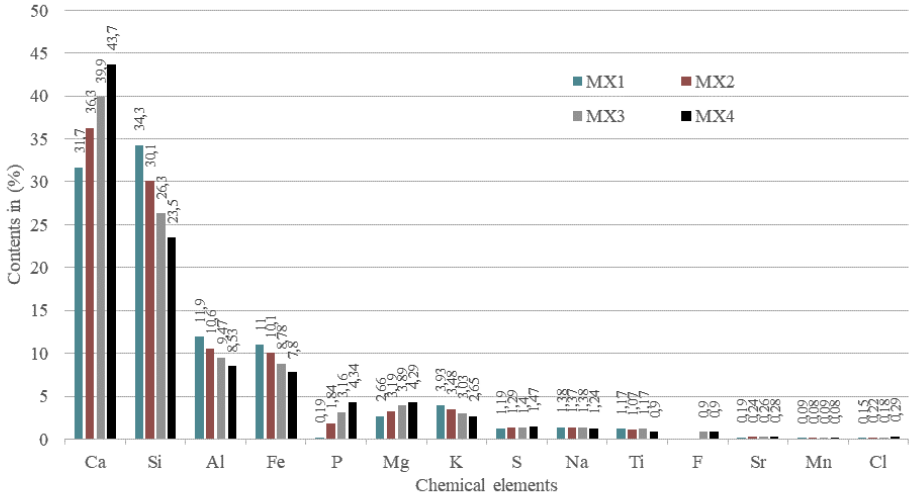

Fig. 4. Chemical composition of the bricks by XRF at a temperature of 850 °C

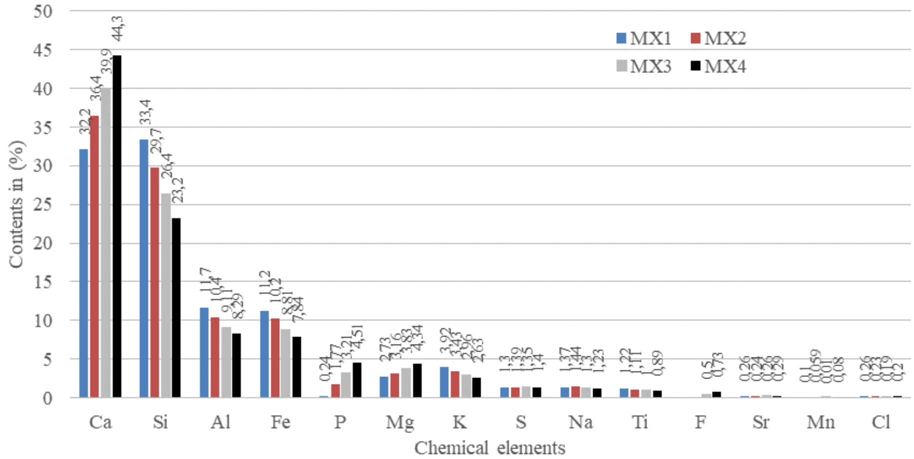

Fig. 5. Chemical composition of the bricks by XRF at a temperature of 900 °C

Table 6

Chemical composition of bricks at 900 °C

Elements	Contents in (%)			
	MX1	MX2	MX3	MX4
Ca	32.2	36.4	39.9	44.3
Si	33.4	29.7	26.4	23.2
Al	11.7	10.4	9.11	8.29
Fe	11.2	10.2	8.81	7.84
P	0.24	1.77	3.21	4.51
Mg	2.73	3.16	3.83	4.34
K	3.92	3.43	2.96	2.63
S	1.3	1.39	1.35	1.4
Na	1.37	1.44	1.3	1.23
Ti	1.22	1.11	1	0.89
F	–	–	0.5	0.73
Sr	0.26	0.24	0.256	0.29
Mn	0.1	0.059	0.01	0.08
Cl	0.26	0.23	0.19	0.2

Table 7

Chemical composition of bricks at 950 °C

Elements	Contents in (%)			
	MX1	MX2	MX3	MX4
Ca	32.2	36.4	40.3	44.3
Si	33.9	29.8	26.5	23.4
Al	11.9	10.6	9.47	8.54
Fe	11	10	8.96	7.82
P	0.19	1.78	3.05	4.38
Mg	2.61	3.06	3.62	4.13
K	3.69	3.24	2.29	2.44
S	1.26	1.33	1.29	1.32
Na	1.45	1.56	1.39	1.31
Ti	1.18	0.97	0.93	0.75
F	–	0.54	0.94	0.94
Sr	0.21	0.23	0.26	0.29
Mn	0.1	0.11	0.07	0.08
Cl	0.14	0.17	0.17	0.15

other mixtures MX2: 3.94 MPa, MX3: 3.76 MPa and MX4: 3.43 MPa. Also, the compressive strength of MX1 (26.76 MPa) is higher than that of the other mixtures MX2 (25.32 MPa), MX3 (19.59 MPa) and MX4 (15.03 MPa).

The results of the flexural and compressive strength of the bricks obtained at 950 °C are shown in Table 10 and Fig. 9. These results show that the flexural strength of 100 % clay bricks (MX1): 3.76 MPa is greater than those of the other mixtures MX2: 3.07 MPa, MX3: 2.63 MPa and MX4: 2.53 MPa. The compressive strength of MX1 (23.50 MPa) is higher than that of the other three mixtures MX2 (19.40 MPa), MX3 (18.26 MPa) and MX4 (12.74 MPa).

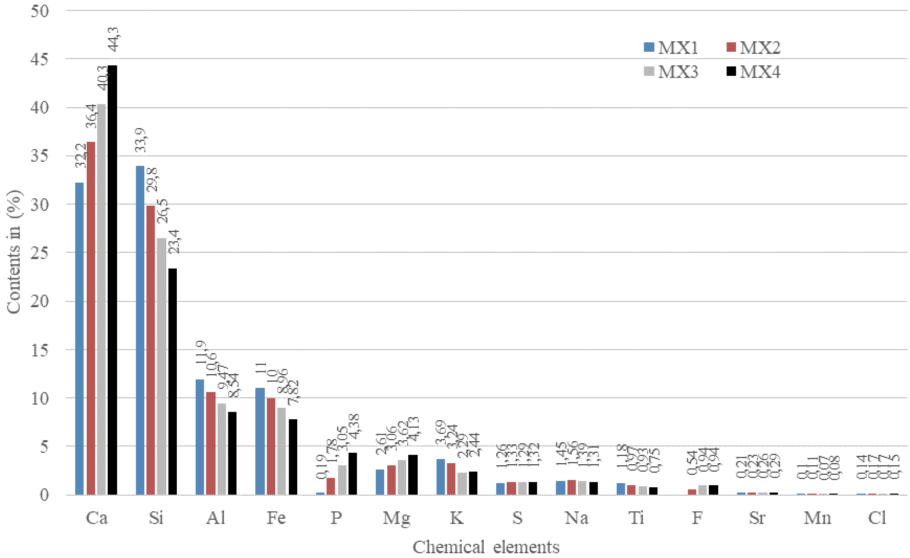

Fig. 6. Chemical composition of the bricks by XRF at a temperature of 950 °C

Table 8

Results of the flexural and compressive strength of the bricks at 850 °C

Flexural and compressive strength in (MPa)	Mixtures			
	MX1	MX2	MX3	MX4
F	4.43	3.62	3.07	2.89
C1	23.25	20.7	15.3	17.23
C2	24.54	19.81	18.94	14.83
CM	23.89	20.25	17.12	16.03

Table 9

Results of the flexural and compressive strength of the bricks at 900 °C

Flexural and compressive strength in (MPa)	Mixtures			
	MX1	MX2	MX3	MX4
F	5.03	3.94	3.76	3.43
C1	29.88	24.31	21.58	12.72
C2	23.64	26.34	17.61	17.35
CM	26.76	25.32	19.59	15.03

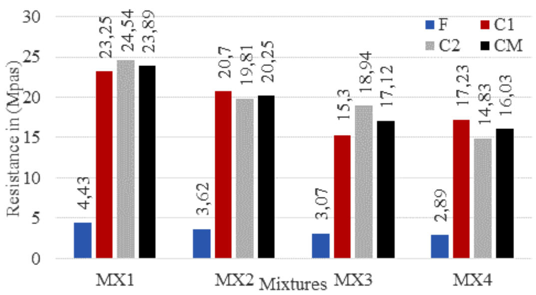

Fig. 7. Histogram of resistance (MPA) to flexural and compressive brick at 850 °C

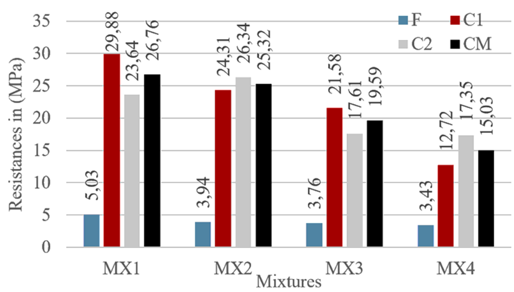

Fig. 8. Histogram of flexural and compressive strength of bricks at 900 °C

The obtained results of the physical tests showed that the best flexural strengths are recorded at a temperature of 850 °C for the four types of bricks (MX1: 5.03 MPa, MX2: 3.94 MPa, MX3: 3.76 MPa and MX4: 3.43 MPa). However, the best compressive strengths are recorded at a temperature of 900 °C for the other bricks (MX1: 26.76 MPa, MX2: 25.32 MPa, MX3: 19.59 MPa and MX4: 15.03 MPa). On the other hand, the best compression result is recorded by the 100 % clay MX1 brick because of the high content of silicon (34.3 %) and low calcium (31.7 %) compared to the other MX2 bricks (30.1 and 36.3 %), MX3 (23.5 and 43.7 %) and MX4 (26.3 and 39.9 %). Silicon played a role as a filler material, generally used in the manufacture of fired bricks to reduce the plasticity and increase the hardness of the brick. Silica is known for its role in the formation of the skeleton of baked products [11].

Table 10

Results of the flexural and compressive strength of the bricks at 950 °C

Flexural and compressive strength in (MPa)	Mixtures			
	MX1	MX2	MX3	MX4
F	3.76	3.07	2.63	2.53
C1	21.98	21.62	19.45	12.89
C2	25.03	17.19	17.07	12.59
CM	23.50	19.40	18.26	12.74

Conclusion. The characterization of our samples made it possible to define the nature of the clay rich in silicon (34.9 %), aluminum (12.2 %), iron (11.6 %) and potassium (4.23 %) compared to the phosphate wastes: silicon (4.82 %), aluminum (1.35 %), iron (1.18 %) and potassium (0.510 %). Phosphate wastes are characterized by high calcium (71.4 %) and magnesium (5.90 %) contents compared to clay: 30.6 % (calcium) and 2.21 % magnesium.

The presence of silicon in high contents plays a role of filler materials, used in the manufacture of fired bricks to reduce the plasticity and increase the hardness of the brick. Aluminum makes it possible to improve the plasticity of the mix-

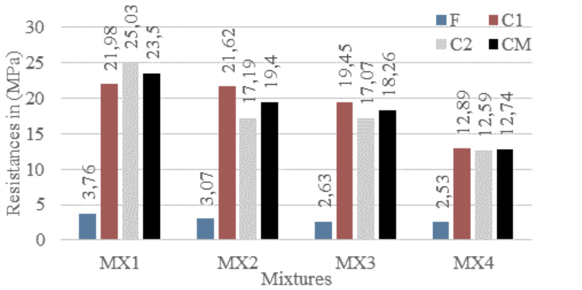

Fig. 9. Histogram of flexural and compressive strength of bricks at 950 °C

tures and to obtain pressed products with significant reliefs. Calcium, potassium, sodium and magnesium are also considered fluxing elements and tend to combine with silicates during firing. The rest of the chemical elements have similar contents. The high calcium content of over 30 % allows it to be categorized as a very workable and plastic calcareous clay.

The obtained results of the mechanical tests made it possible to determine the best flexural strength at a temperature of 900 °C for the four types of bricks (MX1: 5.03 MPa, MX2: 3.94 MPa, MX3: 3.76 MPa and MX4: 3.43 MPa). The same applies to the best compressive strengths (MX1: 26.76 MPa, MX2: 25.32 MPa, MX3: 19.59 MPa and MX4: 15.03 MPa) since the verification mechanisms generally start at around 900 °C and end at around 1,050 °C [11].

On the other hand, the best compression result is recorded by the 100 % clay MX1 brick because of the high content of silicon (34.3 %) and low calcium (31.7 %) compared to the other MX2 bricks (30.1 and 36.3 %), MX3 (23.5 and 43.7 %) and MX4 (26.3 and 39.9 %). Silicon played a role as a filler material, generally used in the manufacture of fired bricks to reduce the plasticity and increase the hardness of the brick. The increase in the quantities of phosphate wastes decreases the presence of silica, alumina and iron and results in a loss in the compressive strength.

It can be concluded that the best flexural strength results are obtained at a firing temperature of 850 °C. For the four types of bricks, the resistance is greater than 2.5 MPa. This meets well the construction standards of American Standard and Testing Materials (ASTM-C674, 1999).

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Використання відходів природного фосфату у виробництві будівельної цегли

Т. Тахрі[1], *Н. Беззі*[1], *А. Бузензана*[2], *К. М. Бенгадаб*[3], *А. Бенселгуб*[4]

1 – Лабораторія матеріалознавства та технології процесів (ЛМТП), Університет Беджаї, м. Беджая, Алжир, e-mail: tahri2008@yahoo.fr

2 – Гірнича лабораторія, Гірничий інститут, Університет Ларбі Тебессі, м. Тебесса, Алжир

3 – Лабораторія металургії та матеріалознавства, Університет Баджі Мохтар, м. Аннаба, Алжир

4 – Відділ навколишнього середовища, моделювання та зміни клімату, Науково-дослідний центр з охорони довкілля, м. Аннаба, Алжир

Мета. Валоризація фосфатних відходів і диверсифікація асортименту цегли за рахунок покращення її механічних властивостей. Наша робота у цьому контексті була зосереджена на використанні відходів фосфату із Джебель Онк (Тебесси) у комбінації із глинами регіону Сіді Айх (Беджая) з метою виробництва нового сорту цегли. Суть ідеї полягає у зміні процентного вмісту цих відходів і температури випалу.

Методика. Після механічної підготовки в лабораторії, що складається з операцій дроблення, подрібнення та просіювання через грохот, відбувається аналіз двох матеріалів «фосфату та глини» декількома методами (XRD, IR та XRF). Випробування цегли, виготовленої з цих двох матеріалів, проводилися при різних вагових відсотках і різних температурах випалу.

Результати. Поєднання цих двох матеріалів являє собою новий підхід до використання природних фосфатів, що мають дуже високу норму відходів. Випробування виготовлення цегли із цих двох матеріалів дали обнадійливі результати. Із механічної точки зору, найкраща міцність на вигин отримана за температури 900 °С, знаходиться в діапазоні від 3 до 5 МПа залежно від типу суміші фосфату/глини. Так само й міцність на стискання, що варіюється від 15 до 27 МПа в залежності від типу суміші. Ці результати відповідають будівельним нормам ASTM-C674 1999 року.

Наукова новизна. Використання відходів природного фосфату із Джебель Онк у різних пропорціях дозволило отримати якість цегли, яка відповідає міжнародним стандартам.

Практична значимість. Механічні властивості цегли, виготовленої із цих двох матеріалів, такі як міцність на вигин та стискання, стають основою дійсно ефективного способу утилізації відходів фосфату із Джебель Онк.

Ключові слова: *відходи фосфатів, глина, виробництво, цегла*

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