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INFLUENCE OF PARTICLE SIZE OF MUSSEL SHELLS IN PHYSICAL, MECHANICAL AND INSULATING PROPERTIES OF FIREPROOF MATERIALS*

Begoña Peceño[1], Bernabe Alonso-Fariñas[2], Celia Arenas[2], Carlos Leiva[2]**

[1]*Universidad Católica del Norte, Larrondo 1281,Coquimbo, Chile*
[2]*Universidad de Sevilla. Camino de los Descubrimientos s/n 41092, Seville, Spain*

Abstract

In this study, the behavior of mussel shell as a component of passive fire protection materials was evaluated. To investigate the performance of mussel shell waste, different gypsum replacement ratios were analyzed from 40 to 80 % wt. Two different kinds of shells, separately or as a mix, were employed. In addition, two different particle size distributions, smaller than 320 μm and bigger than 320 μm, were used. Physical, mechanical, insulating capacity and leaching properties were thoroughly analyzed. Our results indicate that replacements lower than 60 % wt comply with all mechanical demands and have no reduction in insulating capacity. Additionally, no leaching problems were detected. Mortars made by combination of mussel shell and gypsum have the potential to be used as a component in construction materials for passive protection against fire.

Keywords: circular economy, mussel shell, fire insulating, recycling waste valorization

1. Introduction

Generated waste is increasing, becoming a major public health and environmental problem. The aquaculture industry produces some 6.000.000 to 8.000.000 tons of waste worldwide annually and only 25 % of that is recycled (Yan and Chen, 2015). In Chile, mollusk trap industry generated 215.766 tons of waste in 2015 (Servicio Nacional de Pesca y Acuicultura, 2015). Nowadays, most of the shell waste is deposited in landfills. One of the main environmental problems of mollusks waste is bad odor from the decomposition of organic matter (Kwon et al., 2004). On the other hand, when the properties of a waste

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**Corresponding author: e-mail: begopc@ucn.cl

product are useful for applications with high added value, it's possible to obtain new products that can compete with those created from traditional materials (Vilches et al., 2003).

In this research work, the application of medium temperature (250 ºC) calcined mussel shell waste as substitution material of gypsum in fireproofing panels manufacturing is proposed. Fireproofing materials restrict the spreading of fire for certain periods, insulating steel structures from the effects of the high temperatures and provide the firefighting teams with sufficient time to intervene before the fire expands. Gypsum is employed in the construction industry as a fireproof material for walls and ceilings due to its very good mechanical and thermal properties and its high resistance to fire. Endothermic dehydration of gypsum carried out at high temperatures can slow fire propagation through structures made with gypsum materials (Kontogeorgos and Founti, 2012). The high $CaCO_3$ concentration in the shell waste makes it a good candidate to be used as fireproofing materials due to the positive effect of the endothermic reaction of decomposition of $CaCO_3$ to CaO and CO_2 on the fireproofing properties. Under a circular economy approach, this alternative would allow reducing the use of a row material, gypsum, in fireproofing panel manufacturing by recycling, with a low energy and economic cost, mussel shell waste, which is nowadays accumulated in landfills.

2. Material and methods

2.1. Materials

In this study, the performance of shells from Mediterranean mussel (M) was studied along with commercial gypsum (G). In this study, commercial gypsum was used according to (EN 13279-1, 2009).

The chemical composition of mussel and gypsum is shown in Table 1 and was determined for both majority and minority elements, following the ASTM D 3682 standard (ASTM D3682-13, 2013). As can be seen, CaO is the major component in the shells, presented as $CaCO_3$. The calcination losses of mussel is high, mainly because $CaCO_3$ decomposes at 650 °C in CaO and CO_2, the latter representing the calcination losses (LOI).

Table 1. Chemical composition (%wt)

	G	*M*
SiO_2 (%)	0.61	N.D
Al_2O_3 (%)	0.82	0.02
Fe_2O_3 (%)	0.43	0.02
MnO (%)	N.D.	0
MgO (%)	0.24	0.1
CaO (%)	42.7	53.74
Na_2O (%)	0.07	0.46
K_2O (%)	0.24	0.02
TiO_2 (%)	N.D.	N.D.
P_2O_5 (%)	N.D.	0.02
SO_3 (%)	47.92	0.24
PC (%)	6.1	43.43

The pretreatment carried out on mussel was as follows: firstly, the shells were washed with water to eliminate salts on their surface. Subsequently, drying was carried out at 190 °C for 18 minutes. After drying, the shells were crushed by a jaw crusher to a particle size of less than 1 mm. Then, to remove organic matter retained in the shells, calcination was carried out at 250 °C for one hour (Barros et al., 2007). Once the shells were calcinated, they were ground with a hammer. Commercial gypsum is composed mainly by $CaSO_4$, although presents a 6 % wt of calcium carbonate in this composition.

Fig. 1 shows the granulometric distributions of the materials used. To study how particle size of the waste affects the physical, mechanical, and fire-insulating properties of the final product, the mussel shells were passed through a 320 μm sieve, obtaining two granulometric particle size distributions of the two fractions of mussel, coarse (M-C; d_p > 320 μm) and fine (M-F; d_p < 320 μm). Fig. 1 shows the gypsum particle size distribution is similar to M-F.

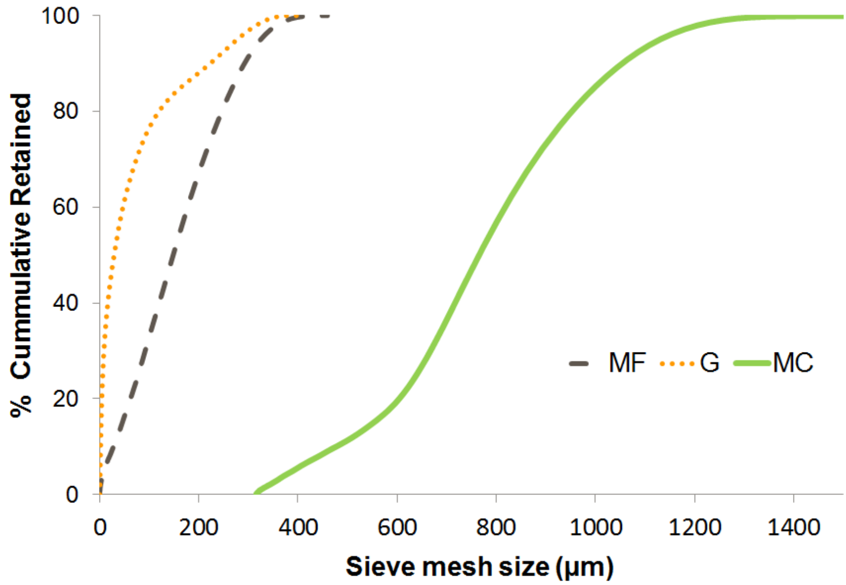

Fig. 1. Particle size distribution

Table 2 shows the average particle sizes of each fraction used (M-F, M-C, and G) and the specific gravity of the materials used. Specific gravity was obtained by (ASTM D854-14, 2014).

Table 2 shows that the average size of the coarse fraction (M-C) was almost 6 times greater than that of the fine fraction (M-F). Specific gravities of M-F and M-C were the same due to specific gravity is a parameter independent of the granulometry of the solids.

Table 2. Average particle size and specific gravity (g/cm^3)

	G	*M-F*	*M-C*
Median size (μm)	29.83	147	765
Specific gravity (g/cm^3)	3.3	2.67	2.67

2.2. Specimen manufacturing method

Product specimens were manufactured following a simple and low-cost method under standard laboratory conditions (temperature: 25 °C and pressure: 1 atm). To prepare the specimens, the materials were weighed and mixed according to the amounts of mussel and gypsum indicated in Table 3. The water/waste ratio was 0.50. Table 3 shows the different compositions generated. Table 3 shows how amount of mussel and particle size affect the physical, mechanical, and passive fire protection properties.

The experiments were carried out with three different types of specimens: cylindrical molds of 4.2 cm diameter and 20 cm height, used for the fire insulating capacity experiments; prismatic test pieces of 10 cm length, 4 cm width and 4 cm height, used to determine flexural resistance; and finally, small cylindrical molds of 34 mm diameter and 40 mm height that were used for the remaining tests performed in this study. Fig. 2 shows the different types of specimens used.

Table 3. Composition (%wt) of specimens

	Compound		***Particle size (µm)***
	G (%wt)	***M (%wt)***	
100-G	100	0	-
40M-C	60	40	d_p > 320µm
60M-C	40	60	
80M-C	20	80	
40M-F	60	40	d_p < 320µm
60M-F	40	60	
80M-F	20	80	

a b c

Fig. 2. Types of specimens manufactured
a. Compressive strength, b. Flexural strength, c. Fire resistance

2.3. Methods
2.4.
2.4.1. Physical and chemical properties

The thermogravimetry (TG) test was carried out using a Mettler-Toledo thermogravimetric analyzer, model TGA/SDTA 851. Between 100 and 150 mg samples were placed in an aluminum tray and brought up to 1000 °C at a heating rate of 20 °C/min in a nitrogen atmosphere (Li et al., 2015).

The density (d) measured according to (ASTM E605/E605M-19, 2019).

The moisture content (W) was measured according to (EN 12859, 2012). The mass of the product was measured at room temperature (M1). Then, it was heated to 105 °C, until the free water was eliminated; that is, until a constant mass was reached (M3). Humidity was calculated by the moisture content (W) as follows:

$$W(\%)= (M2-M1)/ M1 \quad (1)$$

To measure pH, a procedure described in the European standard (EN 12859, 2012) was used. This test measured the pH of a solution composed of 1 gram of the specimen that was obtained by scraping the surface of a specimen and dissolving it in 10 grams of demineralized water.

2.4.2. Mechanical properties.

Compression (S_C) and flexural (S_F) resistance tests were performed. The test machine used for both is a Servosis model MUE-403. The compressive strength and the flexural strength were measured according to (EN 13279-2, 2014).

The surface hardness (D) was measured according to the (EN 13279-2, 2014) standard, with the help of a Shore C durometer. The test was carried out with prismatic specimens and for each face of the specimen 5 essays were done. Surface hardness is the arithmetic mean of ten measurements made for each of the tested specimens, expressed in Shore C units.

2.4.3. Fire insulating capacity

The fire insulating test was carried out on the cylindrical specimens that were subjected to the heating regimen, in accordance with the temperature curve of a fire indicated by the European standard (EN 1363-1, 2020), which follows the expression:

$$T= 20 + 345\log (8t + 1) \quad (2)$$

Where, T is the oven temperature for the tests (°C) and t is the duration (minutes). To measure temperature in the center of the cylinder (T_{int}), a type K thermocouple of 3 mm diameter was used. However, to measure the outside temperature (T_{out}), an S-type thermocouple of 3.5 mm diameter was used, as shown in Fig. 3.

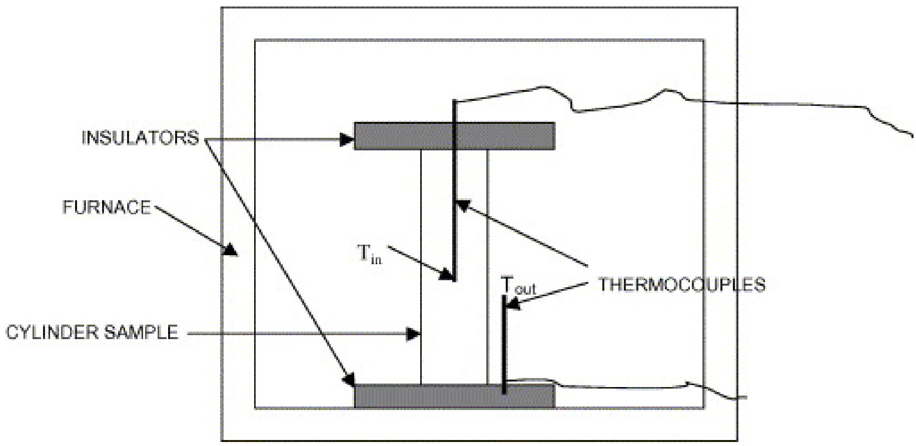

Fig. 3. Experimental set-up for fire resistance test

The insulating capacity is the time necessary to reach 600 ºC in the centre of the cylinder, because at this temperature, the steel protected by the fireproof material would loses its mechanical properties.

2.4.4. Leaching tests

To facilitate the use of mussel waste as a construction material, the product developed in the present study should guarantee low toxicity. Considering this, an environmental study was carried out for a broader characterization of the product generated with mussel and, therefore, a better evaluation of the potential uses of this product.

Leaching methods can be classified as: (1) batch leaching, in which the specimen is placed in a given volume of leach solution, (2) as a column or flow through systems, and (3) as a flow *en masse* or flow around systems for monolithic specimens (Kim, 2005). The chosen leaching method was NEN 7375 (1995) because it works with monolithic specimens that can be assimilated into the final construction product and it is the most similar to rain, the most common leaching conditions. The specimens measured were those with the highest percentage of mussel which is 80M-F, and 100-G. In (EA NEN 7375:2004, 2005), the leaching fluid (water at pH = 7) is renewed 8 times, which is similar to rain, the main leaching factor of construction materials. The metal leachate analysis was carried out using atomic absorption spectrophotometry and inductive coupling plasma techniques. Three different specimens of the final product were subjected to this test (Leiva et al., 2015).

3. Results and discussion

3.1. Physical and chemical properties

Fig. 4 shows the curves obtained from the thermogravimetry test for the mussel, and gypsum. The reduction in mussel weight is due to: (1) the evaporation of moisture at 100 °C (Asako et al., 2004), (2) the transformation of aragonite to calcite, between300 and 500 ºC (Hu et al., 2011), and (3) the endothermic reaction of carbonates ($CaCO_3$ to CaO and CO_2 between 600 and 800 ºC (Payá et al., 1998). This is the most significant drop in weight is observed, which comprises 40% of the total weight loss for mussel.

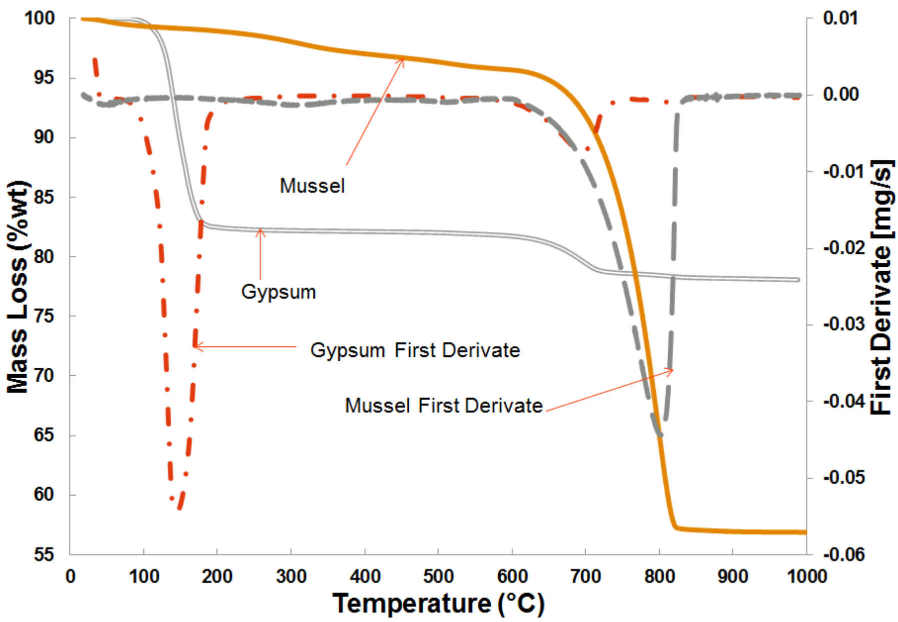

Fig. 4. Mass loss and TG first derivative of raw materials

The mass of the gypsum, however, remains constant up to 100 ºC. It begins to decrease, since it loses moisture and chemically-bound water. During heating, there are two reactions, around 120 °C and around 200 °C, reaction 1 and 2, respectively (Binici and Aksogan, 2017; Thomas, 2002).

$$CaSO_4{\cdot}2H_2O \rightarrow CaSO_4{\cdot}1/2H_2O + 3/2\ H_2O \qquad \text{(Reaction 1)}$$

$CaSO_4 \cdot 1/2H_2O \rightarrow CaSO_4 + 1/2\ H_2O$ (Reaction 2)

Above 600 °C, decomposition of calcium carbonate present in gypsum occurs.
In Table 4, the physical and chemical properties of the products generated are shown.

Table 4. Physico-chemical properties of shell gypsum

	d (kg/m³)	*W (%wt)*	*pH*
100-G	1485	7.81	8.43
40M-C	1440	6.63	6.85
60M-C	1375	5.46	6.82
80M-C	1305	3.95	6.80
40M-F	1465	6.45	6.86
60M-F	1430	5.23	6.82
80M-F	1315	3.96	6.81

The densities of the products generated are between 1305 and 1485 kg/m^3, therefore, according to (EN 12859, 2012), all final products are classified as high density (>1100 kg/m^3). Table 4 shows that increasing the percentage of mussel decreases the density of the product. This is because the specific density of the shell is lower than the specific density of gypsum (Li et al., 2015), and the particle size of gypsum is slightly lower. The densities are slightly lower in the coarse fraction, due to the fact that increasing the particle size decreases the porosity and therefore the density (Muñoz et al., 2013).

With regard to humidity, as the proportion of waste increases, the humidity of the specimens decreases. This is because the amount of free water in the waste is less than the amount of free water in the gypsum, as indicated in Fig. 4. In accordance with the requirements in (EN 12859, 2012), the moisture content (W) must be less than 8 %, therefore, all test pieces manufactured comply with the regulations.

According to the standard (EN 12859, 2012), all the specimens would be classified as normal pH because they are all between pH 6.5 and 10.5. Table 4 shows that as the ratios of shell wastes in the manufactured specimens increase, the pH doesn’t change significant.

3.2. Mechanical properties

An increase in shell composition decreases the compressive strength (Table 5).

Table 5. Mechanical properties of shell-based and commercial gypsums

	Sc (MPa)	*S_F (MPa)*	*Hardness (Shore C)*
100-G	14.20	7.10	94
40M-C	9.68	3.00	85
60M-C	4.15	1.90	80
80M-C	2.65	0.95	49
40M-F	12.30	3.40	92
60M-F	5.17	2.22	81
80M-F	3.11	1.07	61

With respect to 100-G, the compressive strength decreases between 27.5 % and 88.4 %. According to the (EN 13279-1, 2009), all the produced specimens fulfill the requirement that the compressive strength not be less than 2.0 MPa. The same tendency occurs with S_F when the waste composition increases. The addition of shell waste in the specimens causes a decrease of S_F, varying from 47.5 % to 85.0 %, with respect to the 100-G product. The (EN 13279-1, 2009) indicates that the flexural strength in gypsum panels must be higher than 0.83 MPa; therefore, all the produced specimens comply with this requirement. The densest specimens are the ones with the best mechanical properties due to its lower porosity (Leiva et al., 2015).

In Table 5, the average hardness of each manufactured mortar is shown. The surface hardness of the panel decreases with an increase in the composition of the shells. The lowest value of hardness corresponds to the coarse particle size, 49 Shore C. Because all the mortars were classified as high density, as established by the standard (EN 12859, 2012), all must meet a minimum hardness of 80 Shore C. That is why only mortars with 80 %wt mussel shells do not meet this requirement.

3.3. Fire-insulating properties

In Fig. 5, the fire insulating curve for fine granulometric is shown. In both figures the reference curve (100-G) is shown.

Fig. 5 shows that temperature remained approximately constant over time for about 2.5 minutes. After 4 minutes (at 100 ºC approximately) the water presents in the material begins to evaporate, maintaining constant the temperature in the center of cylinder and producing the called evaporation plateau. The duration of the evaporation plateau is one of the most important factors (together with the slope of the curve after the plateau) in the fire-insulating, and it is proportional to the water (free and chemically bound) present in the material.

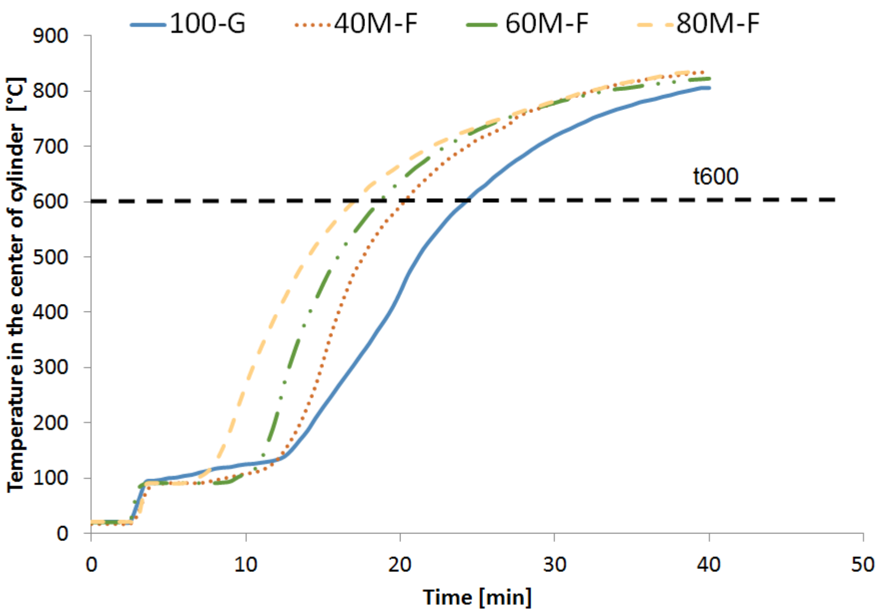

Fig. 5. Fire-insulating capacity of 100-G, 40M-F, 60M-F, 80M-F materials

Fig. 5 shows that the highest evaporation plateau is found in the pure gypsum specimen, and as the concentration of shell waste increases in the specimens, evaporation plateau is shortened. This is because the gypsum specimens have a greater amount of chemically bound water than the shell wastes specimens, as shown in Fig. 4. The other important factor is the slope after the evaporation plateau, when the content of shells is increased the slope of the curve is lower, increasing the t600, it is due to the high calcium carbonate present in the shells, which is decomposed endothermically, but this reaction is produced in a high range of temperature (between 600 and 800 ºC as it can be seen in Fig. 4) and it cannot produce a plateau, only a diminution of the slope of the curve at high temperatures. With the interaction of those two factors as the shell waste concentrations in the specimens increased, the t600 slightly decreased.

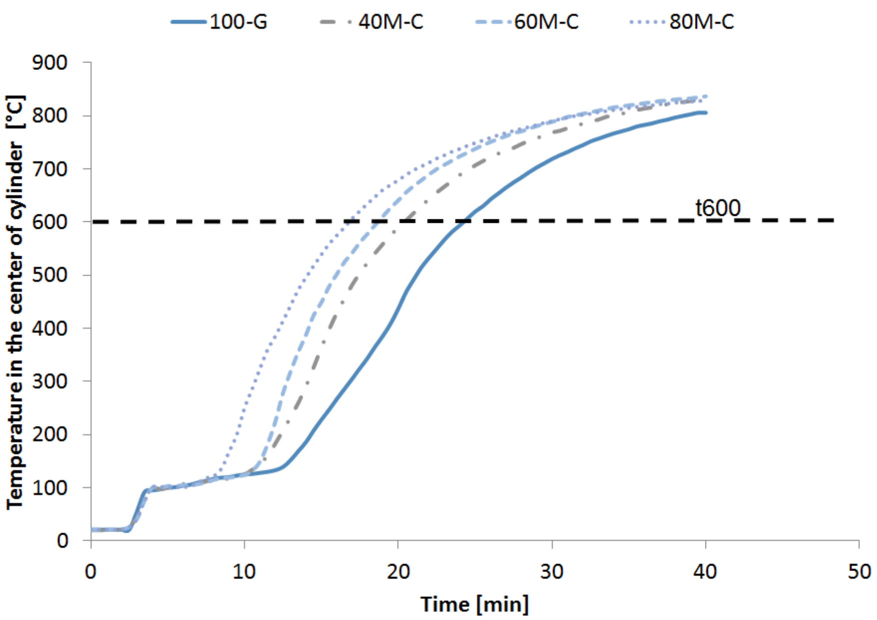

Fig. 6. Fire-insulating capacity of 40M-C, 60M-C, 80M-C and 100-G materials

In Fig. 6, the fire insulating curve for coarse granulometric is shown. In both figures the reference curve (100-G) is shown.

For different granulometries, the evaporation plateau of the specimen 40M-C decreased by approximately 20 % with respect to the specimen 40M-F, materials with a lower density present a high porosity and the evaporated water can escape more easily, decreasing the duration of the evaporation plateau and as the density is lower the amount of calcium carbonate is lower and the slope is higher.

3.4. Environmental properties

In general, when using recycled materials, the main environmental problem can be the release of heavy metals into the environment through leaching. There are various studies showing the presence of heavy metals in mollusks and their shells (Palpandi and Kesavan, 2012; Sivaperumal et al., 2007; de Souza et al., 2016). According to Sivaperumal et al. (Sivaperumal et al., 2007), there is selenium in 93 % of mollusks. There is also a large probability of containing cobalt (83 %) and arsenic (80 %), respectively, as well as other metals such as copper and manganese (Sivaperumal et al., 2007). In Spain, there is no legal requirement to use recycled wastes in fireproof materials. Given the scarcity of existing standards regarding the reuse of waste in the construction industry and their environmental or health risks, the present study compared the results of the leaching test with regulations established in the Dutch Soil Quality Decree (Decree 469, 2007). It establishes norms destined to prevent surface and groundwater contamination from construction material leaching and the monolithic test to carry out, (EA NEN 7375:2004, 2005). Table 6 contains the leaching concentrations of chemical elements accumulated in 64 days for the produced materials with the largest amount of waste (80M-F). In addition, all the materials with mussel wastes meet to the limits set by the (Decree 469, 2007) standard, even, they are considerably smaller than those of the G-100.

Table 6. Results of the leaching test for 80M-F specimen compared to (Decree 469, 2007) limits

	Decree 469 limits	***100-G***	***80M-F***
Hg	1.4	0.68	0.68
Se	4.8	4.5	0.34
Sn	50	2.3	0.34
Pb	400	3.4	0.14
Ba	1500	2.2	1.20
Cd	3.8	0.4	0.03
Sb	8.7	2.3	0.07
Co	60	0.2	0.03
Cr	120	0.2	1.03
V	320	2.3	0.12
As	260	3.4	0.34
Mo	144	1.1	0.14
Ni	81	1.1	0.07
Zn	800	6	0.07
Cu	98	0.4	0.07

4. Conclusions

The results of this study lead to the conclusion that a novel environmental-friendly from gypsum and mussel shell wastes may have the potential to be used as a fireproof material because it presents similar characteristics to those of other traditional products in this industry. In addition, since there are no appreciable differences in their physical, mechanical, insulating and environmental properties, products can be built from different size particle.

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