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Recyclability Process of Standard and Foamed Gypsum

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Abstract

Gypsum is an ancient material, still widely used and suitable for many applications in the constructions, due to its low cost, availability, lightweight, good thermal and sound isolating behavior, fire resistance and low energy consumption. Nowadays gypsum have several applications spacing in different engineering fields. For example, on one hand it can be adopted as pattern material to produce high quality composite pipes via autoclave and on the other it can be adopted as building material for the production of gypsum walls that in some case can be reinforced by vegetable or synthetic fibers. These applications, that can appear completely different and unlinked, are characterized by an important common key point: the possibility of using more and more times the gypsum material. Therefore, considering that nowadays every industrial company needs to decrease the materials waste, increase recyclability and use more eco-friendly materials; this paper aims on the study of the recyclability process of both standard and lightweight gypsum. For the production of the lightweight gypsum, a vegetable protein was adopted as foaming agent and the manufacturing technique was here discussed. Bending and compression tests were preliminary conducted to know the starting mechanical properties of both standard and foamed gypsum. Then, tested specimens were reduced in small pieces and subjected to the recycling process. The recycling process mainly consists in two steps: grinding for the reduction of gypsum in powders of established granulometry size and heat treatment of the powders to pass from dehydrate to hemihydrates phase of gypsum. Then the effects of the grinding time, temperature and time of the heat treatment on the flexural and compression behavior of gypsum up to 4 recycles were investigated. The results showed that if the recycling parameters are properly setted, both foamed and standard gypsum can be recycled up 3 times without significant variation of the mechanical properties and of the gypsum mixture workability.

Keywords: gypsum; recycling; foaming; compression strength; flexural strength.

1. Introduction

Gypsum is an air-hardening binding material produced from calcination calcium sulfate dehydrate, i.e. $CaSO_4{\cdot}2H_2O$, that partially dehydrates producing gypsum plaster, i.e. calcium sulfate hemihydrates, $CaSO_4{\cdot}0.5H_2O$.

Gypsum is one of the earliest building materials and its application history can be traced back to 4000 years ago. Thanks to its various outstanding advantages such as the easy fabrication, low energy consumption and price, good aesthetic appearance, good fire resistance, sound and thermal isolation properties, nowadays gypsum is one of the most important construction materials used in house building applications and often it is preferable to other building materials (wood, plywood etc.) [1,2].

Nowadays gypsum have several applications spacing in different engineering fields. For example, in manufacturing composite field, it can be adopted as pattern material to produce high quality composite pipes via autoclave [1]. Indeed gypsum has high temperature and compression resistance enough to resist at the autoclave cycle, and due to its good workability and high quality surface it allows the production of an internal pipe surface characterized by high quality finishing.

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On the other hand, gypsum can also be adopted as building material for the production of gypsum walls that in some case can be reinforced by vegetable or synthetic fibers as proved in [2].

The above-mentioned applications, can appear completely different and unlinked, but they are characterized by an important common keypoint: the possibility of using more and more times the gypsum material; this means to recycle the material. Especially in last decade, the recycling topic is more and more a relevant issue to solve; indeed the reduction waste materials as well as the reuse of at the end of life of products are ones of the key points of the circular economy.

In this context, gypsum appears to be a valid material. Indeed, the dihydrate and hemihydrates phases of calcium sulfate can be converted into each other under certain conditions, which provides a basic theory for the recycling of waste gypsum [3].

Additionally to conventional gypsum, foamed gypsum is also widely used: for example some authors [4] proved that foamed gypsum showed higher thermal insulation properties, attractive acoustic properties and lower transportation costs than conventional gypsum. Other authors [1] demonstrated the interest in the use of foamed gypsum to produce easily removable pattern used in the autoclave process of fiber glass pipes.

In this context it seems interesting to study in the present work the recycling process of gypsum and evaluate the mechanical properties in terms of flexural and compression strength at the end of each recycle both for conventional and foamed gypsum.

Therefore, the feasibility of the process was investigated and the mechanical properties in terms of flexural and compression strength, before and after the recycles for both the conventional and the foamed gypsum, were studied.

2. Materials and methods

The gypsum used in the present work was provided by Gyproc Saint-Gobain (Milano, Italy) and it is mainly constituted by calcium sulphate hemihydrate. Its chemical and mineralogical composition was assessed by thermal analysis (DTA/TGA) and X-ray diffraction analysis (XRD), and it was elsewhere reported [5].

As foaming agent for the production of foamed gypsum, ISOCEM S/L vegetable protein in water solution (supplied by Isoltech s.r.l. Italia) was used.

For the un-foamed samples (hereafter referred to as TQ), the dry mixture was firstly homogenized and then added to the distilled water (water to gypsum weight ratio = 0.7) and mixed for about 30 s. The mixture was casted into prismatic open molds, 160 × 40 × 40 mm^3, (see Fig. 1a) for producing specimens for the mechanical characterization.

Then the water-gypsum mixture was compacted with a conventional vibration table for about 30 s. The compacted mixture exhibited a setting time of about 20 min and after 2 h from it, the specimens were demolded, then dried for 48 h at T = 40 °C and finally stored in a climatic chamber (MSL Humichamber, mod. EC 125) at 20 °C and RH = 50%, until the time of testing. A similar procedure was adopted for the foamed samples (hereafter referred to FO), where a further step (foaming) was carried out by adding the vegetable protein.

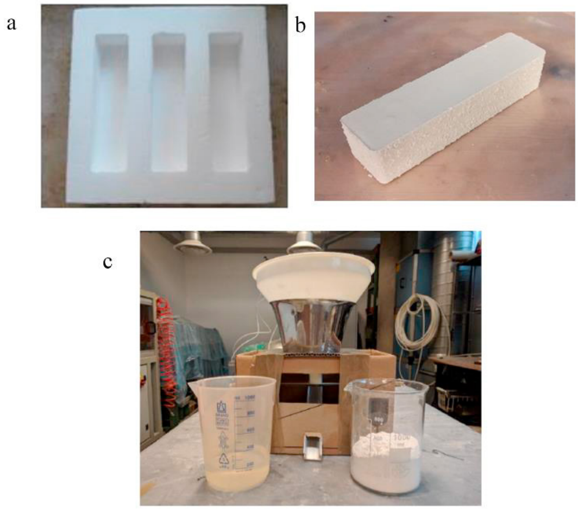


Fig. 1. (a) Mold for prismatic specimens; (b) example of a specimen and (c) homemade V-funnel apparatus

Generally, a protein foaming process, usually employed in porous ceramic materials production, consists in a strongly whipping of the vegetable protein that, added to the inorganic mixture, can act as “meringue” type foam. Since the rate of whipping influences strongly the expansion grade, it has to be suitably selected, in order to obtain a stable foam network, able to maintain itself during the gelling and consolidation processes [6]. In order to allow the entrapping of the bubbles in the gypsum matrix, the foaming step has to take place just before the gelification of the system. Therefore, during the mixing of water and gypsum, the vegetable protein was added (gypsum to protein weight ratio = 0.02).

To compare the “flowability’’ of un-foamed (TQ) and foamed (FO) gypsum mixtures, homemade V-funnel tests generally used for concretes were carried out (see Fig. 1b). These tests consist of filling a funnel-shaped container with a fixed mass of gypsum-water mixture (680 g), then to open the tap door and allow to the mixture to flow under gravity into a cylindrical container placed below the funnel. So, the time required for the discharge of the funnel (t_d) and the mass of mixture (m) present into the cylindrical container were measured.

The mass of material was measured in order to evaluate the amount of lost material, i.e. the material that was remained bonded to the surface of the funnel and that is correlated with the viscosity of the water-gypsum mixture.

For the mechanical characterization of both TQ and FO samples, UNI EN 1015-11 standard was followed: prismatic specimens (40 x 40 x 160 mm^3), as shown in Fig. 1c, were made to perform flexural and out-of-plate compression tests using a universal testing machine (MTS Alliance RT/50).

After tests, the pieces of gypsum were recovery (without mixing FO and TQ types) and subjected to grinding by using a commercial blender.

Different conditions of grinding velocity (10000, 20000 and 30000 rpm) and time (3, 5, 10 and 15 min) were considered.

In order to obtain a gypsum mixture with the adequate value of granulometry, for each condition the granulometry of the gypsum powder was evaluated by using 1000-500-250-125 and 50 μm sieves.

Finally, in order to convert the dihydrate phase into the hemihydrates phases of calcium sulfate (i.e. allow the recycling of gypsum) an heat treatment is required, then by using the climatic chamber (MSL Humichamber, mod. EC 125) both the temperature and time of the heat treatment was considered as variable as reported in Table 1.

Therefore, thermogravimetric (TG, Netzsch, model 409ST Luxx) analyses were carried out in order to choose the correct heat treatment conditions. After the heat treatment, both mechanical and V-funnel tests were carried out in order to study how the recycling procedure affects these properties. In the present work, 4 recycles were carried out.

Table 1. Heat treatment conditions.

Condition name	Time (h)	Temperature (°C)
A1	1	
A2	2	140
A3	3	
B1	1	
B2	2	150
B3	3	
C1	1	
C2	2	160
C3	3	
D1	1	
D2	2	200
D3	3	

3. Results and discussion

In the V-funnel test, lower is the discharge time (t_d) and higher is the mass (m), higher is the flowability of the mixture. For this reason the t_d/m ratio was taken into account. By comparing this value for the un-foamed (TQ) and foamed (FO) mixture appeared that the flowability of the FO gypsum mixture was higher, i.e. a lower td/m ratio value.

Specifically, it passed from 1.1 x 10^{-2} to 8.0 x 10^{-3} s/g for the TQ and FO mixture respectively. This means that the use of the vegetable protein make the mixture more “workable”, increasing the capability of production of complex shape. This could be useful for example for the production of big and complex shape products.

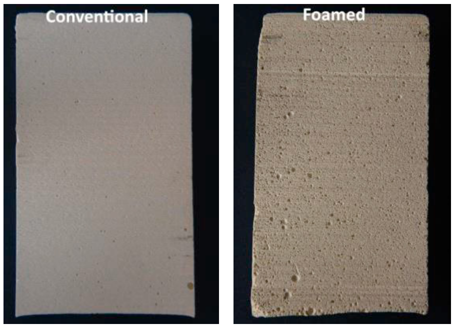
Another advantage in the use of the vegetable protein is its effect on the density. The density of a TQ samples was equal to 1.1 g/cm^3 and a reduction of the 46% was obtained for the foamed (FO) samples.

By way of example, in Fig.2 the cross section of a sample type A using both un-foamed (TQ) and foamed (FO) mixture is shown, the foaming effect of the vegetable protein is clearly evident.

The porosity area percentage was evaluated by using the MATLAB® software; it was equal to 0.79% and 12.4% for the TQ and FO sample respectively.

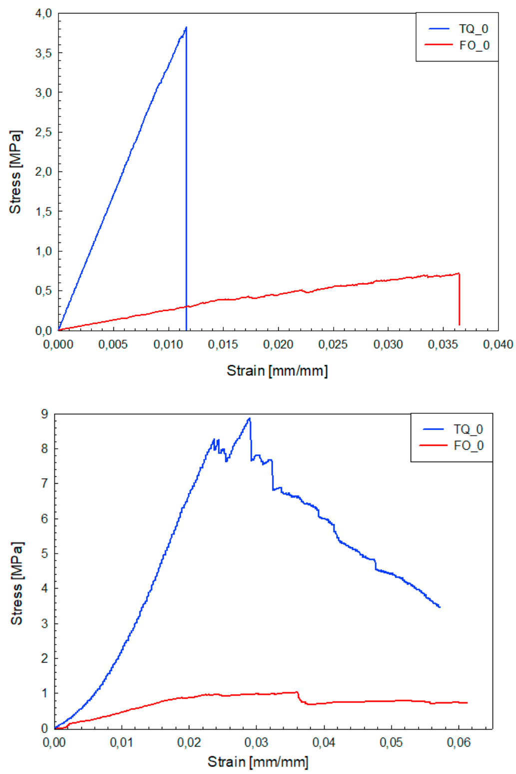
In Fig. 3 the results in terms of stress-strain curves obtained from three point bending and compression tests are reported.

It can be observed that the curves of the FO specimens are characterized by a less regular trend than the ones of the TQ specimens (Fig. 3a) due to the major presence of the porosity.



a b

Fig. 2. Example of (a) TQ and (b) FO cross section sample.



a

b

Fig. 3. Comparison between typical stress-strain curves of TQ and FO carried out from (a) flexural and (b) compression tests.

All curves show a trend typical of the brittle materials, without the presence of the plastic behavior.

If on one hand the addition of the foaming agent increases the strain at breaking, on the others a reduction of about 90% for the flexural modulus and a reduction of about the 80% for the flexural strength was observed.

Similar results were obtained from the compression tests. In Fig. 3b the results in terms of stress-strain curves obtained from out-of-plane compression tests carried out on specimens 40 x 40 x 40 mm^3 are reported.

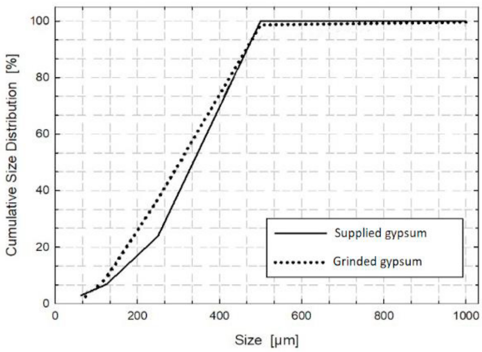
It is possible to note that the curves of the TQ specimens show an initial concavity followed by a linear trait; the compression modulus was evaluated in this linear trait.

Differently, the curves of the FO specimens were characterized by the fact that the maximum stress value was always postponed by a nonlinear trait.

After tests, broken pieces of gypsum were grinded according to the procedure explained in the previous section.

The best result was reached by using a velocity of 30000 rpm and 10000 rpm for a time of 3 and 10 minutes for TQ and FO respectively, therefore these grinding conditions were adopted for the recycling process.

The cumulative granulometry curves of both supplied and grinded gypsum for TQ and FO type are plotted in Fig.4.



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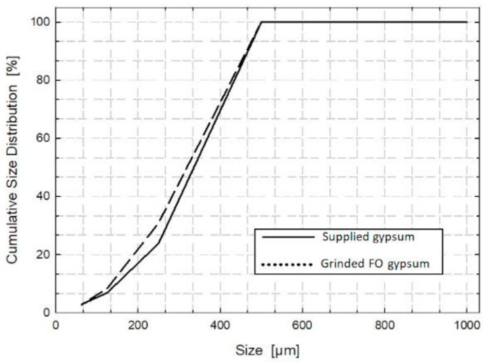
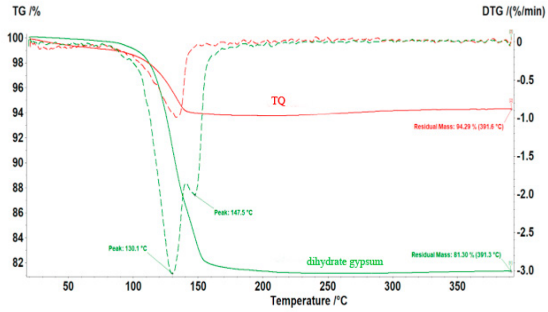


b

Fig. 4. Cumulative size distribution of supplied and grinded gypsum for (a) TQ and (b) FO sample type.

In Fig. 5 the results of the thermogravimetric analyses were reported. In detail, in Fig. 5a a comparison between supplied gypsum and grinded gypsum before the thermal treatment, i.e. hydrate gypsum, is reported.

The supplied gypsum (red curve) has a moderate weight loss (less than 6%) and a single peak of the dTG, related to decomposition (water loss) of the "hemi-hydrate gypsum" phase in "anhydrous gypsum". Hemihydrates phase, is the most reactive phase with water, and it is therefore the most abundant phase typically found in commercial plaster.



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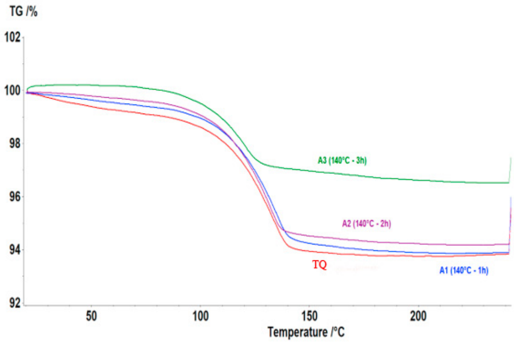

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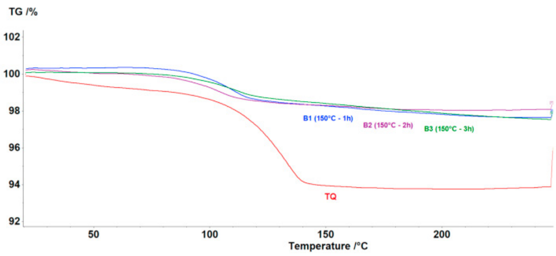

c

Fig. 5. Thermogravimetric curves of (a) supplied and grinded gypsum; and of heat treated gypsum for condition (b) A1,A2 and A3 and (c) B1, B2 and B3

Differently, the sample of hydrated gypsum (green curve) showed more loss of weight (around 20%) and two peaks of dTG, the first concerning the decomposition of calcium sulphate dihydrate into hemihydrates (T≈130 ° C) and the second related to the decomposition of the hemihydrate in anhydrous phase (T≈150 °C).

In Figs. 5b and 5c, the results of the thermogravimetry analyses for each heat treatment conditions are reported.

It appeared that the best conditions were achieved for A2 and A1.

Therefore, for economic reason considering the lower time required in A1 respect to A2, the first condition, i.e. A1, was selected for the heat treatment.

By using the selected grinding and heat treatment condition, V-funnel tests were conducted at the end of each recycle.

In Fig. 6 the results in terms of mass loss percentage (Fig. 6a) and discharge time (Fig. 6b) for both TQ and FO sample type are reported.

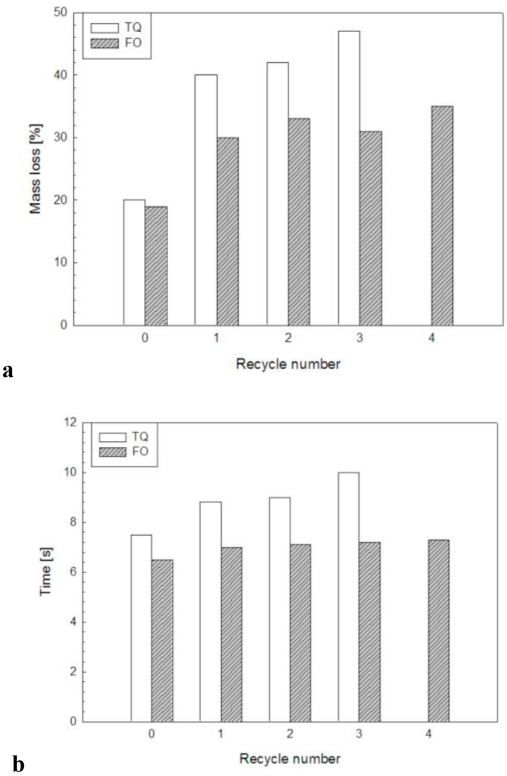

Fig. 6. V-funnel test results in terms (a) mass loss percentage and (b) discharge time for both FO and TQ sample type.

From the results it appeared that the recycling process affected the feasibility process when TQ gypsum was adopted and no relevant variations was observed for FO gypsum.

In detail, it was possible to perform the V-funnel test up to 4 times for the FO gypsum and only 3 times for TQ gypsum.

This is due to the fluent action of the vegetable protein. In any cases, the lower workability, highlighted in the V-funnel tests, did not limit the possibility to produce TQ sample for compression and flexural tests also for 4 recycles.

Finally, mechanical tests after each recycle were carried and stress-strain curves at bending (Fig. 7) and compression (Fig. 8) are plotted in Fig. 7 and Fig. 8 respectively.

From the tests appeared that the flexural and compression strength was not affected so much from the recycling process.

The only exception was represented by the TQ_4 sample, i.e. TQ gypsum recycled 4 times.

The results are in agreement with those obtained in the V-funnel test.

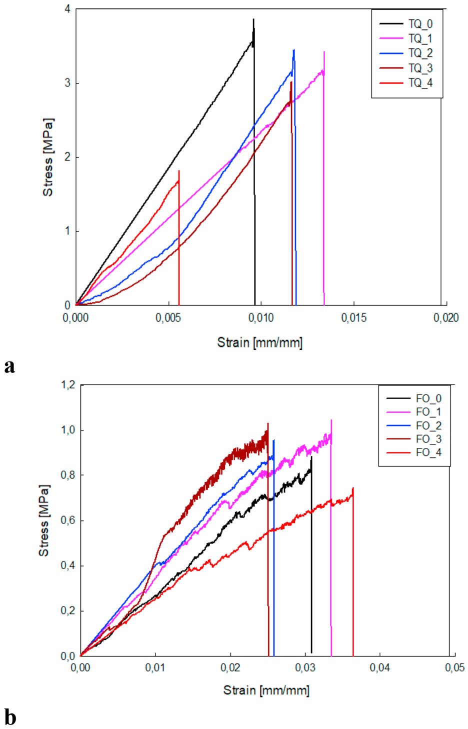

Fig. 7. Stress-strain curves at bending for each recycle for (a) TQ and (b) FO samples.

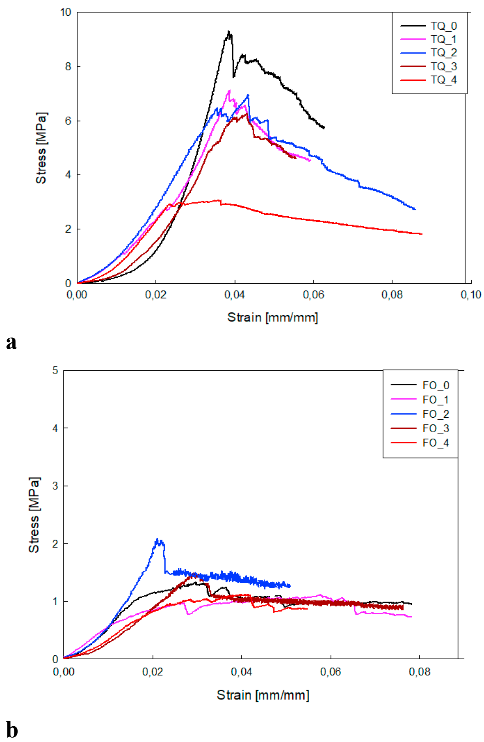

Fig. 8. Stress-strain curves at compression for each recycle for (a) TQ and (b) FO samples.

4. Conclusions

In this work the recycling process of conventional and foamed gypsum was studied. As foaming agent, a vegetable protein (ISOCEM S/L) was utilized (gypsum to protein weight ratio = 0.02); the foamed gypsum showed a lower density then the un-foamed one (reduction of around 46%) and a better workability as highlighted in the V-funnel test. It was proved that if the recycling parameters are properly set in terms of grinding and heat treatment condition, both conventional and foamed gypsum can be successfully recycled allowing 4 reuses. The only problem was showed for the fourth recycle of TQ sample, indeed the gypsum so recycled did not allow the successful execution of the V-funnel tests and both the compression and the flexural strength showed a reduction more than half time the properties of the starting gypsum. In the other cases, the mechanical properties were not affected in a relevant manner.

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