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FILLER CALCIUM CARBONATE INDUSTRIAL APPLICATIONS: THE WAY FOR ENHANCING AND REUSING MARBLE SLURRY

Graziella MARRAS(*), Nicola CAREDDU(*) & Giampaolo SIOTTO(**)

(*) University of Cagliari - DICAAr - Via Marengo, 2 - 09123 Cagliari, Italy
(**) Mediterranea Progetti e Finanza s.r.l. - Via Tola, 30 - 09128 Cagliari, Italy
Corresponding author: graziellamarras@gmail.com

EXTENDED ABSTRACT

Uno dei problemi di maggior rilevanza nel settore delle rocce ornamentali, sia relativamente alle attività di estrazione sia a quelle connesse agli impianti di trattamento, è legato alla mole di sfridi generati, i quali vengono attualmente conferiti nelle discariche. Una soluzione alternativa, fortemente consigliata dall'Unione Europea (direttiva 2006/21/CE del Parlamento Europeo e del Consiglio del 15 marzo 2006 relativa alla gestione dei rifiuti delle industrie estrattive) e dal D.Lgs. n.152 del 03/04/2006 - "Norme in materia Ambientale" e ss. mm. e ii., è quella di concentrarsi su processi di produzione a zero scarti, attuabili attraverso l'integrale riutilizzo delle materie prime secondarie tramite procedure di recupero e riciclaggio.

Il carbonato di calcio micronizzato, proveniente dalle torbide prodotte negli impianti di segagione e trattamento del marmo, non ha ancora trovato rilevanti applicazioni in campo industriale, tali da risultare economicamente vantaggiose per un suo riutilizzo. Il suddetto argomento trova ancor più barriere in realtà industriali "chiuse", come quelle del comparto estrattivo, che spesse volte oppongono resistenza nell'apportare delle modifiche nel loro modus operandi, ormai consolidato da tempo. Fino ad oggi i fanghi disidratati, sono stati erroneamente assimilati a rifiuti da smaltire in discarica, procedura che peraltro rappresenta un onere per le aziende, dati i costi di conferimento. Lo scopo della ricerca è quello di considerarli non più come rifiuti ma come materie prime e seconde da impiegare in ulteriori processi produttivi, perseguendo, in tal modo, il concetto di sviluppo sostenibile. Come è intuibile dalla normativa, il primo passo per valutare la possibilità del riuso di tali sfridi è quello di condurre analisi di caratterizzazione del materiale allo scopo di identificare sia le differenti fasi mineralogiche, sia le caratteristiche chimico-fisiche, sia gli eventuali elementi inquinanti presenti.

Nella fase sperimentale sono stati utilizzati i fanghi provenienti dagli stabilimenti di lavorazione delle rocce calcaree situati nel polo estrattivo di Orosei (NE Sardegna), campionati a valle delle sezioni della filtropressa.

La fase di caratterizzazione chimica fisica e mineralogica dei finissimi ha evidenziato che tale materiale è costituito quasi totalmente da carbonato di calcio micronizzato, e non sono state rilevate sostanze inquinanti tali da comprometterne l'uso in ulteriori campi industriali; pertanto non sono presenti condizioni ostative al suo riutilizzo. Da questo punto di partenza si è affrontata la fase di sperimentazione vera e propria che ha visto concentrare la ricerca nel campo dell'edilizia. Tale scelta è stata fatta partendo dalla considerazione sulle quantità di materiale che sarebbero state potute essere smaltite e riutilizzate in processi produttivi correlati all'ambito del comparto edilizio: poiché il settore dell'edilizia è quello che ha maggiori consumi di materiali si ci è concentrati sulla possibilità di utilizzare tali prodotti in sostituzione od integrazione di altri nella realizzazione di prodotti edili. Nello specifico nel corso di questi anni si è studiata la possibilità di utilizzare il carbonato di calcio per la fabbricazione di mattoni, per la produzione di calcestruzzo e per la produzione di intonaci a proiezione meccanica. In tutti e tre i campi si sono studiati degli impasti con diverse percentuali di sostituzione di specifici componenti con i fanghi di segagione, e si sono ottenuti dei campioni che sono stati sottoposti alle stesse prove meccaniche e analisi fisiche subite dai materiali standard, così da valutare, mediante comparazione, l'incidenza dell'aggiunta del fango di segagione su tali proprietà. I risultati sono stati incoraggianti e sono presentati in dettaglio nella presente trattazione.

Nonostante ciò la ricerca si sta spostando su settori industriali nei quali il carbonato di calcio viene acquistato, trattato come elemento di nicchia e come tale pagato, anche in funzione del grado di micronizzazione richiesto. Questo cambiamento di rotta è stato dettato dal fatto che i materiali che si utilizzano nel settore edilizio sono cosiddetti poveri, anche perché usati in grandi quantità, per cui il ritorno economico del riutilizzo dei fanghi di lavorazione si avrebbe inserendoli in cicli produttivi a valle degli impianti di trattamento, per i quali il raggio di trasporto risulterebbe molto limitato. Lo studio bibliografico ha evidenziato che il carbonato di calcio micronizzato entra in gioco come filler in svariati settori quali l'industria dello pneumatico, l'industria cartaria, la produzione di vernici quale fondente per altoforni, etc.

Ad oggi la ricerca è in itinere e si sta indirizzando sul riutilizzo dei fanghi di segagione nelle mescole per gli pneumatici e negli impasti per la produzione cartaria, settori nei quali si impiegano delle quantità numericamente ridotte, rispetto ai campi civili, ma che consentono di dare un valore economico molto più cospicuo al nostro sottoprodotto. Se si riuscisse a dimostrare la fattibilità dell'utilizzazione dei fanghi di segagione alla stessa stregua del carbonato di calcio prodotto per macinazione e messo in commercio sarebbe un grande passo avanti nella ricerca.

ABSTRACT

Since ancient times marble has been the most praised material due to its multi-use possibilities. The investigation conducted by the authors started several years ago with the aim to outline the study of industrial applications for marble dust deriving from marble processing plants as calcium carbonate. The matter of fact is that this stone slurry is composed primarily of calcium carbonate and has a great potential as secondary raw material or by-product, although it has up to now been considered as waste and treated as such by their producers. Seeing as how calcium carbonate product specifications vary depending on the application, the marble sludge was submitted to a characterization in order to evaluate the compatibility with the specifications required by particular sectors. Firstly, mineralogical, physical and chemical determinations were carried out. Secondly, tests and analyses have been accomplished for the use of micronized calcium carbonate in building industry (brick, plaster and concrete making). More recently, the possibility of using micronized stone powder to produce different commodities for tyre industries has been studied. The data emerging from the analyses ensure that the sludge production can have a potentially high economic value and allow to go along with the concept of sustainable development.

Key words: *micronized calcium carbonate, marble slurry, marble dust, reuse, brick, concrete, plaster*

INTRODUCTION

Calcium carbonate rocks are widespread over practically the whole world. Limestones were the first rocks to be quarried in human history. Due to their wide-spread occurrence they were easy to find, and due to their softness and layered structure they were easy to work. $CaCO_3$ deposits requirements in respect of calcium carbonate as filler are high so the raw material in the quarries meets certain criteria. The first criterion is purity. The calcium carbonate content of a rock must be at least 97 percent. Marbles differ according to the degree of metamorphism and the original composition of the limestone; the multifarious nature of these rocks is considerable (Tegethoff, 2001).

Italy is certainly the leading country detaining the best qualities and varieties of marble and Orosei Marble (NE Sardinia - Italy) is praised and widely used. On the other hand, one of the main environmental impacts of the ornamental stone extraction process is represented by the waste material that can reach a great quantity, even similar to the worked one (Osnet, 2004). There is a diversification in waste generation as the following one: waste deriving from quarries and offcut/waste deriving from processing plants (Careddu *et alii*, 2014). More precisely, there are two types of natural stone processing waste: solid (i.e.: unshaphed blocks, rubble, crushed slabs/strips/tiles) and semi-liquid or slurry (Almeida *et alii*, 2007) which are mainly composed by stone microfine sawdust and water.

All over the world, the ornamental stone industries create vast amount of by-product rock waste that could be used in building production and in particular the rock powder might be suitable to be used in civil engineering construction purposes (Careddu & Marras, 2015). Nowadays, landfill is the actual main destination for rock residues. By considering the point of view of waste – re-use it is important to underline that recycling has a large number of benefits which have made it a more attractive option in this period of greater environmental awareness, more environmental laws.

What this indicates is the important that research have in the waste reuse, from the point of view of environmental aspects, and the sustainable development, since the use of waste products from industrial processes reduces the environmental impact of the new material or product, and from a financial standpoint, in connection with the potential cost savings on treatment or disposal of those waste products. Several companies become to understand the importance of research and development. It's clear that this concept is linked with the innovation and consequently, with the concept of sustainable development and at the light of the entrepreneurs try to recognize and exploit the different opportunities that arise from this concept of research and technology.

At this time there is the necessity of appreciate the potential value of marble and convert it into marketable products, thus doing away with the costs necessarily incurred by the extractive industry for its disposal and to go along with the concept of sustainable development. It's important noticed that calcium carbonate has a great importance not only in building sector but also in manufacturing of paper, plastics and paints, in pharmaceutical and chemical industries, in agriculture and it's also used in rubber and tires production.

INDUSTRIAL APPLICATIONS

The best way to handle the waste is its gainful utilization in industrial sector. For a while, the sector devoted to the fabrication of building products, has been the target for the incorporation of other industries rejects, also for ceramic industry. Today, it is clear that some wastes are similar in composition to the natural raw materials used, and often contain materials that are not only compatible but also beneficial in the fabrication of particular products. Thus, upgrading industrial wastes to alternative raw materials becomes interesting, both technically and economically (Bertolini & Celsi, 1990; Torres *et alii*, 2004; Segadães *et alii*, 2005; Saboya *et alii*, 2006; Acchar *et alii*, 2006; Montero *et alii*, 2008) and is becoming common practice.

Currently, attempts are being made to utilize marble wastes in different applications like road construction, concrete and asphalt aggregates, cement, and other building materials, in particular the filter press mud (Dak, 2000) and so on.

Therefore, some of the most interesting applications studied during our research are stated below.

Brick making

Bricks have been widely used as construction and building material all around the world for a long time. Conventional clay based brick production generally uses the mixtures of clays and shale as raw materials, and requires the processes of shaping, drying and firing at a high temperature (Satcu *et alii*, 2015). Fired clay bricks are mainly construction elements used to make walls of buildings. The incorporation of various wastes in red clay-based ceramic products has been widely investigated, frequently with the aim of reducing the waste environmental impact (Acchar *et alii*, 2006, Saboya *et alii*, 2007; Marras *et alii*, 2010; Bilgin *et alii*, 2012, Eliche-Quesada *et alii*, 2012).

Concrete

Concrete is used more than any other man-made material in the world. Generally concrete is used to make pavements, pipe, architectural structures, foundations, motorways/roads, bridges/overpasses, parking structures, brick/block walls and footings for gates, fences and poles. It is for these reasons that a further investigation of the reuse of marble dust in concrete production appeared to be warranted.

Recently, the use of recycled materials as concrete ingredients has gained popularity because of increasingly stringent environmental legislation. The most conspicuous of these is fly ash, a by-product of coal-fired power plants (Scott & Thomas, 2007; Halstead, 1986). This significantly reduces the amount of quarrying and landfill space required, and, as it acts as a cement replacement, reduces the amount of cement required. Different studies were carried out in order to evaluate the feasibility of substituting aggregates with ornamental stone waste (Almeida *et alii*, 2007; Almeida *et alii*, 2007; Binici *et alii*, 2007; Chitlange *et alii*, 2008; Coriandolesi *et alii*, 2010; Güneyisi *et alii*, 2009; Misra & Gupta, 2008) and also of replacing cement with marble powder (Topçu *et alii*, 2008; Alyamaç & Ince, 2009) but for mortars not for concrete. Limestone and dolomite fines are the most frequently used to increase the content of fine particles in self compacting concretes among non-pozzolanic fillers (Binici *et alii*, 2007; Billberg, 1999)

Gypsum plaster

The general term of gypsum (derived from the greek γύπσος) indicates either two substances are known and used for millennia, since the time of the Egyptians: both a mineral and industrial products derived from it (Turco, 1996). Gypsum is a particularly useful processed material. Its main application is as a building material, its main derived product is stucco or plaster for plastering walls and making decorative features in buildings. In the past few decades, gypsum-based renders and plasters have become the material of choice for indoor finishing in many countries (Arikan & Sobolev, 2002). Excellent performance, attractive appearance, easy application, and its healthful contribution to living conditions have made gypsum a most popular finishing material for centuries (Arpe, 1984; Duggal, 1998; Ragsdale & Raynham, 1972). Recently the possibility to use calcium carbonate as filler in mortars and plasters was studied (Molnar & Manea, 2016).

MATERIALS AND METHODS

The subject of the present dissertation is the marble slurry recovered by the filter press exit, in the marble treatments plan from Orosei marble factories, as a by- product resulted from marble cutting and shaping process. Hereinafter we refer to such materials by naming it marble dust (MD).

Marble dust characterization

The MD, was tested by performing mineralogical, chemical and physical analysis to characterize the material and also to identify the absence of pollutants.

MD was obtained in wet form as slurry therefore it was dried in an oven in the laboratory.

Grain size analysis was conducted on the dry solid cake produced by the filter-press using a Sedigraph 5100 Analyser. Bulk density of a representative sample of the filter pressed material in the processing plant was determined using an Accu Pyc 1330 gas pycnometer manufactured by Micromeritics Instruments.

X-Ray diffraction (XRD) analysis was performed on the dewatered slurry to determine mineralogical composition and mineral phases were determined by comparison with the JCPDS index (JCPDS, 1985).

A representative sample of MD was analyzed by Actlabs – Activation Laboratories LTD (Canada) with the technique of lithium metaborate fusion, and loss on ignition (LOI) was calculated too. The data obtained was listed in Tab. 1 (Marras *et alii*, 2010).

Experimental phase

The experimental program was divided into three sections: brick making, concrete and gypsum plaster. It was used the calcium carbonate micronized characterized in the first part of the experimentation (MD).

COMPONENTS	[%]
LOI	45.13
CaO	53.15
MgO	0.45
SiO_2	< 0.01
Al_2O_3	0.07
Na_2O	0.02
K_2O	< 0.01
Fe_2O_3	0.03
MnO	0.005
TiO_2	0.003
P_2O_5	0.03

Tab. 1 - Major components and LOI for MD

Brick making

In this section, the production of fired bricks from mixtures of brick clay and waste marble powder additive was accomplished. This part of research intends to discuss some technical aspects concerning the use of marble sludge in the ceramic raw material.

The brick-making process generally consists of the following steps: gathering, crushing, grinding, screening, and mixing the raw materials; making the brick; and setting, drying, firing, packaging and inventorying the final product.

For preparing test bricks were used two different types of clays (coded YCM - BCU) and MD. Characterization data are reported in Tab. 2.

Mix-designs preparation were obtained adding MD in brick composition, by trial and subsequent adjustments, containing the maximum reasonable proportion; the influence of its addition on brick properties, such as shrinkage and water absorption was investigated and subsequently was made a comparison with a commercial reference brick.

Taking into account previous studies concerning the incorporation of residues in red clay ceramic formulations (Menezes *et alii*, 2005; Gupta, 2005) several batches, based on the two clays, incorporating up to 10 wt.% of MD were prepared (Tab. 3).

The clays were previously sieved to 200 mesh sieve. The BCU gives the brick structure so the percentage of marble powder added was simultaneously removed from YCM. In the following table are listed the mix-designs:

COMPONENTS	BCU [%]	YCM [%]
SiO_2	63.66	56.00
Al_2O_3	15.95	11.40
TiO_2	0.92	0.65
Fe_2O_3	8.30	5.63
CaO	0.41	8.63
MgO	1.64	2.13
K_2O	4.00	3.53
Na_2O	1.00	0.74
S	nd	0.002
Fl	nd	0.071
Cl	0.48	0.023
Attenberg limits		
L_p	19.8	21.2
L_l	39.1	32.4
I_p	19.3	11.1
Drying sensitivity	7 cm	0 cm

Tab. 2 - Characterization data for clays

MIX-DESIGN	YCM [%]	BCU [%]	MD [%]	WATER [%]
A	50	40	10	17.0
B	53	40	7	16.7
C	55	40	5	16.8
D	57	40	3	16.3
REFERENCE BRICK	60	40	0	16.0

Tab. 3 - Mix-designs

Mixes were prepared, thanks to a paste mixer, by inserting the different percentages of the three components with the addition of water vary depending on the workability (16-17 wt.%). The paste had a starting weight of 1 kg to achieve a final sample of approximately 400 g. The mix design which allowed to obtain better workability with the highest percentage of substitution of clay with MD was mix-design-C, and corresponds to a rate of 5 wt.%.

The paste bricks were cast inside rectangular moulds with dimension of 12 x 8.5 x 2 cm.

The homogenized mixtures were submitted at the production line. Firstly samples were introduced for 8 hours in the dryer (Temperature: 130°C); secondly, the process of firing will take place in several steps. Despites, the bricks were passed in the firing section where are burned in a tunnel type furnace for 36 hours, with a curve that consistently leads the sample from room temperature at the maximum temperature 850°C.

Testing. Test on the obtained bricks was carried out to verify the shrinkage and the properties of the specific absorption rate (as, imbibition, according to EN 771-1:2004) and the water absorption capacity (wa).

At the end of the drying - process, they were analyzed for the shrinkage (Fig. 1) and weight variation.

Concrete

Section two discusses the effect of using MD in concrete production. Technical possibilities of producing concretes containing MD have been studied.

The mixed materials were derived from the combination of four components and water: cement (CEM), coarse aggregates (CA), fine aggregates (FA) and MD.

CEM: Portland cement type I 52.5 R cement (complying with the EN 197-1). CEM physical chemical and mechanical properties are tabulated in Tab. 4.

FA [supplied by CEMEX (Spain)]: nominal maximum size 5 mm, with 0.7% passing the 63 μm sieve. Its relative density

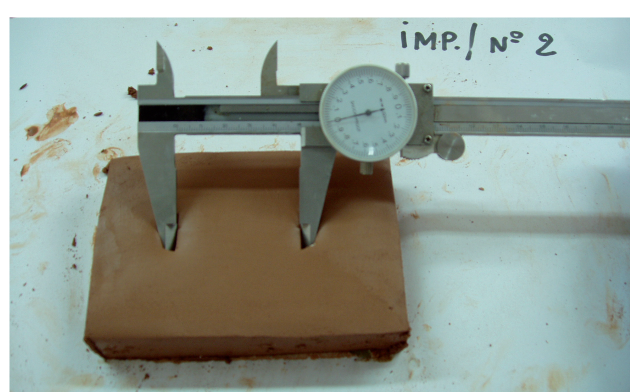

Fig. 1 - Sample engraving

Property	Value
Specific density	3.13 g/cm^3
Specific surface area	5,040 cm^2/g
Initial Setting time	115 min
Final Setting time	165 min
Strength 2 days	39 MPa
Strength 7 days	51 MPa
Strength 28 days	61 MPa
Loss on ignition (LOI)	2.45%
Insoluble Residues (IR)	0.68%
Sulfates	3.18%
Chlorides	0.016%
Expansion	0.5 mm

Tab. 4 - Physical chemical and mechanical properties of CEM I 52.5 R

	FA	CA
Fineness modulus [%]	3.0	1.2
Specific gravity [g/cm^3]	2.59 ± 0.04	2.61 ± 0.02
Water absorption [%]	< 0.5	0.5 ± 0.1

Tab. 5 - Physical properties of the components

(at saturated surface dry condition SSD) was 2.59, and its SSD condition water absorption of <0.5%.

CA [supplied by CEMEX (Spain)]: 20 mm maximum size, with < 0.5% passing the 63 µm sieve. Its relative density was 2.61, and its water absorption of 0.5%.

The sieve analysis of the FA and CA was conducted according the EN 933-1: 2009 and the EN 933-2: 1997, and physical properties are reported in Tab. 5.

This project was concentrated on a general purpose mix, so concrete mixes were designed by assuming a good degree of quality control and moderate exposure conditions. The mixes are designed for CMI_0 CMI_5 CMI_10 CMI_15 CMII_0 CMII_5 CMII_10 CMII_15. For the first calculated mix design we studied several scientific researches and applied the same procedure and the same proportions (Güneyisi *et alii*, 2009). For the second one was used the ACI method (Fernández, 2007), was chosen a strength value of 30 MPa and the associated water-cement ratio of 0.55, and for a finesses modulus of 2.7 was calculated the CA volume to be used in mix-design, and the value obtained was 0.63 for every 10^{-3} concrete volume.

The source data are listed in Tab. 6, the figures obtained are reported in Tab. 7 and the dosage per cubic meter is shown in Tab. 8.

Recapitulating, two different mix-designs were calculated to cover the range of different mixture variations, and a total of 4 concrete mixtures for each mix-design, were proportioned having a constant water-binder (CEM and MD) ratio (Güneyisi *et alii*, 2009).

Test specimens were prepared in the LOEMCO laboratory by direct mixing CEM, CA, FA adding MD in different proportion (by weight) and water. The exact amount of concrete ingredients were weighed and mixed together. The workability of fresh concrete was measured in terms of slump according to EN 12350-2: 2009.

CEM was replaced with MD at various percentages by weight (0%; 5%; 10%; 15%) to prepare the concrete mixtures, fixing wa-

Material	Bulk density [kg/dm^3]	Specific weight [kg]
Cement	3.1	1.12
Water	1	1
Coarse aggregates	2.65	1.57
Fine aggregates	2.65	1.57

Tab. 6 - Physical properties of the components

Maximum size for coarse aggregates [mm]	**20**
Strength at 28 days [MPa]	**30**
Water / cement ratio	**0.55**
Slump [cm]	**3 a 5**
Water absorption [l/m^3]	**185**
Finesses modulus [%]	**2.7**
Specimen volume [cm^3]	**5,301.45**

Tab. 7 - Mix-design II data

Material	Volume [dm^3]	Mass [kg/m^3]	[%]
CEM	**108.50**	**336.36**	**13.70**
Water	**185.00**	**185.00**	**7.54**
CA	**373.25**	**989.10**	**40.30**
FA	**356.25**	**944.06**	**38.46**

Tab. 8 - Mixture proportions of the two mix-designs [kg]

ter to CEM+MD ratio at value of 0.50 (by weight) for the first mix design and 0.55 for the second one. The control concretes for both the mix-designs were made without incorporating MD. All the figures are tabulated in Tab. 9 and graphically represented in Fig. 2 and Fig. 3.

All the components were put in a concrete mixer for 15 minutes. After that, fresh concrete was cast into cylindrical stainless steel moulds (Φ = 150 mm, H = 300 mm) without any vibration. Three cylindrical specimens were prepared from each of the eight concrete mixtures, for a total number of 24. After demolding, they were brought in a moist chamber for curing specimens for strength tests, conforming with the EN 12390-2 (Marras, 2010). Before doing the strength tests the upper ends of the cylinders were struck off with a sulfur mixture method (EN 12390-3).

Testing. Twenty four concrete samples were studied over a 28-day period to determine the effect of the MD addition. Compressive strengths were obtained as per EN 12390-3 and they were evaluated with respect to percentages of waste sludge replaced

MIX-DESIGN I						
	W/(CEM+MD)	CEM	FA	CA	MD	WATER
CMI_0	0.50	8.3	12.5	25	0	4.2
CMI_5	0.50	7.9	12.5	25	0.4	4.2
CMI_10	0.50	7.5	12.5	25	0.8	4.2
CMI_15	0.50	6.7	12.5	25	1.6	4.2
MIX-DESIGN II						
	W/(CEM+MD)	CEM	FA	CA	MD	WATER
CMII_0	0.55	8.6	24.7	24.4	0	4.7
CMII_5	0.55	8.1	24.7	24.4	0.5	4.7
CMII_10	0.55	7.7	24.7	24.4	0.9	4.7
CMII_15	0.55	7.3	24.7	24.4	1.3	4.7

Tab. 9 - Mixture per cubic meter calculated by ACI method

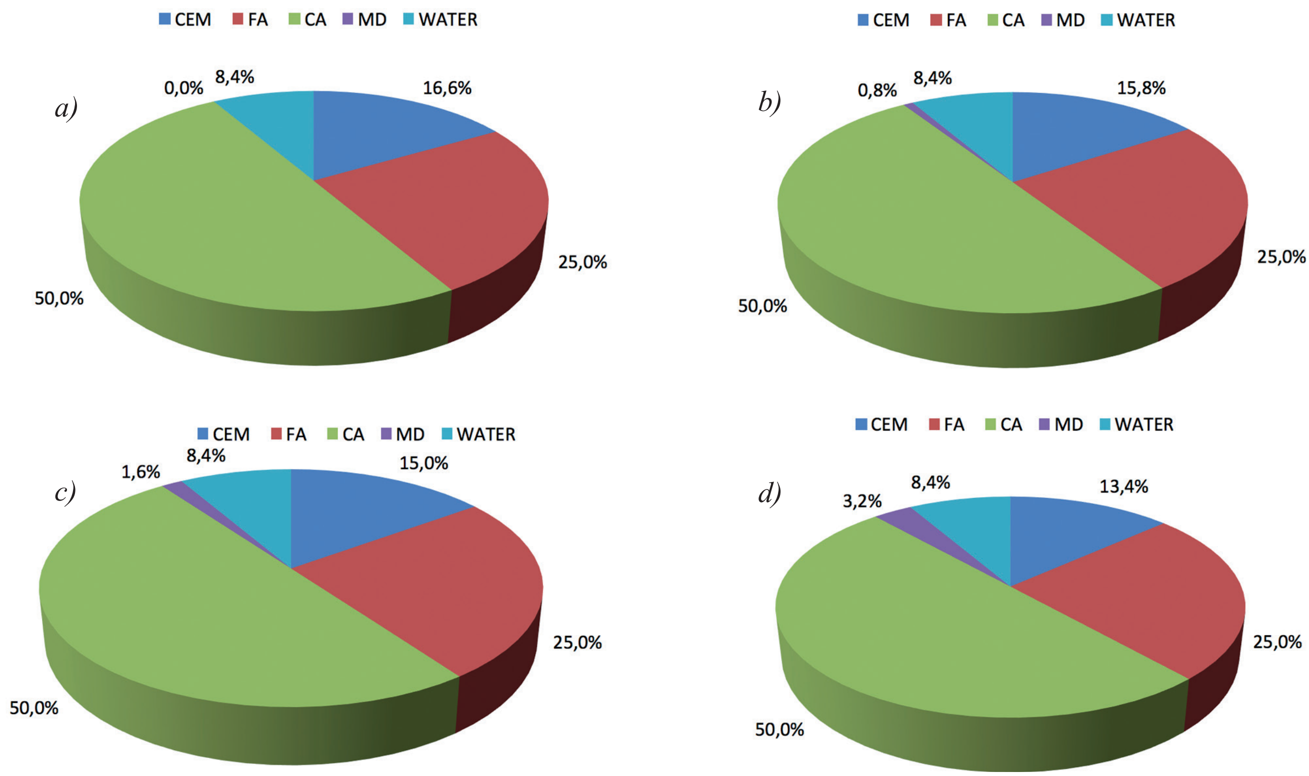

Fig. 2 - Pie charts a) CMI_0 b) CMI_5 c) CMI_10 d) CMI_15

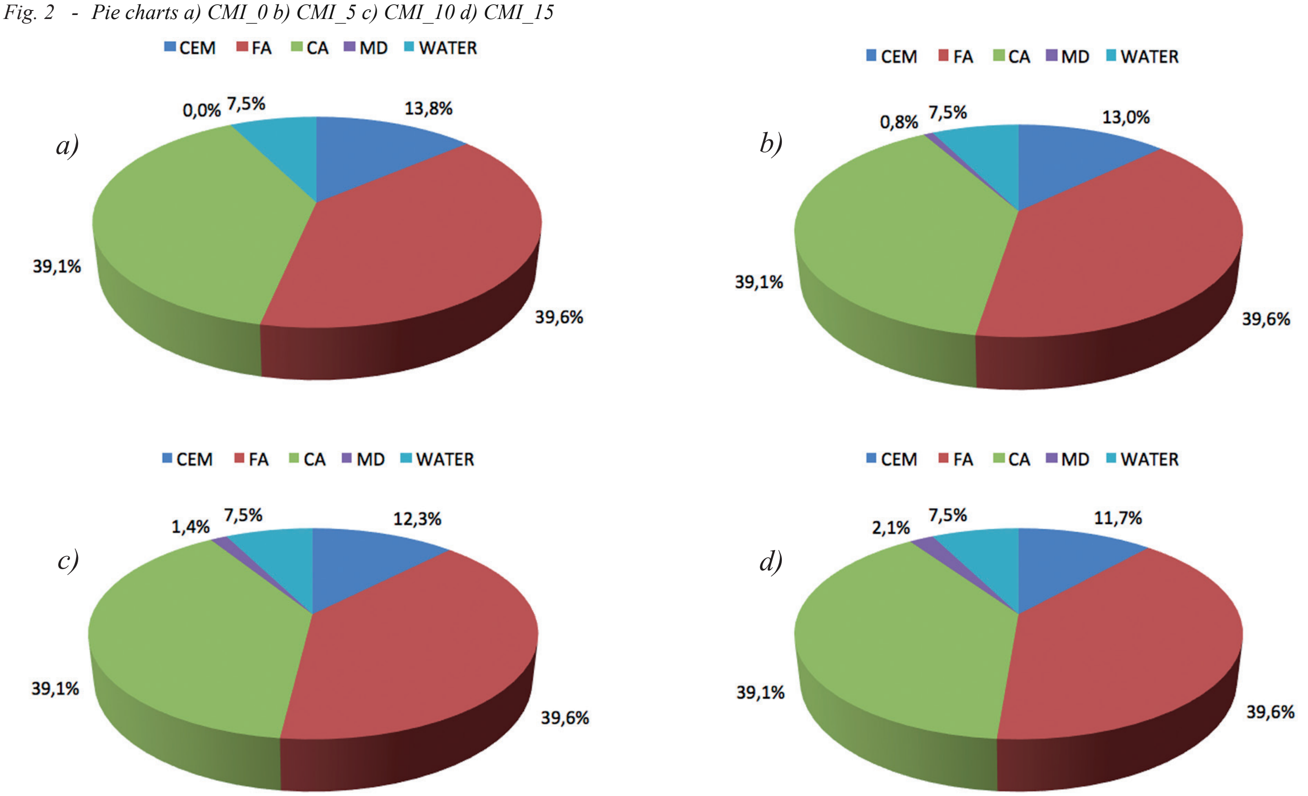

Fig. 3 - Pie charts a) CMII_0 b) CMII_5 c) CMII_10 d) CMII_15

with cement. The tests were determined using a compressive testing machine (SDE: Sistemas de Ensayo, model: CME-300/SDC, 1998) with a maximum capacity of 2,500 kN and were carried out under laboratory conditions. Strains were measured on a base of 150 mm. The specimen was loaded in the machine and preloaded it with a constant rate of loading within the range 0.6 ± 0.2 MPa/s.

Scanning electron microscope (SME) was used to verify the interfacial reaction generated by the hydration process in the different cured specimens.

Gypsum plaster

The feasibility of adding MD in plaster was studied by mixing the components replacing varying proportions of gypsum with MD (Lewry & Williamson, 1994; Singh & Garg, 1997). The experimental phase was carried out at the Department of Gypsum in LOEMCO laboratory (Technical University of Madrid - Spain).

The research had focused on mechanical projection gypsum plaster that is a gypsum plaster formulated for mechanical application, mixed with water to the required consistency and applied by projection machine to the background.

Two different types of materials were used in this research: gypsum (GY) and MD as aggregate.

GY: Type PROYAL MAX, PLACO Company - SONEJA plant (placo.es) was used in the laboratory program, with the technical characteristics tabulated in Tab. 10.

Testing. For this experimental part were carried out the following tests, according to the national standards indicated in the EN 13279-2: 2014 – "Gypsum binders and gypsum plasters", in order to evaluate the mechanical behavior of the created compounds: Consistency (water/plaster ratio by the flow table method) - Flexural strength - Compressive strength - Adhesion to the substrate.

In order to know water/plaster ratio of the GY+MD materials was used the flow table test, for all the mix-designs created (Tab. 11).

The mechanical behavior in terms of flexural strength was studied. For preparing specimens the plasters under test were mixed using the water/plaster ratio determined and adding the different percentages of marble powder 0-5-10-15%. Immediately after being prepared, each plaster paste was transferred using a spatula to press into the sides and corners of the mould which had previously been lightly greased. To eliminate any air-bubble the mould was raised 10 mm by its end and allowed to fall. This was repeated 5 times at each end. The moulds were filled not later than 10 minutes after the start of mixing, and the surface may not be smoothed. After setting was completed the surplus paste was scraped off with a sawing motion of the steel rule. Three prisms for each mix-design were prepared in this way and six for the control one.

Property	Value
Purity index	70 [%]
Grain size	0 – 1.5 [mm]
Water/gypsum ratio	0.5 – 0.6
Surface hardness	≥ 65 (shore C unit)
Efficiency	< 9.5 [kg/cm]
pH	> 6
Thermal conductance coefficient	0.26 [W/mK]
Tensile strength	≥ 1 [N/mm^2]
Compressive strength	> 2 [N/mm^2]
Adhesion	> 0.1 [N/mm^2]
Type of background	Traditional

Tab. 10 - Technical characteristics of Gypsum – Proyal Max

Mix Design	GY wt. %	MD wt. %	Water/Plaster ratio
1	100	0	0.60
2	95	5	0.60
3	90	10	0.57
4	85	15	0.55

Tab. 11 - Mix designs for the experimentation on plasters and relates water/plaster ratio

When an adequate degree of strength had been reached, the prisms, identified with marks, were removed from moulds. The prisms stored 7 days in the standard atmosphere: temperature 23 ±2°C and relative humidity of the air 50±5%. Then, they were dried to constant mass at 40±2°C. After drying, the samples were cooled to room temperature, and the force required to break a plaster prism 160 mm x 40 mm x 40 mm centers was determined.

Compressive strength was determined by applying a load to the broken parts of the test specimens used for the determination of flexural strength. The test specimens were compressed until it fails.

The test pieces were placed between the steel platens so that the sides of the prism which were in contact with the sides of the moulds were in contact with the platens over a section of 40 mm x 40 mm. The upper platen is allowed to tilt so that perfect contact was made between the test piece and the platen. The axis of rotation of the upper platen passes through the centre of the surfaces which are being compressed. The test specimens were loaded until ruptures of the test specimens occurred.

The adhesion of a plaster to a specific background is measured as the maximum load supported when a metal disc fixed to the plaster is pulled perpendicular to the surface. Bricks of size 30 x 14 x 4 [cm] were used as background surfaces (Fig. 4).

Background surfaces were prepared in accordance with good practice or the appropriate code of application (EN 13279-2: 2004).

The plasters were mixed, using the water/plaster ratio determined in Table 11, with the different percentages of marble powder 0-5-10-15%, and applied to the substrate. When the plaster had set the specimens was stored for seven days in the test-atmosphere: temperature 23±2°C and relative humidity of the air 50±5%. Using a circular core cutter (Fig. 5), test areas

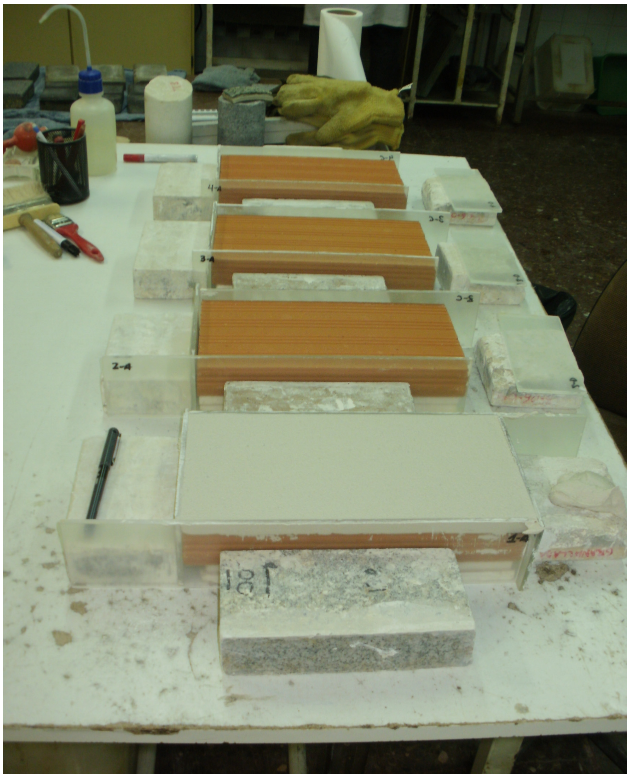

Fig. 4 - Adhesion test samples preparation

Fig. 5 - Core cutter and pull-heads for adhesive test

were isolated from the surrounding plaster. The cut was made to a depth of approximately 5 mm into the background and with 50 mm ± 0.5 mm in diameter. The pull-heads were glued to the isolated area of plaster with the adhesive (Loctite brand., No.454) (Fig. 5). The tensile load was applied perpendicular to the test area using the testing machine. The load was applied at an uniform rate within the range 0.003 N/mm^2 to 0.1 N/mm^2 per second, at the test area of 1.963 mm^2 (ø 50 mm).

Traction device permitting a tensile force to be applied to the steel plates without subjecting the assembly to a flexural stress.

RESULTS AND DISCUSSION

Marble dust characterization

MD characterization showed that the particles are very fine, as resulted in the grain size curve. The main fraction of the material exhibits grain sizes of below 25 µm (Fig. 6).

The XRD spectrum shows calcite to be the only mineral constituent detected. The measured density dry solid from filter press was 2.69 g/cm^3.The marble waste dust contains very minor amounts of impurities; therefore there are no constraints in adding MD as by-product.

Brick making

As written before, the mix design which allowed to obtain better workability with the substitution of clay with MD, was mix-design-C (Tab. 3). Thus, as regards mix-design-C samples, the average shrinkage was measured after drying equal to 5.60% and after firing the value of which was 0.42%. Table 12 illustrates the shrinkage for the brick samples after drying and after firing, average value was reported.

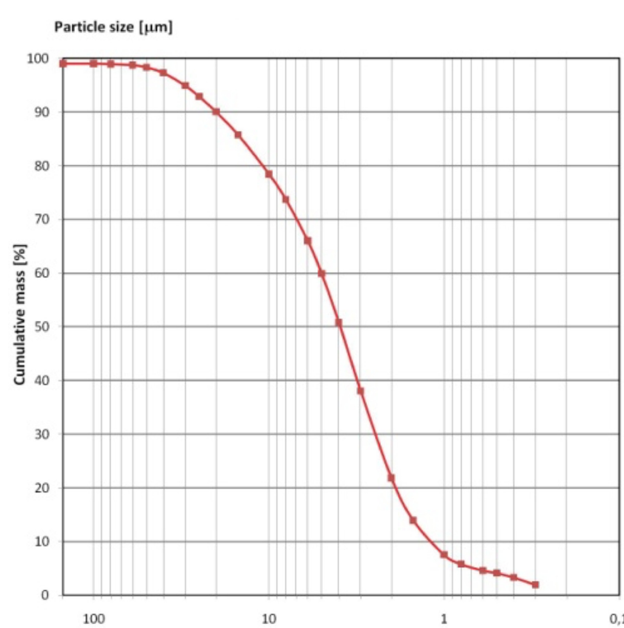

Fig. 6 - MD grain size distribution

Mix-Design C			
Shrinkage Measurement	[mm]	Shrinkage [mm]	Shrinkage [%]
Starting Length	50.0	---	---
Drying Length	47.2	2.8	5.60
Firing Length	47.0	0.2	0.42

Tab. 12 - Shrinkage determination

Shrinkage Measurement		Specific Absorption Rate [g·min^{-1}·dm^{-2}]	Water Absorption [%]
Starting Weigth [g]	3,611	0.31	13.8
Final weigth [g]	3,914		
Bearing Surface [dm^2]	979.4		

Tab. 13 - Water absorption and specific absorption rate on mix-design-C samples

Moreover, the specific absorption rate was checked equal to 0.31 g•min^{-1}•dm^{-2} while water absorption percentage was 13.8% (e.g. Tab. 13). It is important noticed that a standard brick. has the following values: as = 0.37 g•min^{-1}•dm^{-2} and w_a = 14%. The presenting results are the average of 4 tested samples.

As described the expansion due to humidity in structural ceramic materials, is improved by the addition of MD, in a proportion around 5%, to the clay paste. In fact results show that paste prepared using 5% of MD could be well considered, as a matter of fact the outcomes are very hopeful for mixing the MD in the conventional bricks making.

The experimental tests conducted on clay dried and fired sample bricks, in comparison with those of a standard brick, have showed that the use of MD, ensures the maintenance of withdrawal both in drying and firing processes.

Water absorption can be regarded as an indirect method to predict the technological properties of the final products since the measured values are closely related to the microstructure of the manufactured sample. As seen from the results, the mix-design-C sample water absorption is just a little bit lower than the standard brick one, and since the water absorption is one of the most critical properties for bricks, a small decreasing in this factor is a positive consequence.

Concrete

The workability of fresh concrete was measured in terms of slump, slump flow value which is presented in Table 14. Slump flow for mix-design I varying from 4.9 to 4.0 cm and for mix-design II varying from 5.0 to 4.2 cm.

The analyses of the results can be drawn that the consistency of the mixture is plastic (3 to 5 cm), and that this would satisfy the conditions imposed on the mixing. It can be seen that the replacement of cement by MD reduces the slump but within the limits of plastic consistency.

The compressive strengths (R_C) tests are proceeded satisfactorily. Generally, compared to plain concrete with the same W/C ratio and cement type, concrete with high limestone filler content with suitable particle size distribution possesses improved strength characteristics (Sonerbi, 2000; Petterson, 2001).

All the results obtained for the compressive tests are acceptable (Fig. 7).

The strength development characteristics were affected by MD percentages. The results are compiled in Tables 15 and 16;

Fig. 7 - Specimen rupture

	Mix-design I	Mix-design II
CM _0	4.9	5.0
CM _5	4.5	4.7
CM _10	4.4	4.5
CM _15	4.0	4.2

Tab. 14 - Slump test values [cm]

Mix-design I	Specimen 1	Specimen 2	Specimen 3	Average value
CMI_0	41.05	38.85	37.44	39.12
CMI_5	41.29	39.28	38.56	39.71
CMI_10	32.62	35.45	35.42	34.50
CMI_15	33.45	27.90	31.43	30.93

Tab. 15 - Compressive test data for mix-design I [MPa]

Mix-design II	Specimen 1	Specimen 2	Specimen 3	Average value
CMII_0	28.74	27.77	27.88	28.13
CMII_5	27.46	27.61	27.18	27.41
CMII_10	23.69	24.75	23.78	24.07
CMII_15	19.07	18.98	19.16	19.07

Tab. 16 - Compressive test data for mix-design II [MPa] as shown, the maximum compressive value was observed for specimens containing a 5% MD. In the case of 10% of replacement the compressive strength decreased and more in the case of 15%. So it was observed in Fig. 8 that there was a reduction in the compressive strength with increasing MD content.

In Figure 9 is shown the microstructure of the concrete mixture CMI_5, after 28 days of curing and in Fig. 10a) and b) the microstructure respectively of CMI_10 and CMI_0 past 28 days in the moist chamber.

The well bonded interfacial zone is a characteristic of higher strength development of the concrete. Analyzing the SEM photos, it's clearly visible the hydration of cement paste, in particular for CMI_5 (Fig. 9). It can be observed that the marble sludge powder had a high specific surface area; this could mean that is addition should confer more cohesiveness to concrete. The reaction between aggregate and cement-paste is not totally completed in fact is discernible the dark colour in the core which is a symptom of a no-reaction zone, instead of the clear zone that has already got reaction. Nevertheless, the reaction is in progress.

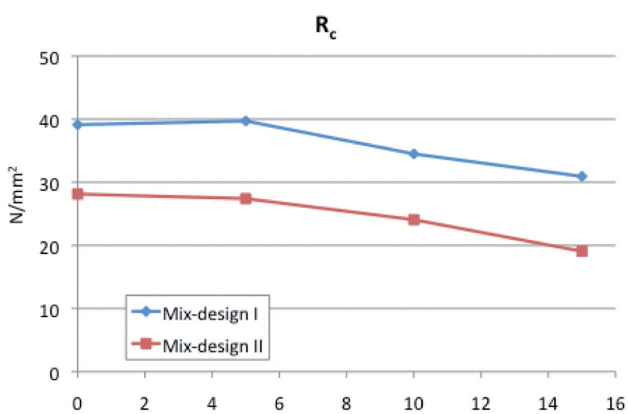

Fig. 8 - Compressive strength curves for both mix-designs

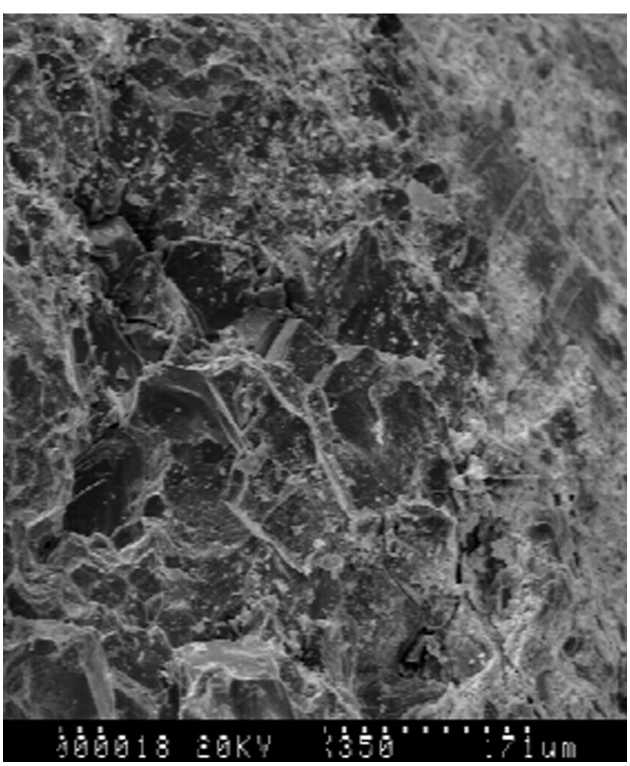

Fig. 9 - Microstructure of CMI_5 at 28 days (80x)

In the CMI_10 the aggregate borders look corroded, and this is a sign of good reaction (Fig. 10a). Despite that pores are visible within the matrix, it creates a reduction in compressive strength. This may be due to the smaller amount of cement used.

Moreover, even if a good bond between CEM - MD matrix and the aggregates is occurred, after 28 days of hydration, the distribution of aggregates after compaction is inhomogeneous due to the influence of manual vibration. As a result, sometimes gradients of strength may be significant.

In terms of mechanical performance 5% substitution of cement by MD provided higher compressive strength, after 28 days of curing, but all the results are acceptable. The replacement of cement with 5% of marble powder gives an excellent result in strength aspect and quality aspect. Then, test results show that these industrial wastes are capable of improving hardened concrete performance, in proper proportions. Generally experimental concrete has relatively high compressive strength.

For the CMII_15 sample was obtained the value of 19.07 MPa. It is a medium strength mix and is known as a C20 mix. This mix is not suitable for house foundations.

Gypsum plaster

Possibility to use MD as filler in gypsum plaster for projection one was studied.

The average values obtained for the rupture in flexural strength (P_F) tests are shown in the Tables 17, 18 and in Fig. 11.

It can be seen that MD has direct relation with the flexural strength of gypsum plaster. In fact, the use of marble powder resulted in increase in flexural strength, the average value rises from 1.32 [N/mm²] control specimens MP0, to 1.68 [N/mm²] MP15.

The effect of MD on the compressive strength (R_C) of set and hardened gypsum plaster at different percentage of substitution is shown in Tables 19 and 20 and in Fig. 12. The mean values of compressive tests were calculated and expressed in N/mm².

Data show that at 10% of MD addition, maximum attainment

	BREAKING LOAD, P [kN]			
Mix Design	**1**	**2**	**3**	**Mean Value**
MP0 - 1	0.63	0.64	0.65	0.56
MP0 - 2	0.54	0.45	0.47	
MP5	0.65	0.55	0.63	0.61
MP10	0.60	0.68	0.65	0.64
MP15	0.77	0.70	0.69	0.72

Tab. 17 - Figures obtained for rupture in flexural strength tests

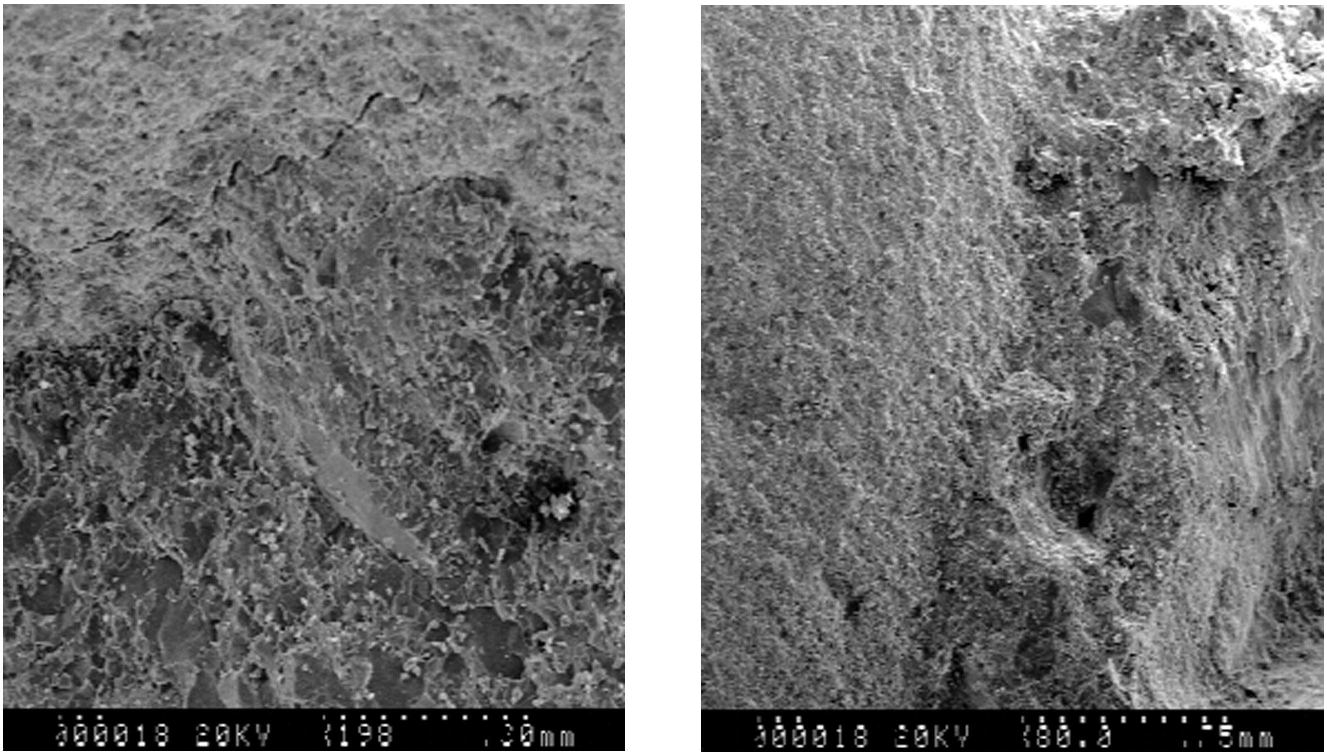

Fig. 10 - a) Microstructure of CMI_10 at 28 days (80x); b)Microstructure of CMI_0 at 28 days (80x)

Mix Design	Test specimen 1 P_F [N/mm²]	Test specimen 2 P_F [N/mm²]	Test specimen 3 P_F [N/mm²]	Mean Value P_F [N/mm²]
MP0 - 1	1.47	1.50	1.52	1.32
MP0 - 2	1.26	1.05	1.10	
MP5	1.52	1.29	1.47	1.43
MP10	1.40	1.59	1.52	1.51
MP15	1.80	1.64	1.61	1.68

Tab. 18 - Figures obtained for rupture in flexural strength tests

of strength generally was achieved which clearly manifests that in terms of compressive strength MD can improve the strength in the plasters.

The adhesive strength (R_U) was calculated as the mean value from the individual values of the specimen to the nearest 0.01 N/mm². The final results are reported in the following Table 21.

In the tests carried out for adhesive strength was impossible to calculate the average values for the different figures obtained, because of the ruptures that occurred.

In fact, in some cases rupture may not occur in the correct way at the interface between mortar and substrate. These values shall therefore be neglected when calculating the mean value. The fracture pattern shall however in each case be reported according to Fig. 13 a), b), c) and d).

When the fracture occurs at the interface between plaster and background (Fig. 13 a) the test value equals the adhesive strength, as in the test MP15. Only for this one, was possible to calculate the

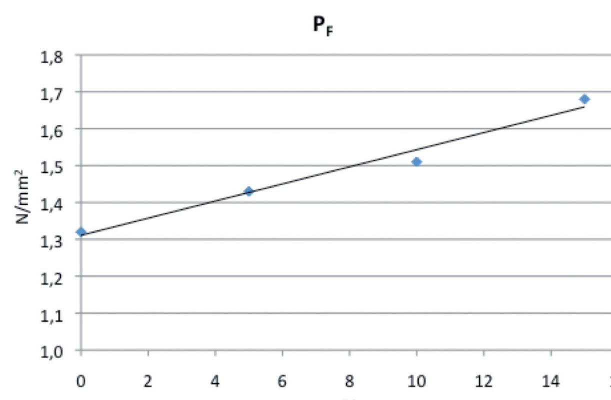

Fig. 11 - Flexural strength march

average value that results 0.17 [N/mm²], that is an acceptable datum. For the other specimens (tests MPO specimens 2 and 3, MP5

MAXIMUM LOAD AT FRACTURE, P [kN]							
Mix Design	1-1	1-2	2-1	2-2	3-1	3-2	Mean Value
MP0 - 1	6.65	6.70	6.84	6.89	7.20	6.99	6.50
MP0 - 2	6.14	6.02	6.14	6.07	6.21	6.11	
MP5	6.49	6.15	6.41	6.65	6.99	6.60	6.55
MP10	7.28	7.89	7.86	7.70	7.39	7.74	7.64
MP15	7.73	7.02	7.61	8.06	6.74	6.85	7.34

Tab. 19 - Figures obtained for rupture in compressive strength tests

Mix Design	Test specimen 1 R_c [N/mm^2]		Test specimen 2 R_c [N/mm^2]		Test specimen 3 R_c [N/mm^2]		Mean Value R_c [N/mm^2]
	1-1	1-2	2-1	2-2	3-1	3-2	
MP0 - 1	4.16	4.19	4.28	4.31	4.50	4.37	4.06
MP0 - 2	3.84	3.76	3.84	3.79	3.88	3.82	
MP5	4.06	3.84	4.01	4.16	4.37	4.13	4.09
MP10	4.55	4.93	4.91	4.81	4.62	4.84	4.78
MP15	4.83	4.39	4.76	5.04	4.21	4.28	4.58

Tab. 20 - Figures obtained for rupture in compressive strength tests

specimens 1 and 2, MP10 specimens 1 and 2 and MP15 specimens 1, 2 and 3) were present a fracture in the plaster itself, that corresponds to an adhesive strength greater than the test value.

As explained, the marble powder significantly increases both flexural and compressive strength in projection gypsum plaster. A compressive strength loss was observed in the case of the application of the 15% of MD, but the value remains considerably higher than the basic sample.

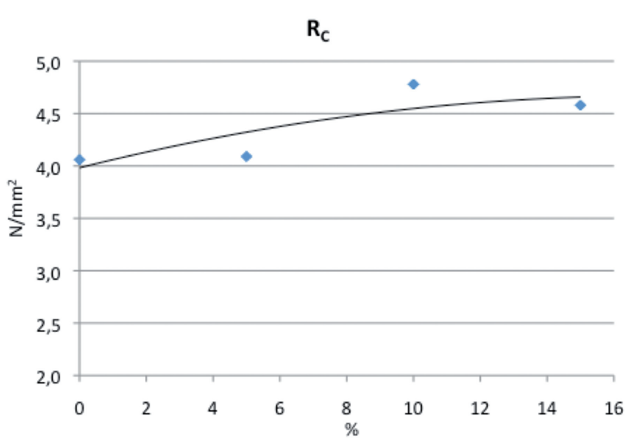

Fig. 12 - Compressive strength march

Mix Design	Test specimen 1 R_u [N/mm^2]	Test specimen 2 R_u [N/mm^2]	Test specimen 3 R_u [N/mm^2]
MP0	0.19	0.17	0.29
	Plaster	Interface	Interface
MP5	0.19	0.23	0.15
	Interface	Interface	Plaster
MP10	0.19	0.20	0.23
	Interface	Interface	Plaster
MP15	0.17	0.18	0.16
	Interface	Interface	Interface

Tab. 21 - Figures obtained for adhesion tests

It was found that the adhesive tests may not be sufficient for the determination of adhesive strength, because of the false ruptures, as analyzed.

CONCLUSIONS

The results presented and discussed along this work have shown the necessity of reducing the burden of the waste management, avoiding the negative impact associated with landfill, and of preserving natural resources, for example, by reusing industrial residues, should be regarded as a challenge and a great opportunity to suitably reformulate the traditional building products and keep or even improve their properties.

The high calcium carbonate content, the absence of heavy metals and pollutants, the particle size with high percentages of ultrafines confirm that the marble slurry could be used in commercial applications and can be economically exploited as by-product for many different industrial processes.

The main result obtained in this paper has shown that marble process residues could be used in several industrial field without any previous treatment. What's more, the outcome presented, shows the feasibility of using this by-product in the mix composition of concrete, bricks and mechanical projection gypsum plaster. According to this research, MD should be recycled as partial substitute of paste components. This means that these inexpensive residues can be regarded as good substitutes for the costly raw materials, therefore preserving the mineral resources, while lowering the production costs. The causes related to problems with materials are linked with the set of mechanical, physical and chemical characteristics. Consequently, improving properties is the only way to ensure their good execution and proper functioning when applied in the building.

Based on the investigation reported in this study, the following conclusions can be drawn.

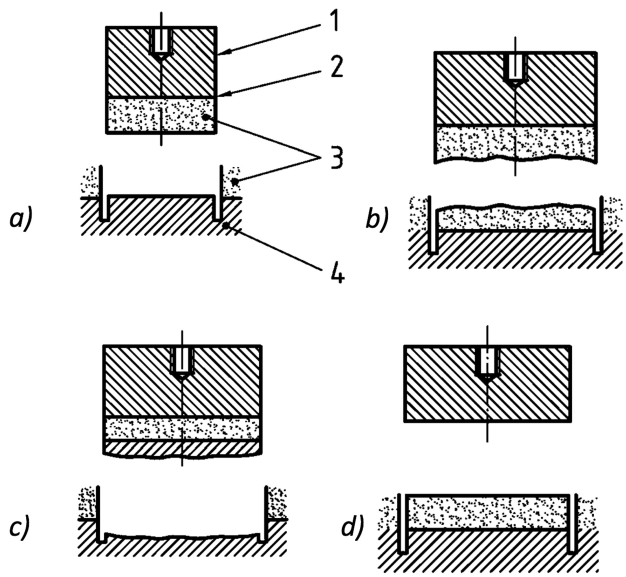

Fig. 13 - Examples of the failures (EN 13279-2) a) Adhesion fracture (at the interface between plaster and background); b) Cohesion fracture (in the plaster itself); c) Cohesion fracture (in the background material); d) Fracture in the adhesive layer

Brick making. The use of marble sludge in the production of ceramics is an option for reuse. Results arisen from this experimentation part have demonstrated that marble wastes can get help the problem of the expansion due to humidity in ceramic materials. The application appears most interesting in this industrial for substituting of the calcium carbonate, by the marble waste mud as additive to the clay paste.

Concrete. Technical possibilities of producing concretes containing MD have been studied with positive results. Thus it can partially replace cement, pursuing the aim of minimize waste generation and disposal and also of producing revenue for companies. In fact cement is the most expensive concrete component, as a result if can replace cement can obtain an economical revenue. Due to its high fineness of the MD it provided to be very effective in assuring very good cohesiveness of concrete. The results presented and discussed along this work enable to draw the conclusions that the high tonnage of raw materials consumed in the fabrication of concrete make their formulations suitable matrixes to incorporate this kind of residue replacing nonrenewable natural resources.

Gypsum plaster. The feasibility of adding MD in plaster was studied. The results have demonstrated that MD, added to gypsum plaster before gauging, modifies some physical properties of the binder after hardening, e.g. compressive and flexural strengths, increasing both of them.

In summary, until now, the fines shall be considered as an expensive item as waste products to be delivered to approved landfills. Looking into the research from economic point of view the economy can further improve, in fact it can be noticed that the final products consume large amount of raw materials, in addition to its benefits in saving landscape as well as natural resources, it can be, obviously, cheaper than commercial products.

In these years different industrial sector were investigated but all deriving from construction area, now the interest is passing to possible industrial applications having high value added. An experimental phase is currently carried out in order to join this by-product in tyre industry. Simplified rubber formulations filled with reused calcium carbonate and cross-linked by vulcanization have been investigated. From this, a lot of analyses are being carried out for the use of micronized calcium carbonate in rubber and tyre industries. The research is ongoing.

As a result, to effectively utilize of these wastes as raw material, filler, binder and additive in developing alternative materials, detailed physical and chemical engineering, thermal, mineralogical and morphological properties of these wastes are to be evaluated and accurate data made available. Durability and performance of the newer products and dissemination of technologies emphasizing costs-benefits analyses and life cycle assessment report will significantly contribute to successful commercialization of innovative processes.

In this respect, for the development of materials with waste materials, further research and development is necessary. Besides, not only on the technical, economic and environmental features but also on standardization, government policy and public education related to waste reusing and sustainable development is required for wide production and application.

In conclusion, by considering the added value of the waste products generated by dimension stone sawing and processing, future research directions will aim to enhance the quality of the fine material in industrial field applications. So what until now was considered as a waste, really is an important economic resource capable of promoting the sustainable industry.

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