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MEASUREMENTS TO EVALUATE THE POSSIBILITY TO PRODUCE BRICKS FROM WASTE CUBILOT SLAGS

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Abstract

In this paper, the typical measurement techniques and production procedures defined in the UNI-EN guidelines for ceramic manufacture are taken into consideration to evaluate the properties and the characteristics of the bricks produced by using a mixture containing cubilot slag. The results of the experimental tests demonstrate that these measurement techniques are not enough to define the safety of the waste bricks. Up to now, UNI-EN guidelines for ceramics do not define crystallinity measurement for the products and then do not foreseen to determine the interaction between slag and clay. In this paper, a solution for this lack is proposed. In particular, the Thermal Analysis, X-Ray Diffraction, and the measurement of dangerous ions releasing are considered. The experimental results suggest that these techniques should be included UNI-EN guidelines because allow the definition of specific properties to the waste materials.

Key words: biomass, biorefinery, systems design, zero emissions

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1. Introduction

Social needs to preserve the natural environment and the growing economic pressure have renewed interest in the use of products employing waste as raw materials. Such materials are also called secondary raw materials (Andreola et al., 2005; Dino et al., 2017; Wiemes et al., 2015).

The researches on utilization of waste materials to produce bricks can be divided into three general categories based on the production methods: firing, cementing and geo-polymerization (Zhang, 2013). In this regards, the firing ceramic process can be considered an ideal method to accomplish the inerting of hazardous waste materials (Bingham and Hand, 2006; Coronado et al., 2015). Therefore, many researchers have studied the production of bricks from waste materials based on firing, in the following simply called waste bricks (Zhang, 2013). Although many researches have been conducted on this topic, the commercial production of bricks from waste materials is still very limited. The possible reasons of this limited production are related to: (i) the difficulties to add the waste materials in the production line of traditional bricks, (ii) the scary to have a contamination of the environment from the bricks including waste, and (iii) the slow acceptance of waste bricks by industry and public, and mainly (iv) the absence of relevant standards or guidelines for the use/recycle of waste in bricks. On the other hand, the replacing of natural raw materials by wastes to produce bricks can configure an opportunity to ameliorate waste management of today (Rawlings et al., 2006). The valorisation of industrial wastes and their reuse as alternative raw materials, especially in ceramics technology, can presents several advantages

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when compared with the use of primary natural resources, namely reduction in the extraction volume of clay (Junkes et al., 2012). In addition, encapsulating the waste slags in a ceramics matrix is the best solution for achieving the inertizing and neutralization of the waste (Couto et al., 2001; Ribeiro et al., 2002). Moreover, sometimes a collateral benefit can be derived from the energy properties associated with the content of organic matter in the wastes (Zhang, 2013).

Based on the results showed in previous papers (Acchar et al., 2009), at the typical measurement techniques used to characterize the building materials, and described in the UNI EN guidelines (UNI EN 14411, 2016), further measurement methods of the produced materials must be added to evaluate the safety for humans. In particular, in this research measurement methods based on thermal analysis, x-ray powder diffraction, and dangerous ions releasing are proposed to be included in the UNI EN guidelines. These measurement methods are devoted to define the acceptance properties of the clay with respect to the slag (Lamonaca et al., 2017). As a consequence, experimental results are performed to validate the effectiveness of such method, by considering the use of different amount of cubilot slag added to a Calabrian clay and two different preparation methods of the bricks: extrusion and pressing.

2. Experimental setup

The measurements to qualitatively evaluate the major crystalline phases were performed utilizing the X-ray diffractometry (XRD) techniques. The XRD measurements were carried out by CuKα radiation using a Philips X-Ray Powder Diffractometer PW 1730/10 with a vertical goniometer (30 mA and 40 kV), with 2θ degree from 5° to 70° at 0.02°/s. The chemical compositions were carried out with an Energy Dispersive X-ray Analysis (EDAX) by Link installed on SEM Cambridge 360 microscopy. The thermal behavior of the materials was evaluated by utilizing a NETZSCH STA 409. The STA 409 allows the simultaneous evaluation of the: thermogravimetry (TG), differential thermogravimetry (DTG), and differential scanning calorimetry (DSC) curves. The simultaneous acquisition of TG and DSC data allows to easily compare the curves by assuming same experimental condition in the analysis of the sample. A computer controls all the measurement instruments, the calibration, the optimization of the experimental conditions and the hardware description are reported elsewhere (Carni et al., 2013; Lamonaca et al., 2015).

The Particle Size Distribution (PSD) was determined by using a series of sieve as defined in the UNI-EN guidelines. Two series of bricks were prepared: the first extruded utilizing an extrusion laboratory brick moulding machine MVP/O series G. The amount of water was automatically defined by the machine. The volume of the hopper is about 5 l and were utilized about 5 kg of charge for each mixture. The size of the samples was 135x22x11 ± 2 mm. A second series of bricks were performed utilizing 75 g of dry mixture with about 10% of water. The mixture was pressed at 300 bars with a laboratory press by Gabrielli. The pressure was maintained for 15 minutes. The size of the stainless steel moulds is 110.0x55.0x5.0 ± 0.1mm. The amount of powder pressed was constant and the size of the pressed bricks is about 0.2% different by the size of the mould after the shakeout. After the preparation the green body, bricks and tablets, were dried into an oven at 100 ± 5 °C for about one day.

The method of preparation is in agreement with procedures utilized by others (Andreola et al., 2005; Coronado et al., 2015; Vasile et al., 2016; Zhang, 2013). Then the samples were normalized and burned into a furnace by Nabertherm mod lh/15-12 for 180 min at 1000 °C for a first series and at 1100 °C for a second series. The heating and cooling cycles were controlled by computer. After any treatment, each sample, normalized for 24 hours at room temperature, was weighed and measured to evaluate the shrinkage and the weight loss. The linear shrinkage was derived from the length of ten samples before and after firing using a calibre with a resolution of ±0.01 mm, the results are reported as mean of the measurements. The bulk density was calculated as the ratio of the dry mass of the brick and its normalized volume.

The clay utilized in this research was mining from the area called Perrotta in the Rosarno town (RC, Italy), located at about 80 m on sea level. The cubilot slag was extracted from the storage platform of Pertusola farm in Crotone (Vasile et al., 2016)

Different amounts of slag were added to the raw clay body. About 5 kg of clay and of cubilot slag were collected and homogenized into a blender. The samples were dried at 60 °C and desegregated into a ball-mill for about 20 min and then sieved before being utilized. In Table 1 the composition of clay and slag used is reported. In Fig. 1 the particle size distribution of the dry slag and of the dry clay after the desegregation are reported considering the amount passing and the amount retained on each sieve in agreement with UNI-EN 933-1 procedures (Eliche-Quesada et al., 2011).

Table 1. EDAX chemical analysis of the slag and clay. % in weight

	SiO_2	*$Al2O_3$*	*Fe_2O_3*	*CaO*	*MgO*	*K_2O*	*Na_2O*	*ZnO*	*SiO_2/ Al_2O_3*	*CuO*	*MnO*	*PbO*	*Others*	*Ignition loss*	*SO_x*
Slag	6.72	1.05	40.24	1.39	1.39	0.18	15.48	20.21	6.40	1.33	1.12	6.14	0.66	---	4.09
Clay	55.50	18.20	6.62	4.22	2.15	2.94	1.55	---	3.05	---	0.96	---	1.09	6.99	---

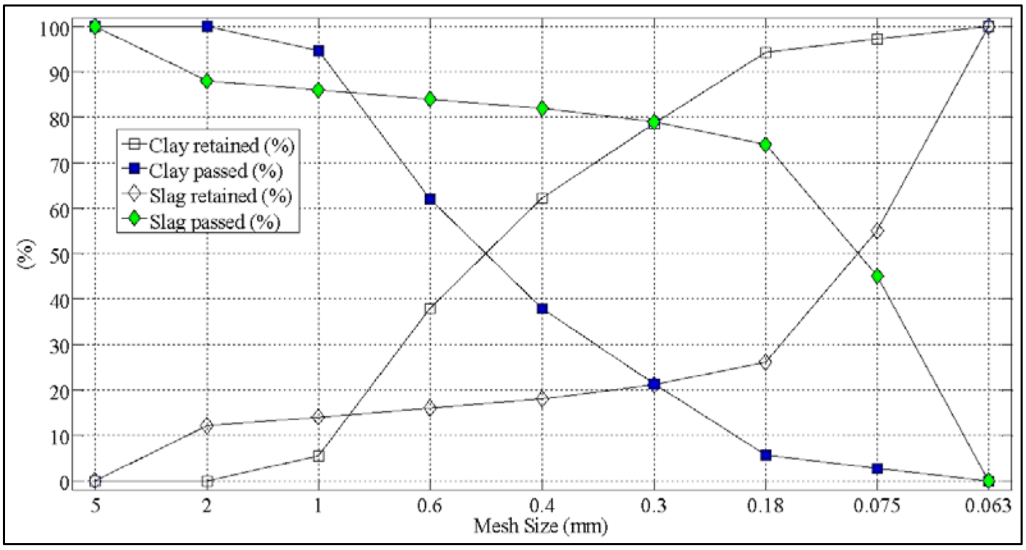

Fig. 1. Size distribution of clay and slag after 20 minutes of desegregation. Mesh size (mm) as reported on the used sieve

Five samples of extruded bricks and five samples of pressed bricks, after the mechanical characterization, are immersed into a beaker containing 800 ml of water to evaluate the ability of the bricks to release metals. In particular, after ten days of immersion at room temperature the water was filtered and analysed with atomic absorption analysis utilizing a Shimadzu AA 660 spectrophotometer.

3. Results and discussions

The chemical analyses of the clay and of the slag (Table 1) confirm the compatibility of the powders. In fact, the addition of slag to the mixture does not modify substantially the SiO_2/Al_2O_3, $Na_2O + K_2O/SiO_2$ and $Na_2O + K_2O/Al_2O_3$ ratios. In this regards, the addition of slag does not modify, in particular, the firing temperature of the bricks and the technical properties of the ceramics products obtained after the firing. Also, the mixture slag and clay can be proposed because the curves reported in Fig. 1 show that the clay and the slag are able to be desegregated and homogenized together in a blender having a compatible behaviour. This kind of measure is proposed because it is fundamental in a way to establish the compatibility between the slag and the clay.

Fig. 2 reports the X-ray patterns:

- A, B, C mixtures of clay and slag,
- D curves of the clay fired at 1100°C,
- E, F clay and cubilot slag as made,
- G, H curves of slag after firing at 1000°C and 1100°C.

The patterns clearly show that during the firing the slag does not modify the crystalline product of the bricks and this is the proof that the slag is just incorporated into the clay body. Moreover, the X-ray pattern of the *as made slag* (curve F) does not show the presence of Quartz (there is no peak at about 26° 2θ degree) additionally, there are not peaks detectable with appreciable intensity, related to heavy metal silicates. It is due to the possibility that part of heavy metals can be linked with silicon but the amount of their silicates is very low and the sensibility of the diffractometer is not enough to detect this very low concentration. The peaks attributed to the carbonates or sulfates have not an intensity sufficient to define the type of gypsum or carbonates. In the diffractograms A, B and C, peaks at low intensities attributed to the typical products normally detected in bricks fired at 1000°C and 1100°C are detected as reported in (Carter et al., 2007).

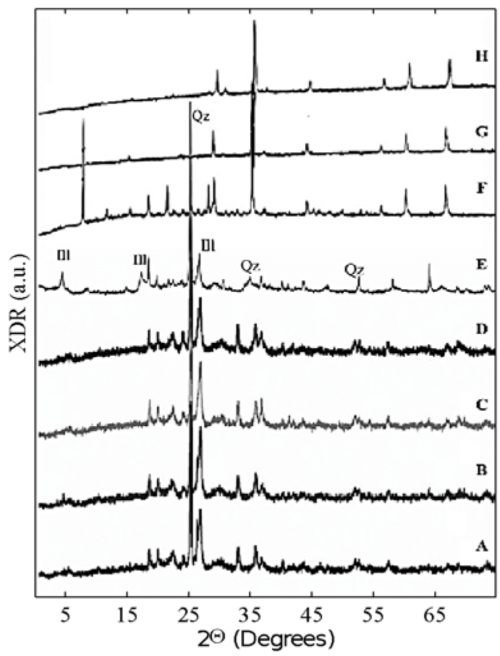

Fig. 2. XRD patterns of materials used to produce bricks and product after firing at 1000°C and 1100 °C. Curves E and F clay and cubilot slag as made; curves G, H slag after firing at 1000°C and 1100 °C respectively; curves A, Clay fired at 1100°C; curves B, C and D, mixtures clay slag after firing at 1100°C containing 2, 4, 8 % of slag respectively. Qz indicates the quartz peaks and III indicates the Sulphates and/or Carbonates

The salts of heavy metals, as sulfates and carbonates, with peak at about 12° 2θ degree, detected before the thermal treatment, are destroyed and transformed into oxides during the heating at 1000°C in air. It is an indication that the SiO_2 present in the slag is not involved, or at least marginally involved, in the formation of the bricks. In this regard the addition of slag to the clay does not produce a variation of the SiO_2/Al_2O_3 ratio in the bricks and it does not produce a variation of the fired ceramics products. The formation of some spinel cannot be excluded. These measurements confirm that the slag is practically inerting and merging into the ceramic matrix of the bricks. This behavior is observed with other type of industrials residues by others (Coronado et al., 2015; Zhang, 2013). Utilizing a firing temperature higher than 1100°C the X- ray patterns are very similar, just a variation of some peak intensities is observed due to the formation of a vitreous phase. The bricks produced after 120 min of firing at 1100°C are slightly deformed and then the use of a temperature higher than 1100°C is not possible with this type of clay. This preliminary conclusion is also supported by DSC curve of clay (Fig. 4) where is evident that at temperature of 1000°C start a fusion of the clay.

The amount of the weight losses evaluated for the slag and for the clay corresponding at the DSC curves of Figs. 3-4 are reported in Table 2.

The thermograms of Figs. 3-4 confirm the hypothesis formulated with the measurements with X-ray. Indeed, the DSC of cubilot slag (Fig. 3) does not show peaks that can be attributed to the formation of silicates of heavy metals. Moreover, the TG and the DTG curves show only weight losses confirming the degradation of the salts and their dehydration. In Fig. 3 the peaks of DTG and of DSC between 100°C and 150°C are linked and can attributed to the loss of the humidity of the slag (water no strongly linked with slag), in fact the DSC peak is endo. The peak of the DTG associated to a small peak of the DSC at about 350°C is attributed to the loss of water strongly linked to the salts and other micro components of slag. The peak at about 800°C is attributed to the thermal degradation of heavy metals salts, as sulphates and carbonates, detected by X-ray analysis. This degradation transforms the salts to metal oxides. The very small effects detected in DSC curves correlated with the small amount of the weight loss, confirm the entity of the effect and the small quantity of these salts. This is in agreement with analysis showed in Table 1. It is the explanation of the disappearance of the peak at about 12° 2θ degree in the X-ray slag pattern after firing (Fig. 2).

As concerning the clay, the thermograms in Fig. 4 show just 3 peaks: the first (about 100°C) attributed to the humidity, the second (about 500°C) is linked to the degradation of the hydrated silicates of the clay, and the third peak (very small at about 800°C) related to the decomposition of traces of carbonates and at the combustion of some organics normally presents in the mines.

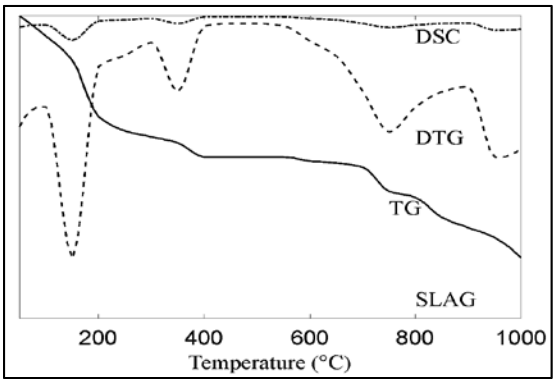

Fig. 3. Thermograms of cubilot slag

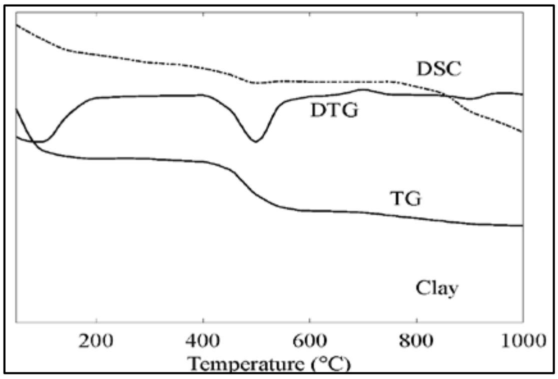

Fig. 4. Thermograms of clay

Also, the modification of the slope of DSC curve, at high temperature, indicates that the fusion of the silicates of the clay starts. In this regards the maximum temperature at which it is possible fire the bricks are 1100°C. In fact, the samples obtained at higher temperature result deformed as above desumed with the XRD patterns.

The intensities of the thermograms peaks is not indicative of the intensity of the phenomena at which are associated, because their intensities are automatically defined by programs to better visualize the effects. It is the reason that ordinates are without a scale. This technique is well known at the users of simultaneous thermal analyzer. The results reported in Table 3 confirm the good effect of the addition of slag to the clay mixture because the weight losses of the formed bricks are reduced as function of the percentage of the added cubilot slag.

Table 2. Weight losses of clay and cubilot slag

	1° DSC peak 129°C	*2° DSC peak 362°C*	*3° DSC peak 686°C*	*Total loss*
Clay	3.13 ± 0.2%	3.58 ± 0.2%	0.85 ± 0.2 %	7.79 ± 0.3 %
Slag	0.54 ± 0.2%	0.23 ± 0.2%	0.58 ± 0.3 %	1.14 ± 0.3 %

Table 3. Weight losses after drying at 110°C and after firing at 1000 and 1100 °C

	Extruded				*Pressed at 300 Barr*			
% Slag	*0*	*2*	*4*	*8*	*0*	*2*	*4*	*8*
Green body weight as made (g)	83.72 ± 1.55	79.20 ± 1.45	80.66 ± 1.49	77.75 ± 1.54	74.30 ± 0.13	74.51 ± 0.12	74.69 ± 0.13	74.02 ±0 .14
Weight losses after drying								
Weight loss %	23.30 ± 1.05	22.85 ± 1.15	21.52 ± 1.13	20.13 ± 1.09	11.01 ± 0.99	11.01 ± 0.89	9.77 ± 0.90	7.95 ± 0.79
Weight losses after firing								
Loss after firing at 1000°C, %	10.10 ± 0.10	10.10 ± 0.06	10.10 ± 0.08	10.10 ± 0.10	10.10 ± 0.12	10.35 ± 0.11	8.78 ± 0.09	7.50 ± 0.09
Loss after firing at 1100°C, %	10.10 ± 0.08	10.10 ± 0.10	10.10 ± 0.05	10.10 ± 0.07	10.15 ± 0.12	10.40 ± 0.14	8.90 ± 0.10	7.61 ± 0.88
The pressed bricks, after the forming shown a sizes variation of about 0.2 %.								

It is evident that the extrusion process needs about the same amount of water (automatically defined by the machine) independently by the amount of slag added. In other hand, in the pressed bricks process the amount of slag modify and reduce the amount of water that is necessary to form the pressed brick. These results are in agreement with the results reported in the following (Figs. 5-7).

The amount of slag in the mixture is not higher than 8%. In fact, during the drying and the firing, the added slag does not lose weight as supported by the results of TG and DSC (Fig. 3 and Table 2). This is because the slag is obtained by a thermal process. These reduction of weight losses are supported by a small shrinkage after the drying and after the firing of the formed bricks. These effects are observed on bricks extruded or pressed. The results are showed in Fig. 5. It is evident that the shrinkage of the pressed bricks is very low. This is due to the small amount of water added to the mixture prior to the molding process. The density of the extruded bricks is not influenced by the addition of slag. In fact, the density is about 1.59 ± 0.15 kg/ml for bricks without slag and 1.70 ± 0.15 kg/ml for bricks with 8% of slag, then there is a low variation. In the same manner, the density of pressed bricks is 2.66 ± 0.55 kg/ml without slag and 2.86 ± 0.56 kg/ml for bricks with 8% of slag, confirming the low influence of the slag addition in both cases.

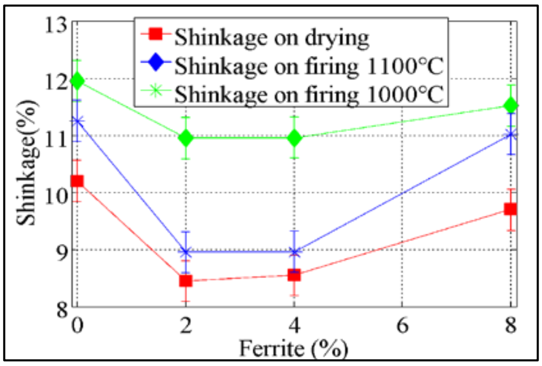

Fig. 5. Shrinkage of the slag-bricks extruded after drying and after firing

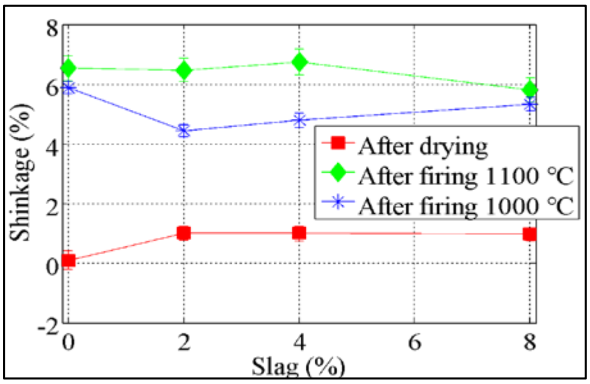

Fig. 6. Shrinkage of slag bricks pressed at 300 Bar after drying and after firing.

The addition of the slag to the clay modifies the shrinkage of the brick. In particular, for bricks extruded, a high reduction is observed when 2% of slag is added to the clay. By increasing the amount of slag the shrinkage increases because of the amount of water that the extrusion machine automatically adds to the mixture in order to have a better plasticity and a good extrusion.

This explanation is supported by the weight loss of the bricks after the drying process. In fact, the increasing of the added slag (more than 2%) produces an increasing of the water loss as confirmed by the results of the porosity reported in Fig. 7.

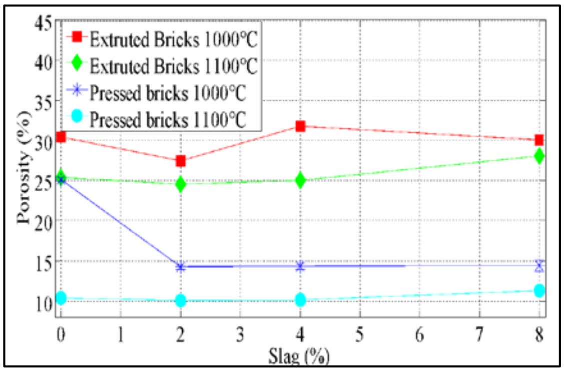

Fig. 7. Porosity of extruted and pressed bricks after firing at 1000 and 1100°C for 120 min. (UNI-EN 8942)

Different trend is observed for pressed bricks. It is due to: (i) the constant small amount of water added to the mixture, (ii) the effect of the pressure, and (iii) because the process is carried out with a constant amount of mixture's powder and forming pressure.

In any case the high variation of the properties above reported, due to the addition of the 2% of slag to the mixture, are quite in agreement with the results observed with other waste materials added to the clays reported in literature (Eliche-Quesada et al., 2011).

The results reported in Table 4 are in agreement with the results of porosity (Fig. 7). Indeed, the porosity of the pressed bricks is at least 50% of the porosity of the extruded bricks. Considering wasted bricks as traditional bricks following the UNI-EN guidelines, the results for pressed define these bricks as ceramics of type B11b (UNI-EN 178), because the water absorption is between 6 and 10% (Table 4). Than at least these materials can be proposed as not antifreeze tile flooring. The extruded presents a low absorption property with respect to the extruded traditional bricks and then can be proposed as bricks for wall because the gelivity is in agreement with properties required by UNI-EN guideline as reported in Table 4. In addition, the results reported in Tables 3-4 and in Figs. 5-7 suggest that the firing temperature equal to 1100°C can be considered a better cooking temperature with respect to 1000°C, and that the 2% of added slag can be considered the best adding percentage to have mechanical properties in agreement with EN-UNI guidelines, used for the traditional bricks.

The mechanical properties of the extruded and pressed bricks are carried out with different procedures due to the different size of the samples and the different producing procedures. In Table 5 are reported the mechanical properties of the extruded bricks. All reported results are measured and calculated following the procedure of the guideline UNI 8942 about completely transposed in the UNI-EN guidelines. From these results, it is evident that the inclusion of slag does not produce a variation of the mechanical properties. Moreover, it is evident that cannot be added, to this kind of clay, an amount of slag higher than 4% because the mechanical properties are not able to satisfy the properties suggested by UNI-EN guidelines for traditional bricks.

The inclusion of the slag in the mixture does not produce bricks releasing heavy metal. Just a concentration of iron and zinc are detect as reported in Table 6. These concentrations are lower than the concentration established by Italian laws and by the European suggestions. For the slag, the concentration of heavy metals as Cd, Hg, and Sn is reported. For each heavy metal is reported the mean value evaluated on 10 samples of the same stock. After the drying of the samples immersed in water, no efflorescence is observed. After the immersion in the distilled water, the pressed bricks have shown a concentration of heavy metals that is at least the 30% lower than the extruded bricks. This result is justified by the lower porosity and then the lower capability to adsorb water. In other hand the firing of bricks carried out in oxidizing environment produce heavy metal completely oxides and/or linked with silicon than are practically insoluble in distilled water. In fact, the ceramization is commonly used to fix the heavy metal, reducing the environmental risk.

Table 4. Absorption and imbibition properties of waste bricks extruded and pressed fired at 1100°C for 120 min (UNI-EN 99; 176-177-178)

	Extruded Bricks			*Pressed Bricks*		
% of slag	***Absorption of water %***	***Saturation %***	***Imbibition of water %***	***Absorption of water %***	***Saturation %***	***Imbibition of water %***
0	12.67 ± 0.31	0.86 ± 0.22	6.80 ± 0.41	4.94 ± 0.51	0.88 ± 0.41	2.80 ± 0.42
2	11.40 ± 0.32	0.88 ± 0.31	5.48 ± 0.61	9.02 ± 0.62	0.87 ± 0.40	4.28 ± 0.61
4	10.72 ± 0.51	0.74 ± 0.52	9.07 ± 0.60	6.03 ± 0.43	0.87 ± 0.31	3.07 ± 0.60
8	6.66 ± 0.32	0.93 ± 0.31	12.36 ± 0.52	6.60 ± 0.51	0.88 ± 0.51	3.36 ± 0.52

Reducing the firing temperature at 1000 °C, these values are increased of about 20 %. It is due to the increasing of the open porosity

Table 5. Mechanical properties: values obtained from the correlation formulas reported in UNI-EN guidelines

Slag %	***Average Decline (kg_f/cm^2)***	***Average Tensile (kg_f/cm^2)***	***Avarege Compress (kg/cm^2)***	***Cylindrical resistance (kg_f/cm^2)***	***Young modulus E compress (kg_f/cm^2)***	***Young modulus E tensile (kg_f/cm^2)***
0	125.23±0.15	62.61±0.12	336.07±0.22	1513.01±0.13	69926.87±0.15	63933.13±0.16
2	148.53±0.13	74.27±0.18	387.20±0.25	1871.16±0.18	77730.86±0.14	71068.21±0.15
4	144.17±0.18	72.09±0.14	377.74±0.28	1803.03±0.15	76308.29±0.17	69767.58±0.13
8	114.55±0.17	57.28±0.15	312.11±0.23	1354.17±0.14	66169.26±0.13	60497.60±0.19

Table 6. Concentration of heavy metals in distilled water after 10 days of immersion of extruded bricks

Slag %	***Fe, ppm***	***Ni, ppm***	***Pb, ppm***	***Zn, ppm***	***Mn, ppm***	***Cd, Hg, Sn, ppm****
0	0.090 ± 0.003	absent	absent	absent	absent	absents
2	0.097± 0.002	-------	------	0.011 ± 0.001	-------	-------
4	0.325 ± 0.002	-----	-----	0.015 ± 0.003	------	------
8	0.362 ± 0.003	------	------	0.044 ± 0.002	-----	------
Slag	0.121 ± 0.002	0.372 ± 0.003	1.084 ± 0.001	5.163 ± 0.002	0.120 ± 0.003	0.051 ± 0.004

4. Conclusions

The measurement techniques typically applied in the paper to characterize traditional materials are used for the characterization of building materials that include waste. These procedures for the evaluation of some characteristics and parameters of the material are standardized for every material in the UNI EN guidelines.

For the building material including waste the guideline must enriched with further measurement methods to evaluate material acceptability as concerning the safety for the human. In fact, if on one side the addition of cubilot slag to the clay can produce some benefit to the environment because reduces the amount of raw material (clay) to take from mine and produce a ceramic in capsulation of this dangerous slag, on the other side, a not proper inclusion of waste in bricks can provokes the generation of dangerous materials, not well intertied in the clay, with hazard for humans.

The combination of the: (i) measurements for material characterization defined by guideline, and (ii) the physical-chemistry measurements presented in this paper can also be effectively used to characterize the properties of traditional bricks. Therefore, it is suggested to add in the UNI-EN guideline for ceramics building materials, in order to extend them also for waste-materials, the (i) thermal analysis, (ii) X-ray powder diffraction and (iii) dangerous ions releasing.

In this paper the effectiveness of the proposed measurements is evaluated by considering several cooking temperatures, production procedure, percentage of included slag. From the experiments and the performed measurements it is possible to asses that bricks including the 2% of cubilot slag, and fired at 1100°C have mechanical properties in agreement with the present UNI-EN guidelines (can be used instead of traditional materials) and by using the proposed adjunctive measurements it is also possible to assess that these materials are not dangerous for human.

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