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Sludge valorization from wastewater treatment plant to its application on the ceramic industry

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ABSTRACT

The main aim of this study is to assess the effect of incorporating waste sludge on the properties and microstructure of clay used for bricks manufacturing. Wastewater treatment plants produce annually a great volume of sludge. Replacing clay in a ceramic body with different proportions of sludge can reduce the cost due to the utilization of waste and, at the same time, it can help to solve an environmental problem. Compositions were prepared with additions of 1%, 2.5%, 5%, 7.5%, 10% and 15% wt% waste sludge in body clay. In order to determine the technological properties, such as bulk density, linear shrinkage, water suction, water absorption and compressive strength, press-moulded bodies were fired at 950 °C for coherently bonding particles in order to enhance the strength and the other engineering properties of the compacted particles. Thermal heating destroys organic remainder and stabilizes inorganic materials and metals by incorporating oxides from the elemental constituent into a ceramic-like material. Results have shown that incorporating up to 5 wt% of sludge is beneficial for clay bricks. By contrast, the incorporation of sludge amounts over 5 wt% causes deterioration on the mechanical properties, therefore producing low-quality bricks.

1. Introduction

The considerable growth of the waste generated and the global environmental situation in which we find ourselves make it possible for projects on waste reuse to be implemented, provided they are profitable (Andreola et al., 2005; Elías, 2008).

By purifying wastewater, large quantities of sludge are generated. In case this final sludge is not disposed correctly, it can considerably contribute to environmental contamination (Cusidó and Cremades, 2005). Usually, sludge from wastewater treatment plants has been placed in landfills, but many problems, such as acceptance landfill sites, capacity limitations. Incineration may be an alternative solution to reduce its volume, but substantial amounts of ash are produced. Sludge is usually a heterogeneous solid material.

The construction industry is the most indicated technological activity sector to absorb solid wastes, due to the large quantity of raw materials and final products used.

The prospective benefits of using sludge as the brick or tile additive in the fired matrix, oxidizing organic matter and destroying any pathogens during the firing process have been studied by others authors (Alleman et al., 1990; Tay and Show, 1992; Weng et al., 2003; Jordán et al., 2005; Espejel et al., 2006; Merino et al., 2007; Monteiro et al., 2008). Jordán et al. (2005) studied the substitution of clay for sewage sludge in different proportions (0–10 wt%) in a ceramic body. Ceramic bodies were prepared by uniaxial pressing and fired. The authors concluded that the incorporation of sludge gives rise to a decrease of the bending strength, therefore the selection of a adequate percentage of sludge (4–5 wt %) to be added to the body clay to meet the standards. Monteiro et al. (2008) studied the influence of firing temperature on the technological properties of red ceramics prepared with incorporation of 0, 3, 5 and 10 wt% of sludge into the clayey body. The authors concluded the incorporation of sludge must be done in low percentage (3, 5 wt%) to avoid the damage the ceramic processing and the quality of the ceramic.

Ceramic products, bricks and tiles, also have a heterogeneous composition, being formed by clay raw materials with a very wide range composition (Couto et al., 2003). For this reason, this industry sector is suitable for valuation and use of different wastes, among which we can find sludge coming from wastewater treatment plant (Dondi et al., 1997a,b).

Considering that in the province of Jaen (South Spain), the ceramic industry has a great economic importance, the possible application of this recycling technique can be same benefits as

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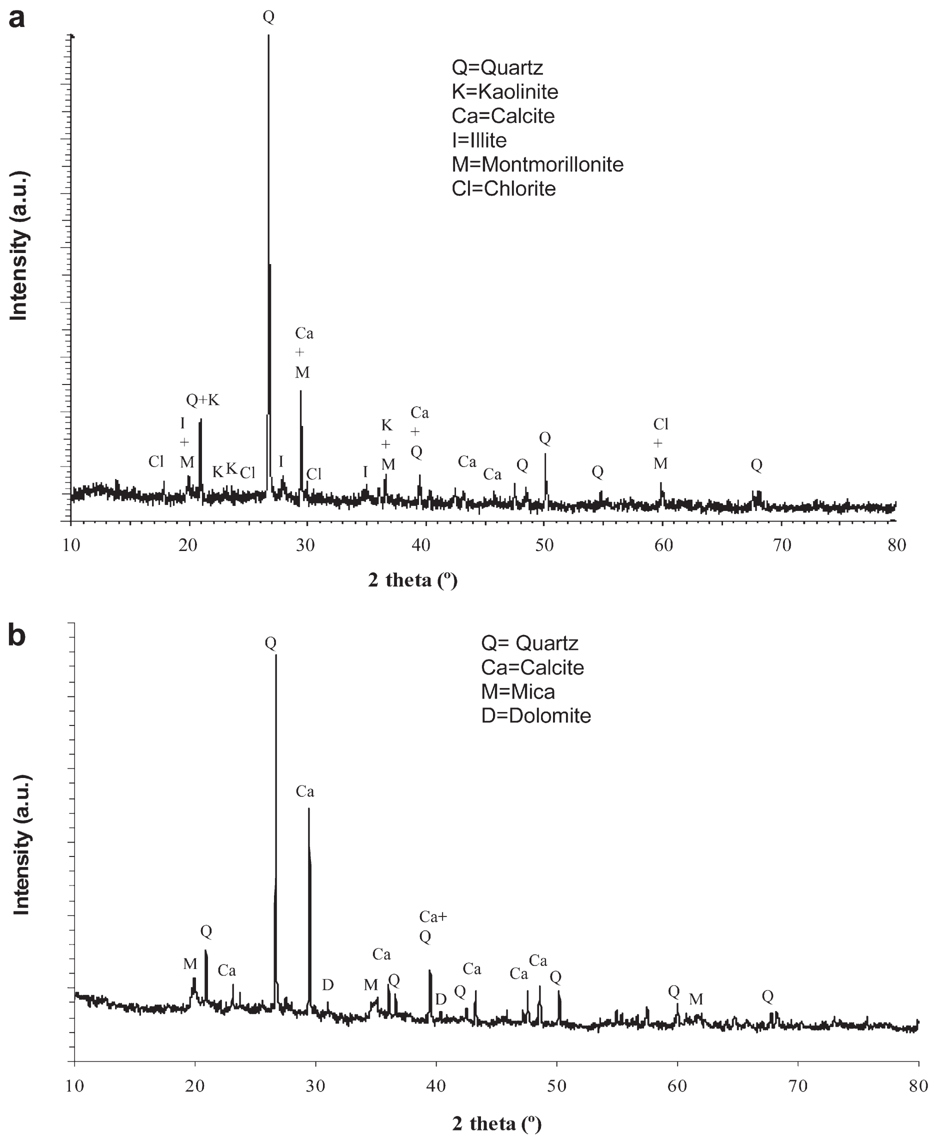

Fig. 1. DRX patterns for powdered (a) clay and (b) sludge.

(i) saving a resource, raw material and energy; (ii) positive effects on the brick-making processes and (iii) reducing the cost of final product and environmental problem due to using waste additive in the process.

This work assesses the use of sludge wastewater treatment plant, normally is placed in landfill or used for agricultural purposes (Directive 86/278/EEC, 1986), to manufacture structural ceramics as an alternative means of sludge disposal. The raw materials were characterized by physical–chemical methods and final ceramics products physical and mechanical properties were investigated, taking into account the composition.

2. Materials and methods

2.1. Materials characterization

Materials used in the present study are clay and sludge from Jaen (South Spain). In order to get a uniform particle size, both sludge and clay were crushed and grounded until a powder with a particle size suitable to pass through a 150 μm sieve was obtained.

Table 1
Chemical composition of the clay and the sludge ash and metal content of the sludge.

Oxide content (%)	Clay	Sludge	Metal content (%)	Sludge
SiO_2	55.82	46.37	Na	1.580
Al_2O_3	12.13	20.33	Mg	0.990
Fe_2O_3	4.83	8.55	Al	2.870
MnO	0.03	0.28	K	0.170
MgO	1.49	2.19	Ca	3.550
CaO	9.21	11.15	Mn	0.160
Na_2O	0.49	0.36	Fe	2.110
K_2O	2.78	3.25	Zn	0.512
TiO_2	0.83	0.85	Si	0.027
P_2O_5	0.12	5.89	Cr	0.090
Zr (ppm)	279.3	161.7	Ni	0.023
LOI	10.55	0.05	Cu	0.048
			Sn	0.015

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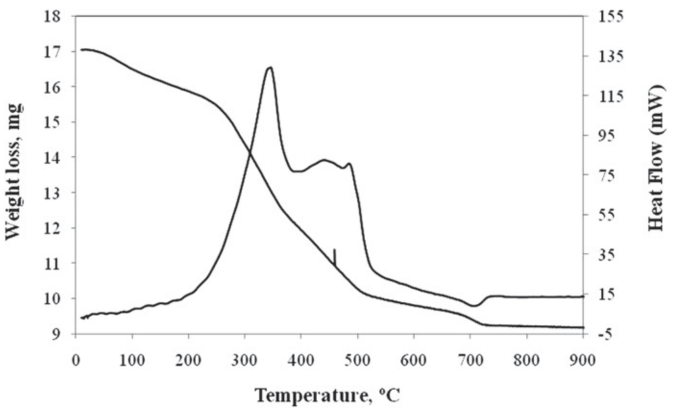

Fig. 2. TGA/DTA curves of sludge.

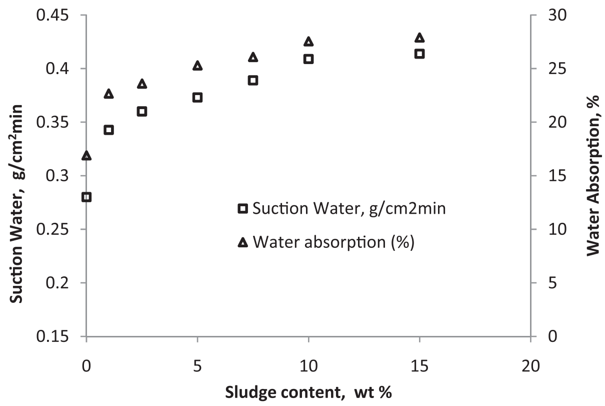

Fig. 3. Effect of sludge content on the water suction and water absorption as a function the amount of sludge addition.

Sludge was previously dried with an electric heater during 48 h at 90 °C in order to remove moisture.

2.2. Sample preparation

The sludge content in the clay mixture varied from 1% to 15% of dry weight. In order to obtain comparative results, series of ten samples were prepared for the tests. Mixtures were moulded under 10 MPa of pressure using a uniaxial laboratory-type pressing Mega KCK-30 A. The necessary amount of water (10 wt%) for mixing was added to obtain adequate plasticity and absence of defects at the compression stage. Solid bricks with 30 × 10 mm cross sections and a length of 60 mm were obtained. The shaped samples were dried during 48 h at 110 °C in an oven to reduce moisture content. Dried samples were fired in a laboratory-type electrically heated furnace at a rate of 10 °C/min up to 950 °C for 24 h. The samples conformed are appointed like *C* for the brick of clay and *M* for the mixtures of clay and sludge in different proportions from 1 to 15 wt% (samples, M1, M2.5, M5, M7.5, M10 and M15).

2.3. Raw materials and conformed materials characterization

Qualitative determination of major crystalline mineralogical phases present in the clay and sludge was achieved by using a Philips X'Pert Pro automatic diffractometer equipped with a Ge (111) primary monochromator, which provides a strictly monochromatic radiation CuK1. Chemical composition was determined by X-ray fluorescence (XRF) in a Philips Magix Pro (PW-2440) equipment. The metal content of the sludge was determined by ICP-ms Agilent series 7500 with internal patron. Thermal behaviour was determined by thermogravimetric analysis (TGA) and differential thermal analysis (DTA). These analyses were conducted simultaneously using a Mettler Toledo 851^e equipment under oxygen. The content of carbonates in clay and sludge were determined by calcimetry.

Weight loss on ignition was obtained by measuring the weight after drying stage at 110 °C and after firing stage at 950 °C. Linear shrinkage was obtained by measuring the length of the samples before and after the firing stage, using a calliper with a precision of ±0.01° mm. Bulk density was obtained according to standard procedure (UNE-EN-772-13). Water absorption was determined according to standard procedure (UNE 67-027). Test on determining water suction was implemented according to standard procedure (UNE 67-031). Compressive strength was measured for fired samples according to standard procedure (UNE 67-026) in a Suzpecar CME 200 SDC laboratory Testing Machine. The effect of freezing was measured according to standards procedure (UNE 67-028). In order to determine the risk of leaching in samples, a lixiviation test was performed following procedure (DIN 38414-S2, 1995). Samples' microstructures were observed with scanning electron micrographs (SEM) by using a JEOL model JSM-5800.

3. Results and discussion

The mineralogical composition of raw material and sludge used to design body compositions has been determined by X-rays diffraction (XRD). The crystalline components of the clay are: quartz (SiO_2), kaolinite (aluminium silicate), calcite ($CaCO_3$), illite (potassium aluminium silicate), montmorillonite (aluminium magnesium

Table 2
Technological properties of construction bricks made from sludge.

Sample	Sludge content (wt%)	Weight loss on ignition (%)	Linear shrinkage (%)	Bulk density (g/cm^3)
Clay	0.0	16.90 ± 0.13	0.33 ± 0.08	1.615 ± 0.090
M1	1.0	14.75 ± 0.34	0.48 ± 0.04	1.600 ± 0.121
M2.5	2.5	15.11 ± 0.75	0.47 ± 0.09	1.546 ± 0.854
M5	5.0	15.68 ± 0.64	1.40 ± 0.07	1.423 ± 0.098
M7.5	7.5	16.25 ± 0.42	0.90 ± 0.04	1.410 ± 0.096
M10	10.0	17.55 ± 1.12	1.88 ± 0.06	1.409 ± 0.103
M15	15.0	18.06 ± 0.96	1.16 ± 0.05	1.340 ± 0.965

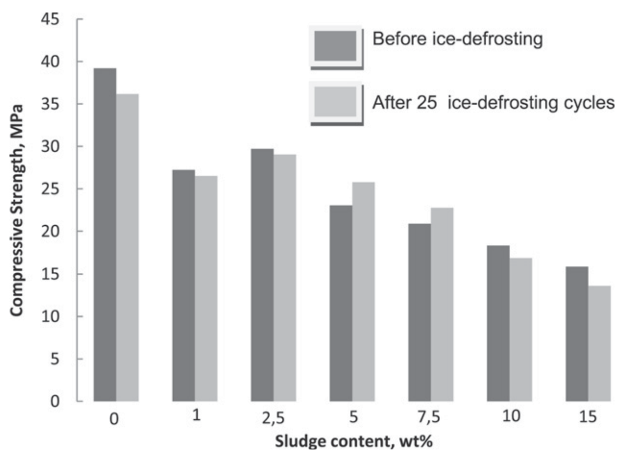

Fig. 4. Compressive strength of the clay as a function the amount of sludge addition before and after freezing.

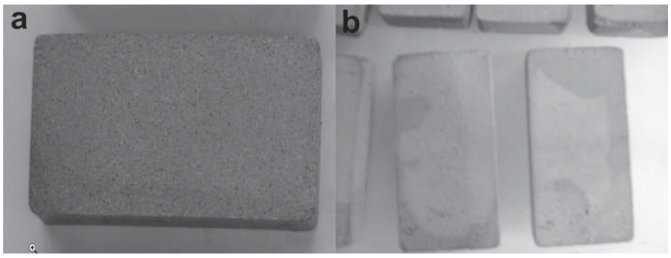

Fig. 5. Effect of freezing on the compressive strength. (a) before ice-defrosting; (b) after 25 ice-defrosting cycles.

silicate) and chlorite (Fig. 1a). From the corresponding XRD graphs of the sludge (Fig. 1b), the presence of quartz (SiO_2), calcite ($CaCO_3$), and phyllosilicates as mica (K–Mg–Fe–Al–Si–O–H_2O) and dolomite ($CaMg(CO_3)_2$) can be concluded as main mineral phases. XRF analysis indicates that after the firing process, the sludge ash is basically composed of high amounts of silica, alumina and iron oxide, mainly due to the presence of phyllosilicates and calcium oxide from the decomposition to calcite phase (Table 1). The inorganic fraction of the sludge showed high contents of iron and aluminium partly due to the flocculating reagent added during the wastewater processing. The present of calcium, magnesium and sodium was likely caused by the sediments from the urban sewage system (Table 1). The sludge contained materials similar to the clays which would indicate the possibility to replace one raw material with another.

Thermal behaviour of sludge was analysed by thermogravimetric (TGA) and thermodifferential (DTA) analysis, as shown in Fig. 2. The heated from room temperature to 200 °C with a weight loss of 4.8%, produces the release of physically adsorbed water, an endothermic peak has been found at about 100 °C. The decomposition of organic matter occurs between 200 and 550 °C, with a weight loss of 36.5%. The first exothermic peak between 200 and 400 °C was associated with biodegradable materials, undigested organics and dead bacteria, together with the emissions of semi-volatile compounds (Calvo et al., 2004; Conesa, 2000; Font et al., 2001). The second exothermic peak between 400 and 550 °C was associated to the oxidation of other oxidizable material in the sample. Finally, the last endothermic peak, at 700 °C could be due to the decomposition of calcium carbonate (calcite) with release of CO_2.

After assessing and discussing analytical data, some experimental tests have been carried out with mixtures of different proportions of sludge in order to study their technological properties. The quality of the bricks can be further guaranteed according to the degree of firing linear shrinkage. Good quality bricks usually show shrinkage below 8%. Shrinkage percentage grew up with increasing sludge additions (Table 2). On the contrary, samples with 7.5 and 15 wt% showed lower linear shrinkage. Since levels of swellability and organic content of sludge are higher than those of clay, the addition of sludge to the mixture should enlarge the degree of firing linear shrinkage. As a result, the quality of the bricks is downgraded. The no clear tendency in the results appeared in others works, which confirmed that there was no relation between this technological property and the percentage of applied sludge (Jordán et al., 2005; Montero et al., 2009).

When sintering the brick, a loss of variable weight, according to the percentage of sludge added, is observed, probably due to the combustion of organic matter as well as to the loss of humidity. In the case of a normal clay brick the loss of weight after firing at 950 °C is 16.9%, which could be mainly attributed to the calcium carbonate (11.8 wt%) and organic matter content in clay (Table 2). It assumed that, as temperature was increased, carbonate in clay decomposed into CO_2. The results were superior to those reported in other studies (Tay and Show, 1992; Chiou et al., 2006), this is due to high carbonate content of the clay used. This calcareous clay contributed to a greater lightening of the materials obtained. However, upon the addition of sludge to the mixture, the loss of weight increased, but only mixtures containing higher sludge additions showed higher weight loss on ignition than clay. Weight loss should increase due to the high contribution of organic mass from sludge. These results could indicate, furthermore, the brick weight loss on ignition also depended on the inorganic substances in both clay and sludge being burnt off during the firing process.

The bulk density of the clay bricks was 1.615 g/cm^3. The addition of increasing amounts of sludge causes a decrease in bulk density (Table 2). The main reason for this tendency is the combustion of the matter organic of residue during the sintering period, which forms open and closed pores in the body clay. According to these observations, high sludge waste addition to the clay body improved the thermal properties of the material but also had negative effects on the mechanical resistance of materials and could give rise to products with low compressive strength.

The experimental data of water suction of green and firing samples show an increase in water suction after sintering at 950 °C. Results were expected and, consequently, when the pieces were acted upon by high temperatures, the superficial interconnected porosity was developed. A light increase of this parameter could be observed with increasing sludge additions (Fig. 3). Water suction was 0.34 g/cm^2 min for the test tubes with a 1 wt% of sludge and 0.41 g/cm^2 min when the content of sludge increased up to 15 wt%. Suction water affects quality and durability of the final material. High contents of sludge could cause defects in bricks, a clear tendency on water suction and, therefore, lower durability.

The sludge addition caused an increment in water absorption of the clay body (Fig. 3). For example, increases in the amount of sludge varied from 1 to 15 wt%, the absorption water changed from 22.67% to 27.90%. The results were similar to those obtained by others authors (Montero et al., 2009; Monteiro et al., 2008) using

Table 3
Results lixiviation test.

Sample element ppb	M1	M2.5	M5	M7.5	M10	M15	ppb max. Test TCLP
Cr	924.10 ± 2.34	882.9 ± 2.13	706.6 ± 2.61	809.5 ± 2.44	478.8 ± 2.53	341.3 ± 2.22	5000
Ni	2.17 ± 0.32	10.74 ± 1.11	4.03 ± 0.68	2.11 ± 0.65	25 ± 2.44	1.65 ± 0.34	2000–400
Cu	17.67 ± 1.21	29.75 ± 1.54	18.8 ± 1.12	23.8 ± 1.11	39.27 ± 3.07	29.32 ± 1.33	10,000–2000
Zn	1.19 ± 0.08	5.05 ± 0.64	86.15 ± 2.03	1.17 ± 0.55	84.59 ± 2.22	15.92 ± 1.43	10,000–2000
As	26.22 ± 1.08	33.48 ± 1.81	78.62 ± 2.01	32.23 ± 1.98	137.4 ± 3.76	5.47 ± 0.71	1000–200
Se	1.72 ± 0.06	9.47 ± 0.88	1.46 ± 0.77	1.52 ± 0.62	14.44 ± 2.10	4.71 ± 0.99	1000
Ag	0.81 ± 0.02	5.22 ± 0.93	0.54 ± 0.09	0.66 ± 0.07	4.84 ± 0.69	2.4 ± 0.11	5000
Cd	0.53 ± 0.01	6.42 ± 0.66	0.07 ± 0.02	0.08 ± 0.02	6.78 ± 0.99	0.15 ± 0.03	500–100
Pb	1.87 ± 0.23	11.16 ± 0.94	0.66 ± 0.01	0.33 ± 0.01	13.12 ± 1.01	0.66 ± 0.06	2000–400

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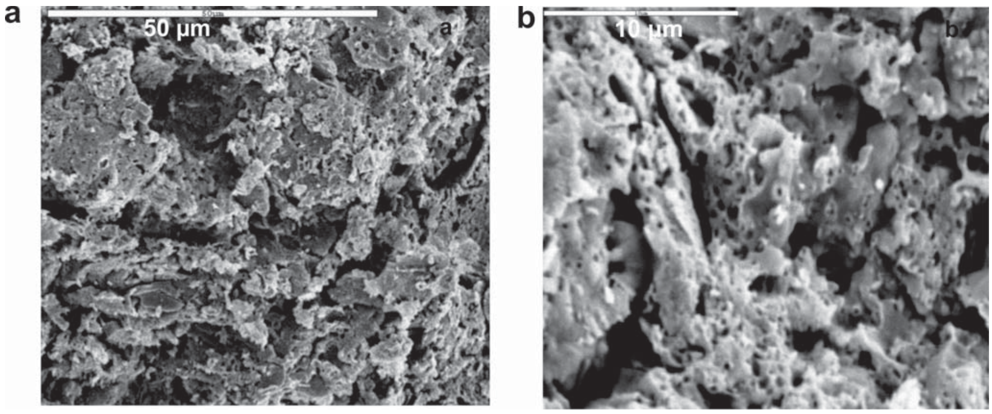

Fig. 6. (a) SEM micrographs of the clay and (b) clay containing 5 wt% of sludge.

the same sludge proportions. They observed that the incorporation of sludge was limited due to the increase of the water absorption and decrease of the mechanical strength. The results of suction and absorption water indicate that an increment in the sludge content produced a more porous material with lower mechanical resistance. Therefore, it has also been noticed that sludge contents above 5 wt% in the clay body could produce some negative effects on technological properties of the bricks obtained.

The sludge content had a significant influence on the mechanical strength of the compositions (Fig. 4). Sludge residue addition reduced the compressive strength of clay samples. In all the samples studied, compressive strength of clay (58.2 MPa) is decreased with sludge additions. Samples with 2.5 wt% residue addition had the highest compressive strength. However, incorporating sludge additions of up to 5 wt% decreased the compressive strength of pure clay by 40%. The overall decrease in the compressive strength over sludge content addition could be attributed to the bulk density reduction (Table 2) along with the increase in water absorption, both connected with the presence of a high porosity level in bodies. Such high porosity level was produced by the combustion of waste organic matter, what is known to have a marked detrimental influence on mechanical strength of ceramic (Carty and Senapati, 1998). The results of samples with 5 wt% of residue were better than those obtained using other wastewater sludge (Tay and Show, 1992; Weng et al., 2003) with values of compressive strength lower than 30 MPa for the same sludge content.

The freezing resistance is defined by the decrease of samples compressive strength before and after undergoing 25 ice-defrosting cycles. After the 25 ice-defrosting cycles, we proceeded to the eyepiece inspection of the probes. During the test no cleavage, fissure or scalping were encountered in samples with sludge content lower than 15 wt%. Superficial deterioration may be clearly observed in the case of samples with higher sludge content (Fig. 5). Then, compression comparative test on samples was conducted again. Samples with sludge content of up to 10 wt% showed a slight decrease in the compression resistance. The highest decrease was observed in sample with 15 wt% of sludge content, so it shows scalping after 25 ice-defrosting cycles (Fig. 4).

Results obtained from the lixiviation test indicated that all the samples within the range of compositions of sludge subject to study would not classify as dangerous and met the current legislation. None of the concentrations of the specific elements override the ones indicated by the standard (Table 3). These results indicated that the degree of metal immovilization achieved by the brick manufacturing processes was high. Therefore, no environmental problems due to heavy metals are expected for the unrestricted use of the sewage-clay bricks.

Morphological study of samples containing clay, as well as that of sample containing 5 wt% of sludge was obtained by means of SEM (Fig. 6). The micrograph of 5 wt% sludge sample showed clearly that waste agglomerates were distributed in the microstructure of clay. Also, the presence of sludge increased clay porosity according to data regarding bulk density (Table 2) and water absorption (Fig. 3).

4. Conclusions

This work showed that the ceramic sector could be a receptor in different types of wastes as sludge wastewater treatment plant. The proportion of sludge in the mixture has been proven as a key factor in altering bricks quality, affecting technological properties of the final ceramics products. Increasing proportions of sludge have been shown to clearly increase water suction and water absorption. On the other hand, sludge addition entails a reduction in compressive strength due to increased porosity caused by the decrease of bulk density. For this reason, selecting the appropriate percentage of sludge to be added to the clay body will be controlled. Therefore, this type of waste should be incorporated in low percentages in order to produce good quality ceramic bricks. In all, the recommended proportion of sludge in brick is up to 5 wt%.

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