Valorization of ceramic sludge waste as alternative flux: A way to clean production in the sanitary ware industry

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ABSTRACT

The ceramic industry consumes a large amount of raw materials, much of which consists of fluxes. In view of the growing demand for infrastructure and sanitation, the waste valorization is a viable alternative for recycling materials, mitigating the extraction of natural resources. In this context, the present work studied the feasibility of replacing feldspathic flux by ceramic sludge waste (CS) in the sanitary ware production. The waste was characterized by particle size distribution, chemical composition, and mineralogical phases. The replacement ratios (by mass) were set in a wide range of 5, 10, 25, 50, 75, and 100%. The ceramic mass in fluid aspect was evaluated by means of density, viscosity, thixotropic index, setting rate and drying time. The hardened state was investigated through shrinkage, pyroplastic deformation, compressive and flexural strengths, water absorption and microstructure. The results showed that CS can be a stronger flux than granite. One of the proportions with total replacement (B-100) showed promising results, being the best option in terms of rheological behavior and physical-mechanical properties. An important finding concerns water absorption, which decreases with increasing the CS content. It is suggested that CS valorization on industrial scale may be sufficient to eliminate the entire volume of waste. This approach represents an eco-friendly solution, promotes the closing of waste loops and can be a way to cleaner production in the sanitary ware industry.

1. Introduction

As new and more restrictive environmental practices are implemented to achieve sustainable development in industrialized societies, there is increasing demand for alternative and efficient methods to replace waste disposal in landfills. The recycling of wastes has been studied by researchers aiming to apply it in industries as a method to reduce costs, improve relationships with staff and society, and mainly, to ensure the existence of natural resources for future generations (Singh et al., 2017). Also, industrial sustainability presupposes holistic changes to achieve long-term eco-efficiency, which are closely related to waste valorization and closing material loops (Bocken et al., 2014).

In terms of the production processes sustainability, there are some approaches to promote more efficient industrial systems. Among those most accepted today is the circular economy, which has been successfully practiced in the industrial scenario (Walker et al., 2021), includes waste management for resource recovery (Ghaffar et al., 2020) and can

be defined as the integration between reduction, reuse and recycling (Kirchherr et al., 2017). This concept is based on two main approaches. The first refers to the proposition of a circular model, in which waste is considered a resource that can be valued and reinserted in the production chain, in contrast to the linear "take-make-dispose" model; the second refers to the strategies of narrowing, slowing down and closing the material and energy loops, which correspond to reducing the use of raw materials, prolonging the product's useful life, and promoting the recycling of waste (Baldassarre et al., 2019). In view of its characteristics, the circular economy can, synergistically, stimulate economic development and restrict the use of resources, which reconciles economic and environmental objectives (Saavedra et al., 2018). This entire theoretical-practical framework is in line with the goals for sustainable development proposed by the United Nations in Agenda 2030) (United Nations, 2015), especially in terms of responsible consumption and production (goal 12), which are essential to minimize waste generation and avoid resource depletion.

Materials recovery is an important tool to balance the raw materials and energy flows. With a focus on industrial processes, the closing of loops becomes particularly relevant (Chojnacka et al., 2021). As mentioned, waste can become useable resources, after proper valorization. Much attention has been paid to recycling, not only because it constitutes an alternative route to waste disposal, but, notably, because it promotes the transformation of unusable materials into raw materials, mitigating impacts on the environment (Zanelli et al., 2021). The possibilities for valuing waste are many, covering a wide variety of topics, for example, recovery of copper from ore processing wastes (Perez et al., 2020), synthesis of nitrogen-doped carbon from biomass (Paul and Kurian, 2021), sustainable aviation fuel using sewage sludge (Farooq et al., 2020) and adsorbent materials for water decontamination from industrial waste (Janbooranapinij et al., 2021). In terms of ceramic materials, the recycling of glass waste (Bagheri and Moukannaa, 2021) and agro-industrial waste (Salleh et al., 2021) to produce alternative cements and ceramic products can be mentioned. In an even narrower focus is the sanitary ware industry, which can have its production systems optimized (Cuviella-Suárez et al., 2018, 2019).

The production of sanitary ware through slip casting generates three types of wastes: ceramic sludge, plaster molds and ceramic shards. The first is a mix of effluents from mold-washing and glazing, along with burrs from the finishing stage. The next is the result of plaster molds being discarded after losing their water-absorption capacity. Finally, fired sanitary ware that has been discarded by the quality-control sector constitutes the third type (Cabral et al., 2010). The present contribution describes an attempt to recycle ceramic sludge, a waste generated daily in sanitary ware industries. One challenge to achieve this goal is that, in general, the composition of this type of ceramic waste differs from that of the original ceramic slips used for casting; these differences are related to the fact that this type of waste contains materials from different stages throughout the production line (Almeida et al., 2016).

Usually, all solid raw materials come from mining sources that are consumed in the production of ceramic ware, which consist of water-based suspensions of clays, phyllite and granite; the latter is employed as a source of alkaline feldspar. High-feldspar rocks, such as granites and pegmatites, serve as traditional fluxing agents for a variety of porcelain bodies (Dondi, 2018). Other types of fluxes can also be used (*e.g.* quartz sand and bone ash), however, their composition can affect the sintering behavior (Sokolář et al., 2017). The primary function of fluxes during the sintering of white ware is to form a liquid phase, which promotes densification by filling pores, further increasing strength (Kyonka and Cook, 1954). In general, fluxes account for approximately 20% of the dry weight of white wares and are an essential component of sanitary ware production. Although many companies in this sector keep their production numbers secret, it was estimated that in 2017 a total of 19,434 t of sanitary ware were exported from Brazil, while 6,745 t were imported by the same country (MME - Ministério de Minas Energia, 2018), which demands a large, and usually demographic-dependent, supply of fluxes.

In terms of a global vision, Dondi, (2018) compiled several information about feldspathic and alternative fluxes applied to ceramic products. Initially, an accumulated production of 575 Mt was observed between 1971 and 2016 (45 y lapse), with a large portion of these materials being used to feed the ceramic and glass industries. For decade of 2007–2016, the production resulted in 255 Mt, that is, approximately 44% of the amount in the whole period of analysis, indicating expressive growth. Fig. 1 identifies the 10 countries that produced the most mineral fluxes (feldispathic and alternative) between 2007 and 2016. Brazil was the fourth largest producer, which represented 5.3% of world production, showing its relevance. Even if Earth's crust is mainly composed of feldspars, the rising demand for fluxes, and given their crucial role in this industry, ought to create concerns about their availability.

There are several investigations in the scientific literature that address cleaner production, sustainability, and environmental concerns in the sanitary ware sector. Table 1 shows a summary of the main
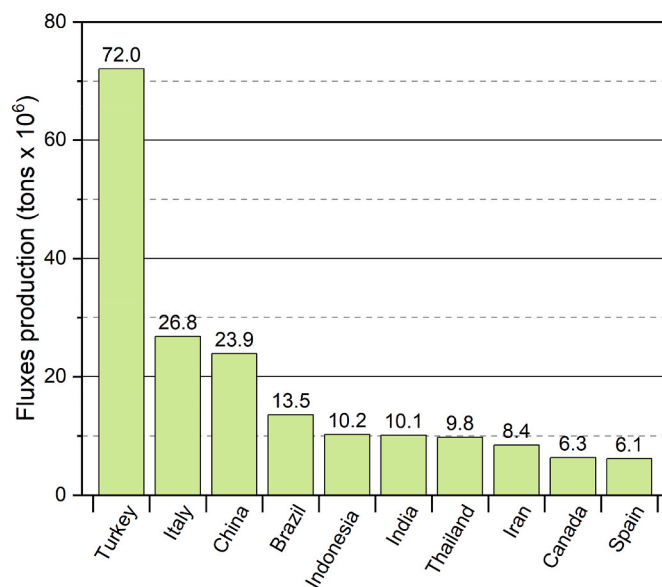

Fig. 1. Main producing countries of flux. Note: cumulative amount for the period 2006–2017 and source data from Dondi (2018).

studies, along with approaches and some relevant findings.

Some studies have associated the search for alternative fluxes with the practice of recycling wastes. Waste-glass has long been studied as a substitute for conventional fluxes, and some works were successful in applying small amounts of waste glass to improve liquid phase formation. Rambaldi et al. (2007) incorporated soda-lime glass wastes into whiteware bodies. Waste content (6% by weight) was defined by the unity molecular formula (UMF) and the molar ratios of the flux–alumina–silica system $[(R_2O + RO) – Al_2O_3 – SiO_2]$, which maintained the glassy phase composition constant. The bodies with replacement have characteristics and properties similar to the standard composition. Pyroplastic deformation was significantly lower with waste incorporation. Kalirajan et al. (2016) investigated the crystallization kinetics and microstructure of glass-ceramic glaze with addition of glass residues. The proportions were 5%, 10% and 15% (by weight) and two types of waste glass were used (light tube and borosilicate). The main phase was zircon and the average crystallite size decreased in the glaze with addition. The crystallization activation energy increased, and surface crystallization/diffusion growth mechanisms were suggested to be dominant. Higher opacity and whiteness were obtained for glaze with 15% of wastes. Martini et al. (2017) produced a sanitary ware body with waste glass and recycled materials, called *Sanitser*. The waste mixture represented approximately 43% of the ceramic mass. The oxide chemical composition and physical properties of *Sanister* slip were equivalent to industrial slip, and the firing temperature of the ceramic bodies was reduced from 1250 to 1170 °C.

Other researchers have investigated "*less common*" wastes. Nandi et al. (2015) evaluated the manufacture of tiles using ceramic sludge from a wastewater treatment plant and recycled glass. The main objective was to immobilize the wastes in a glassy matrix. A wide range of formulations were produced, with 20–80% ceramic sludge and 12–48% recycled glass (both by weight). The results of several tests showed that it is feasible to produce glazed ceramic tiles with waste addition, in line with industry quality standards. Tarhan et al. (2017) investigated the use of sanitary ware waste (SWW) in the production of grazed porcelain tiles. An important aspect concerns the industrial condition in which the study was carried out. The wastes were in different concentrations (5%, 10% and 15%, by weight), replacing pegmatite and feldspar. The main finding was the reduction of expansion coefficients, which indicates dimensional stability. Some proportions showed increased flexural strength and reduced water

Table 1
Summary of main studies that investigated sanitary ware.

Study	Approach	Findings
Rambaldi et al. (2007)	Incorporation of soda-lime glass wastes into whiteware bodies (6% by weight).	Similar characteristics to the standard composition and less pyroplastic deformation.
Halicka et al. (2013)	Concrete with SWW to replace coarse and fine aggregates.	Absence of defects and preservation of shape and cohesion after high temperature (1000 °C)
Medina et al. (2014)	Leaching in concrete with partial replacement of coarse aggregate by SWW.	Leached solution with reduction in the concentration of B, Si, Cl and Mg, and increase in alkalinity (Na and K).
Farinha et al. (2015)	Replacement of fine aggregates by SWW in mortars.	Greater resistance and lower water absorption.
Nandi et al. (2015)	Tile manufacturing from ceramic sludge (20–80%) and recycled glass (12–48%), both by weight.	Ceramic mass showed suitable properties for manufacture of tiles. The formulation with highest ceramic sludge content obtained results comparable to the reference.
Kalirajan et al. (2016)	Addition of waste tubular light glass and borosilicate glass in glass-ceramic glazes (5%, 10% and 15% by weight).	Zircon as main phase, reduction of average crystallite size, increase of crystallization activation energy and higher opacity.
Vieira et al. (2016)	Durability of concrete containing crushed bricks and SWW	High abrasion resistance for mixtures with SWW
Martini et al. (2017)	Glass waste and recycled materials in the formulation of sanitary ware bodies (43% by weight).	Equivalent physical-chemical properties and significantly reduced firing temperature.
Tarhan et al. (2017)	Production of glazed porcelain tiles with replacement of pegmatite and feldspar by SWW.	Reduction of thermal expansion coefficients, which indicates dimensional stability. Greater flexural strength and reduced water absorption.
García-Villén et al. (2018)	Synthesis of zeolites using SWW in hydrothermal condition.	Transformation of quartz and mullite present in the SWW into zeolites
Gungor et al. (2019)	Production of Sanitary ware bodies with galvanizing wastes.	Less deformation, greater whiteness, and reduction in sintering temperature.
Lv et al. (2019)	Life cycle assessment (LCA) and life cycle costing (LCC) in the sanitary ware production.	The drying and firing processes generate greater environmental impact because of the burning of fuels, and material costs are higher in the preparation stage of ceramic bodies.
de Medeiros et al. (2019)	Replacement of feldspar by quartzite in the ceramic mass production.	Promising results for strength, water absorption and firing shrinkage.
Silvestri et al. (2020)	Life cycle assessment (LCA) in the sanitary ware production.	Green-based manufacturing can promote the reduction of environmental impacts by recycling water and using energy-saving technologies.
Muthukumaran et al. (2021)	Use of SWW particles in abrasive jet machines.	The alternative represented an effective and economical way in contrast to commercial abrasives.
Pitarch et al. (2021)	Production of binary binder from Portland cement and SWW.	The binary binder has pozzolanic activity and can contribute to CO_2 reduction.
Reig et al. (2021)	Replacement of Portland cement by SWW (range 0–50%).	There was no significant change in workability, curing time improved and pozzolanic activity was slow.

Note: SWW – Sanitary ware waste.

absorption.

There are also many studies reporting the effects of incorporation SWW in concrete. Halicka et al. (2013) evaluated coarse and fine aggregates from SWW in concrete. After high temperature tests (1000 °C), the concrete with residues showed no defects or cracks, as well as preserved its shape and cohesion. Medina et al. (2014) performed leaching tests and reported that concrete with partial replacement of coarse aggregate by 25% of SWW decreases the concentration of elements B, Si, Cl and Mg in leached water, but increases the alkaline concentration (Na and K). Ogrodnik et al. (2018) reported that concrete with SWW has high abrasion resistance and the application of annealing at 800 °C can improve strength when using calcium aluminate cement. Farinha et al. (2015) added fine aggregates of SWW in cement mortars. The highest content (20%) of addition showed higher strength and lower water absorption. Vieira et al. (2016) evaluated concrete containing crushed bricks and SWW from the perspective of durability. Total (100%) and partial (20% and 50%) replacements of fine aggregates were evaluated. The results indicated that there was an increase in the carbonation depth and greater chloride migration coefficients, caused by the greater demand for water and increased porosity.

Several other works were found in the literature, each one with its specificity, however, all with the common objective of promoting cleaner technologies. García-Villén et al. (2018) reported that it is possible to synthesize zeolites from SWW. After hydrothermal processing under alkaline conditions, the presence of P-zeolite, Na-faujasite, sodalite, analcime, natrolite, and A-zeolite was observed, from the transformation of quartz and mullite present in the SWW. Gungor et al. (2019) reported that sanitary ware bodies with galvanized waste have less deformation and superior whiteness, for sintering at 1200 °C. This finding is relevant because there was a 50 °C reduction in the sintering temperature, which represented 4.57% of energy savings. Lv et al. (2019) performed life cycle assessment (LCA) and life cycle costing (LCC) for the sanitary ware production. The drying and firing processes generate greater environmental impact because of fuel burning, and material costs are higher in the preparation stage of ceramic bodies. The authors suggest the use of green materials to enable environmental and economic sustainability. de Medeiros et al. (2019) reported that quartzite wastes can be added to the ceramic mass to replace feldspar. The 15% replacement content showed promising results for strength, water absorption and firing shrinkage. Silvestri et al. (2020) reported that green-based manufacturing can promote the reduction of environmental impacts by recycling water and using energy-saving technologies. These findings were based on a life cycle assessment that covered manufacturing processes, raw materials, transportation, and energy supply. Muthukumaran et al. (2021) reported that SWW particles can be used as abrasive material in abrasive jet machines. The solution represented an effective and economical way in contrast to commercial abrasives. Pitarch et al. (2021) indicated that replacing Portland cement by ceramic waste (including SWW) in up to 25% has pozzolanic activity, which can contribute to the reduction of CO_2 emissions. Reig et al. (2021) also replaced Portland cement with SWW in the range of 0–50% and observed that there was no significant change in workability, the curing time improved, however, the pozzolanic activity was slow.

For these reasons, there is a worrying scenario regarding waste management in the ceramic industry. Final disposal is commonly carried out in decantation ponds and landfills. This harmful flow to the environment can be changed from the waste valorization and reinsertion of raw materials in the production chain. The present work emerges as an alternative for cleaner production in the sanitary ware sector. The ceramic sludge waste (CS) was characterized in terms of particle size, chemical and mineralogical composition. The rheological behavior of the fluid mass and the physical-mechanical properties of the hardened ceramic were investigated. The feasibility of application was evaluated by the production of hollow ceramic blocks. Additionally, the interrelationship between empirical results and sustainable development was explored.

2. Sanitary ware manufacturing

In the industry where this study was conducted, the manufacturing process of sanitary ware starts by mixing one of the clays and a portion of the phyllite in a mixing-tank with water and deflocculant. The slurry produced is directed to a ball mill alongside the other raw materials and water. After milling, the ceramic slip is passed through a 250 μm sieve; then, the suspension is directed to a stirring tank where the technical laboratory controls the slip properties to ensure the quality requirements. Once analyzed, the slip is used for casting in plaster molds, followed by a slow drying process. In sequence, the dried pieces have their burrs removed and are polished by hand before being guided to glazing cabins. When fully glazed, the pieces are fired in industrial kilns. During this production process, liquid effluents are generated and are all directed to a settling basin, where the ceramic sludge that was formed is retained long enough so its suspended solids can descend to obtain reusable water; however, when the settling basin fills with solids, its efficiency for water clarification diminishes, requiring removal and disposal of the solids in landfills. This waste disposal involves considerable costs and environmental impact and is the reason for developing a project to study the possibility of recycling these solids on site without the need for outside entities. Fig. 2 shows a general flowchart that illustrates the main steps in the manufacturing process and notes where effluents are generated.

After each round of casting, following demolding of the pieces, the plaster molds are washed to remove any remaining ceramic mass. This process generates the effluent identified as mold-washing effluent (WE), which is composed mainly of raw materials that form the slip, and

plaster to a smaller extent. The glazing effluent (GE) is produced at spray booths, as a result of excess glaze that did not adhere to ceramic pieces. This waste-glaze sticks to the walls and floor of glazing booths, from where it is washed to gutters. In addition, the removal of burrs and polishing of dried pieces results in a waste that is also washed to industrial gutters. The composition of this finishing effluent (FE) should be similar to the WE. The merging of these three effluents, after settling, forms the final ceramic sludge (CS), which accounts for approximately 5% of the mass of solid raw materials consumed by the ceramic slip manufacturing. In a previous study by our research group, crushing yard waste (fired ceramic pieces) was proven able to be incorporated in the production of ceramic slips within the same plant (Silva et al., 2019). To pursue the goal of closing the loop on waste generation, the CS is considered as raw material to produce ceramic slips and fired specimens, and their properties and performance in comparison with the standard formulations is assessed in the present study.

3. Materials and methods

In this study, the main objective is to assess the possibility of recycling the ceramic sludge (CS) as an alternative fluxing agent that contains portions of all three types of effluents previously cited. Quantifying the share of each effluent type in the CS proved to be infeasible, as the rate of each process is constantly changing due to 24 h shift work and changes in the manufacturing process to comply with production needs. The initial idea was to collect the effluents at the final chute positioned above the settling basin; however, the uncertainty as to what type of effluent would be flowing out at a specific time and the possible mixing
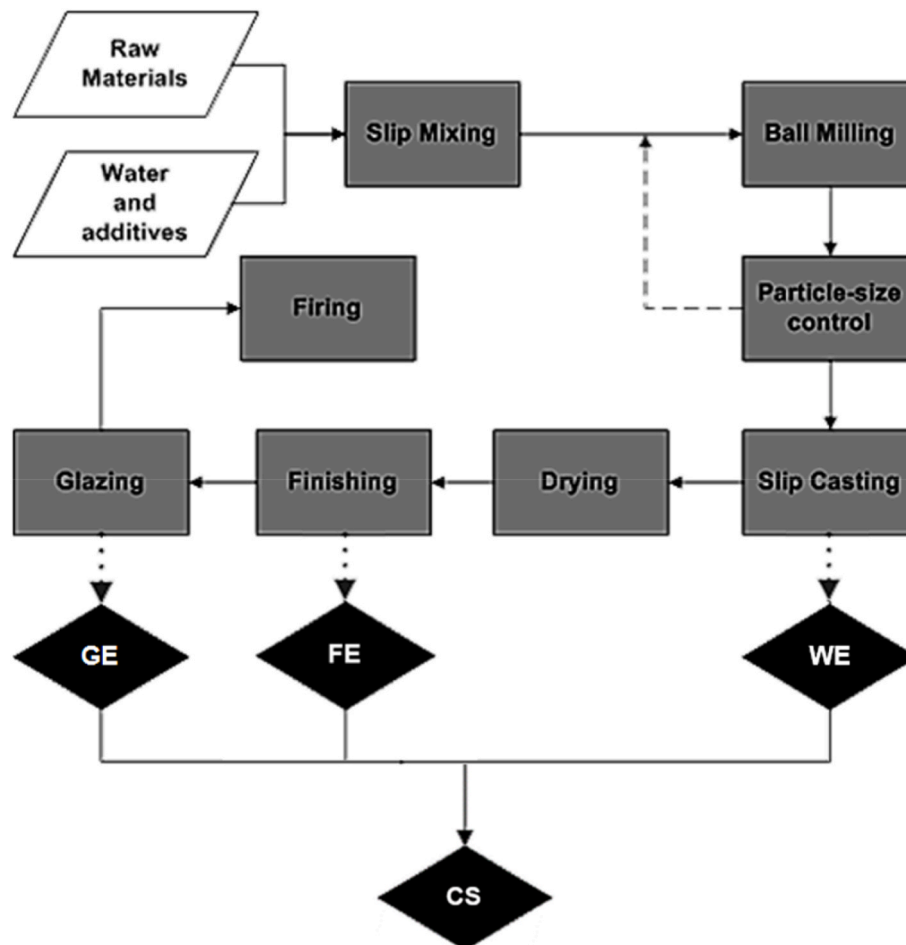

Fig. 2. General steps of sanitary ware manufacturing and effluents generated: WE – mold-washing effluent; FE – finishing effluent; GE – glazing effluent; CS – ceramic sludge.

with another type along the gutters leading to the chute would have resulted in inconsistent samples with combined properties. The method adopted was to sample the settling basin according to ASTM D6907-05 (ASTM, 2016) and ASTM D4687-14 (ASTM, 2014) at different points along the beach formed by decantation.

3.1. Ceramic sludge (CS) characterization

The material collected from the settling basin was oven-dried at 100 °C for 24 h; this step was performed to assist with characterization and handling and to ensure all mixtures would have the same water content. After drying, the CS was manually deagglomerated into a fine white powder using a mortar and pestle. The particle-size distribution was determined by a CILAS-1090 laser granulometer using water as the media, with 15% obscuration and 30 s of ultrasound sonication. The oxide composition was determined for all raw materials by energy dispersive X-ray fluorescence spectroscopy (EDX) in a Shimadzu EDX-720. Crystalline phases were revealed by X-ray diffraction (XRD) with a Shimadzu XRD-7000 using Cu Kα radiation ($\lambda = 1.54056$ Å), 2θ range from 5° to 80°, at 2°/min. Finally, scanning electron microscopy (SEM) images were acquired with a Hitachi TM-3000.

Fig. 3 shows the cumulative particle-size distribution (PSD) curve of the CS. The particle size is generally in the range of 0.1–70 μm, with 90% (D_{90}) under 31.81 μm. The average particle size was 12.21 μm. At this point, the powder had only been dried and deagglomerated. However, it was already within a similar PSD range of materials used to produce porcelain in other works. This was expected since the CS consists of particles that have already undergone industrial milling. Andreola et al. (2010) suggested that raw materials used in industry should have particle size <63 μm and their experimental results showed characteristic diameters of 3.73 μm (D10), 18.51 μm (D50) and 61.10 μm (D90). In a more recent study Andreola et al. (2016) that finer waste glass with particle size <100 μm had higher strength and lower water absorption than particles <400 μm. Kobayashi et al. (2000) used quartz and feldspar powder in different granulometries, 1.2–8 μm and 1.2–4 μm. Tallon et al. (2010) prepared suspensions with different solids contents and nanometric particle sizes (11, 44, 190 and 600 nm). Tarì et al. (1998) used particles within the colloidal range (0.1–10 μm). In another work Tarì et al. (1999) the same interval was reported. Yokota et al. (2006) adopted a mean diameter of 0.53 μm.

The oxide compositions for raw materials and CS are listed in Table 2. As anticipated, the CS composition reflects a mix of raw materials and glaze used by the industry. The main deviations in Table 2 concern calcium, zirconium and zinc oxides. Because of the EDX limitations, one of the identified components was calcium oxide; however, the XRD analysis in Fig. 4 presents the calcium phase as Bassanite, a

calcium sulfate hemihydrate (Hand, 1994) commonly found in plaster molds, and in this case, derived from the degradation of plaster molds throughout slip casting and mold-washing (Morín et al., 2014). It might be important to note that after casting, the calcium phase present in the plaster is most likely gypsum, but since the sludge was dried prior to the XRD analysis, the predominant phase in the sample was the original Bassanite from the molds (Geraldo et al., 2017). In addition, the higher contents of zirconium and zinc oxides come from the glazing effluent since it is very common to use these oxides in ceramic glazes as opacifiers, as well as for thermal expansion. Kalirajan et al. (2016) reported that zinc oxide and zirconium silicate are used as opacifiers and added 1.08% and 9.71% in the experimental ceramic mixture. Özer et al. (2016) evaluated the industrial zinc oxide production for use in ceramic glazes. Other phases identified were quartz, illite and kaolinite, minerals commonly found within the rocks and clays that serve as raw materials for this industry.

After initial characterization, the CS was submitted to a fusion cone test, alongside the raw materials used by the industry. For this assessment, the materials were manually shaped into cones, which were then fired in an industrial kiln under the same firing curve. The goal of this test is to evaluate which of two distinct behaviors the ceramic material presents when fired. In the first behavior, typical of flux materials, the particles fuse together and form a compact structure with a glassy surface. In the second, seen in refractory materials, the particles do not fuse, and the cone either crumbles apart or presents a sandy texture (Bragança et al., 2010). The cone for CS was compared to the ones obtained for each raw material, and it was determined to be compact with a glassy finish, a behavior of flux material, and a decisive factor when determining that CS should replace granite.

Another important parameter for this study was to determine how the incorporation of CS in ceramic pieces could alter casting and firing properties. To assess this, granite was replaced with CS in six proportions: 5, 10, 25, 50, 75 and 100%. Table 3 shows standard ceramic slip formulas employed by the industry; in the code, the letter defines the formulation group, while the number describes the substitution degree. Formulas with the number zero correspond to the standard slips used in the industry (no CS), and their properties serve as comparison points for the other mixes.

3.2. Slip characterization

The list of all prepared ceramic slips is A-0, A-5, A-25, A-50, A-75, A-100 and their respective numbers for group B. The only parameter changed between different formulas within the same group was the granite content, which was replaced by CS. This was important to ensure that variations in properties would reflect CS incorporation and its interactions with the other raw materials. In regard to manufacturing, all ceramic slips were prepared in the industry's laboratory, simulating the industrial process. The mixture of raw materials consisting of clay-I, clay-II, phyllite and granite/CS, together with water, was directed to a ceramic ball mill operating at 70 rpm, where the mix was milled for 1.5 h. The slip produced was passed through a 250 μm sieve, resulting in the final ceramic slips that were used for casting of test specimens in plaster molds.

Prior to casting, the slips were characterized according to parameters adopted by the industry's quality control, which involved analysis of pH, density, viscosity, thixotropic behavior, drying time and setting rate. The pH analysis was performed on-site using a Hanna HI2002-01 pH-meter. The density was verified by weighing 100 ml of slip in a volumetric flask. The viscosity and thixotropic behavior were assessed using a Brookfield RV-series dial-reading viscometer equipped with n° 2 spindle. This viscometer has a conversion table, which is used to obtain the viscosity in centipoise (cP) by multiplying it by the value shown in the dial reading. Two different measurements were performed for viscosity, the first one with a higher stirring rate (50 rpm), then 5 min of resting followed by another reading at a lower stirring rate (2.5 rpm). In
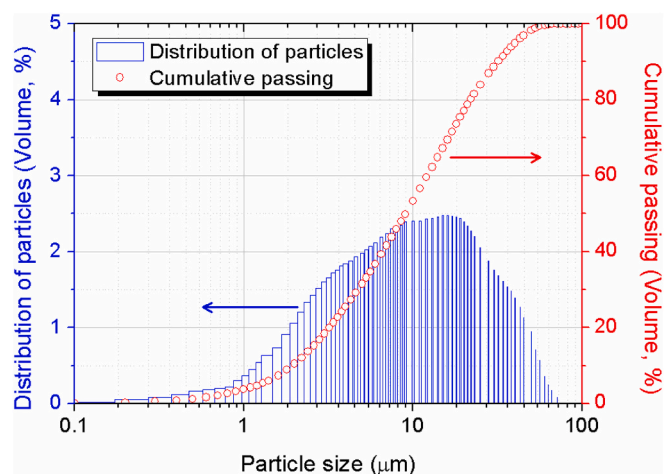

Fig. 3. Particle size distribution curve with histogram for CS.

Table 2
EDX analysis of the CS and raw materials for oxide composition (in wt. %).

Material	SiO_2	Al_2O_3	CaO	ZrO_2	K_2O	ZnO	Fe_2O_3	TiO_2	BaO	Cr_2O_3	Others
	%	%	%	%	%	%	%	%	%	%	%
Clay-I	50.80	31.4	–	0.33	4.56	0.05	10.22	1.95	0.50	–	0.25
Clay-II	63.36	30.45	0.15	0.44	0.96	–	2.23	2.35	–	–	0.06
Phyllite	56.73	24.34	–	0.23	8.56	–	5.46	3.22	–	–	1.46
Granite	71.40	13.39	1.58	0.41	9.32	0.01	1.88	–	–	–	2.01
CS	52.97	13.04	10.92	8.72	4.99	4.01	1.56	0.58	0.26	0.06	2.89
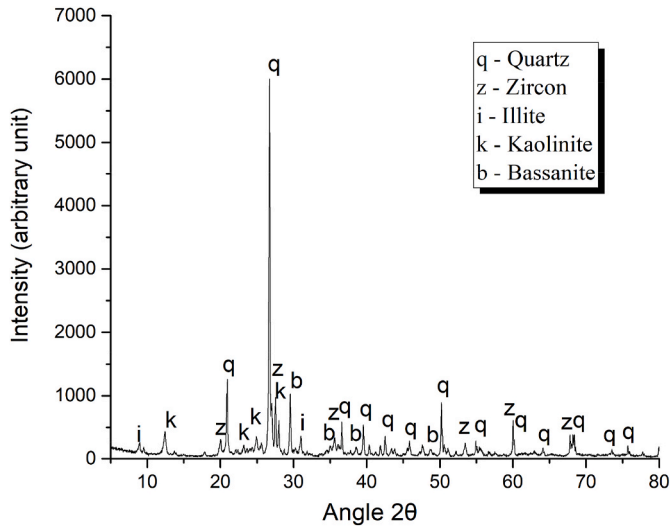
Fig. 4. X-ray diffraction spectra of the dried ceramic sludge.

Table 3
Formulation of standard ceramic slips.

Material	Slip A-0	Slip B-0
Clay-I	8.5%	8.5%
Clay-II	5.0%	8.0%
Phyllite	64.0%	64.0%
Granite	22.5%	19.5%
Water/Solid ratio	1/4	1/4

this case, with the spindle nº 2, and stirring rates of 50 and 2.5 rpm the conversion factors were 8 and 160. The ratio between these two viscosity measurements is known as the thixotropic index, which determines a fluid's behavior as pseudoplastic (index ≥1) or dilatant (index <1). King (2002) reported that thixotropic properties of ceramic slip can be evaluated from viscosity measurements, and Chhabra and Richardson (2011) showed the mathematical models related to the index, based on the shear-thinning and shear-thickening behaviors of non-Newtonian fluids. In addition, tests of drying time and setting rate were performed; the first one assessed the time required for a plaster mold to drain water from the slip until the fluid lost its pearl shine, gradually turning it into a matte finish, while the setting rate test evaluated the speed of slip deposition on the surface of a porous plaster cast. Both tests were performed using a bowl-shaped plaster cast, with an internal surface area of 509 cm^2 and 1527 cm^3 of volume, which was filled with the ceramic slips and left to settle for 2 min before draining. After evaluating the drying time, the dry solid mass was determined by weighing. Then, the setting rate was calculated by Equation (1):

$$SR = \frac{M_{ds}}{S \times DT} \left[g/\left(cm^2 \times \min\right) \right] \tag{1}$$

where, SR is the setting rate, M_{ds} is the dry solid mass, S is the internal

surface area of the mold and DT is the drying time. This approach removes the mold dimension effects and characterizes the behavior of material more reliably.

The pH and density tests were performed in duplicate. For the viscosity test, one measurement was performed for each speed (2.5 and 50 rpm). Drying time and setting rate were performed only once.

3.3. Test specimens

Once the slip properties were determined, the mixes were used to cast three types of test specimens in plaster molds. The first type consisted of six cylinders for each slip formula; these specimens were 200 mm long and 15 mm in diameter after casting. For each formula, three of these specimens were fired in an industrial kiln, while the others were only dried; they were all subjected to three-point flexural strength tests in a DL-30000 universal testing machine, with a loading rate of 50 N/s. For the second type, three specimens were casted for each formula; these specimens were tabular-shaped, with dimensions of 300 × 40 × 12 mm after casting, the lengths measured with a digital caliper, and they are: initial length (Li), dried length (Ld) and fired length (Lf), which were used to determine linear shrinkages (DS, FS and TS) according to Equations (2)–(4).

$$DS = \frac{Li - Ld}{Li} \times 100(\%) \tag{2}$$

$$FS = \frac{Ld - Lf}{Ld} \times 100(\%) \tag{3}$$

$$TS = \frac{Li - Lf}{Li} \times 100(\%) \tag{4}$$

The final specimen type had the same measurements for length and width (300 × 40 mm), with doubled height (24 mm) for the initial 100 mm in length (head). For this type, five specimens were casted for each formula, and they were used to assess two properties: pyroplastic deformation and water absorption. Pyroplastic deformation (PD) is defined as the distance between the fired tail-end and its original position, and it is directly associated with flux content (Bernardin et al., 2006). These specimens were positioned in the industrial kiln with their heads between two refractory bricks, while their tails remained suspended; this allowed them to bend as the liquid phase was formed during firing. Subsequently, a 100-mm section was cut from each specimen's tail to perform water absorption tests, according to ASTM C373-18 (ASTM, 2018).

4. Results and discussion

4.1. Ceramic slips

The influence of CS addition on pH was assessed for each slip. This analysis helps comprehend variations in viscosity for all proposed slips. Table 4 shows a higher pH for the CS compared to the standard slips used by the industry. Because of the higher pH of the CS, Fig. 5 shows that slips prepared with the CS became more alkaline as the CS further replaced granite.

To avoid the corrosion of plaster molds, ceramic slips should be

Table 4
Comparison of the pH values for CS and standard ceramic slips.

| Sample	pH
CS	9.25 ± 0.04
Slip A-0	8.12 ± 0.03
Slip B-0	8.81 ± 0.05

castable at intermediate pH values (Hauth, 1949, 1950). However, the slips should be stable over the long term, which means industries usually work at high or low pH values to ensure the particles have a high surface potential, producing repulsive forces between them (Bellotto et al., 2018). The interactions of the CS with other raw materials, in terms of the pH and its implications on the zeta potential (Anderson and Murray, 1959), are yet unknown. Nevertheless, the results from the rheology analysis shown in Table 5 demonstrate how an increase in the CS affects each of the slips' parameters. The viscosity values shown in Table 5 were previously converted to cP from the dial readings using the viscometer's specific factors for the correspondent spindle nº and stirring rates, while the thixotropic index corresponds to the ratio between the slip's viscosity at 2.5 rpm and at 50 rpm.

In both groups of slips, replacing granite with the CS did not significantly alter the density since all of the slips were within the range adopted by the industry and by other works. Pishch et al. (2006) measured density at 1.83 g/cm^3, Sal'Nik et al. (2013) evaluated two slip mixes at 1.80 g/cm^3 and 1.77 g/cm^3, Martini et al. (2017) reported 1.8 g/cm^3 and Ochoa et al. (2017) 1.82 g/cm^3. By contrast, the viscosity and thixotropic results showed a clear correlation with the amount of CS, with a general increase in viscosity and thixotropy as more CS was added to the systems. The CS contains substances from the mold washing

effluent (WE), including plaster mold material or Bassanite as identified by the XRD analysis. During the slip casting process, this mineral can be responsible for absorbing water from the slips in the reversible aqueous transformation of Bassanite into Gypsum (Brandt and Bosbach, 2001). This process could also be a mechanism to increase the setting rate, as the water retained by the Bassanite in the CS would still be in the slip, causing a reduction in fluidity while increasing the humidity of the mass settled in the mold. Another possible (or complementary) explanation is linked to the surface interactions between the minerals in the slips, which could be affected by the increase in pH (Reed, 1995) observed in Fig. 5 and would explain the minimal differences in drying times since it was expected that slips with a higher Bassanite content should have dried faster (Scherer, 1990).

4.2. Test specimens

The drying, firing and total shrinkage and pyroplastic deformation results are presented in Table 6. The results show that even a five percent substitution of granite by the CS consistently increases the total linear shrinkage, with the effect being subtler during the drying phase and more pronounced at the firing stage (Sokolář et al., 2017). This also proves that the addition of the CS is responsible for altering the plasticity of the slips, as seen in Table 5, because higher plasticity generally results in higher drying and firing shrinkages (Silva et al., 2017). In general, firing shrinkage is linked to the volume of fluxing agents (Rambaldi et al., 2007), as a higher flux content means that a greater number of pores could be filled by the liquid formed during sintering. Gültekin et al. (2017) reported that the densification of ceramics can be carried out by sintering in liquid phase and that one of the mechanisms is related to the viscous flow in the pores. Salem and Salem (2014) observed that there is a reduction in total porosity and refinement of the porous
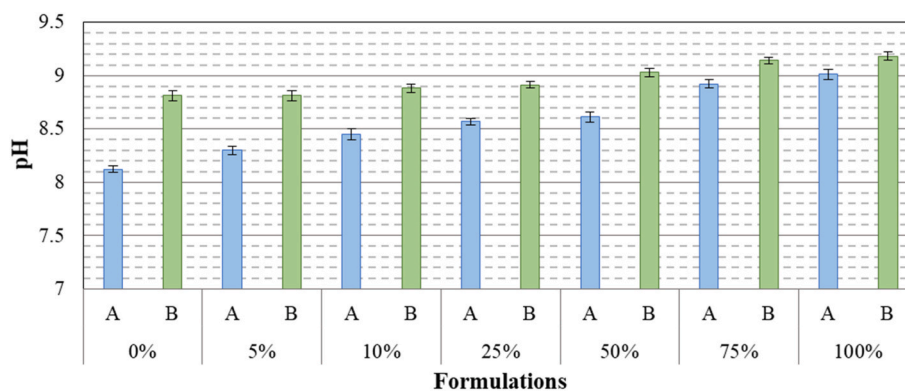
Fig. 5. pH of the ceramic slips.

Table 5
Rheological properties of the ceramic slips.

Slip	Density* (g/cm^3)	Viscosity 50 rpm* (cP)	Viscosity 2.5 rpm* (cP)	Thixotropic Index	Setting rate g/(cm^2 x min)	Drying time (min:s)
A-0	1.81	412	2592	6.3	0.28	01:38
A-5	1.82	421	2320	5.5	0.29	01:41
A-10	1.81	405	2464	6.1	0.30	01:37
A-25	1.80	465	2912	6.3	0.38	01:40
A-50	1.81	482	2944	6.1	0.33	01:38
A-75	1.79	523	4064	7.8	0.34	01:40
A-100	1.79	588	4448	7.6	0.35	01:43
B-0	1.81	402	2322	5.8	0.29	01:39
B-5	1.82	441	2496	5.7	0.29	01:41
B-10	1.81	426	2560	6.0	0.29	01:37
B-25	1.80	421	2704	6.4	0.29	01:40
B-50	1.81	472	3248	6.9	0.30	01:38
B-75	1.80	523	3264	6.2	0.31	01:40
B-100	1.81	521	3216	6.2	0.32	01:39

Notes: * Relative standard deviation $\leq$ 5%.

Table 6
Thermal parameters of test specimens.

Slip	Drying Shrinkage (%)	Firing Shrinkage (%)	Total Shrinkage (%)	Pyroplastic Deformation (mm)
A-0	3.08	8.01	10.85	60.0
A-5	3.12 [0.36]	9.99 [0.28]	12.79 [0.15]	65.8 [1.64]
A-10	3.15 [0.23]	10.40 [0.18]	13.22 [0.05]	69.9 [1.50]
A-25	3.22 [0.25]	10.29 [0.19]	13.19 [0.04]	75.0 [0.77]
A-50	3.39 [0.17]	10.26 [0.13]	13.30 [0.11]	79.5 [0.74]
A-75	3.62 [0.21]	10.13 [0.23]	13.38 [0.24]	82.4 [2.35]
A-100	3.04 [0.28]	9.32 [0.28]	12.07 [0.22]	84.5 [4.06]
B-0	3.38	8.52	11.58	58.0
B-5	3.06 [0.10]	9.20 [0.09]	11.98 [0.11]	57.6 [0.89]
B-10	3.07 [0.09]	9.39 [0.02]	12.17 [0.09]	59.5 [0.44]
B-25	3.19 [0.14]	9.71 [0.07]	12.59 [0.15]	61.6 [1.91]
B-50	3.18 [0.05]	9.51 [0.12]	12.39 [0.13]	62.7 [1.06]
B-75	3.88 [0.27]	9.10 [0.13]	12.63 [0.15]	66.1 [0.75]
B-100	3.47 [0.15]	9.67 [0.39]	12.81 [0.43]	67.8 [2.38]

Notes: Standard deviation in brackets and ≤5% for A-0 and B-0.

network during the sintering phases. Also considering the firing cone test and pyroplastic deformation (PD), the results indicates that CS has a stronger flux behavior than granite.

Although there is not a standard limit to PD, lower values decrease the chances of defects in the final ceramic piece (De Miranda et al., 2015). The clear correlation between PD and the CS content means that the sanitary ware produced from slips with lower degrees of substitution are expected to present fewer imperfections after firing (Almeida et al., 2016). Additionally, while there is not a standard for total shrinkage (TS), the industry usually sets its values according to its own mold design to comply with the dimensions required by the market. In this case study, the industry works with TS values from 10% to 13% to avoid cracks and to agree with the standards in product size; an ideal slip should fall within this range, so no redesign of molds is necessary, which initially excludes the formulas from A-10 to A-75 in terms of technical usability, but further development in slip design, or in mold design, could address that limitation for slips with higher CS content.

Figs. 6 and 7 show the results of three-point flexural strength (FS) tests for green and sintered specimens. Figs. 6 and 7 show that the flexural strength of the specimens initially diminishes as CS replaces granite in the mixtures, after which it starts to regain strength. For raw pieces, the minimum FS adopted by the industry is 1.5 MPa. The green specimens that showed FS higher than the minimum were A-100, B-50, B-75 and B-100, with only the latter being comfortably above the requirement. Considering FS for fired specimens, the minimum accepted
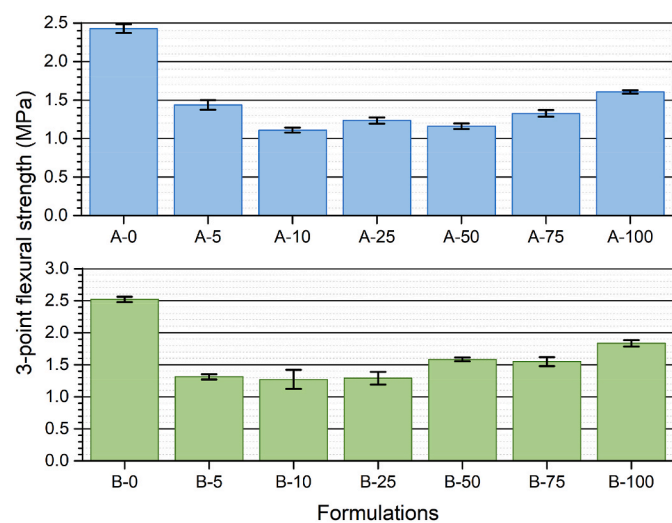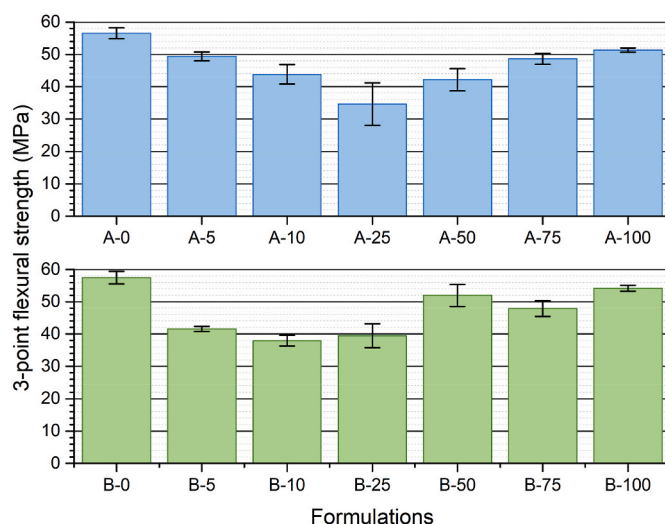
Fig. 7. Three-point flexural strength for fired specimens.

flexural strength is 40 MPa. As a result, there were a few specimens that did not meet the requirement, with A-25, B-10 and B-25 under 40 MPa. Additionally, specimens A-10, A-50 and B-5 did not meet the requirement when accounting for their standard deviations, that is, one or more values within the sample of five specimens resulted in a strength lower than that required.

The mechanisms that cause this initial decrease, followed by a recovery of strength, could be linked to porosity and phase formation. Generally, the more pores a ceramic specimen has, the lower its strength will be. In addition, the dispersion strengthening hypothesis proposed by (Hasselman and Fulrath, 1966) states that well-dispersed crystals of mullite and quartz in the glassy matrix increase the strength of ceramic specimens. Fig. 8 shows six SEM images for each group's standard specimen, also the lowest and highest strength specimen (excluding the standard). The phases were identified with support from the literature and were found to be primary mullite (PM), residual quartz (RQ) and the main structural component, the glassy phase (GP). Stathis et al. (2004) observed the correlation between residual quartz and porosity after firing. Marinoni et al. (2011) reported primary mullite in the form of small cubic-like crystals (~0.1 μm) and secondary mullite with an interlocking structure, needle-shaped and size >1 μm. Gralik et al. (2014) observed quartz, primary mullite, secondary mullite, alumina and glass phase. López et al. (2013) identified residual porosity, quartz, primary mullite, secondary mullite and vitreous matrix. Tunçel and Özel (2012) observed glassy phase, residual quartz, primary mullite and secondary mullite.
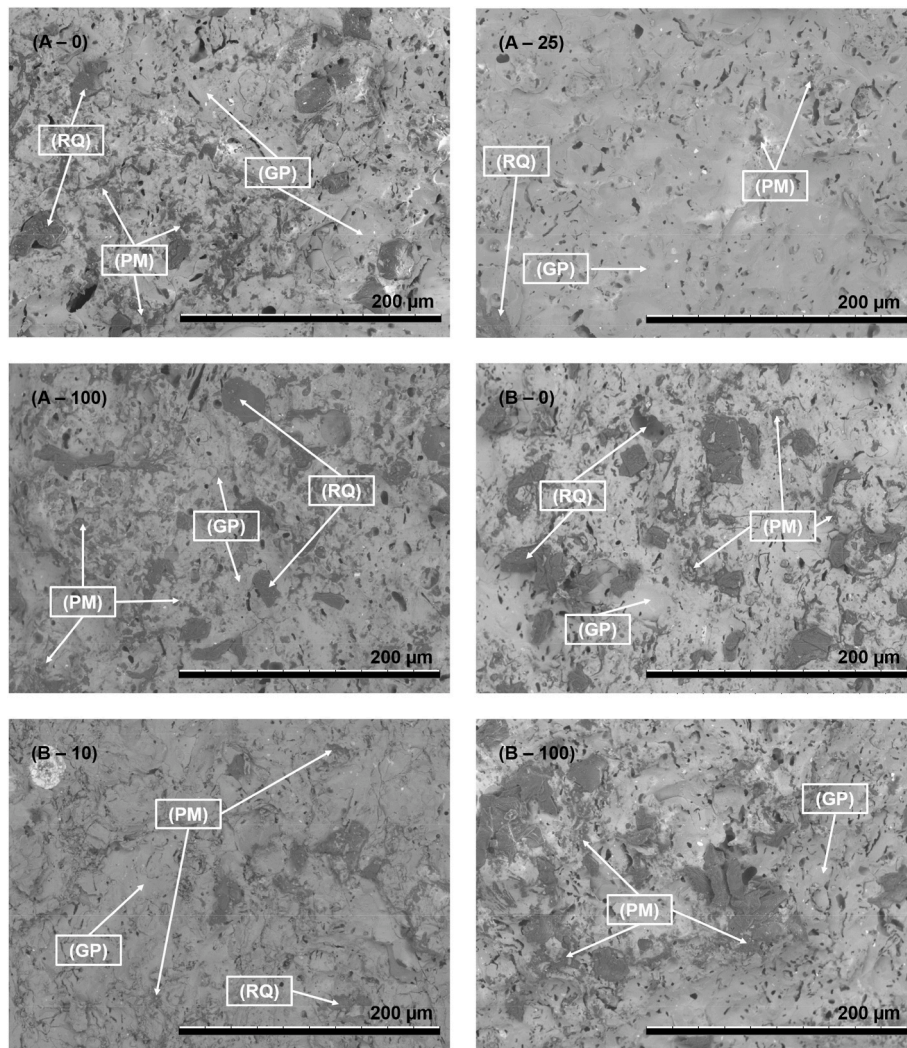
Fig. 6. Three-point flexural strength for green (non-fired) specimens. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

Fig. 8. SEM images of a few fired specimens.

The results of the flexural strength, combined with their corresponding phase dispersion shown in Fig. 8, agree with the dispersion strengthening hypothesis, which states that well-dispersed crystals in a glassy matrix can limit the size and number of Griffith flaws, increasing the strength (Hasselman and Fulrath, 1966). The cause of such differences in phase dispersion regarding different amounts of CS and granite is yet unknown since they were all sintered in the same industrial kiln under identical firing curve adopted for manufacturing.

Sanitary ware must comply with a standard for water absorption (ASTM, 2018), which states that this value should not be greater than 0.50%. This test measured the surface porosity, and although standardized tests of water absorption are not always able to ensure the complete saturation of test specimens (Vieira et al., 2017), they are commonly used to qualitatively correlate the flux content in ceramic specimens. Rambaldi et al. (2007) used the water absorption and shrinkage results to define the ideal firing temperature, which is related to the flux content. Andreola et al. (2010) reported that increasing waste content decreased water absorption and densified the microstructure, possibly caused by higher flux and liquid phase contents during sintering. In another study, Andreola et al. (2016) observed that the increase in strength and resistance to freeze-thaw cycles correspond to the lowest values of water absorption. Fig. 9 shows the average water absorption values obtained for each fired specimen. The results show that increasing the CS content in the mixtures lowers the water absorption. As proposed in this study, the CS acts as a stronger fluxing agent than
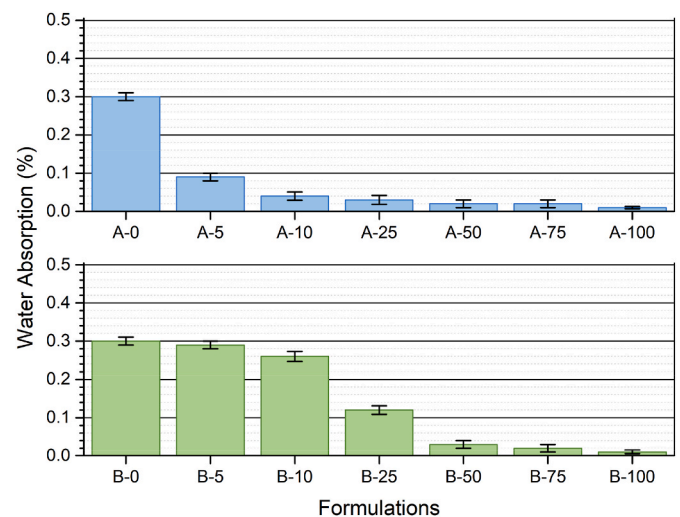

Fig. 9. Water absorption values for fired specimens.

granite, the lower values of water absorption, which indicates that more pores were filled with the liquid phase that formed during sintering. This hypothesis agrees with the higher results found for linear shrinkage

because as more pores are filled, the ceramic specimens should contract; however, this difference in surface porosity is not evident in the SEM images (Fig. 8) since they correspond to internal structures that were exposed.

5. Application feasibility and clean production

So far, the results reflect the ceramic mass behavior at the material level. The analysis can be extended to two converging perspectives. The first is related to application feasibility, which bridges engineering properties with real manufacturing situations. The second refers to the interrelationship between empirical results and sustainable development, promoting cleaner production.

5.1. Application feasibility

To investigate the feasibility of application, hollow ceramic blocks called *cobogó* were produced using slip casting technique. The option for blocks was based on the need for a relatively large piece to assess the change in the color of the enamel and the presence of unwanted air voids, which can be caused by escaping gases in the firing process. The molds used for the large parts were not practical for laboratory testing. Hollow block molds were used (Fig. 10a), a product commonly manufactured in the ceramic industry.

The production of these pieces was more complex than the test specimens, since the blocks are hollow, as the sanitary ware on the production line. However, it is important to remember that the shaping process for sanitary ware is automated, with automatic filling and drainage, facilitating casting, in addition to the injection of compressed air that increases the cohesion of the walls during drying. In the blocks production, these steps were carried out manually, except for compressed air, which was not used. To guarantee the stability of the walls, it was necessary to apply slip diluted in a proportion 1/10 in the plaster molds before slip casting (Fig. 10b). In the center of the mold, talc was applied to facilitate the detachment of the piece. The block mold has two holes used for filling (top) and draining (bottom). After closing the mold,

one of the holes was closed and a tube segment was placed around the other. The slip was poured into the mold until it was filled (Fig. 10c). The time for forming the walls was set at 15 min and the drainage of the remaining liquid mass inside the mold was performed (Fig. 10d). After draining the residual fluid mass, the cast element remained in the mold for another 2 h, was detached and dried naturally (Fig. 10e). After drying, the color was checked, and half of blocks was cut to check the wall thickness (Fig. 11). The other half was finished and placed in the
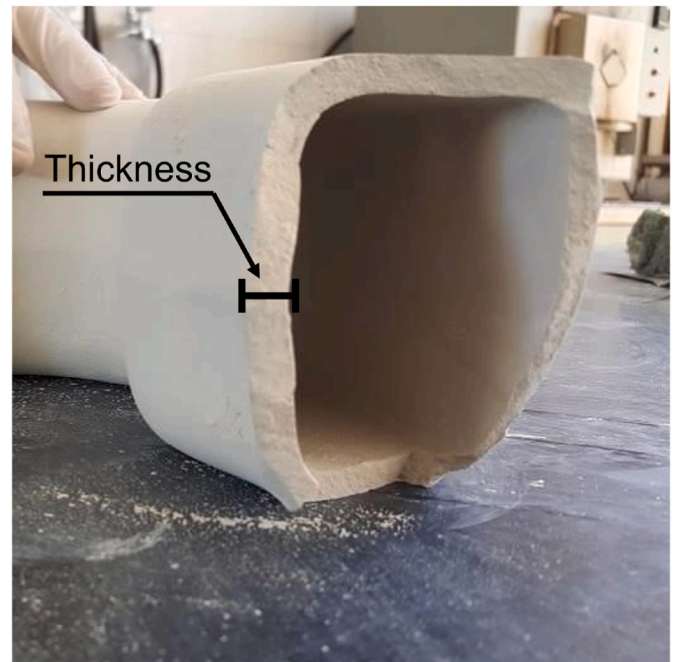

Fig. 11. Ceramic block cross-section and wall thickness.
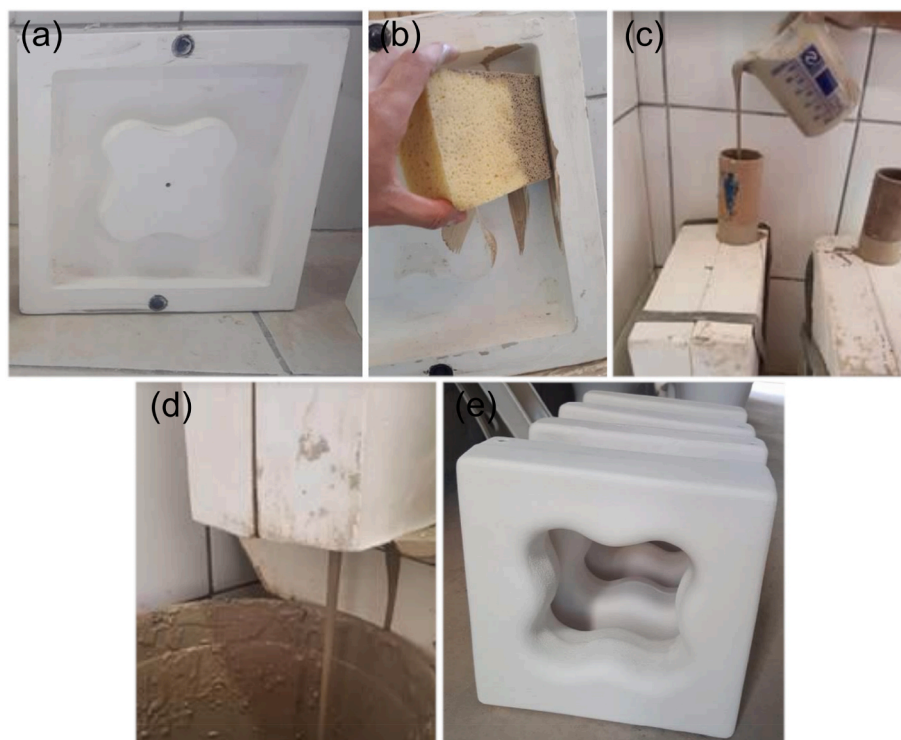

Fig. 10. Manufacture of hollow ceramic blocks.

oven for 24 h. After this period and subsequent cooling, the piece was enameled and proceeded to the firing oven under the same conditions as the sanitary ware.

Fig. 12 shows the wall thickness results for the ceramic blocks. It was observed that higher proportions of replacement resulted in thicker wall formation. The greatest thickness was achieved by the A-100 formulation (11.2 mm), which represents a 12% increase compared to A-0. This result is related to the wall formation during slip casting. The liquid dispersion showed low viscosity, high homogeneity, and no agglomerates, which ensured a good casting. The stability of the aqueous suspension is maintained by electrostatic repulsive forces, which ensures separation between particles. During casting, part of the solvent from the dispersion in contact with the mold is absorbed by the porous plaster, destabilizing the suspension, and promoting the deposition of the ceramic mass, once attractive forces become dominant (Gadow and Kern, 2014). This process is accelerated by draining the remaining dispersion, which further reduces the solvent fraction. The addition of CS may have increased the solids content of the aqueous suspension, which reduced the distance between particles and the energy barrier by electrostatic forces that prevent coagulation. This mechanism also explains the porosity reduction since closer particles promote denser packing. The results are in accordance with the setting rate (see Table 5).

None of the tests showed air holes, visible glaze disagreement or color difference. This indicates that a reformulation of the finishing process would not be necessary for the CS-containing ceramic body. Some of the *cobogós* can be seen in Fig. 13, which belong to formulation B and showed lower values of pyroplastic deformation. Those of formulation A, except A-5, had some defects on the walls, that is, the surfaces were not completely flat. The justification for the appearance of such defects only in group A may be related to both casting and firing. The greater pyroplastic deformation presented in formulation A may be responsible for the appearance of surface discontinuities. Such defects can also be the product of poor casting and operator experience. This latter, if it is the source of the defects, can be solved by automating the process, as is done with larger pieces of sanitary ware. However, if the reason for the defects is the greater pyroplastic deformation, it will be necessary to change the formulation A.

5.2. Cleaner production in the sanitary ware industry

The applicability of the ceramic mass with replacement was confirmed by the production of hollow ceramic blocks with wall thickness equal to or greater than the blocks without CS. This fact indicates
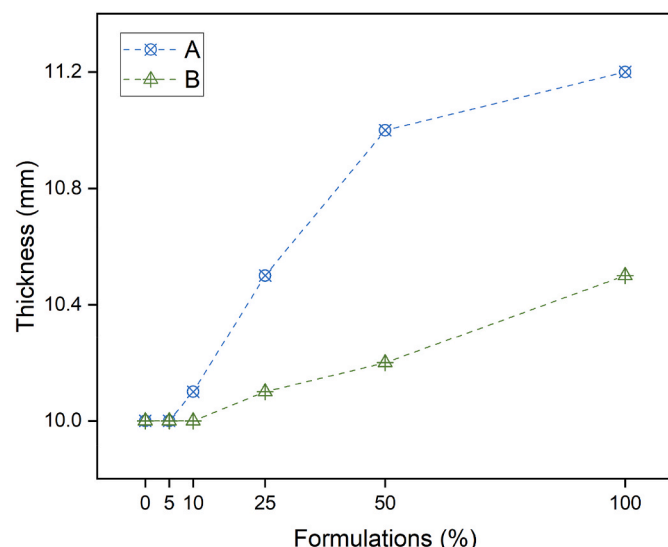

Fig. 12. Wall thickness of the ceramic blocks.

that the ceramic suspension formed a consistent layer along the mold wall. An important finding refers to porosity. A smaller amount of pores indicates voids filling and greater densification of the microstructure. The effectiveness of sintering and the absence of gases escaping during heating were also achieved. All these aspects indicate good durability and adequate life cycle of the finished products, which saves resources and reduces harmful effects on the environment. In addition, the results can be useful for engineers in the sanitary ware sector, particularly for production line waste recovery and dosage optimization.

Since the best formulation results is that of B-100, CS could be recycled as an intermittent solution, replacing granite to reduce solid particles from the sedimentation basin, or as a separate, smaller production line. As the fraction of particles that end up in the sedimentation basin is estimated at approximately 5% of the total raw materials used for production, the application of a waste valorization process could be sufficient to consume all the CS, which would reduce environmental impacts and long-term disposal costs (Fig. 14). This on-site ceramic sludge recycling approach, along with a previous attempt to recycle discarded ceramic pieces (after fire), represents an environmentally friendly solution, which should be less cost-prohibitive than recycling the waste elsewhere, opening the way for cleaner production in the sanitary ware industry.

6. Conclusions

From the experimental program, results and discussion, the following main conclusions can be drawn:

1. Ceramic sludge waste (CS) was valorized and reinserted into the production chain as a replacement for granite.
2. CS was characterized as a stronger flux than granite.
3. Replacement of granite by CS influences the rheological behavior and the physical-mechanical properties.
4. Rheological behavior was influenced by pH and CS content. All proportions of group B and the 5% and 50% of group A showed a rheology similar to the standard mixture.
5. B-100 proportion showed promising results in all tests.
6. Porosity decreases with increasing CS content.
7. Feasibility of the CS application was confirmed by the production of hollow ceramic blocks with a wall thickness greater than standard without waste.
8. Waste valorization process could reduce solid particles in the sedimentation basin and consume all CS.
9. The findings open the way for cleaner production in the sanitary ware industry.

7. Future perspectives

The following aspects of the present work can be expanded:

- Study new compositions of the ceramic mass, by replacing equivalent raw materials.
- Use proportions with a higher substitution level in a separate production line and smaller proportions to the existing process.
- Evaluate the efficiency of solid-liquid separation processes for effluent and define specific applications for waste.
- Examine the economic feasibility of implementing the waste recovery process.
- Investigate the factors that influence the viscosity and setting rate of the fluid mass to stabilize the wall thickness.

Fig. 13. Finished ceramic blocks.
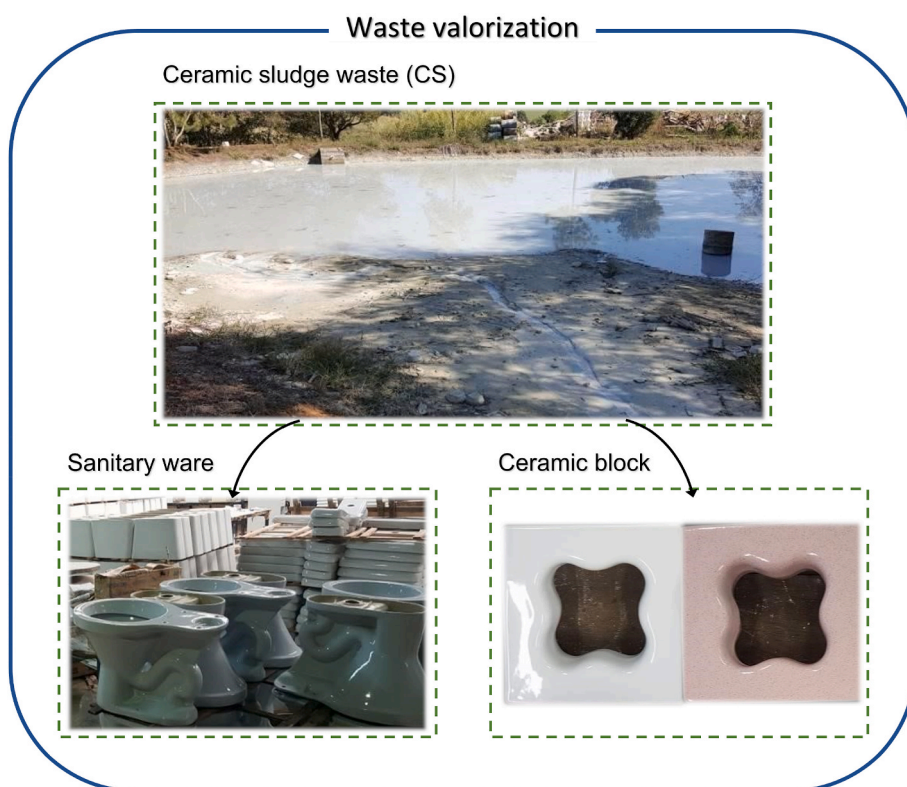
Fig. 14. Waste valorization in the sanitary ware industry.

Declaration of competing interest

The authors declare no competing financial interests or personal relationships that could appear to influence the work reported in this paper.

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