

Reuse of Sanitary Ceramic Waste in the Production of Vitreous China Bodies

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ABSTRACT: An experimental investigation of Sanitary Ceramic Waste (SCW) for use as raw material in the manufacture of vitreous china bodies is presented. The gradual substitution of feldspar by sanitary ceramic waste and its effects on the technical and physical properties of sanitary bodies have been studied. The rheological behavior of sanitary slip is improved using Na-electrolyte. The characterization of the fired vitreous bodies at 1230 °C shows that 10 wt. % SCW substitution is the ideal value for the composition of a vitreous china body. FT-IR and DRX analyses confirmed that the crystalline phases (quartz and mullite) are stable during the addition of sanitary ceramic waste with a significant increase in their intensities. SEM micrographs show an increase in the porosity when the addition of sanitary ceramic waste exceeds 10wt. %, as a result of the reduction of the vitrified phase. From physical-mechanical characterization, an improvement in flexural strength (33 to 41MPa), and a reduction in water absorption (0.36 to 0.31 %) were recorded. These positive results open very promising prospects for the valorization of sanitary ceramic waste, with many technical, economic, and environmental benefits.

KEYWORDS: SCW; Physical properties; Flexural strength; Vitreous china bodies.

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INTRODUCTION

Sanitary ceramics are considered among the most demanded goods in our daily life and building industry [1, 2]. Sanitary ceramics have two main parts, the inner ceramic body and the surface glaze layer which improve the chemical and mechanical properties of the final product [3]. Moreover, sanitary bodies have a complex chemical composition consisting of various oxides making raw materials [4]. Fireclay, fine fireclay and vitreous china bodies are the most used types in the production of sanitary ceramic [5]. Vitreous china is the most produced; it has a low deformation after casting and firing [6]. Typical compositions consist of a mixture of clay-kaolin, feldspar and quartz [7]. We can add that the manufacturing processes of vitreous china bodies are more or less the same as other ceramic products. The composition and particle size of the raw materials has a significant effect on the rheological behaviour of clay slip. First, a clay slip is produced by mixing various raw materials with water. A small amount of electrolytes is usually added to the slip, to improve its rheological behavior [8, 9]. Afterwards, plaster molds are used to absorb slip water by capillarity, which leads to the formation of a green ceramic body before the removal of the molds. After the casting process, the bodies are dried, glazed and fired generally in a tunnel kiln. Finally, the products are colored according to the desired decoration [3, 10]. Generally, mullite, quartz and vitreous phase are the main phases in vitreous china bodies [11]. Clay is well-known for providing plasticity to the mixture. Quartz improves the size control of the product after firing. Feldspar is the melting mineral containing SiO_2 , Al_2O_3 and an alkaline oxide (Na_2O , K_2O or CaO) that produces the liquid phase during sintering [12].

Today, the production growth of various industrial goods causes more generation of solid wastes; these are not always biodegradable. For this reason, the integration of some industrial wastes as substitution materials in various ceramic compositions has been the subject of many studies. These are mostly: red clay [13, 14], fly ash [15, 16], glass waste [17, 18], blast furnace slag [19, 20] and solid ceramic waste [21, 22].

Global sanitary ware production increased by 61.3% between 2004 and 2014, with an annual growth rate of 4.9%. For example in 2014, the production was 349.3

million pieces worldwide [23]. Sanitary Ceramic Waste (SCW) originates from defective pieces which have deformation in their technical properties such as shrinkage 20-30 tons of SCW are discarded every month from the sanitary ware factories [24].

In the literature, much research has been done about the best way to use SCW as a substitute material in the various ceramic industries. The replacing of 15 wt. % kaolin with SCW in the production of wall tiles reduced moisture expansion, increased density and flexural strength (202.5–205.5 kg/cm^2) [25]. Moreover, water absorption is reduced (0.22 to 0.15 %) and flexural strength is increased (580.2–603.2 kg/cm^2) when 10 wt. % of pegmatite are replaced by SCW in glazed porcelain tile production. In the same work, the partial replacement of 15wt. % feldspar by SCW reduces the bulk density and increases water absorption (0.22-0.44%). Besides, in contrast, the addition of SCW which has a thermal expansion coefficient much lower than of feldspar and pegmatite, contributes to decreasing the thermal expansion coefficients of glazed porcelain tiles [26]. Another research concluded that 5wt. % SCW replacement, — allows commercial vitreous china production at 1150 °C [24]. The addition of 15 wt. % SCW in the place of feldspar in porcelain stoneware tiles production, is found to decrease the sintering temperature and leads to better mechanical properties (50-70 MPa) [22]. Also, the incorporation of SCW in floor tile formulations, enhances the physical-mechanical properties as well as thermal expansion [27]. The temperature interval has been reduced by the partial replacement of 11 wt. % quartz and 4 wt. % feldspar with SCW; the bending strength is also improved (57-67 MPa) [28]. In another study, the substitution of 5wt. % granite by SCW, reduces water absorption and improves the rheological behaviour of sanitary ceramic slip [29].

This work aims to investigate the effects of using Sanitary Ceramic Wastes (SCW) as a substitute material for sodium and potassium feldspar mixture on the properties of vitreous china bodies. The benefit of course is reducing the environmental damage of industrial waste, recycling these wastes and reducing the production costs.

EXPERIMENTAL SECTION

Sample preparation

All specimens were prepared under industrial conditions. All vitreous china body compositions contain

Table 1: Formulations (in wt. %) of ceramic compositions for VC, VC5, VC10, VC15 and VC20 samples

Raw materials	VC	VC5	VC10	VC15	VC20
Clay (Hycast VC)	28	28	28	28	28
Parkaolin	12	12	12	12	12
kaolin RMB	12	12	12	12	12
Sodium feldspar	12	9.5	7	4.5	2
Potassium feldspar	11	8.5	6	3.5	1
Silica Sand	25	25	25	25	25
SCW	0	5	10	15	20

clay-kaolin mixture, feldspar and quartz. An industrial vitreous china product formulation was selected as the standard (VC) composition. SCW was used in place of sodium and potassium feldspar mixture in VC5, VC10, VC15 and VC20 bodies at concentrations of 5 wt. %, 10 wt. %, 15 wt. % and 20 wt. %, respectively. Table 1 presents all formulations used in sanitary slip preparations. SCW pieces were collected and crushed in an alumina mill jar. The chemical compositions of SCW and raw materials were obtained using an X-ray fluorescence spectrometer (Rigaku ZSX Primus IV). The Loss of Ignition (L.O.I) was determined by firing materials at 1000°C for 2 hours.

To prepare the vitreous china slip, for each composition, 3000g of dry raw materials with 1000 ml of water and an amount of electrolytes (0.1 wt. % Na_2SiO_3 + 0.075 wt. % Na_2CO_3) were milled in a porcelain jar for 4 hours to obtain a residue less than 1.8 % using a sieve of 63 μm . The rheological properties of the slip, including fluidity, density and residue were checked each 30min. Fluidity was determined by measuring the time required for the slip to fill a 100 ml Ford cup (2.6 mm opening). The residue is measured after passing 100 ml of slip through a sieve (63 μm); then, the residue mass is dried and weighed. Afterwards, in a plaster mould, the resulting slip was cast at room temperature. Test pieces (70 × 20 × 10 mm) and (120 × 20 × 20 mm) were prepared to measure water absorption, linear shrinkage and flexural strength, respectively. Finally, the green bodies were dried and fired at 1230 °C in a tunnel kiln. The values recorded represent the median achieved with eight measurements for the same sample.

Methods of characterization

The fired bodies have been physically characterized; the apparent density (D_A) has been determined by Archimedes' method; the total porosity (P %) has been determined by measuring the true density (D_T) using

the Candlot- Le Chatelier densimeter; the total porosity is determined by Eq. (1)

$$P (\%) = \frac{(D_T - D_A)}{D_T} * 100 \quad (1)$$

The values of Linear Shrinkage (LS) are found by measuring the distance between the lines recorded on the test pieces before and after sintering. The linear shrinkage is computed by Eq.2

$$LS = \frac{(L_i - L_f)}{L_i} * 100 \quad (2)$$

Where L_i is the length value recorded before sintering (10cm); L_f is the length after sintering.

The amount of Water Absorption (WA) is estimated with ASTM C373-88. 2006, by using the difference in weight between the dried samples (m_i). These were immersed for 2 hours in the boiling water, then cooled for 12 hours and the surface dried with a wet towel (m_f). The water absorption is determined by Eq. (3) [30].

$$WA (\%) = \frac{(m_f - m_i)}{m_i} * 100 \quad (3)$$

To measure the flexural strength (fs) of the sintered bodies, the three-point bending method was utilized using the NETSZH global testing machine; the measurements are converted to flexural strength by Eq. (4).

$$fs = \frac{3}{2} \frac{B * S}{w * t^2} \quad (4)$$

Where B is the breaking load (N); S is the space between two supports (10cm); w is the width of the body and t is the thickness of the body.

X-Ray Diffraction (XRD) is an analytical technique based on the interaction of X-rays with the matter, especially when the material is crystallized; its goal is to study the different constituent phases of the material. In our work, X-Ray Diffraction (XRD) has been used to characterize the different crystalline phases of the fired samples, with a siemens D5000 diffractometer, by using $\text{Cu-K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$) at the Brentano Bragg pattern. The specimens were scanned from 2 θ =5 to 80°, at a scanning rate of 3°/min.

To get more information on morphology and microstructural properties, the microstructures of the vitreous ceramic bodies were studied by scanning electron microscopy (SEM, WD S, JEOL JSM 6360LV, Biskra University, Algeria). The micrographs were obtained using an accelerating voltage of 10 kV with a working space of 11.0 mm; the backscattered electron images were taken at 750x magnification.

Table 2: Chemical analysis of SCW and raw materials.

Oxides (%)	Clay Hycast	PAR	Kaolin	Sodium	Potassium	Quartz	SCW
	VC	KAOLIN	RMB	feldspar	feldspar		
SiO ₂	52	48	48	70.74	69.5	96.35	69.2
Al ₂ O ₃	31	37	37	17.92	17.3	0.52	22
TiO ₂	1	0.06	0.05	0.26	0	0.05	0.439
CaO	0.2	0.07	0.07	0.5	0.5	1.19	1.14
MgO	0.4	0.3	0.3	0.2	0.2	0.08	0.279
K ₂ O	2.1	1.9	1.75	0.4	9	0.17	3.17
Na ₂ O	0.2	0.1	0.1	9.6	3.5	0.08	1.64
Fe ₂ O ₃	1.2	0.19	0.85	0.08	0.16	0.24	1.42
ZnO	0	0	0	0	0	0	0.181
ZrO ₂	0	0	0	0	0	0	0.406
L. O. I	12	11.8	12.1	0.5	0.4	1.29	0.8

Table 3: Chemical analysis of ceramic slip modified by added SCW.

Oxides(%)	VC	VC5	VC10	VC15	VC20
SiO ₂	70.21	70.16	70.12	70.08	70.03
Al ₂ O ₃	24.15	24.37	24.59	24.81	25.03
TiO ₂	0.37	0.39	0.40	0.42	0.43
CaO	0.49	0.53	0.56	0.59	0.62
MgO	0.27	0.27	0.28	0.28	0.29
K ₂ O	2.24	2.16	2.09	2.01	1.94
Na ₂ O	1.77	1.52	1.28	1.03	0.79
Fe ₂ O ₃	0.61	0.67	0.74	0.8	0.87
ZnO	0	0.01	0.01	0.02	0.03
ZrO ₂	0	0.02	0.04	0.06	0.08

The IR transmission spectra of SCW and the fired bodies, were obtained using a sample powder with a wavenumber resolution of 1 cm⁻¹ using KBr pellets (purity: 99.9%, spectral grade). The results were registered by a spectrophotometer of Shimadzu Japan, in the interval 4000-500 cm⁻¹. In addition, to obtain a good signal-to-noise ratio, 32 scans were run and averaged.

RESULTS AND DISCUSSIONS

Raw Materials and SCW Analyses

The chemical analysis of SCW and the raw materials is given in **Table 2**. One type of clay and two types of kaolin were used in this work; their compositions consist of a large amount of SiO₂ and Al₂O₃. The main reason that clay Hycast, Parkaolin and kaolin RMB were used is plasticity, whiteness, green strength, productivity and total shrinkage. Sanitary ceramic waste (SCW) is made of

a large amount of SiO₂ and Al₂O₃. Na₂O and K₂O characterize sodium and potassium feldspar, respectively. Silica Sand contain a very high amount of SiO₂ with some traces of CaO. The composition of each formulation is summarized in Table 3. From the table, we notice a slight increase in Al₂O₃ with a decrease in SiO₂ and alkali oxides (Na₂O and K₂O), following-the addition of SCW.

After the grinding process, a white ceramic waste powder is formed. The particle distribution was irregular. The amount of humidity was very satisfactory, not exceeding 3%. Fig.1 shows a medium particle diameter of 30.71µm; 90% of the sample particles are smaller than 100µm. These particles dimensions are comparable to those in raw materials used in the ceramic industry.

The FTIR spectrum of SCW (see **Fig. 2**), reveals the presence of five bands at 780.89, 1079.34, 1634, 2358.52 and 3449.12cm⁻¹. Quartz peaks related to Si-O and Si-O-

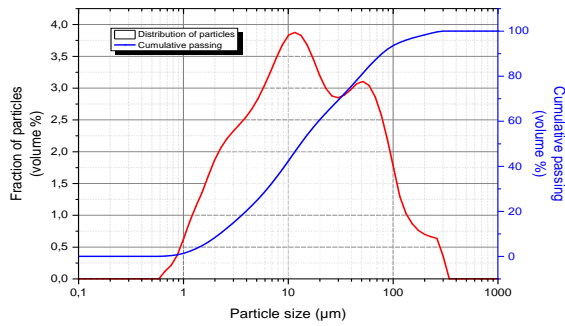


Fig. 1: Particle size distribution curves of SCW powder.

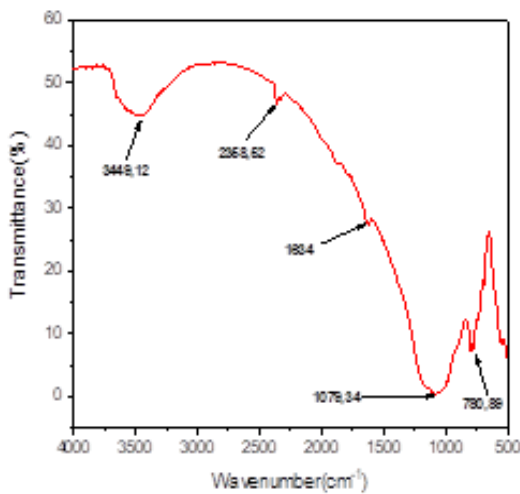


Fig. 2: FT-IR spectrum of SCW.

XRD analyses of SCW and raw materials are shown in Fig. 3. Clay, kaolin RMB and Parkaolin contain illite, quartz and kaolinite. Quartz is the main component of silica sand with a trace of calcite and hematite. Sodium feldspar contains albite and quartz. However, potassium feldspar is composed of orthose and quartz. As expected, SCW is formed by mixing raw materials to form the ceramic body, with the addition of glazing materials. SCW spectrum shows the presence of mullite and quartz. Mullite is the stable phase responsible for mechanical resistance in the fusion of clay minerals [29].

Si vibrations, were recorded at 780.89 and 1079.34 cm^{-1} [31, 32]. The bands situated at 1634 and 3449.12 cm^{-1} , indicate the H–O–H bending vibration of adsorbed water [32, 33]. The band located at 2358.52 cm^{-1} reveals the Si–C stretching [34].

Table 4: Evolution of the fluidity, residue on sieve and density of slip as a function of added SCW concentration.

	VC	VC5	VC10	VC15	VC20
$\frac{\text{Na}_2\text{CO}_3}{\text{Na}_2\text{SiO}_3}$			0.25		
$\frac{\text{Na}_2\text{CO}_3}{\text{Na}_2\text{SiO}_3}$ (wt. %)			0.375		
Density (g/cm ³)	1.771	1.780	1.773	1.776	1.775
Residue on sieve (63 μm) (%)	1.6	0.6	0.9	0.6	0.5
fluidity(s)	30	25	24	24	20
Drying shrinkage (%)	5.7	8.56	7.59	8.44	8.97

The effect of SCW on rheological behavior of slip

The incorporation of SCW enhanced the slip properties. Furthermore, the addition of some electrolytes was necessary to enhance the rheological behavior of the slip. To do that, a mixture of sodium carbonate and sodium silicate was used. The combination ratio $\frac{\text{Na}_2\text{CO}_3}{\text{Na}_2\text{SiO}_3} = 0.25$, with a total amount of 0.375 wt. % had a positive effect on the fluidity of the different slips; this amount of electrolytes is the same in all formulations as appears in Table 4. The findings prove that fluidity is reduced by the addition of SCW powder and Na-electrolytes. This effect can be attributed to the excessive amount of sodium cations Na^+ in the slip, which decreases the thickness of the diffusion layer [8]. The residue on the sieve (63 μm) (%) was also decreased by the addition of SCW. That could be due to the small size of the SCW particles compared to the size of the feldspar particles. Generally, The rheology of the slip, greatly affects the surface charge of the ceramic particles, which leads to a change in the slip behavior [35]. In this context, the utilization of several electrolytes has been found more effective to improve the slip properties compared to the utilization of sodium silicate only, the same observation has been also reported by many authors [36].

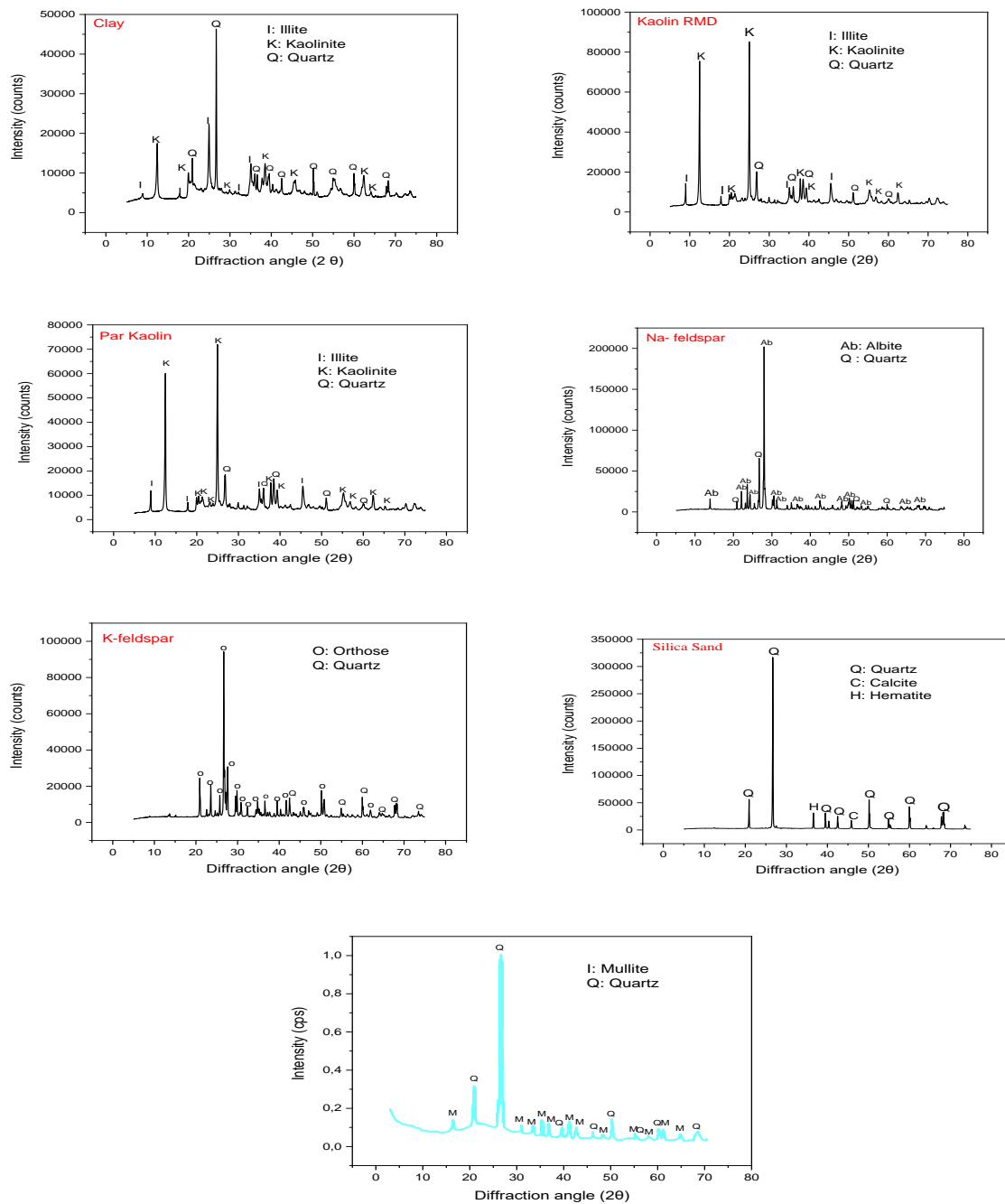


Fig. 3: XRD patterns of raw materials and SCW.

Effect of SCW on technological properties of the fired samples

Bulk density and total porosity measurements for the ceramic bodies, are shown in Fig.4. The highest density was recorded in sample VC5 where feldspar is replaced with 5 wt. % SCW. Also, the addition of 10 wt. % SCW makes the ceramic body VC10 slightly denser than the

standard ceramic VC; that is can be due to the formation of the liquid phase resulting from the alkali oxides ($K_2O + Na_2O$) of feldspar in combination with the alkaline earth oxide (CaO) originating from SCW. The liquid phase improves the firing process and fills the voids between the crystalline phases. The gradual substitution of feldspar by SCW, contributes to the reduction of the mixed alkaline content;

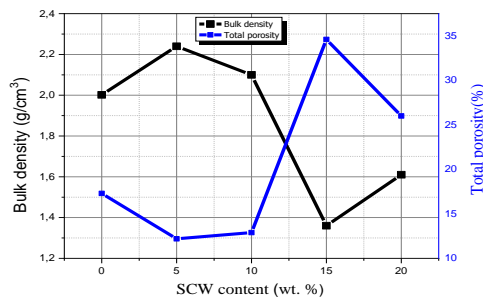


Fig. 4: Variation of bulk density and total porosity with SCW content, for a vitreous ceramic heated at 1230°C.

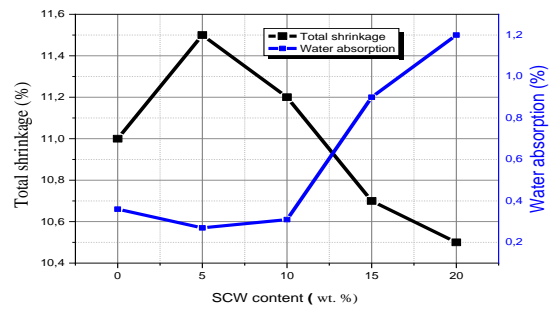


Fig. 5: Variation of water absorption and total shrinkage with SCW content, for a ceramic heated at 1230°C.

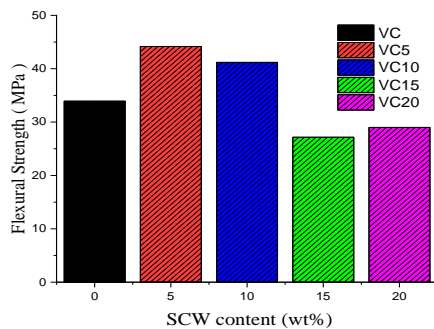


Fig. 6: Variation of flexural strength with SCW content for our samples heated at 1230°C.

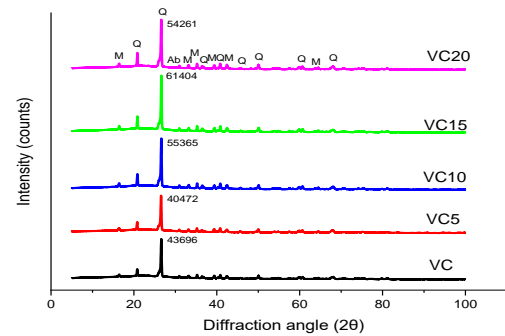


Fig. 7: XRD patterns for heated samples at 1230 °C (M: mullite, Q: quartz, Ab: albite).

thus, the formation of the liquid phase is diminished and the presence of the viscous SiO₂ melt from the SCW, is increased. For this reason, porosity goes up in VC15 and VC20, which have 15 wt. % and 20 wt. % SCW respectively [19, 37, 38]. The values of shrinkage and water absorption are given in Fig.5. Usually, the decrease in shrinkage is due to the reduction of the liquid phase that causes more opening of the pores, which leads to more water absorption of the ceramics containing SCW [39]. According to ASTM C 373-88, the conditions shrinkage < 12% and water absorption < 0.5% are necessary for a longer cleanliness of the final product [31, 40]. SCW contains less residual quartz than feldspar (Table 2), and its coefficient of thermal expansion is also much lower than that of feldspar [26]. The decrease in quartz content results in a lower coefficient of thermal expansion. This result is important for meeting the dimensional and deformation requirements of the final product, indicating that final products with high dimensional stability and suitable deformation properties can be obtained [26].

Effect of SCW on flexural strength of fired ceramics

The influence of SCW on the flexural strength of vitreous ceramic bodies, is illustrated in Fig.6; a significant increase was observed in the case of 5wt. % SCW addition. The increase in VC10 bodies (44MPa) compared to standard ceramics (33MPa) can be explained by a higher formation of crystalline phases, in particular mullite which is formed by the rich content of SiO₂ and Al₂O₃ in SCW; another reason is the high liquid phase formed by the rich alkali oxides in the composition [25]. However, when the substitution feldspar by SCW exceeds 10 wt. %, the flexural strength decreases because of the smaller amount of alkali oxides; this reduces the liquid phase and creates more porosity [26].

X-ray analysis of fired ceramics

X-ray diffraction analysis of the fired vitreous china bodies at 1230°C, is shown in Fig.7. The major crystalline phases for all VC bodies consist of a glassy phase, mullite and quartz. The high Al₂O₃ content in the SCW together with the alkali oxides of feldspar, enhance the formation of mullite. Similarly, the high SiO₂ content results

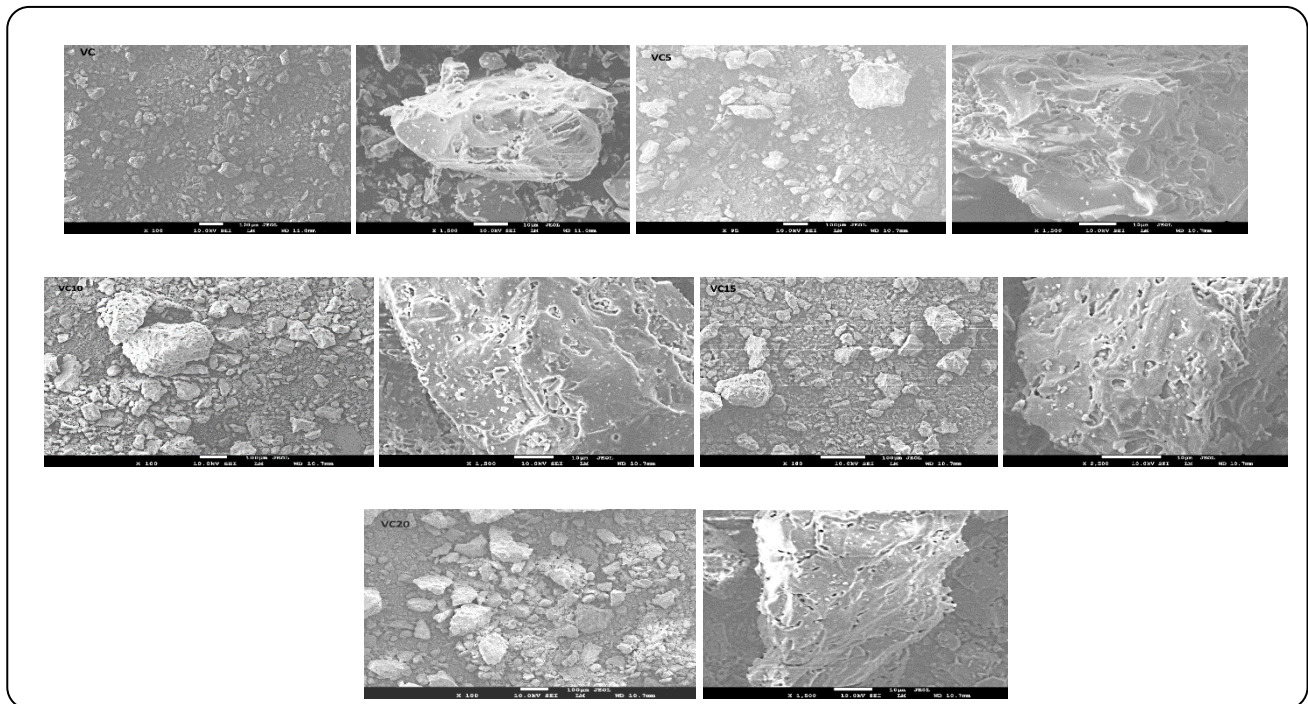


Fig. 8: SEM micrographs of VC, VC5, VC10, VC15 and VC20 samples.

in a larger intensity of the quartz phase. The diversity chemical compositions of ceramic bodies, leads to physical-chemical transformations during the firing process [26]. Water is removed from the body at about 100°C. The reverse transformation of α - β quartz of silica at 573 °C where kaolinite transforms to metakaolinite ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) happens when its OH group is lost between 450°C and 600°C. Gradually, mullite peaks appear at 1000 °C, and the spinel phase is formed. Albite is mostly invisible due to its dissolution to form a glassy phase above 1100°C [41, 42]. The relation among density, flexural strength, chemical composition and microstructure was explained in detail in the SEM analysis section. Mullite obtained from SCW is the main phase in vitreous china bodies and contributes to the increase in flexural strength. The strength increases with mullite content in the presence of alkali rich feldspar (K_2O and Na_2O , where the percentage of ceramic waste does not exceed 10 wt. %.

Microstructure characterization

To demonstrate the relationship between the technological properties and the microstructure, SEM analyses were conducted on the investigated bodies. Fig.8 presents the SEM micrographs for the fired bodies at 1230°C. All ceramics present a heterogeneous particle size

distribution; with various crystals present in a glass matrix formed by dissolved feldspar-penetrated clay. These are mostly mullite and quartz crystals. VC5 body exhibits a higher density than the other bodies with few closed pores on the surface of the crystals. SCW has a small few amount of CaO oxide mixed with the alkali oxides of feldspar (K_2O and Na_2O); this leads to the development of a large amount of liquid phase that fills the gaps between the particles, causing a reduction of the porosity. To a lesser extent, the morphology of VC10 was better than the standard body in terms of bulk density and apparent porosity [43, 44]. The increase of dissolved SCW leads to a smaller amount of alkali oxides in the VC15 and VC20 compositions. This reduces the formation of the liquid phase, and leads to the opening of the pores as clearly seen on the surface of the molecules. These results are very consistent with the XRD analysis and physical-mechanical tests [17, 45].

FT-IR Spectroscopy

FT-IR spectra of the five ceramic bodies, were recorded to evaluate their mineralogical phases, shown in Fig. 9. The absorption peaks at 781 cm^{-1} can be ascribed to the presence of Si-O-Al vibrations [46]. It was found that broad bands near 1084 cm^{-1} correspond to the Si-O-Si asymmetric stretching vibration which confirms the

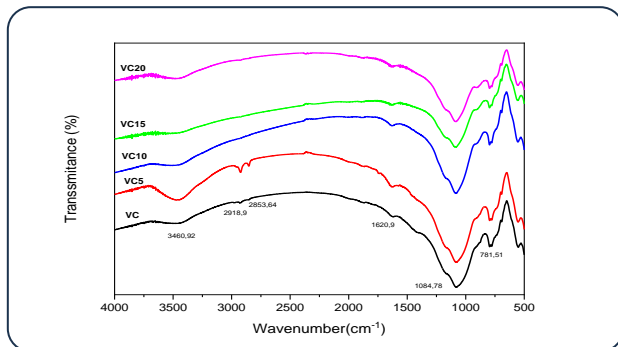


Fig. 9: FTIR analysis of the mixtures with 0, 5, 10, 15 and 20 wt. % SCW.

enhanced high formation of quartz by the addition of SCW [34, 47]. Noteworthy, weak absorption bands at 1620 cm^{-1} are assigned to H–O–H bending vibrations of adsorbed H_2O molecules [33]. The bands that appear in VC and VC5 samples, at 2853 and 2918 cm^{-1} can be attributed to Si–O and Al–O symmetric stretching vibrations in the structure; this leads to the formation of mullite and enhances the strength of VC5 [48, 49]. Finally, the broad bands at 3460 cm^{-1} reveal the H–O–H bending vibrations of adsorbed water molecules [32]. These findings are fully consistent with physical-mechanical tests and DRX analyses.

CONCLUSIONS

This study investigated the effects of sanitary ceramic waste (SCW) on vitreous china bodies. As the amount of SCW waste increased up to 10 wt. %, flexural strength, firing shrinkage and bulk density increased whereas water absorption and total porosity decreased. However, flexural strength and bulk density of investigated bodies containing more than 10 wt. % SCW reduced. According to FTIR, DRX and SEM analyses, the use of SCW increases the intensity of quartz and mullite with a reduction of the liquid phase. Findings show that the use of SCW in place of feldspar is most effective at ratios less or equal 10 wt. % for the production of vitreous china bodies. The most important result of this work was to define the suitable amount for SCW in vitreous china bodies composition, to obtain competitive products with good dimensional stability and good mechanical-physical properties.

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