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Utilization of rhyolite and red syenite rocks as an alternative of potash feldspars in manufacturing the sanitary ware bodies

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Abstract

Sanitary wares were prepared from rhyolite lava and red syenite replacing k-feldspar in the ceramic batches. quartz, albite, microcline and anorthoclase minerals were identified in the k-feldspar, rhyolite and syenite rocks. The thermal behavior indicated that the softening temperatures were 1202°C for k-feldspar,1208°C for rhyolite and 1178°C for red syenite whereas the melting points were 1309°C for k-feldspar,1312°C for rhyolite and 1303°C for red syenite. After sintering the batches at 1200°C/20 h, quartz with mullite was developed in sanitary ware containing k-feldspar, but in that consisting of rhyolite lava or red syenite sillimanite was developed. The microstructure of sintered samples produced with rhyolite lava and red syenite replacement shows irregular pattern containing submicron and nano-size particles. For physicals testes, samples treated at 1200 °C/20h gave nearly similar values. The coefficient of thermal expansion (CTE) which refers to the rate at which a material expands with the increase in temperature at 800 °C were 5.29 x 10^{-6} $°C^{-1}$ for k-feldspar containing body, 6.84 x 10^{-6} $°C^{-1}$ for rhyolite and 5.7 x 10^{-6} $°C^{-1}$ for red syenite based ones. The modulus of rupture for samples before firing were 34.9 kg/cm^2 for k-feldspar, 34.3 kg/cm^2 for rhyolite and 33.57 kg/cm^2 for red syenite and their corresponding sintered samples after firing at 1200°C/20 h were 482.87 kg/cm^2 for k-feldspar, 445.27 kg/cm^2 for rhyolite and 481.6 kg/cm^2 for red syenite because of solidification. The ratio of water absorption values were 0.29% for k-feldspar, 0.33% for rhyolite and 0.19% for red syenite, while the shrinkage ratios were 10.3% for k-feldspar, 10% for rhyolite and 10.83% for red syenite The present characteristics and properties indicate that both rhyolite lava and red syenite are suitable for the replacement of k-feldspar in sanitary body.

Keywords: Sanitary ware bodies, alkali feldspar, sintering

1. Introduction

Feldspar is a common raw material used in glassmaking, ceramics, and to some extent as a filler and extender in paint, plastics, and rubber. In ceramic industry, sanitary wares are made by the supporter layer (sanitary body) which composed of stoneware, and the surface layer is of glaze which covers the supporter layer [1,2]. However, the constituent of sanitary body is: ~ 56 % kaolin, 32 % feldspar and 12% silica sand which are mixed with 0.25 water glass (sodium silicate) and about 30 % water [3-5].

Feldspars are a group of rock-forming aluminum tectosilicate minerals, containing also other cations such as sodium, calcium, potassium, or barium. This group are spread mainly in igneous rocks, and in metamorphic and sedimentary rocks which cover about 50 % of the earth's crust. Feldspar formation includes the ability of silicon tetrahedral to be substituted by aluminum and the defective positive charge is adjusted by alkali or alkaline earth elements. In ceramics, the alkalis in feldspar (calcium oxide, potassium oxide, and sodium oxide) act as a flux, lowering the melting temperature of a mixture. Fluxes melt at an early stage in the firing process, forming a glassy matrix that bonds the other components of the system together [6]. The feldspar-containing alkalis are considered as fluxing agents, not only to lower the melting temperature in the glass but

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also to facilitate sintering in ceramic materials [7,8]. Sintering is a critical stage in the production of ceramic bodies. By controlling the density and microstructure formation, sintering now emerged as a processing technology of ceramic materials. Tailoring the structural, mechanical, electrical, magnetic and optical properties is widening the application of ceramics in various fields [9]. Therefore, feldspars are extensively used as raw materials in the glassmaking (~60 %) and ceramic production (~35 %) [6]. Furthermore, alkali feldspar is considered as a backbone in ceramic industries and the researchers usually search for these alkali rocks. Alkali feldspar either sodium-rich or potassium-rich are used in glassmaking or ceramic manufacturing while calcic-feldspar can be employed in fiberglass that strengthens the consequent plastic composite [10].

In the ceramic industry, granite pegmatite is the traditional source of feldspar, however, other sources of feldspar comprise acid- and sub-volcanic rocks, syenites, phonolites, feldspathic arenites, metamorphics, albitites or epithermal alterations [11,12]. Feldspars are thought as the backbone of ceramic and porcelain industries. They are used in the production of sanitary-wares, floor-and wall tiles, enamel, tableware, kitchenware, etc...

The main aim of the present research is to engineer sanitary ware bodies with using optimal alkali igneous rocks. In this study, the traditional k-feldspar used in the manufacture of sanitary bodies was replaced by rhyolite lava or red syenite. For this purpose, different sanitary ware and rock raw material samples have been collected, characterized and processed with the starting raw materials and sintered samples.

2. Materials and methods

The present research based on the preparation of sanitary bodies using rhyolite lava and red syenite instead of K-feldspar. The starting raw materials were collected from rhyolite and syenite rocks in the northern part of the Eastern Desert of Egypt, being about 60 km southwest of Ras Gharib at the Red Sea.

The chemical analysis of the raw materials were done in the special laboratories of Research Institute of Natural Sciences (RINS), Okayama University of Science, Okayama, Japan using Philips X-ray fluorescence (XRF) spectrometer Model PW/2404 equipped with Rh tube (**Table 1**). The composition of mix constituents of the sanitary bodies for both standard containing k-feldspars (ST) and rhyolite lava (RL) - and red syenite (RS) ones are listed in **Table 2**. However, all batches of sanitary bodes were fired in a production kiln to have the same firing conditions at 1200 °c /20 h, then fast and slow cooled as indicated in firing curve (**Fig. 1**)

Identification of the minerals of starting raw materials containing as received rocks, standard k-feldspars (ST) and both rhyolite lava (RL) and red syenite (RS) and also heat-treated mixtures to get the end product was made using X-ray diffraction analysis (XRD) (Analytical BV - cubix3, X-ray powder diffraction, Holland). The reference data for the interpretation of X-ray diffraction patterns were obtained from ASTM X-ray diffraction file index and some relevant publications.

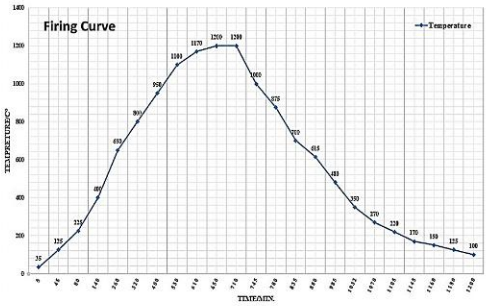


Fig. 1. Sanitary ware firing curve

The differential thermogravimetric analysis (DTG) for the studied raw materials were conducted using Netzech - STA449 F3 Jupiter, Germany, to demonstrate the mass changes and thermal effects between 100°c and near 1100°c. The sinterability and melting had been measured using hot stage microscopy HSM 1400–Misura, ESS, Italy with heating rates of 10°c/min.

The microstructure and microanalysis of the sintered samples, pre- etched using 1% HF + 1% H_3NO_3 solution (15 seconds), were considered using scanning electron microscopy (SEM/EDX, FE-SEM, Model FEI, QUANTA, FEG, 250, Holland).

CTE of the sanitary bodies containing standard k-feldspar (ST), rhyolite lava (RL) and red syenite (RS) ones, after firing at 1200 °c, were determined using dilatometer (Dilatometer Misura @ HSM-ODHT, Italy). The sintered sample was free of edges (2x1x1cm in dimensions) and subjected to heating rate of 10 °c/min in the dilatometer up to 800 °c.

The modulus of rupture (MOR) test of the sanitary wares (kg/cm^2) was performed on the green (before firing) and fired specimens using two different testing machines. The MOR test for green bodies was performed using CHST002, E.J payne limited, United Kingdom and for sintering using MOR/100 Kg E.J payne limited, United Kingdom. For these tests ten rods pieces (~12 cm length ~ 1.11 cm diameter) shaped by casting the prepared slip in gypsum mold then dried in a dryer (105 °c/3h) then cooled down to room temperature. The test was done for five dried samples and other five sintered at 1200 °C/20 h.

Water absorption and shrinkage tests were done for the sanitary specimens (k-feldspar-, rhyolite lava- and red syenite-containing bodies) sintered at 1200°c. The test of water absorption was conducted according to ASTM C373-18, (it should be ≤0.5%). For water absorption test, the weighed dry sample (Md) was immersed in hot water left to boil for 2 hours and then allowed to cool down to room temperature for 24 hours followed by drying with wet clothes piece then weighed as wet weight (Mw), consequently the water absorption can be given by E = (Mw – Md)/Md) x 100 formula. The shrinkage test was carried out according to ASTM C326. The shrinkage of sanitary ware after firing should be range between 10 to 12%**.** For shrinkage test, three wet bodies shaped as ruler (15 cm length with a sign of 10cm) were dried at 100°C/3h then cooled in desiccator for five hours (length after dryness is referred as Ld) and finally they sintered at 1200 °C/20h (length after sintering is referred as Ls). The ratio of shrinkage is determined by = (Ld – Ls / Ld) x 100.

3. Results and discussion

3.1. Characteristics of the raw and processed samples

3.1.1 Chemical Characteristics

The starting raw materials were chemically analyzed. The batch of the standard sample was designed according to Seger formula by calculating the total percentage of total SiO_2 and total Al_2O_3 for all components of body recipe to determine (SiO_2/Al_2O_3) ratio. The calculated ratios for K-feldspar body (ST), rhyolite lava (RL) and red syenite (RS) ones were 5.68, 5.69 and 5.56 respectively, which are in the possible range of Seger ratio. It must be mentioned that the alkali (Na_2O, K_2O, CaO and MgO) weight percentage are 9.37 for Rhyolite Lava, 14.56 for Red syenite and 10.11 K-feldspar containing ones.

Table 1: Constituents of the sanitary ware batches from different raw materials

Sample	**Constituents of the sanitary batches in weight %**									
	K - Feldspar	**Rhyolite lava**	**Red syenite**	**Na feldspar**	**Clay Imported**	**Clay Local**	**Kaolin Imported**	**Kaolin Local**	**Silica' sand**	**Pitcher**
Standard Feldspar (ST)	**17**	**00**	**00**	**10**	**19**	**5**	**19.5**	**4**	**20.5**	**5**
Rhyolite lava (RL)	**00**	**17**	**00**	**10**	**19**	**5**	**19.5**	**4**	**20.5**	**5**
Red syenite (RS)	**00**	**00**	**15**	**10**	**19**	**5**	**19.5**	**4**	**22.5**	**5**

Table 2: Chemical Composition (wt%) of the raw materials

Raw Material	**Chemical** composition									
	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	IL
K feldspar	72.00	15.70	1.42	0.20	0.00	0.26	1.39	4.00	4.46	0.06	0.51
Rhyolite lava	72.41	15.64	1.91	0.18	0.05	0.32	0.36	5.21	3.48	0.03	0.41
Red syenite	61.22	18.46	4.83	0.42	0.09	0.46	2.06	6.77	5.26	0.10	0.32
Na Feldspar	69.10	15.70	1.33	0.51	0.00	0.28	0.91	9.18	1.20	0.12	0.46
Clay Imported	55.07	28.34	1.33	0.75	0.01	0.29	0.12	0.18	1.3	0.04	12.42
Clay Local	52.8	28.40	3.58	1.22	000	0.35	0.52	0.45	0.14	1.34	11.20
Kaolin Imported	84.10	35.63	1.10	0.51	000	0.46	0.03	0.00	1.83	0.01	12.3
Kaolin Local	48.60	35.18	1.10	0.90	00	00	00	00	2.30	0.22	11.70
Silica sand	99.30	0.01	0.04	0.01	000	0.01	0.43	0.01	0.05	000	0.04
Pitcher	71.41	19.3	1.38	0.53	00.01	1.42	0.93	2.86	2.16	000	000

1.1.2. XRD, DTA and SEM/EDX analysis

The X-ray diffraction analysis (XRD) was carried out to determine the crystalline phases of fired bodies as well as the raw materials. The followed procedure was based on the description by [13]. The X ray diffraction analysis (XRD) of k-feldspars (FR) and both rhyolite lava (RLR) and red syenite (RSR) show the mineral pattern of alkali feldspars, microcline, albite, anorthoclase and quartz (**Fig. 2**). Anorthoclase [(Na,K) (Si_3 Al)O_8] was the major mineral in red syenite that is rich in both Na_2O and K_2O ratios whereas quartz and albite ($NaAlSi_3O_8$) are the major minerals in both k-feldspar and rhyolite lava. Microcline ($KAlSi_3O_8$) is presented as secondary mineral in k-feldspar and red syenite (**Fig. 2**).

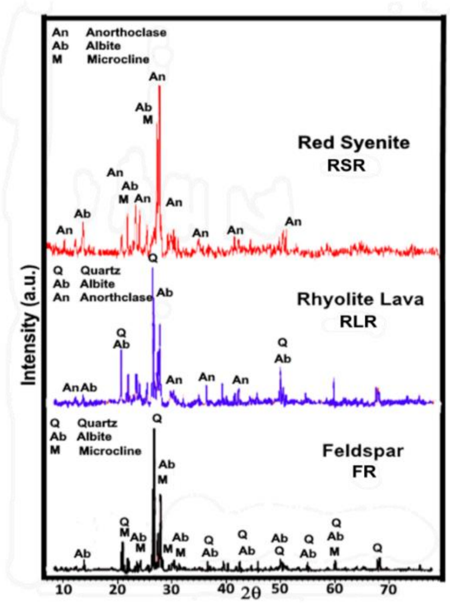


Fig. 2. X ray diffraction patterns of K-feldspar, rhyolite lava and red syenite.

The DTG of as received k-feldspars (FR) and both rhyolite lava (RLR) - and red syenite (RSR) show clear weight loss with temperature starting from 100 up to 1200°c (**Fig. 3**). The weight loss greatly attributed to both the release of molecular water (H_2O) and hydroxyl groups (OH) [14]. The later weight loss reflected as intense endothermic peak at 204°C in k-feldspar and 230°C in red syenite. Other intense exothermic appears at 1026°C in rhyolite lava and 1073°C in red syenite which reflects the start of softening the crystalline minerals into glass phase, however, this temperature is higher in k-feldspar (Fig. **3**).

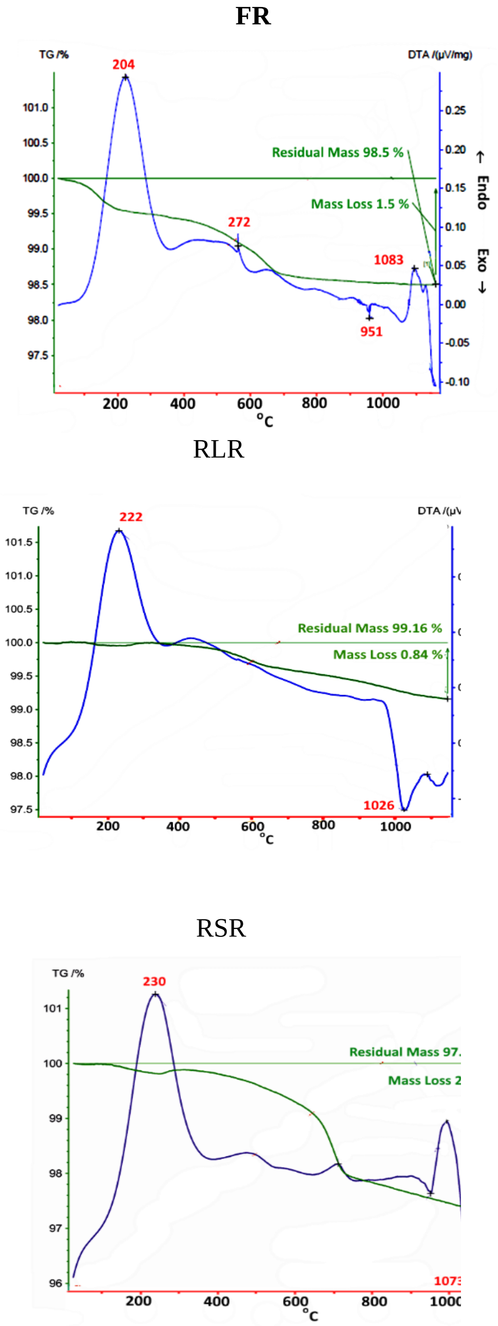


Fig 3 . DTG analysis of as received K-feldspar (FR) , rhyolite lava (RLR) and red syenite (RSR) rocks

The X-ray diffraction analysis after well mixing and sintering the sanitary batches (ST, RL and RS) at 1200 °C for 20 hours indicated the crystallization of quartz and aluminum silicates phases. appears as mullite ($Al_{4.59}$ $Si_{1.41}$ $O_{9.7}$) in ST and RL samples or as sillimanite (Al_2SiO_5) in RS as a high temperature phase (**Fig.4**).

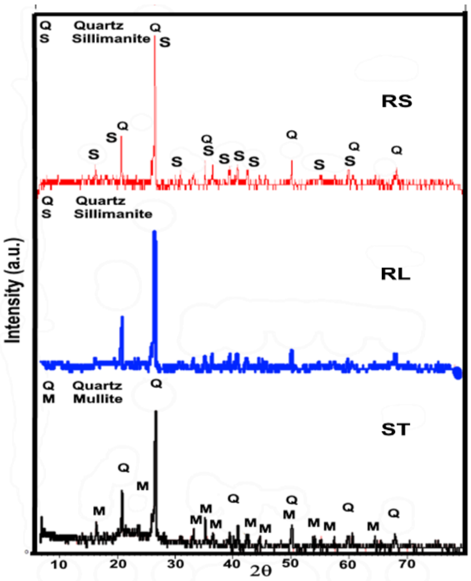
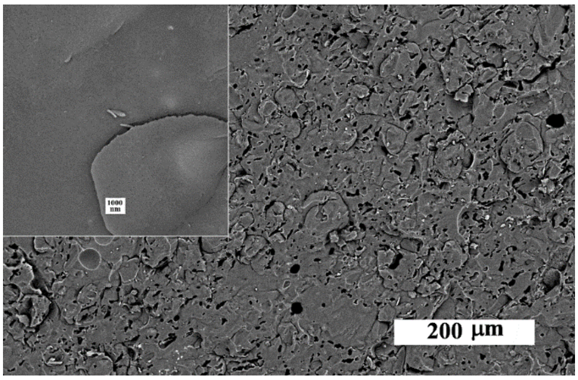


Fig. 4. X ray diffraction patterns of ST , RL and RS sintered samples at 1200 °C/20h.

The microcrystalline structure of both standard feldspar sample (ST) and rhyolite lava (RL) sintered at 1200°c for 20h show similar microstructures (**Fig. 5**), demonstrating irregular patterns containing submicron and nano-size particles spread in little glassy matrix. The particles grain size ranges between 50 and 100 nanometer which scattered in glassy groundmass. Some pores were scattered between the crystalline phases.



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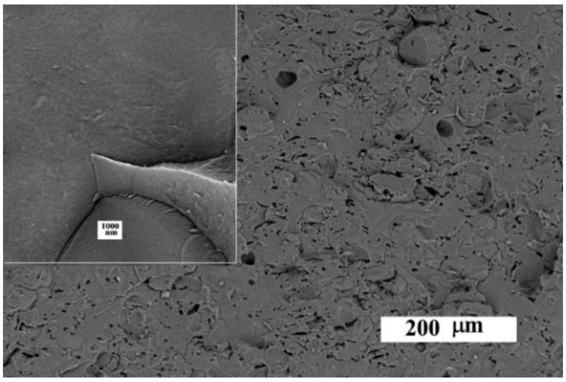

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Fig. 5. The SEM micrographs of the sanitary bodies containing standard feldspar sample (ST) and rhyolite lava (RL) sintered at 1200 °C/20h.

The EDX microanalysis of k-feldspar (ST) and rhyolite lava (RL) after their mixing and sintering (1200°c /20 h) show the chemical constituents in the sintered sanitary samples. The Si/Al ratio of ST and RL sanitary samples were 2.50 and 2.67 respectively. (**Fig. 6**)

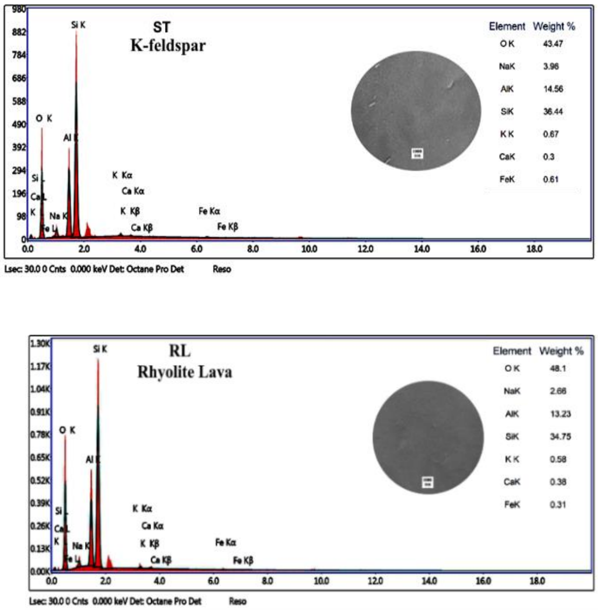
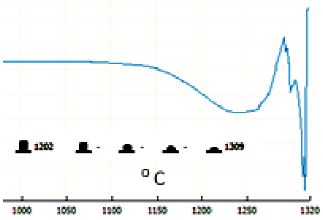
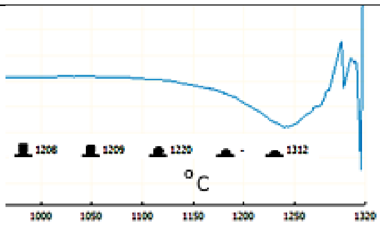
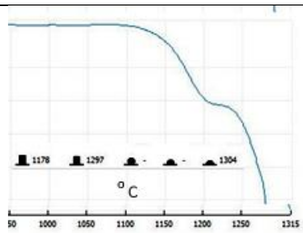
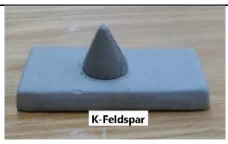
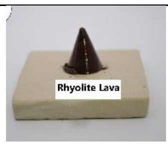
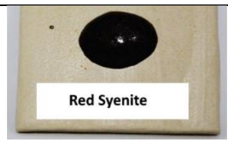


Fig. 6. The EDX microanalysis of the sanitary bodies containing K-feldspar (ST) and Rhyolite Lava (RL) after sintering at 1200 °C/20h.

3.1.3. Fusibility Tests

The fusibility is a change of the materials state from solid to liquid state. In the ceramic materials this test is done either by hot stage microscope (HSM) or by cone method. The fusibility temperature of K-feldspar, rhyolite lava and red syenite are listed in Table 3. The alkali ratios are nearly similar in both K-feldspar (FR) and rhyolite lava (RLR). The softening and melting points are nearly parallel. In the case of red syenite (RSR), the ratio of alkalis is higher than in that of rhyolite lava, therefore both the softening and melting points are lower than in samples (FR and RLR) (Table 3). One question which was raised is why the partial melting occurs although some major present minerals have high melting point. Through eutectic systems, the mixes of crystalline minerals can melt at lower temperatures than the individual ones [15].

Table 3. The fusibility results of FR, RLR and RSR as received rock samples.

Fusibility Testes	**Tested raw materials**		
	FR **K-feldspar**	**RLR** **Rhyolite lava**	**RSR** **Red syenite**
HSM results			
softening point (sintering)	**1202**	**1208**	**1178**
Melting point (fusion)	**1309**	**1312**	**1303**
Cone method at 1200°C Results			
	Clear sintered cone	Clear sintered cone	Clear fusion

The tests of the bodies after firing at 1150°c, 1180 °c, 1200°c, 1210 °c indicated that the optimum degree of firing is between 1200 °c and 1210 °c, however all samples sintered at 1200°c /20 h at production kiln.

3.2. Properties of the sintered samples

3.2. Properties of the sintered samples

3.2.1 Coefficient of thermal expansion (CTE).

CTE of the sanitary bodies containing the standard k-feldspar (ST), rhyolite lava (RL) and red syenite (RS) after firing at 1200°c/20 h, were caried out according to ASTM C1300-95 test for linear hermal expansion of sanitary ware by Interferometric Method; Determination of coefficient of linear thermal expansion were determined using dilatometer. The examinations indicated that the thermal properties (%) are near the standard sanitary ware body. The CTE shows different values at lower temperatures (300-700°c), whereas it becomes nearly similar at higher temperatures (>700 °c) (Table 4) presenting similar behavior of the produced (RL) and (RS) samples in comparison to the standard one.

Table 4. CTE of the sanitary ST, RL and RS bodies sintered at 1200°C/20 h

Temperature	CTE of sintered sanitary bodies at 1200 °c/20h x 10^{-6}°c^{-1}		
	ST	RL	RS
300 °c	2.69	4.55	3.45
400 °c	3.76	5.47	4.40
500 °c	4.48	6.21	5.05
600 °c	5.48	7.39	6.09
700 °c	5.38	7.19	5.93
800 °c	5.29	6.84	5.70

3.2.2. Modulus of Rupture (MOR) (kg/cm^2)

The MOR of the investigated green (before firing) sanitary samples were tested according to **ASTM** C689-09 (2019) standard test method for modulus of rupture of unfired clays. It was 34.3 (RL), 33.57 (RS) and 34.90 (ST) kg/cm^2 which show similarity and also in the sintered samples which tested according to ASTM C1211, they were measured as 482.87 Kg/cm^2 for (ST), 445.27 Kg/cm^2 for (RL) and 481.6 Kg/cm^2 for (RS) (Table 5 and Fig. 7). However, the results were more or less similar to the standard ST sample.

Table 5. MOR, water absorption and shrinkage (%) of the ST, RL and RS sanitary samples.

Sanitary bodies	Property tests*			
	MOR (Kg/cm^2) Green sample (Before firing)	**MOR (Kg/cm^2)** Sintered samples at 1200 °C/20h	**Water absorption %** Sintered samples at 1200 °C/20h	**Shrinkage %** Sintered samples at 1200 °C/20h
ST	**34.90**	**482.87**	**0.29**	**10.30**
RL	**34.30**	**445.27**	**0.33**	**10.00**
RS	**33.57**	**481.60**	**0.19**	**10.83**

*The property test values represent the average of three samples for each test

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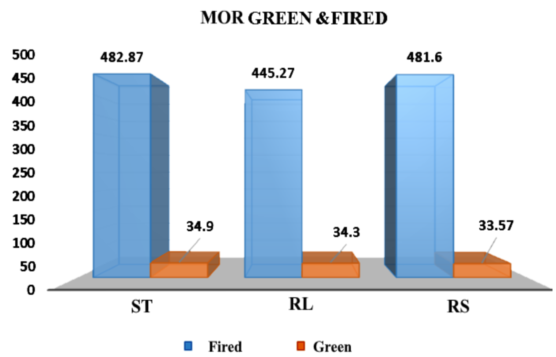
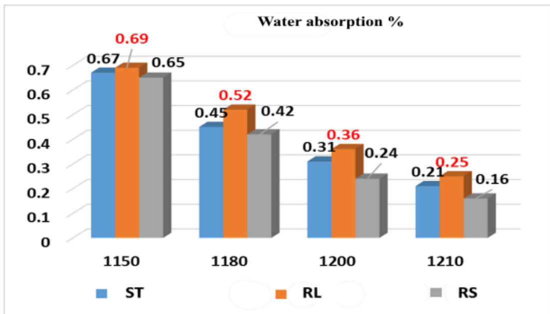


Fig. 7. The modulus of rupture (MOR) of the investigated green (before firing) and after sintering sanitary samples.

3.2.3. Water absorption and shrinkage (%)

The water absorption and shrinkage results of the pre-sintered samples fired in laboratory kiln in various sintering temperatures are presented in **Fig. 8**. It is noticed that water absorption decreases with increasing temperature and the shrinkage increases with increasing temperature. The water absorption and the shrinkage of (ST), (RL) and (RS) indicate that the optimum degree of firing is between 1200°c and 1210°c, it is clear from the results that there is similarity of the values of water absorption especially for samples that sintered at 1150°c. Also, the shrinkage values are similar in the samples sintered at 1210°c. The water absorption and shrinkage results of the sintered samples at 1200°c are given in Table 5 and Fig. 9. It is obvious from the results that there is similarity of the values of water absorption and shrinkage.



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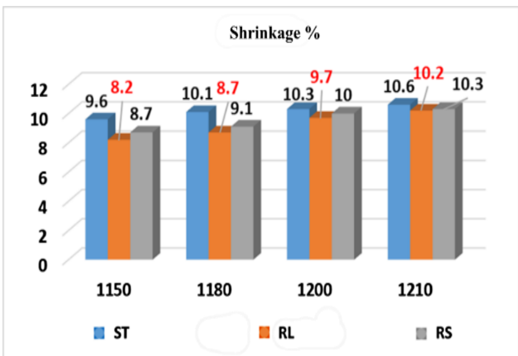

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Fig. 8. The water absorption (A) and shrinkage (B) values of sanitary body samples sintered for 20 hours at various temperatures.

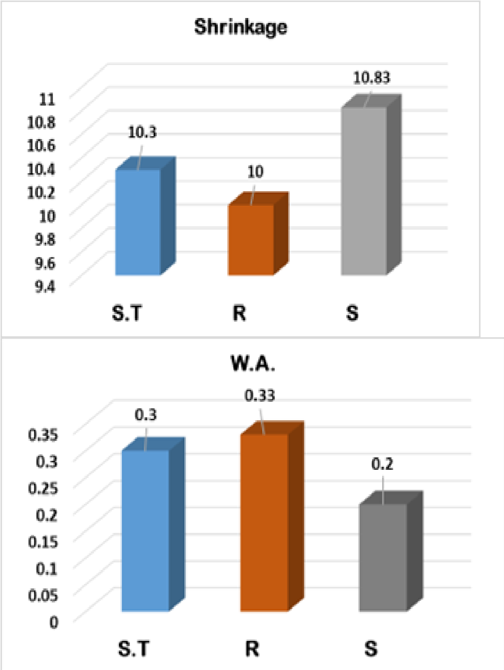

Fig. 9. The water absorption and shrinkage ratios of sanitary body samples sintered at 1200°C/20h.

Conclusions

Rhyolite lava and red syenite were used instead of traditional k-felspar in the manufacture of sanitary bodies. The X-ray diffraction analysis (XRD) of the investigated K-feldspars (F) and both rhyolite lava (RL) and red syenite (RS) show the mineral pattern of alkali feldspars, microcline, albite, anorthoclase and quartz. The thermal behavior of as-received the later samples shows that the softening temperature were 1202°C for k-feldspar,1208°c for rhyolite and 1178°C for red syenite whereas the melting point were 1309°c for k-feldspar,1312°C for rhyolite and 1303°C for red syenite. After sintering the sanitary bodies at 1200°C/20h, quartz and mullite were developed in k-feldspar containing sanitary body and with sillimanite in that containing either rhyolite lava or red syenite. The microstructure of sintered samples of the rhyolite lava and red syenite shows irregular pattern containing submicron and nano-size particles, which refers to the rate at which a material expands with the increase in temperature at 800 °C were 5.29 x 10^{-6} $°C^{-1}$ for k-feldspar, 6.84 x 10^{-6} $°C^{-1}$ for rhyolite and 5.7 x 10^{-6} $°C^{-1}$ for red syenite. The modulus of rupture for samples before firing were 34.9 Kg/cm^2 for k-feldspar, 34.3 Kg/cm^2 for rhyolite and 33.57 Kg/cm^2 for red syenite and their corresponding sintered samples, after firing on 1200°C/20 h were 482.87 Kg/cm^2 for K-feldspar, 445.27 Kg/cm^2 for rhyolite and 481.6 Kg/cm^2 for red syenite because of solidification. The ratio of water absorption values was 0.29% for k-feldspar, 0.33% for rhyolite and 0.19% for red syenite, while the shrinkage ratios were 10.3% for k-feldspar, 10% for Rhyolite and 10.83% for red syenite. The present characteristics and properties indicate that both rhyolite lava and red syenite are suitable for the replace the K-feldspar in sanitary body.

Conflicts of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. “There are no conflicts to declare”.

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