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Utilization of muscovite granite waste in the manufacture of ceramic tiles

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Abstract

Granite waste is by-product from a decorative rock industry. The present study aims to investigate the effect of muscovite granite waste on the physico-mechanical properties of ceramic tiles to demonstrate its suitability for industrial production. A series of flooring- and facing ceramic tiles were prepared by adding 20, 25, and 30 wt% muscovite granite waste into the batch compositions. The sintering behavior and degrees of densification of two kinds of ceramic tiles were evaluated by determining their physico-mechanical properties and characterizing them by scanning electron microscopy (SEM) and X-ray diffraction (XRD), respectively. As expected, the facing ceramic tile (FacCT1) sample, containing wollastonite, hematite, anorthite, quartz, and cristobalite, with 20 wt% muscovite granite waste showed lower physico-mechanical properties than those of the flooring ceramic tile (FloCT3) sample, containing mullite, calcium aluminosilicate, quartz, and cristobalite, with 30 wt% muscovite granite waste. The reason is that lower firing temperatures cannot accelerate a complete fusion of the granite waste which behaves like an inert non-plastic material similar to quartz. These results illustrate the prospects of utilizing muscovite granite waste in ceramic tile production.

Keywords: Ceramic tile; Muscovite granite waste; Physico-mechanical properties; Waste utilization

1. Introduction

Granite is extensively distributed throughout the continental crust of the earth and is the most abundant intrusive felsic igneous rock formed from magma [1]. Because of its unique characteristics, such as high durability and resistance to scratches, cracks, stains, spills, heat, cold, and moisture, granite has found diverse applications in today's life. Due to a rapid growing demand for granite, the processing activities of natural granite rocks have been generating tremendous amounts of granite waste. So far, granite waste has been considered as replacements for conventional ceramic raw materials that are becoming scarce and will be eventually exhausted. For instance, reformulated roofing tiles, in which sand was replaced by granite waste, present lower plasticity that reduces the risk of dimensional defects [2]. According to Binici et al. [3], granite waste aggregates could be used to improve the mechanical properties, workability, and chemical resistance of conventional concrete mixtures. Moreover, granite waste was also investigated and found suitable as a feldspathic raw material that forms glassy matrix at lower temperatures for the manufacture of floor tiles [4,5], porcelainized stoneware and tiles [6,7], red-clay ceramics [8], bricks [9], glass [10], and glass-ceramics [11], and glaze [12,13].

From the economic and environmental points of view, utilization of granite waste in the ceramic industry has proved to be highly efficient because of the resulting reduction in production cost, energy saving, and waste utilization. However, the chemical and mineralogical compositions of granite can vary widely from deposit to deposit, and thereby granite wastes vary in their applications consequently. In this work, the effect of muscovite granite waste on the physico-mechanical properties of the ceramic tiles was therefore investigated. Sintering behaviors and microstructural characterization of the flooring and facing ceramic tiles are presented.

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2. Experimental

Muscovite granite waste with a mean particle size of 34 μm was obtained from Tashkent Marble Factory, Uzbekistan. The chemical compositions of the starting materials used in this work are listed in Table 1. Three flooring ceramic tile (FloCT) compositions were formulated using a mix of kaolin, grog, bentonite, and muscovite granite waste, and similarly, three facing ceramic tile (FacCT) compositions were formulated using a mix of kaolin, grog, loess, and muscovite granite waste as clay and fluxing raw materials, respectively. The batch compositions prepared are given in Table 2. One kilogram batches of each of the above-noted compositions were prepared by the following ceramic process. The raw materials were thoroughly mixed by wet ball milling for 12 h in a porcelain jar using agate balls. The slurry obtained was screened (screen size is 63 μm), dried at 110 °C overnight, powdered, and granulated to small nodules for better compaction using 7% moisture. The batch was uniaxially pressed by a standard laboratory hydraulic press at 30 MPa in the form of rectangles (100 mm × 100 mm × 10 mm) and cylinders (100 mm × 15 mm). The pressed green bodies were dried at 110 °C overnight and fired in the temperature range of 800–1150 °C in an electric muffle furnace. The heating and cooling rate was 5 °C/min, and soaking time at the maximum temperature was 1 h. The physico-mechanical properties were determined using ten specimen bodies for each composition in order to average the data. Bulk density, water adsorption, linear shrinkage, and compressive strength were quantified according to the ASTM Standard Methods [14–16]. The four-point bending strength of the as-prepared specimens was measured using an Instron 8562 universal testing machine (Instron, Canton, MA, USA) at a crosshead speed of 0.5 mm/min, according to the standard procedure [17].

The linear thermal expansion coefficients of the samples were measured using an Orton 1600B dilatometer (The Edward Orton Jr. Ceramic Foundation, Westerville, OH, USA). The sample sizes were 5 mm × 5 mm × 10 mm, and the measurements were performed in the range of 25–500 °C. The microhardness of the samples was measured using an HMV-2000 microhardness tester (Shimadzu, Japan) on polished samples with an indentation load of 200 g for 15 s. The chemical compositions of the samples were analyzed using a RIX2000 X-ray fluorescence spectrometer (Rigaku, Japan). The crystalline phases were identified by X-ray powder diffraction (XRD) using an XRD-6100 diffractometer (Shimadzu, Japan) with monochromated CuKα. The differential thermal analysis (DTA)/thermogravimetry (TG) was carried out on a TG-8120 Thermo Plus Analyzer (Rigaku, Japan) in the temperature range of 20–1000 °C at a heating rate of 10 °C/min in flowing air. The microstructures of the samples were examined using a JSM-5310 scanning electron microscope (JEOL, Japan).

Table 2
Batch compositions and densities of the facing (FacCT) and flooring (FloCT) ceramic tile bodies (wt%).

Sample	Kaolin	Grog	Bentonite	Loess	Muscovite granite waste	Density (g/cm^3)
FloCT1	65	10	5	–	20	1.51
FloCT2	65	5	5	–	25	1.53
FloCT3	65	0	5	–	30	1.57
FacCT1	60	10	–	10	20	1.52
FacCT2	60	5	–	10	25	1.54
FacCT3	60	0	–	10	30	1.56

3. Results and discussion

The average chemical composition of muscovite granite waste is summarized in Table 1. The chemical composition data indicate that muscovite granite waste predominantly contains SiO_2, Al_2O_3, CaO, MgO, Na_2O, and K_2O. In addition to these major constituents, it has also a considerable amount of iron oxides (4.34 wt%). The fluxing (Na_2O and K_2O) and auxiliary fluxing oxides (CaO, MgO, and Fe_2O_3) in the granite waste constitute nearly 18 wt%. The XRD pattern of muscovite granite waste (Fig. 1) shows that the main crystalline phases are

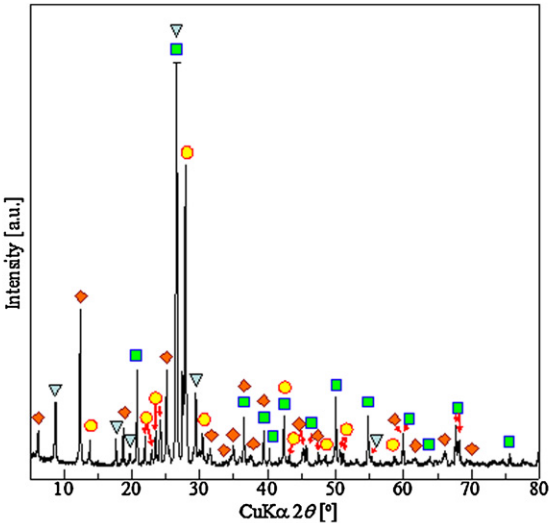

Fig. 1. XRD pattern of muscovite granite waste. *Keys:* rectangle – quartz; circle – anorthite; diamond – chamosite; reverse triangle – muscovite.

Table 1
Chemical compositions of raw materials (wt%).

Product	SiO_2	Al_2O_3	$FeO + Fe_2O_3$	TiO_2	CaO	MgO	Na_2O	K_2O	L.o.I.
Muscovite granite waste	65.10	14.00	4.34	–	6.04	1.12	3.51	2.75	3.14
Kaolin	57.30	26.91	1.10	0.39	0.46	0.45	0.48	0.74	12.17
Grog	42.27	52.08	1.43	1.13	1.44	1.65	–	–	–
Bentonite	67.32	18.96	2.56	0.53	0.84	0.72	1.88	3.19	4.00
Loess	55.00	10.56	7.76	0.48	8.77	4.13	1.59	2.44	9.27

quartz (SiO_2, JCPDS card no. 46-1045), anorthite ($CaAl_2Si_2O_8$, JCPDS card no. 20-0528), chamosite ($(Fe,Al,Mg)_6(Si,Al)_4O_{10}(OH)_8$, JCPDS card no. 21-1227), and muscovite ($KAl_2Si_3AlO_{10}(OH)_2$, JCPDS card no. 07-0025). Probably, the presence of quartz, anorthite (the calcium-rich end-member of the plagioclase feldspar), and chamosite (the most common form of chlorite in granites) originated from the fractional crystallization of granitic rock. Muscovite is the only mica found in the fine-to-medium-grained granites, and its appearance suggests that it may be of primary magmatic origin. According to Neiva et al. [18], muscovite granite might have originated by a thermogravitational diffusion. The DTA–TG curve of muscovite granite waste appears in Fig. 2. The DTA curve shows a wide endothermic effect centered at 893 °C between 750 °C and 1000 °C due to the dehydroxylation of the minerals present in the granite waste. Corresponding to this peak, the TG curve indicates a maximum weight loss of 5.18 wt% at 1100 °C indicative of total dehydroxylation. Mineralogical characterization leads to the conclusion that muscovite granite waste has characteristics similar to the conventional non-plastic ceramic raw materials, namely, feldspar and quartz. Assuredly, the use of muscovite granite waste instead of high-grade conventional raw materials results in a reduction in the production costs of ceramic materials of the desired technological properties.

Water adsorption, bulk density, linear shrinkage, bending and compressive strengths are the main parameters, which are generally used for drawing sintering curves, enabling an optimization of firing cycles to obtain the desired mechanical properties in ceramic tiles. The physico-mechanical properties of the ceramic tiles are listed in Table 3. It can be seen that water adsorption shows average values of about 3.66–4.22% for flooring ceramic tile (FloCT) samples sintered at 1150 °C and 12.35–12.68% for facing ceramic tile (FacCT) samples sintered at 1050 °C. Further, the lowest water adsorption values appearing in FacCT1 (12.35%) and FloCT3 (3.66%) are because of the high degree of vitrification and low porosity caused by raising the firing temperature. Shrinkage is an important property of ceramic tiles, and small alteration that may occur in this property because of sintering temperature can ultimately lead to the related changes in mechanical properties. Table 3 also shows that the densification behavior in terms of bulk density is a function of sintering temperature of the specimens. Densification of the samples reaches the maximum values at 1150 °C and 1050 °C for FloCT3 (2.16 g/cm^3) and FacCT1 (2.02 g/cm^3), respectively. The lower density values observed for the specimens at lower temperatures are directly related to the higher porosity and low particle packing, which with rising temperature are modified by the formation of the glassy phase. The maximum values of linear shrinkage appear in FloCT1 (2.11%) and FacCT3 (2.31%). In contrast, these values are slightly higher than those of ceramic tiles based on carbonate loam (1.0–1.3%) [19] and far lower than those of ceramic tiles containing paper mill sludge and glass cullet [20]. The bending and compressive strengths of the samples dramatically rise with an increase in temperature despite the quantity of the added granite waste. Clearly, the maximum

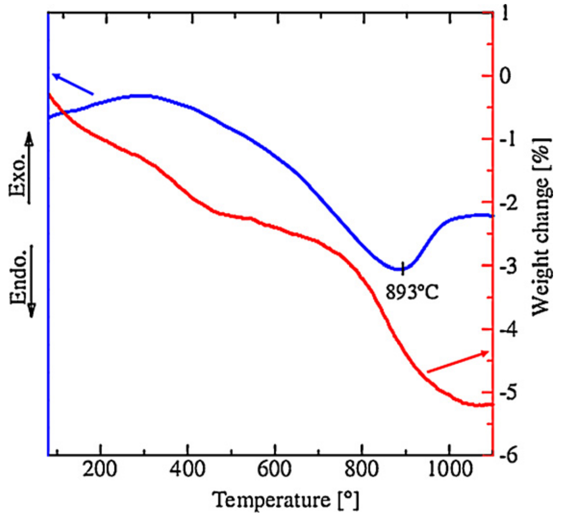

Fig. 2. DTA–TG curve of muscovite granite waste.

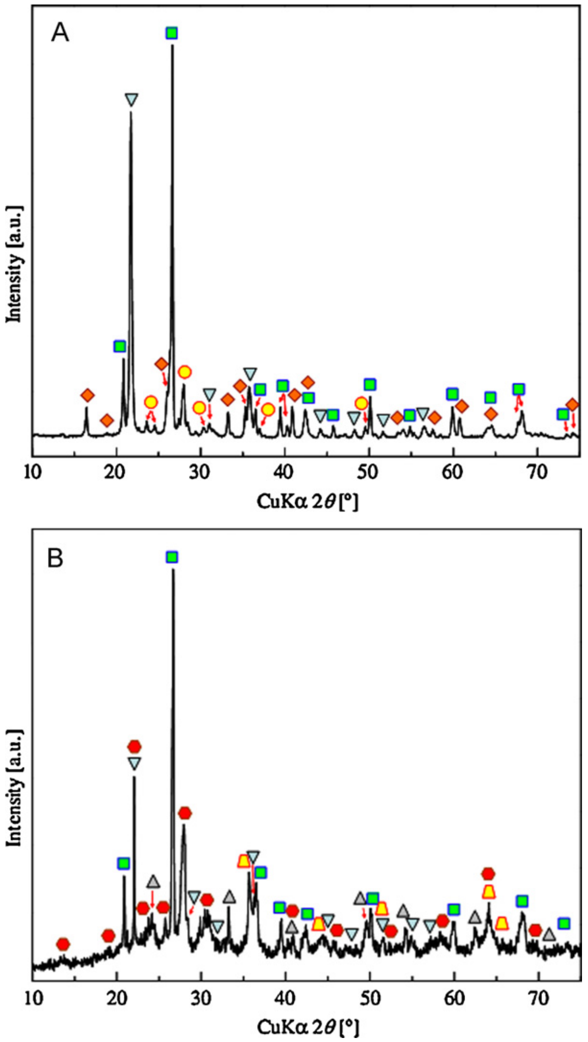

Fig. 3. XRD patterns of the FloCT3 (a) and FacCT1 (b) ceramic tile samples. *Keys:* rectangle – quartz; circle – calcium aluminium silicate; diamond – mullite; reverse triangle – cristobalite; hexagon – anorthite; trapezoid – wollastonite; triangle – hematite.

Table 3
Physico-mechanical properties of the facing and flooring ceramic tile bodies.

Sample	Temperature (°C)	Water adsorption (%)	Density (g/cm^3)	Linear shrinkage (%)	Bending strength (MPa)	Compressive strength (MPa)
FloCT1	800	21.40	1.68	0.20	7.43	15.62
FloCT2		21.21	1.77	0.17	9.04	19.00
FloCT3		20.11	1.90	0.15	9.75	20.49
FloCT1	900	19.71	1.82	0.92	8.83	18.56
FloCT2		19.45	1.85	0.90	9.87	20.75
FloCT3		19.27	1.95	0.84	10.69	22.47
FloCT1	1000	13.42	1.98	1.94	14.28	30.02
FloCT2		12.72	2.03	1.92	16.48	34.64
FloCT3		12.23	2.09	1.80	19.08	40.11
FloCT1	1150	4.22	2.10	2.11	21.06	44.27
FloCT2		3.84	2.12	2.05	23.84	50.12
FloCT3		3.66	2.16	2.00	32.04	54.24
FacCT1	800	21.01	1.84	0.88	6.76	21.03
FacCT2		22.23	1.76	0.90	6.48	18.00
FacCT3		22.54	1.58	0.97	5.89	12.62
FacCT1	900	17.20	1.94	1.22	11.03	30.12
FacCT2		17.82	1.83	1.32	9.94	25.94
FacCT3		17.94	1.72	1.63	8.19	18.84
FacCT1	1050	12.35	2.02	1.98	26.80	48.21
FacCT2		12.49	1.98	2.09	25.56	36.84
FacCT3		12.68	1.96	2.31	21.48	32.35

values of bending and compressive strengths for the FloCT3 sample containing 30 wt% granite waste are 32.04 MPa and 54.24 MPa, respectively. For the FacCT1 sample containing 15 wt% granite waste, the corresponding values are 26.80 MPa and 48.21 MPa, respectively. Probably, the lower firing temperature is responsible for the uncomplete fusion of granite waste, which in FacCT samples then acts as an inert non-plastic material similar to quartz in mechanical strength, and therefore diminishes when the quantity of granite waste is introduced. Similarly, this behavior is consistent with that of the FloCT samples. Considering the relationship between the bending and compressive strengths of the samples sintered at higher temperatures, higher correlation is found in the FloCT samples ($r = 1.0$) rather than in the FacCT samples ($r = 0.9862$). From Table 3, it is found that the FloCT3 and FacCT1 samples demonstrate favorable physico-mechanical properties. Hence, the linear thermal expansion coefficient and Vickers microhardness were determined only for these samples. The linear thermal expansion coefficients (in the range of 20–500 °C) and microhardness of the FloCT3 and FacCT1 samples are 6.3×10^{-6} °C and 6.1 GPa and 7.2×10^{-6} °C and 5.3 GPa, respectively. That the microhardness value of FloCT3 is superior to that of FacCT1, and it might be resulted from the crystalline phases formed besides the densification and vitrification occurring during sintering. The greater difference in linear thermal expansion coefficient could be related to the origins of the different lattice vibrations in both samples. The results presented above confirm that the ceramic tiles prepared utilizing muscovite granite waste satisfy the State Standard (no. 6787-2001) for ceramic flooring tiles and the State Standard (no. 6141-91) for ceramic facing tiles.

The values of linear shrinkage, water absorption, bulk density, bending and compressive strengths correlate with the crystalline phases formed during the sintering process. The XRD pattern shown in Fig. 3a establishes that the FloCT3 sample contains four predominant crystalline phases, mullite ($Al_6Si_2O_{13}$, JCPDS card no. 15-0776), calcium aluminium silicate ($Ca_{0.88}A_{0.12}Si_{2.23}O_8$, JCPDS card no. 52-1344), quartz (SiO_2, JCPDS card no. 04-007-0522), and cristobalite (SiO_2, JCPDS card no. 01-082-0512). Meanwhile, the XRD pattern of the FacCT1 sample (Fig. 3b) shows the presence of wollastonite ($CaSiO_3$, JCPDS card no. 01-088-1922), anorthite ($CaAl_2Si_2O_8$, JCPDS card no. 39-1425), quartz (SiO_2, JCPDS card no. 99-000-3084), cristobalite (SiO_2, JCPDS card no. 39-1425), and hematite (Fe_2O_3, JCPDS card no. 33-0664) as the major crystalline constituents. The XRD patterns of these samples differentiate slightly from each other. The major difference is the absence of the peaks belonging to mullite phase in the FacCT1 sample. Probably, a small amount of mullite was formed in FacCT1 by the reaction of the metakaolinite caused by the dehydroxylation process of kaolin. However, this mullite phase was difficult to detect due to the volume fraction and extremely small size. That might prove to be evidence of the formation of a primary mullite that did not sufficiently develop at lower temperatures. Additionally, it was proven that the formation of anorthite crystals occurs coupled with incipient or primary mullite [6]. This difference in the formation of crystalline phases in the samples is largely attributed to the differences in the characteristics of raw materials and sintering temperatures.

The SEM micrographs of the FloCT3 and FacCT1 samples are represented in Fig. 4a and b, respectively. A study of the

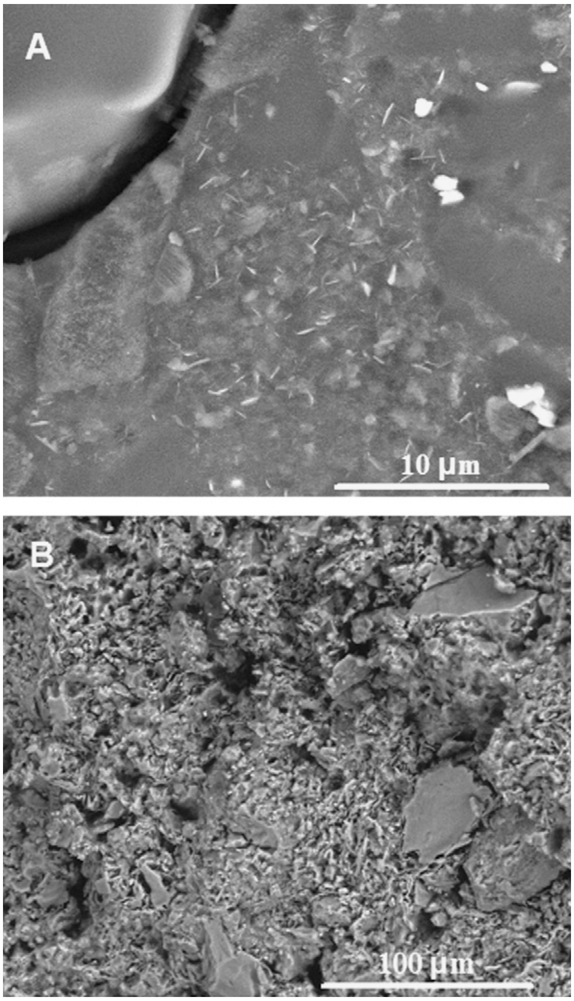

Fig. 4. SEM micrographs of the FloCT3 (a) and FacCT1 (b) ceramic tile samples.

microstructural evolution of both samples reveals that the FloCT3 sample contains small acicular mullite crystallites embedded in the glassy matrix and large undissolved quartz grains remained at the core of mullite clusters, whereas in the FacCT1 sample a large number of wollastonite crystals and residual quartz grains are present. This is the factor that contributes to the higher mechanical strength of the sintered ceramic tiles. The formation of anorthite and wollastonite in the FacCT1 sample, which improves the physico-mechanical properties of the specimens, is the result of the interaction between the CaO and the kaolinite present in the composition. The higher strength of the ceramic tiles can also be explained by an intense process of the formation of mullite, anorthite, and wollastonite during the firing along with the lower concentration of cristobalite (Fig. 3a and b).

The chemical composition of granite waste, apart from the contents of SiO_2 and Al_2O_3, shows a relatively large amount of alkali and alkaline-earth oxides. This evidences the flux potential of granite waste; however, a lower sintering temperature does not support the large amount of muscovite granite waste (25–30 wt%) in the FacCT sample, which behaves as a flux and interacts with the glassy phase to slow down considerably the formation of the crystalline phases. The significant amount of Fe_2O_3 present in the FacCT1 gives the specimen a relatively reddish color after it is fired.

As mentioned earlier, granite waste does not always produce better mechanical performance. Although the granite reduces open porosity, the median pore diameter increases markedly, and coupled with a high amount of quartz, it might introduce large enough pores and micro-cracks that compromise the mechanical strength [21].

It is well known that at a temperature of 650 °C, decomposition of kaolinite with the formation of metakaolinite becomes thermodynamically feasible

$$Al_2O_3{\cdot}2SiO_2{\cdot}2H_2O \rightarrow Al_2O_3{\cdot}2SiO_2 + 2H_2O.$$

The transformation of metakaolinite to mullite becomes thermodynamically favorable at temperatures above 962 °C

$$Al_2O_3{\cdot}2SiO_2 \rightarrow (1/3)(3Al_2O_3{\cdot}2SiO_2) + (4/3)SiO_2.$$

So, mullite begins crystallizing at around 1000 °C, and the β-quartz is transformed to α-cristobalite. Crystallization of cristobalite mostly occurs at the edges of the quartz grains. It must be noted that the addition of alkali oxides to kaolinite retards or even stops crystallization of cristobalite and lowers the formation temperature of mullite [22]. The addition of alkaline-earth oxides likewise lowers the temperature of mullite formation and leaves crystallization of cristobalite unaffected. In the presence of alkali cations, the coordination of Al changes from six to four and the Si–O–Al bonds form in the melt, which do not assist in constructing the lattices of various modifications of silica. When such a structure is present in the melt, the conversion of silica to cristobalite is slowed down.

The formation of anorthite, the most thermodynamically stable product among calcium aluminosilicates, can be explained by the reaction of metakaolinite with calcium oxide, as follows:

$$Al_2O_3{\cdot}2SiO_2 + CaO \rightarrow CaO{\cdot}Al_2O_3{\cdot}2SiO_2.$$

The reaction of metakaolinite with calcium oxide can reduce the formation of mullite crystals. Moreover, the reaction between the mullite and the excess of calcium oxide in the presence of silica forms anorthite phase according to the following reaction:

$$3Al_2O_3{\cdot}2SiO_2 + 4SiO_2 + CaO \rightarrow 3[CaO \cdot Al_2O_3{\cdot}2SiO_2].$$

Along with the formation of anorthite crystals, wollastonite crystals, in turn, precipitate in the FacCT1 sample. Utilization of muscovite granite waste as a fluxing raw material enables a minimization in the consumption of scarce and expensive raw materials, a reduction in production cost and an improvement in the economic and environmental parameters of production.

4. Conclusions

The results presented and discussed in this work enable drawing the following conclusions:

(1) Muscovite granite waste is composed of quartz, muscovite, anorthite, and chamosite. According to its characteristics,

muscovite granite waste resembles conventional non-plastic ceramic materials, and therefore can be beneficially used in the production of ceramic tiles.

(2) The flooring ceramic tile (FloCT3) sample, containing mullite, calcium aluminosilicate, quartz, and cristobalite, with 30 wt% muscovite granite waste showed higher bulk density, bending and compressive strengths and lower water adsorption due mainly to the major crystalline phases and glassy matrix formed during the firing process.

(3) The facing ceramic tile (FacCT1) sample, containing wollastonite, hematite, anorthite, quartz, and cristobalite, with 20 wt% muscovite granite waste exhibited lower physico-mechanical properties compared to those of the FloCT3 sample. The reason was that lower firing temperatures could not accelerate a complete fusion of granite waste, which behaves like an inert non-plastic material similar to quartz.

(4) The ceramic tiles prepared by utilizing muscovite granite waste satisfied the requirements of the State Standard (no. 6787-2001) for ceramic flooring tiles and the State Standard (no. 6141-91) for ceramic facing tiles. Therefore, it can be effectively used in ceramic tile industry as a low cost raw material.

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