Ceramic Wall and Floor Tiles Containing Local Waste of Cement Kiln Dust- Part II: Dry and Firing Shrinkage as well as Mechanical Properties

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Abstract The effect of using the local electrostatic precipitator cement kiln dust waste (EPCKD) collected from Tourah Portland cement factory on the production of ceramic wall and floor tiles was studied. The EPCKD as received from the factory was used to replace gradually a part of the total ceramic batch composition of a traditional wall and floor tile composition (clay, feldspar, limestone, quartz). The thermal properties in terms of dry and firing shrinkage as well as mechanical properties in terms of bending strength of the resulting wall and floor tiles were investigated. The results showed that the EPCKD can be used with an amount of 5 up to 25 wt. % from the total batch as previously illustrated (Part I) without any dangerous adverse effects. In the second part, the results showed that the dry shrinkage was nearly unchanged, while the firing shrinkage increased as the firing temperature as well as the EPCKD content increased to reach 2.8-7.9 % at 1170-1200 °C. The green bending strength was improved and enhanced by the addition of EPCKD to reach 23.43 kg/cm^2 compared with that of the control batch (C0) 12.24 kg/cm^2. The bending strength of the fired articles was also improved and enhanced to reach 474.31 kg/cm^2 compared with 249.09 kg/cm^2 of the control mix. Furthermore, an excellent ability to coloration of the tiles was detected.

Keywords: *cement kiln dust, wall tiles, floor tiles, clay, feldspar, quartz, water absorption, bulk density, apparent porosity*

1. Introduction

Nowadays, the use of Portland cement and the electrostatic precipitator cement kiln dust waste (EPCKD) becomes a matter of a great importance. The electrostatic precipitator cement kiln dust waste (EPCKD) is the main source of air pollution or generally environmental pollution. It resembles the present day problem in many industrial countries due to the accumulation of large quantities of this dangerous waste material that is often characterized by its light weight and very fine particles. It can be easily transported by simple air winds or storms to very far agricultural and/or population areas. In a general sense, ceramics are characterized by its high mechanical strength that makes them more suitable for many specialized structural applications and uses. On this basis, Watchman [1,2] classified ceramics into several categories (Figure 1). Most of the traditional white wares particularly foor tiles falls into the category of polycrystalline aggregates in a glassy matrix for which the mechanical strength as well as physical and thermal properties are greatly influenced by the stresses developed in the glassy phase and corroded relics of quartz grains and numerous small mullite crystals [1,3].

On the other hand, the strengthening of the glassy matrix of the tile bulk composition was increased by increasing its Al_2O_3 and decreasing K_2O contents [4], whilst those of MgO, K_2O and Na_2O are factors governing the maturing temperature, liquid phase formation, crystallization process. As a result, the various properties are developed.

Salteveskaya [5] studied the firing shrinkage and bending strength of ceramic tiles containing some industrial by-products as waste slurry resulting from the production of water glass. This waste material has a high content of SiO_2 but a low content of Al_3O_2. He found that the tiles exhibited a firing shrinkage of 1-1.5 % and bending strength of 13.8-14.5 MPa. Some research studies [6,7] investigated the influence of mining tailings and metallurgical wastes on the physical and mechanical properties of ceramic floor tiles containing it. They indicated that the produced floor tiles had a low porosity and good mechanical strength. The nature and composition of EPCKD waste which contains a high CaO content together with alkali oxides (Na_2O & k_2O) with a considerable part of Fe_2O_3, the EPCKD waste has often a powerful fluxing effect. So, it could be more suitable to be used as a component of ceramic porcelain bodies [8,9]. So, the main objective of the current study is the production of ceramic floor tiles containing a local waste of cement kiln dust (EPCKD) with satisfactory mechanical properties as

well as dry and firing shrinkages to be in the line with the   International as well as Egyptian standards.
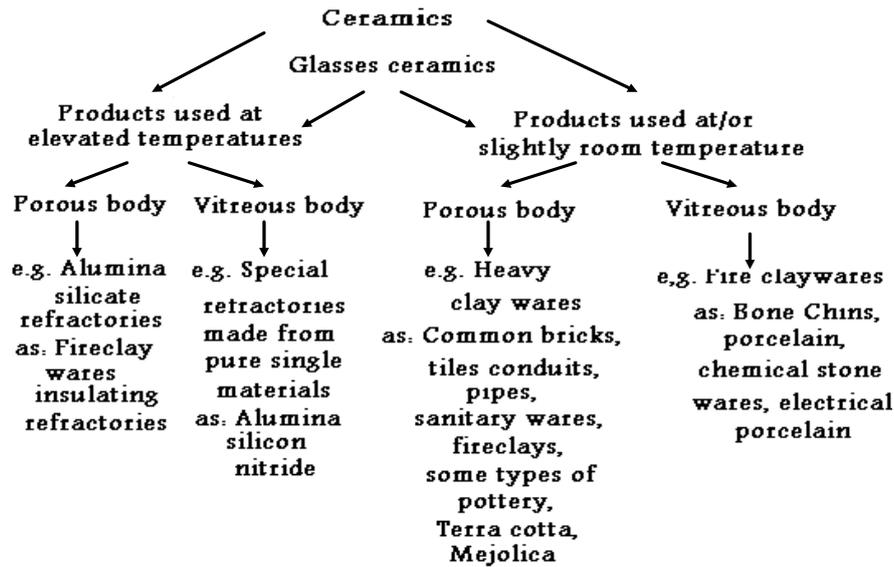

Figure 1. The Classification of Ceramic products

2. Experimental

2.1. Raw materials

The clay sample was taken from Toshka region (TC). Toshka region is located on latitude 20° 30^- N and longitude 31° 53^- E at 250 km south of Aswan which was related to the Upper Cretaceous age. The selected clay deposit is belonging to El-Dakhla Shale Formation (Figure 2). About 20 kg clay was collected from the 85th km north of Aswan/Abu-semple asphaltic road. It is a dark yellowish grey. The clay sample was first well dried in an open air for 2 days and in a suitable furnace at 110 ^{o}C for another 2 days, then crushed, ground and quartered to have a representative sample which was fine ground to pass 200 mesh sieves [10]. Feldspar (F), quartz (Q) from El-Hekma firm, limestone (L) from Samalout district were obtained from the Arab Ceramic Company which is commercially known as Aracemco, Cement kiln dust (EPCKD) which was provided by Tourah Portland Cement Company and a broken fired ceramic waste with the commercial name Grog (G) from the Aracemco ceramic factory, Egypt. The chemical analysis using the X-ray fluorescence (XRF) technique and the particle size distribution of the starting raw materials as well as the suggested ceramic batch composition from the above mentioned raw materials is are previously published in the first part [10].The base batch composition was well mixed in an agate ball mill for one hour using the wet method, let to dry and thoroughly crushed and ground well again to pass through 200 mesh sieve to be the stock base ceramic tile batch. The chemical analysis using the X-ray fluorescence (XRF) technique and the particle size distribution of the starting raw materials are shown in Table 1 and Table 2, respectively. The suggested ceramic batch composition from the above mentioned raw materials is given in Table 3. The base batch composition was well mixed in an agate ball mill for one hour using the wet method, let to dry and thoroughly crushed and ground well again to pass through 200 mesh sieve to be the stock base ceramic tile batch.

Table 1.The chemical composition of the raw materials, wt. %.

Materials Oxides	T-Clay (TC)	Feldspar (F)	Sand (S)	Limestone (L)	Homra (H)	C kiln dust (EPCKD)
L.O.I	9.72	0.67	1.78	42.63	-	24.51
SiO_2	53.47	75.37	93.63	0.08	58.22	12.84
Al_2O_3	26.78	13.62	3.64	0.03	28.25	1.86
Fe_2O_3	3.99	0.41	0.08	0.04	8.16	1.53
CaO	0.60	0.53	0.18	56.84	0.79	52.51
MgO	1.38	---	---	0.10	0.46	1.84
MnO	0.03	0.03	0.02	---	---	---
K_2O	1.18	5.84	0.14	0.05	1.46	1.65
Na_2O	1.15	3.44	0.17	0.12	1.32	1.83
TiO_2	1.12	0.05	0.16	0.01	1.34	---
SO_3	---	0.02	0.14	0.02	---	2.43
P_2O_5	0.51	---	---	---	---	---
Cl^-	---	0.02	0.06	0.08	---	---

Figure 2. Map indicates the area from which the clay sample was taken

Table 2. The particle size distribution of the raw materials, wt. %

Range Raw materials	$< 71\ \mu m$	$100\text{-}71\ \mu m$	$>100\ \mu m$
TC	87.33	7.78	4.89
Feldspar (F)	89. 41	9.52	1.07
Silica (S)	98.67	1.21	0.12
Limestone (L)	96.78	2.93	0.39
Grog (G)	99.42	0.36	0.22
EPCKD	93.25	5.21	1.54

Table 3-The base batch composition, mass %

Materials	Clay	Quartz	Feldspar	limestone	Grog
Wt.%	46.04	18.71	18.71	9.35	7.19

2.2. Preparation and Methods

Six ceramic batches containing TC, F, L, Q, G and EPCKD were prepared as 100:0, 95:5, 90:10, 85:15, 80:20 and 75:25 mass % having the symbols C0, C1, C2, C3, C4 and C5, respectively. The batches were mixed well in a gate ball mill for one hour using the wet method, dried at 105°C for 72 hours and then ground to pass a 200 mesh sieve to obtain the same homogeneity of all batches. Five disc-shaped samples of 2 cm diameter and 2 cm thickness were prepared for the physical properties in terms of water absorption (WA), bulk density (BD) and apparent porosity (AP), five rod-shaped samples of 2.5 x 2.5 x 7 cm^3 for dry and firing bending strength and five cubes of 2.5 x 2.5 x 2.5 cm^3 for crushing strength were moulded. The molding of specimens were carried out under a shaping pressure of 20 KN/mm^2 using water as a binder. After demoulding, the samples were let to dry in air (23 ± 2°C) for 48 hours and then dried to a constant weight at 105°C in a suitable oven to ensure the complete elimination of the free water and to avoid the cracks during firing. The firing process was carried out by a slow rate furnace Mod. VECSTAR with a heating rate of 5°C/min. The firing temperatures were in the range of 1000 and 1250 ºC with one hour soaking time. The fired specimens were left to cool slowly inside the furnace over night to room temperature [10].

The optimum firing temperature of each ceramic batch was also estimated.

2.3. Mechanical Properties

The physical properties in terms of water absorption, bulk density and apparent porosity were investigated in a previous study [10]. The mechanical properties in terms of bending strength (BS) [11,12] of the green and fired units could be carried out using the three point adjustments system (Figure 3) and is calculated from the following equations:

$$\mathbf{B}.\,\mathbf{S}, \mathbf{kg/cm} = 3(\mathbf{BS})/2(\mathbf{W})(\mathbf{T})\,\mathbf{Kg/cm^2}/10.2\ \mathbf{MPa}\ (1)$$

Where,

B. S is the bending strength, kg/cm^2, B is the load of rupture, kg, S is the Span (the distance between the two lower beams, 5 cm), W is the width of the sample, cm and T is the thickness of the sample, cm.
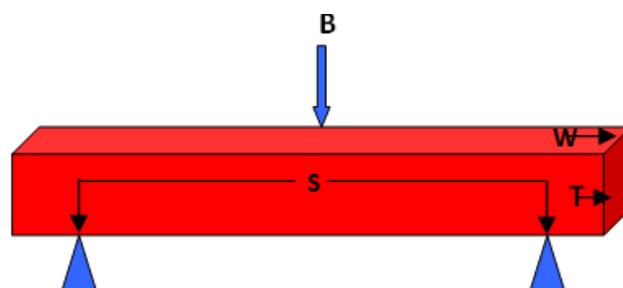

Figure 3. Schematic diagram of the bending strength, B: beam, S: span, W: width and T: thickness,

3.4. Dry and Firing Shrinkage

The tested specimens of the base batch (C0) and those containing 5-25 wt. % EPCKD waste (C1-C5) were subjected to drying and firing processes to follow the drying and firing shrinkages for each batch. At least 30 measurements for three representative samples of each firing temperature were taken. The dry and firing

shrinkage [13] of the dried and fired units could be measured from the following equation:

$$F.S,\% = (Lo - L)/(Lo) x100 \qquad (2)$$

Before firing, the diameter of each disc specimen was measured from more one side which was represented by (Lo). The measurements on the same disc after firing were done by the same way for each temperature interval and the average value was designated by (L) according to ASTM-Standards-C326 [13].

2.5. Coloration Ability

In order to test and determine the ability of each batch to coloration, suitable ceramic pigments, color spinel and stains were mixed with the raw mix of the best ceramic batches mixed by a quantity of 3-5 wt. %, then pressed and fired at a temperature lower than that obtained before owing to the fluxing effect of the pigment, spinel or stain additives. The fired specimens were tested for different colors to obtain the ability of numbers of colors as yellow, blue, blue, brown and violet [14,15,16,17].

3. Results and Discussion

Generally, the characterization of the raw materials and the densification parameters in terms of water absorption, bulk density and apparent porosity were previously studied [10].
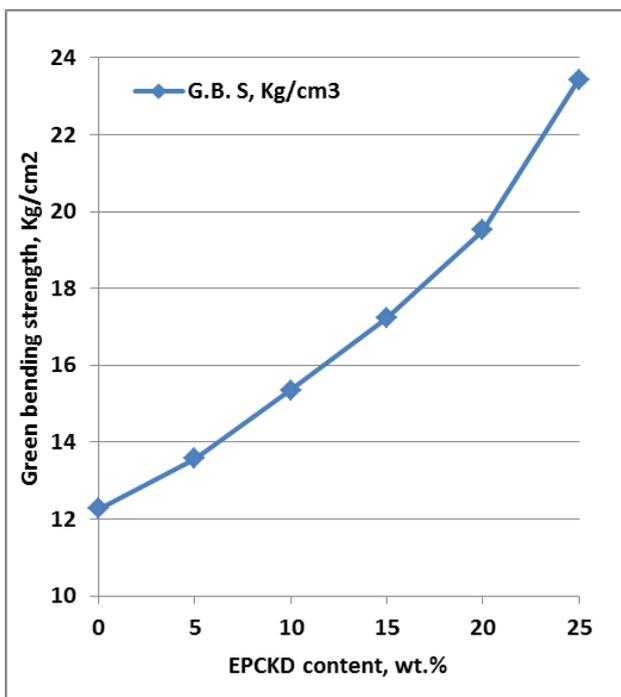

Figure 4. The Green bending strength of the dried samples containing 0-25 wt. % EPCKD waste

3.1. Mechanical Properties

The bending strength of the green (unfired or dried) specimens was plotted as a function of EPCKD content in Figure 4, while that of the fired bodies was plotted as a function of firing temperature in Figure 5. The results revealed that the green bending strength of the control (C0) was slightly enhanced and much improved with the gradual addition of EPCKD waste. The green bending strength of the base batch (C0) recorded 12.27 Kg/cm^2, while that of the batch containing 25 % EPCKD waste (C5) was 23.43 Kg/cm^2. This represents a higher improving rate (Figure 4). This is mainly attributed to that the EPCKD waste contains bonding phases and fluxing oxides that increased both workability and bending strength of the bodies containing it [2,16,18,19].
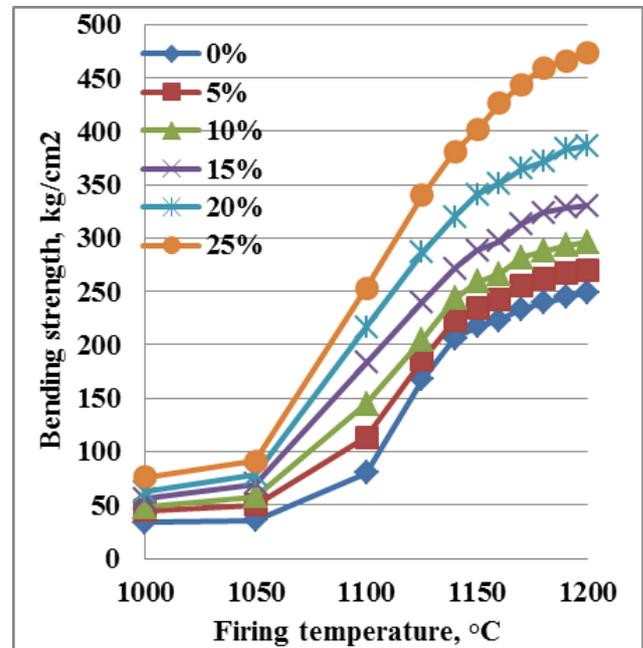

Figure 5. Bending strength of fired ceramic bodies containing 0-25 wt. % EPCKD waste as a function of firing temperatures from 1000-1200 °C

On the other side, the bending strength of the fired bodies increased as both the EPCKD waste content increased up to 25 wt. % and also as firing temperature increased up to 1200°C. The fired ceramic bodies containing 25 wt. % EPCKD waste (C5) at the maturing temperature of 1150 °C achieved bending strength value of about 401.32 kg/cm^2 compared with that of the base batch (C0) fired up to 1200°C which recorded only 249.09 kg/cm^2. This means that the improvement and enhancement in bending strength value is equal to 61.11 %. It is well known that the mechanical properties of the fired ceramic tiles are dependent on the porosity and crystalline grain size. Hence, the grain size of the starting raw materials must be very fine to give considerable low particle boundaries which in turn resulted on low porosity [10,16,20,21,22]. This would be enhanced with the formation of glass phases which may be containing mullite, gehlinite and anorthite crystals [2,16,21].

Moreover, the rate of either densification or strength of the fired ceramic bodies could be increased by the addition of EPCKD waste to reduce the total porosity of the formed glassy matrix during firing which solidifies on cooling. This in turn could cement all the unmelted and/or unreacted particles and crystals together to give good mechanical properties for the resulted bodied [22,23]. The influence of alkalies and earth alkalies in EPCKD waste can alter the relative amounts of the formed mullite and liquid phases and lower the viscosity of the liquid. The used intimate mixing improves the latter process and reduces the maturing temperatures [2,16,21,22,23].

3.2. Dry and Firing Shrinkage

The dry and firing shrinkage of the dried and fired ceramic tiles are graphically represented as a function of firing temperature in Figure 6. The results indicated that the values of dry shrinkage of the tested samples are so very low (0.02 up to 0.05 %) that it seems to be zero As a result, it could be neglected. The firing shrinkage on the other hand increased slightly with firing temperature as well as with EPCKD waste content. Moreover, all tested samples were displayed the same trend either with firing temperature or with EPKD content. Also, it could be noticed that the values of firing shrinkage are being low with the lower temperatures (1000-1100 °C) and increased more and more with the increase of it (1100-1200 °C). This is mainly attributed to the increase of the alkali content of the EPCKD waste (Na₂O + K₂O = 3.48 %). The presence of the high content of CaO (52.51 %) as carbonates, sulphates, silicates reduced the melting point and increased the formation of glassy phase during firing which in turn increased the firing shrinkage by increasing both firing temperature and the EPCKD content [2,17,24].
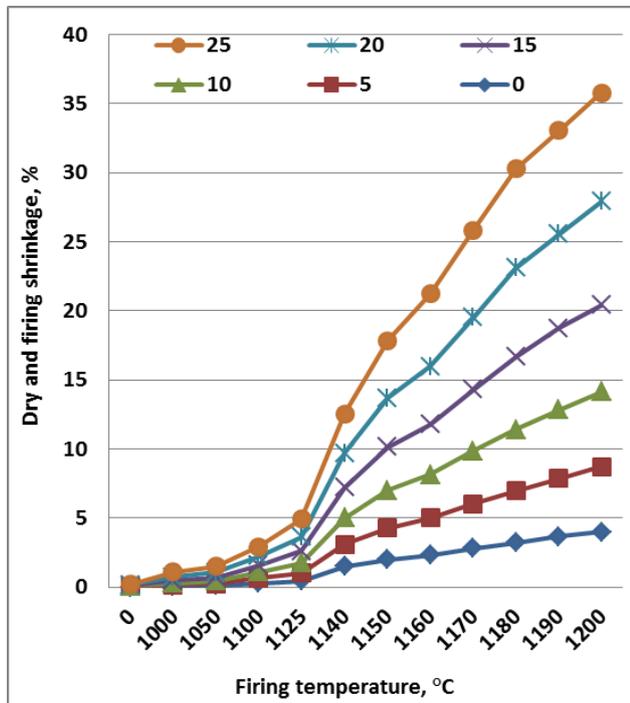

Figure 6. Dry and firing shrinkage of ceramic tiles as a function of firing temperature

3.3. Ability to Coloration

The ability of the prepared ceramic batches containing 0-25 wt. % CKD waste to coloration was investigated by the addition of 3-5 wt. % ceramic color pigments and/or glaze spinel colors during milling and mixing process before moulding and firing. The used colors are blue, yellow, brown, and green. The moulding discs containing coloring oxides were fired to their maturing temperatures. So, it is clear that the ability for coloration was successful for all ceramic batches containing CKD waste. The obtained colors were well distributed, very intense and with no change in its shading or gradation. So, the composition has no effect on the colors produced, i.e. the Fe_2O_3 and TiO_2 were bleached and compensated to be

controlled without any influence on the color of the used pigments which in turn proved the combination of Ca with Fe during firing. Therefore, it is prevented its effect on the coloring of used pigments.

3.4. General Discussion

Generally, clay minerals are often formed from the decomposition of igneous rocks as granite which is composed mainly of roughly equal proportions of potash mica ($K_2O. 3 Al_2O_3. 6SiO_2. 2H_2O$), quartz ($SiO_2$) and potash feldspar ($K_2O. Al_2O_3. 6SiO_2$). Kaolinitic clay was due to the kaolinization of feldspar in presence of air and water [1,14,25,26] as follows:
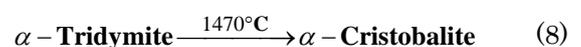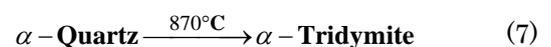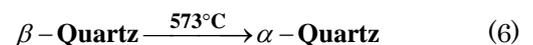
$$K_2O.3Al_2O_3.6SiO_2 + 2H_2O$$
$$\rightarrow Al_2O_3.2SiO_2.2H_2O + K_2O + 4SiO_2 \qquad (3)$$
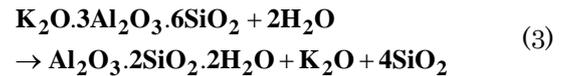
The most important clay mineral for potters is kaolinite ($Al_2 Si_2 O_5 (OH)_4$ or $Al_2O_3. 2SiO_2. 2H_2O$ or AS_2H_2), which appears as flat and extremely minute hexagonal plates. The crystal size may vary from 5 μ to 1 μ (or 10^{-4} meters), which in turn is responsible for their extreme properties [14,22,27,28,29,30,31] as the plasticity or workability which simplifies the manufacture of clay wares and also account for the dry strength of the unfired article. So, this assists in reducing handling losses. At the temperature range 100-200 ºC, the volume of certain argillaceous minerals shrinks as a result of the water loss causing a dimensional change. The oxidation of organic materials occurs at the temperature range 200-300 ºC [1,26].The Kaolinite is a decomposition product of feldspar which starts to decompose in the temperature range 450-500 ºC to metakaolin ($Al_2O_3. 2SiO_2$ or AS_2) and water vapor[25,31]as follows:

$$Al_2O_3.2SiO_2.H_2O \rightarrow Al_2O_3.2SiO_2 + 2H_2O \qquad (4)$$

The reaction is accompanied by an expansion, i.e. the ware becomes larger. At about 980 ºC, a sudden evolution of heat accompanies the complete breakdown of the structure with the formation of mullite ($3Al_2O_3. 2SiO_2$ or A_3S_2) and quartz minerals as follows:

$$Al_2O_3.2SiO_2 \rightarrow 3Al_2O_3.2SiO_2 \qquad (5)$$

The transformation of quartz to cristobalite is slow and dependent on the temperature and time of firing. The amount of formed cristobalite for a particular firing temperature depends largely on the size of the quartz particles. At 573 °C, β-quartz converts to α–quartz which converted to α–Tridymite at 870 °C. The last phase starts to convert to –Cristobalite from 110-1470 °C. The general scheme of the polymorphous transformation of quartz at different temperatures [26,32] can be represented as follows :-

$$\beta-\textbf{Quartz} \xrightarrow{573°C} \alpha-\textbf{Quartz} \qquad (6)$$

$$\alpha-\textbf{Quartz} \xrightarrow{870°C} \alpha-\textbf{Tridymite} \qquad (7)$$

$$\alpha-\textbf{Tridymite} \xrightarrow{1470°C} \alpha-\textbf{Cristobalite} \qquad (8)$$

Also, there are two feldspars namely, orthoclase-potash feldspar, $KAlSi_3O_8$ or $K_2O. Al_2O_3. 6SiO_2$ and albite-soda feldspar, $NaAlSi_3O_8$ or $Na_2O. Al_2O_3. 6SiO_2$, in addition to small amounts of mica and quartz. Feldspar starts to melt

at a high temperature depending on the total alkali content and the particle size of the material.

Limestone is an important ingredient in wall tiles due to its ability to produce bodies of very low moisture expansion which it is particularly very important in tiles, where the highly porous body would otherwise be prone to absorb moisture and expand [1,25]. Limestone is often used at about 10-12 wt. % in tile bodies to reduce the amount of glassy phase during firing. Its behavior during firing is more complex and so, it cannot be classified simply as a filler or flux. On 700-900 °C, it decomposes to CaO and $CO_2\uparrow$ as follows:-

$$CaCO_3 \rightarrow CaO + CO_2 \uparrow \qquad (9)$$

Then, the resulting CaO can react with other constituents to form anorthite-lime feldspar ($CaAl_2Si_2O_8$) rather than enter into the glassy phase. The reduction in glass content virtually eliminates firing shrinkage [33].

4. Conclusion

The total substitution amount of EPCKD waste is limited by 5-25 wt. %. The dry shrinkage is too small and therefore it could be neglected. The obtained firing shrinkage is very small with the lower firing temperatures (1000-1100°C). At higher firing temperatures (1170-1200°C), the firing shrinkage lies in the range 2.8-7.9 %. The values of the dry and firing bending strengths as well as the maturing temperature of each batch at every firing temperature are gradually increased with the increase of EPCKD content and also with the increase of firing temperatures.

The ability for coloration was successful for all ceramic batches containing EPCKD waste. The resulting colors were very intense and well distributed. The used ceramic composition has no effect on the colors produced, i.e. the Fe_2O_3 and TiO_2 were bleached and compensated to be controlled without any influence on the color of the used pigments which in turn proved the combination of Ca with Fe during firing. Therefore, it is prevented its effect on the coloring of the used pigments.

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