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AN INVESTIGATION ON UTILIZATION OF DOĞANHİSAR/KONYA CLAYS IN GLAZED PORCELAIN TILE BODIES

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Highlights

- Technical properties of 3 different clays from Doğanhisar/Konya region were determined in order to investigate their usage potential in glazed porcelain tile production.
- Use of Konya clays instead of Ukrainian clay in tile body recipes increased the strength of fired bodies.
- Konya based clays could be used in bodies up to 6 wt.% of tile recipes.

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ABSTRACT: In this study, technical properties of 3 different clays from Doğanhisar/Konya region were determined to investigate their usage potential in glazed porcelain tile production of Yurtbay Seramik Company. For this purpose, mineralogical, chemical, and thermal characterization of the clays was performed using XRD, XRF, and dilatometric analyses. Furthermore, the use of Konya clays instead of Ukrainian clay in the porcelain tile compositions was studied. Ukrainian clay was substituted gradually by Konya clays in different tile body recipes. Samples, having these new compositions, were prepared by uniaxial pressing, and exposing to a firing regime at 1190 °C for 55 min. Fired samples were then characterized based on their dry strength, fired strength, water absorption, color, and firing shrinkage properties. Experimental results proved that the use of Konya clays instead of Ukrainian clay increased the strength of fired bodies. It would be possible to use Konya clays in tile bodies up to 6 wt.% with some modifications in the body compositions and firing regime.

Keywords: *Ceramic, Konya Clay, Tile, Ukrainian Clay*

1. INTRODUCTION

Porcelain tiles are produced using naturally occurred raw materials in nature such as quartz, clay, and feldspar. Among these raw materials, clay is widely abundant in the world and is one of the oldest crafting and building materials [1]. On the other hand, clay minerals used in the production of porcelain tiles can vary in structure and chemical content since they are natural materials having different origins. As an example, Ukrainian clays were characterized with their low iron content, good sintering properties, and high plasticity whereas eastern and central Europe-based clays were characterized with their medium plasticity and weak sintering properties [2]. Hence, mostly Ukrainian clays are preferred by the ceramic industry in Turkey. Porcelain tile body compositions generally consist of 25-40 wt.% plastic clay. So, the high price of the Ukrainian clays and of their transportation has a significant effect on the manufacturing cost of porcelain tile products. In addition, the production quantities of ceramic tile industry grow year by year, and as a result the need for raw materials also increases at the same rate.

Due to the decrease in raw material reserves, the supply of quality raw materials in the ceramic tile industry is becoming increasingly difficult. Therefore, ceramic tile industry in Turkey is in a search of local clay sources to minimize their dependency on foreign raw material import, to decrease raw material and product costs, and to secure their clay sources for prudential reasons. But the recent geopolitical uncertainties in the supply of Ukrainian clays as well as the decrease in the quantity of local clays and environmental problems related to their mining lead the ceramic industry to search for alternative clay sources.

There are some research studies for the alternative clay sources that can be utilized in the production of ceramic products, such as Nevşehir- [3, 4], Afyon- [5, 6], Çanakkale- [7], Denizli- [8], and Konya-based clays [9]. Apart from the tile products, Konya-based clays were also tested for ceramic primer applications in artistic ceramics [10]. Usage of waste materials as alternative raw materials in ceramic products was also studied in literature [11, 12]. Ceramic tile manufacturers supply clay mainly from Şile (İstanbul) and

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Söğüt (Bilecik) regions, which are the two largest clay fields of Turkey. Apart from these regions, the regions with secondary clay potential are Afyon, Kütahya, Çanakkale, and Konya. Konya-based clays are generally used in the production of bricks, pottery, and tiles. Aim of this study was to investigate the possibility and suitability of regional clays (Doğanhisar/Konya, Turkey) for fabrication of porcelain tile products by replacing the currently used Ukrainian clay in the tile recipes. 3 different Konya-based clays were added to the standard porcelain tile body recipe of Yurtbay Seramik (Eskişehir) instead of Ukrainian clay. Physical, chemical, mechanical, and thermal properties of both the clays and the tile bodies were investigated. Tarhan also studied the use of a Doğanhisar region clay in porcelain tile body compositions [9]. But the clay used had a different chemical composition and physical properties compared to the clays used in this study.

2. MATERIAL AND METHODS

2.1. Characterization of Clay Samples

In this section, mineralogical, thermal, and physical analyses were performed on 3 different clays (K1, K2, and K3) collected from Doğanhisar region of Konya. X-ray diffraction (XRD) was used for the phase identification of clay samples. Prior to measurement, samples were ground in an agate mortar and sieved through 125-micron (120 mesh) sieve. A Europe-Gnr model diffractometer was used with Cu-Kα radiation; the diffraction data were collected over the 2θ range from 5° to 70° with a scanning rate of 2°/min. Quantitative analysis of the phases detected by XRD was performed using the MDI JADE software package. It was assumed that the phase content of the clays was entirely accounted for by the XRD patterns. In addition to XRD, chemical analysis was also performed to identify the mineralogical content of clay samples. Bulk chemical composition was determined using a Panalytical/Axios MAX model wavelength dispersive X-ray fluorescence spectrometry (WD-XRF). Loss on ignition (LOI) values were obtained via mass data monitored before and after a calcination process performed at ~1000 °C.

Physical properties of fired clays were investigated by preparing disc samples pressed at 40.79 kg/cm^2 in a cylindrical die with 50 mm diameter. Then, these discs were fired at 1190 °C for 39 min in a roller kiln at Yurtbay Seramik Company. Water absorption of the fired samples was tested using water impregnation method (ISO 10545-3) in immersion under vacuum (Essepienne VSVD/120) [13]. Firing shrinkage values were calculated by measuring sample dimensions before and after the firing process. An X-rite Ci62 model spectrophotometer was used to measure the chromatic coordinates (L*, a*, and b* values) of the fired clay samples. Thermal expansion coefficient (TEC, α) of clays were determined using a Toledo SDTA841 model thermomechanical analyzer. Measurement was performed by heating the pressed clay samples from 20 °C to 600 °C with a heating rate of 10 °C/min, and calculating the TEC value at 400 °C.

2.2. Characterization of Ceramic Bodies

New porcelain tile body formulations were prepared to determine the effect of Konya clays on the technical properties of ceramic bodies. Using these new formulations, suitability of Konya clays instead of Ukrainian clay was also investigated. For this purpose, Konya clays were added in different ratio instead of Ukrainian clay in the standard tile recipes of Yurtbay Seramik. Ratio of raw materials (in weight percentage) in the standard (Std) and modified recipes were listed in Table 1.

Ceramic slurries were prepared according to the body recipes by wet milling. Then, they were dried, ground, sieved to <63 μm, and moisturized with 5-6 wt.% water to obtain granulated powders. Samples were formed by pressing the powders in a 100 x 50 x 5 mm rectangular die under 50.99 kg/cm^2 using a Gabrielli brand press. Some of these samples were used to determine the sintering behavior and dry (green) strength values. Other samples were fired at 1190 °C for 55 min in the industrial roller kiln. Fired samples were tested with respect to their phase content, fired strength, and thermal expansion. 3 samples were prepared for each recipe for the strength and thermal expansion tests to increase the reliability of the data.

Table 1. Raw material fractions (wt.%) of the tile body recipes.
(*Other Raw Materials: clays & feldspars (from different regions in Turkey), kaolinite, silica sand, magnesite, and Na_2SiO_4)

Recipes	Ukrainian Clay	K1	K2	K3	Other raw materials*
Std	17	-	-	-	83
R1	11	-	-	6	83
R2	6	-	-	11	83
R3	-	-	-	17	83
R4	11	6	-	-	83
R5	6	11	-	-	83
R6	-	17	-	-	83
R7	11	-	6	-	83
R8	6	-	11	-	83
R9	-	-	17	-	83

Granulated powders were also pressed in a 50 mm die at 40.79 kg/cm^2 to obtain disc shaped samples, to be used for the investigation of physical properties such as water absorption, firing shrinkage, and color developed after firing. Phase content of fired bodies were analyzed using a Europe-Gnr model XRD with Cu-Kα radiation; the diffraction data were collected over the 2θ range from 10° to 70° with a scanning rate of 2°/min. Quantitative analysis of the phases detected in the fired bodies was performed using the MDI JADE software package. Dry strength tests were performed with a 3-point bend testing machine in Yurtbay Seramik (Gabrielli Flexi 150). A Shimadzu AGS-X model universal testing machine was used for the fired strength measurements. Water absorption, firing shrinkage, and color of fired samples were determined using the same methods mentioned in Section 2.1 (Characterization of Clay Samples). TEC values were measured with a mechanical dilatometer (DIL 402 PC Netzsch) using cylindrical samples with 6 mm diameter and 25 mm length. Sintering behavior of ceramic bodies were investigated via an optical dilatometer (Misura 3 ODHT). For the measurements, samples were heated from room temperature to 1300 °C using 50 °C/min heating rate.

3. RESULTS AND DISCUSSION

3.1. Clay Samples

XRD patterns of Konya clays as well as Ukrainian clay are presented in Figure 1. They prove that the main phases of Konya clays are quartz (JCPDS no:05-0490), illite (JCPDS no:26-0911), and kaolinite (JCPDS no:06-0221). On the other hand, Ukrainian clay is rich in kaolinite and poor in illite content.

Contrary to the similar XRD patterns of Konya clays, their appearances are significantly different from each other as seen in the photograph given in Figure 2. Especially K1 clay has a darker color than the other 2 clays. A similar color formation is also observed in subclays at coal fields. In a research study on Ilgın/Konya region coal fields, a similar XRD pattern (to the pattern of K1 clay) was obtained for the lower clays formed underneath the coal level and named as "black subclay" [14]. The pattern mainly contained quartz, illite, and kaolinite as well as pyrite mineral (FeS_2). Black color was due to the existence of pyrite mineral in the subclay. Phase content of K1 clay (collected from Doğanhisar) and subclays (in Ilgın) might be similar since these two regions are very close to each other (~30 km). However, a clear peak of pyrite phase cannot be observed in the XRD pattern of K1 clay. This may be due to the pyrite content in K1 clay probably being lower than the XRD detection limit. Elemental compositions of clays were measured by XRF, and the results are shown as oxides in Table 2. K1 clay has higher amount of sulfite, compared to other 2 clays. This proves the possible existence of compounds, such as pyrite, in K1 clay causing a darker appearance.

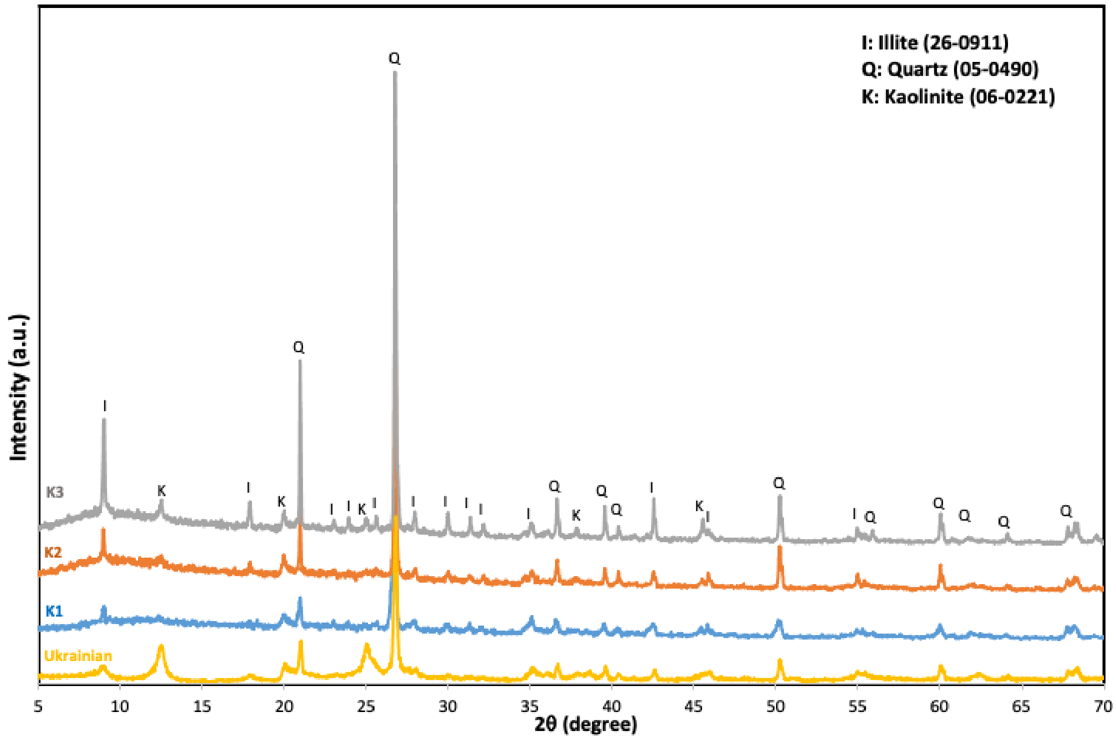

Figure 1. XRD patterns of clay samples.

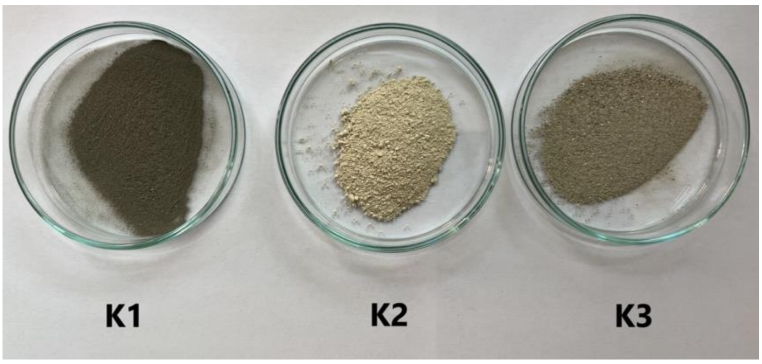

Figure 2. Clay samples collected from Doğanhisar/Konya region.

Physical properties of clay samples after firing are listed in Table 3. Water absorption value for Ukrainian clays in literature is given as a range between 0.2% and 2.0% [15]. Konya clays exhibit higher water absorption than the Ukrainian clay used in this study. This is due to the differences between Konya clays and Ukrainian clay in their phase content and structure. Clays are classified as layer- and chain-structured based on the arrangement of silica and alumina layers. In layer-structured clays, this arrangement can be either in an order or disorder. In the latter case, mixed-layer clay minerals are formed. Ukrainian clays exhibit a mixed-layer formation [16]. As shown in Figure 1, Konya clays are rich in illite mineral which has a 3-layered structure (2:1 type) and a theoretical chemical formula of $K_{1-1.5}Al_{4-3.5}(Si_{7-6.5}Al_{1-1.5})O_{20}(OH)_4$ [17]. On the other hand, kaolinite, the main constituent of Ukrainian clays, is a 2-layered (1:1 type) mineral. Compared to 2-layered minerals, clays with 3-layered structure can hold more water between these layers. Among the 3 Konya clays investigated in this study, K1 clay has a higher water absorption value than that of other 2 clays.

Table 2. Oxide content and loss on ignition (wt.%) of Ukrainian and Konya clays.

	K1	K2	K3	Ukrainian Clay
SiO_2	65.49	68.75	77.62	59.68
Al_2O_3	21.20	18.81	13.17	26.56
K_2O	2.98	2.41	1.65	2.00
TiO_2	1.00	0.99	0.65	1.25
Na_2O	0.97	0.31	0.24	0.57
Fe_2O_3	0.84	1.52	0.82	1.09
MgO	0.54	0.63	0.42	0.39
P_2O_5	0.16	0.40	0.12	-
CaO	0.11	0.32	0.09	0.62
ZrO_2	0.06	0.08	0.04	-
SO_3	0.09	0.04	0.02	-
LOI	6.56	5.69	5.13	7.84

Table 3. Physical properties of fired clay samples.

	Ukrainian Clay	K1	K2	K3
Water Absorption (%)	1.0-1.5	8.5-9.0	7.2-7.9	7.0-7.5
Firing Shrinkage (%)	7.5-8.0	3.4-3.7	3.3-3.9	3.0-3.5
Chromatic Coordinates (L*-a*-b*)	82.00-2.20-13.00	82.36-0.88-12.71	78.49-2.75-21.65	78.00-3.20-20.00
TEC (α_{400}x10^{-7} °C^{-1})	61.80	78.25	80.08	78.80

Firing shrinkage value for 3 clays are in a range of 3-4% and it fits well with the standard value desired for the clay raw materials used in the ceramic tile industry. Chromatic coordinates are also given in Table 3. K1 clay has lower a* and b* values than that of other 2 clays. Therefore, a different color development can be expected for the fired K1 clay sample, compared to the color of other 2 clay samples. Besides, K2 clay has the highest b* value (more yellowish appearance). Both conditions are also in good agreement with the clay appearances shown in Figure 2. According to the quantitative analysis of the phases in XRD patterns in Figure 1, amount of quartz in clays is in order of K2>K3>K1>Ukrainian. Since crystalline quartz has a very high thermal expansion coefficient [18], Konya clays have higher TEC values than that of Ukrainian clay as given in Table 3.

3.2. Ceramic Bodies

Among the ceramic body recipes prepared in this study, R2, R5, and R8 (recipes with a mixture in which the amount of Konya clay is higher than Ukrainian clay) were selected for the phase analysis of ceramic bodies. XRD patterns of these mentioned recipes and the standard recipe are shown in Figure 3.

Microstructure of a porcelain-ware mainly consists of newly formed phases after firing (e.g., mullite) as well as residual phases (quartz and feldspars) dispersed in a glassy (vitreous) phase [19]. There is an obvious increase in the intensity of mullite peaks with the addition of Konya clays in the bodies instead of Ukrainian clay. This increase is more significant for R2 (K3 clay) and R8 (K2 clay) bodies. In addition, there is also an increase in the intensity of peaks belonging to residue feldspars after firing. McConville et al also observed a much higher proportion of fluid amorphous phases for 2:1 type clays (e.g., illite) than the kaolinite based clays. This result was related to the existence of feldspars and the fluxing action of the cations located between 2:1 layers. This caused the formation of larger mullite crystals than the mullite in kaolinite samples, at temperatures above 1000 °C [20].

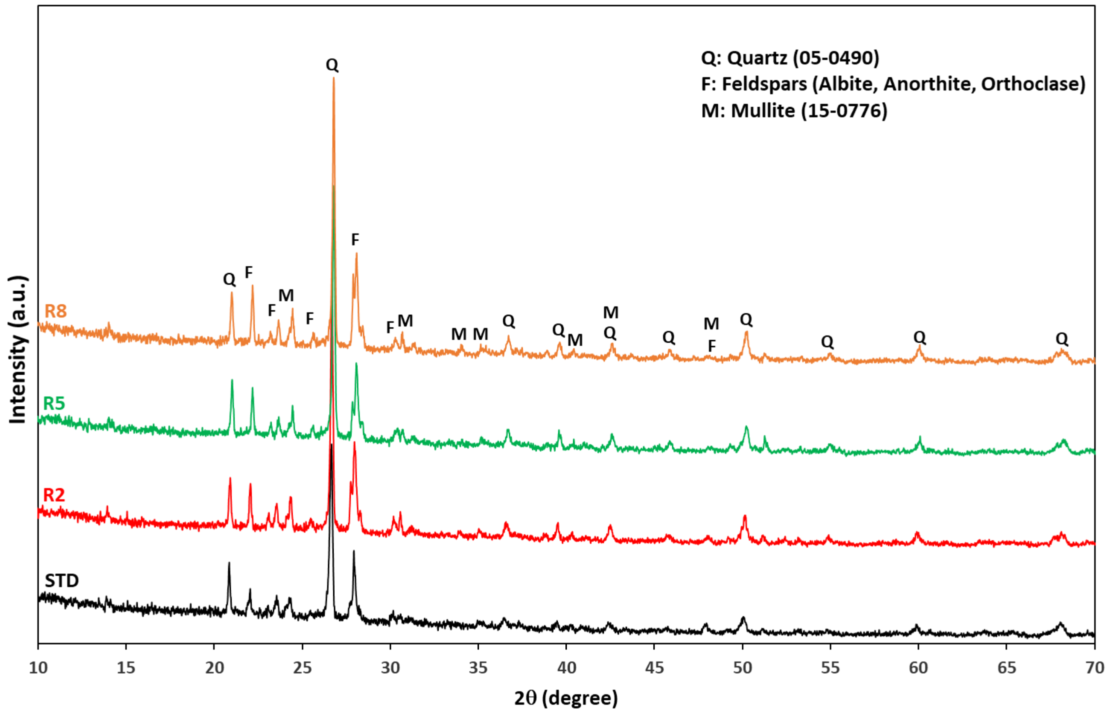

Figure 3. XRD patterns of fired ceramic bodies belonging to Std, R2, R5, and R8 recipes.

Mechanical and physical properties of ceramic bodies prepared according to the standard and modified recipes are given in Table 4. Dry strength values decrease with the increase in clay amounts compared to the standard recipe. The dry strength values of the recipes coded as R1 and R7 are close to the standard recipe value and are within the tolerance range of floor tile standards. Dry strength acceptance standard of the company is in the range of 15-16 kg/cm^2.

Firing shrinkage values increase with the increase in amount of Konya clays. This increase can be interpreted as an improvement in the sintering properties of the bodies. However, considering all recipes, firing shrinkage values ranging between 8.21-9.27% did not show any negative deviation from that of the standard body. Firing shrinkage acceptance standard of the company is around 8.5-9.0%. According to the firing color results, although there is a decrease in lightness (L*) values as the amount of Konya clays increase, the L*-a*-b* values of all 9 recipes are generally similar to that of the standard recipe.

The fired strength values of the bodies obtained using K3 (R1-R3) and K2 (R7-R9) clays are higher than that of the standard recipe. This increase can be associated with the amount of mullite phase formed during firing. Mechanical strength of a porcelain body is directly related to the interlocking mechanism of the fine mullite needles in its microstructure. Therefore, as the amount of mullite in the body decreases, the bending strength is expected to decrease. According to the results of the quantitative analysis performed on the XRD patterns in Figure 3, the lowest amount of mullite was determined in the body containing K1 clay (R5). As a result, the lowest fired strength values were obtained for recipes containing K1 clay in Table 4. In addition, the increase in fired strength values can also be related to vitrification period during firing. As already stated for XRD patterns in Figure 3, amount of residue feldspars increased for bodies containing Konya clays. High amount of residue feldspars implies the high alkali content of starting raw materials. Alkali oxide and alkaline earth oxide based raw materials decrease the viscosity in the order $MgO < CaO < SrO < BaO < K_2O < Na_2O < Li_2O$ [21]. The decrease in the viscosity of the liquid phase with the increasing alkali content increases the rate of condensation during firing and contributes to the increase in the amount of mullite crystals, and as a result, provides denser and high strength structures [22].

Water absorption is one of the most important physical properties of ceramic tile products. Water absorption value mainly depends on the raw materials used in the production of tiles. Especially for floor tiles used at outdoor surfaces, a water absorption value lower than 3% is desired. Higher absorption values can cause breaking and cracking of outdoor floor tiles [5]. Furthermore, ISO 10545-3 standard for porcelain tiles imposes a water absorption value of ≤0.5%. All the recipes used in this study exhibit water absorption values lower than 0.1% (not given in Table 4) and they have been evaluated to comply with the standards.

Table 4. Mechanical and physical properties of ceramic bodies.

Recipes	Dry Strength (kg/cm^2)	Firing Shrinkage (%)	Chromatic Coordinates (L*-a*-b*)	Fired Strength (kg/cm^2)
Std	17.30	8.45	64.3-3.4-11.1	512.63
R1	15.54	8.25	64.2-3.3-11.2	533.69
R2	10.24	8.45	63.8-3.3-11.4	579.10
R3	6.84	9.17	61.9-3.6-11.5	569.48
R4	13.60	8.35	65.1-3.3-11.4	469.61
R5	10.47	8.71	63.2-3.5-10.3	428.77
R6	6.57	9.27	65.2-3.1-10.7	416.74
R7	14.96	8.21	63.0-3.6-11.4	517.54
R8	13.06	8.45	62.7-3.5-11.5	566.39
R9	10.80	8.83	61.2-3.4-11.5	615.11

Thermal expansion coefficients of fired ceramic bodies are listed in Table 5. Thermal expansion values increase -compared to the standard recipe- with the increase in the amount of all 3 clays. The quartz phase content of Ukrainian clays is less compared to Konya clays. It is well known that the thermal expansion coefficient of crystalline quartz is higher than that of amorphous quartz [18]. As the amount of Konya clay increases instead of Ukrainian clay in the bodies, the amount of crystalline quartz increases, and this causes the coefficient of thermal expansion to increase.

Table 5. Thermal expansion coefficients of fired ceramic bodies.

Recipes	TEC (α_{400}x10^{-7} °C^{-1})
Std	74.00
R1	76.04
R2	78.02
R3	78.10
R4	75.85
R5	76.49
R6	76.83
R7	73.63
R8	78.42
R9	78.77

"Flex point" temperatures at which the highest dimensional change is achieved during rapid sintering were determined using the optical dilatometer analysis data. Flex point of standard, R2, R5, and R8 bodies are 1220, 1222, 1223, and 1194 °C, respectively. In the bodies (R2 and R5) where K3 and K1 clays are used, the flex point temperature is close to the standard body. There is a decrease in the flex point value in the R8 body where K2 clay is used. According to this result, it can be concluded that the body containing K2 clay is rich in terms of alkaline-earth content compared to other bodies. It has also been stated in similar studies in the literature that raw materials containing Fe_2O_3 and alkaline-earth oxides such as CaO and MgO, with their fluidizing (fluxing) effects, improve vitrification in ceramic bodies and the liquid phase is formed at lower temperatures [11, 23].

4. CONCLUSIONS

In this study, the suitability of 3 different clays from Doğanhisar region of Konya (K1, K2, and K3 clays) for porcelain tile production was investigated. When Konya clay is added instead of Ukrainian clay in tile body recipes, the packing (compressibility) feature of green bodies is negatively affected, and dry strength values decrease. Ukrainian clays, with their high plasticity property, enable easier pressing and thus enable a body with high green density to be obtained. Dry strength acceptance standard for porcelain tiles is in the range of 15-16 kg/cm^2, and the use of K2 and K3 clays up to 6 wt.% instead of Ukrainian clay can provide the desired dry strength standard.

An increase in firing shrinkage and thermal expansion values was observed with the use of Konya clays. Crystalline quartz has a higher coefficient of thermal expansion compared to amorphous quartz. Hence, the coefficient of thermal expansion increases with the addition of Konya clays (especially K3 clay with high SiO_2 content) in the recipes. On the other hand, the use of Konya clay (K2 and K3) instead of Ukrainian clay has a positive effect on the fired strength of the bodies. This result is related to the increase in the amount of mullite phase formed after firing, with the use of Konya clays.

Konya clays (since they are illite-based clays) cannot be used as a complete replacement for Ukrainian clay in the recipe. However, with optimizations in the recipes and firing regime to keep the firing shrinkage and thermal expansion values at the desired standard values, it is possible to partially use Konya clay (especially K3 clay) instead of Ukrainian clay.

Declaration of Competing Interest

The author(s) stated that there are no conflicts of interest regarding the publication of this article.

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