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RESEARCH ARTICLE

INVESTIGATION OF THE USE OF KÜTAHYA REGION KAOLIN IN WALL TILES

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Abstract

The main raw materials in the ceramic wall tiles are clay, kaolin, calcite, and feldspar. The technical properties of the tiles (water absorption, total shrinkage, dry firing strength, fracture strength) and the desired physical properties (L, a, b, surface smoothness) vary depending on the raw material content in the recipe. Rheological parameters are crucial in the formulation development stages to avoid production problems and to ensure the final desired product parameters. Density, viscosity, and sieve analysis have therefore been included in the developed formulations.

The use of raw materials from the region close to the production facility facilitates easy access to these materials. Thanks to this advantage, the use of domestic raw materials is becoming more widespread. In addition to the use of alternative raw materials, it contributes to the reduction of greenhouse gas emissions.

The aim of this study, is to formulate a recipe for wall tiles using kaolins from the Kütahya region instead of clay from the Afyon region. Recipes containing 2%, 4%, 6%, and 8% of Kütahya region kaolin, with a corresponding reduction in clay from the Afyon region, were developed and sintered at 1130-1150°C with a 29-minute firing cycle.

By analyzing the technical properties of the developed formulations, it was found that a maximum of 2% of Kütahya region kaolin could be used in the wall tile formulation. It was found that exceeding this percentage was not feasible due to the associated increase in water absorption, expansion of product size, and decrease in dry strength.

Keywords

Kaolin,
Wall tile,
Characterization,
Technical specifications

Time Scale of Article

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1. INTRODUCTION

For the ceramic industry, which is rapidly developing in our country and has an important position in the world market, raw material accesibility, location of raw material resources, raw material reserves of our country and usage characteristics of reserves of our country and usage characteristics of reserve areas are important issues. In the globalizing world and increasing competitive environment,

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companies that produce at low costs without compromising quality and keeping customer satisfaction high tend to constantly grow in the sector. It requires the research and development of quality raw materials resources, which are depleted day by day, the discovery of new raw material areas and the more efficient use of existing raw materilas resources. Additionally, the increasing need for raw materials suitable for producing this material [1, 2].

In the basic components of ceramic wall tiles establishing an optimum clay composition is important for companies in terms of standardising properties such as plasticity, raw strength and firing colour. In addition to quartz, iron oxide and titania are present as impurities in the clay mixture, which is generally formed by combining kaolinitic based clays. The geological formation, structure, mineralogical and other physico-chemical properties of various clays used in recipes have been widely investigated and discussed in the literature [3, 4].

In parallel with the increase of population in the world and in our country, clays are constantly on the agenda as one of the most important raw materials in every period of the ceramic sector [5,6]. Based on these reasons, the properties, applications and production characteristics of kaolinite group clays were examined in this study, and the possibilities of using kaolins in Kütahya region in wall tiles were studied

In the ceramic industry, kaolins containing 75-80% kaolinite minerals are generally preferred. Ceramic kaolins are required to have properties such as firing colour, viscosity, friction resistance, low Fe_2O_3 and TiO_2 ratios and 83-91% brightness, high water dispersibility, plasticity, low firing shrinkage, white firing colour. In addition, physical and chemical properties such as purity, whiteness, grain size distribution, thermal behaviour and high Al_2O_3 content [refractoriness] are important factors affecting that influence the location and amount of kaolin used in ceramics [7].

Clay is a very fine-grained sediment with a grain size of less than 0,02 mm. The elements that make up clay are minerals with a high Al content. Kaolinites, which are the most important of these minerals, are hydrosilicates with Al_2O_3, $2SiO_2$, $2H_2O$ composition, SiO_2 is 46,54% Al_2O_3 39,50% H_2O 13,96%, Kaolinite group clay minerals are white in colour when pure and have a matt appearance, its hardness is 1, specific gravity is 2,1 gr/cm^3, It loses water at 330-450 ℃. The main constituent is aluminum silicate. In case of its decrease, a small amount of iron sulphur or potassium is mixed into the composition [8].

One of the most important determinants of quality is silica. Intensive removal of silica from the structure, produces high quality kaolin ore. The kaolin that remains as free silica grains gains the quality of leachable kaolin as it is easier to separate. One of the most important criteria is that the iron in the original rock should not be present in kaolin. However, during the chemical process of kaolinization, some of the iron remains without being removed. Kaolinisation occurs as a result of the decomposition of the feldspars mentioned in the formation of kaolin. The more the feldspar decomposes, the more K_2O is removed from the environment, the more this is removed, the higher the Al_2O_3 ratio which determines kaolinisation [9,10].

Clay-kaolin, quartz and pegmatites are the main raw materials of the wall tile structure. Kaolin is the most important raw material for porcelain and wall tiles. For this reason, the raw material properties of kaolin have a significant impact on its field of application. The technological value of kaolin is closely linked to the oxides that affect the firing colour and the feldspar and quartz that can be removed by washing [11].

The limits of kaolin use in the ceramic industry are determined according to TSE (10545 standards. According to these standards, kaolin is expected to be fired white up to 1300 ºC, maximum solid concentration limited to 67-71%, deformation of 5 mm, total shrinkage 0,07%, maximum water absorption 0,2% and dry strength 25 kg/cm^2 Al_2O_3 15-32%, SiO_2 48% [max ,] Fe_2O_3 1% [max ,] is expected.

Clay and kaolin group raw materials provide strength in aqueous mixtures as plastic raw materials, being plastic, they increase the raw strength required during moulding. DTA analysis, chemical and mineralogical analysis are carried out to determine the reactions that will take place in the clay such as moisture content and grain size distribution, iron and titanium content [directly affecting the firing colour], control of carbonates, sulphate and carbon impurities and organic matter content, slurry density, viscosity and pH, plasticity and dry strength, firing shrinkage at firing temperatures, determination of thermal expansion coefficient [12, 13].

Within the framework of this study, research was conducted to develop new recipe compositions that exploit the effectiveness of kaolins from Kütahya region kaolin in wall tile composition recipes. The chemical, physical, and mineralogical properties, dry strength, particle size, viscosity, firing color, rheological properties, moulding parameters, drying behavior, and firing behavior of the kaolins from Kütahya region were investigated in order to fully understand the characteristics of the region.

2. MATERIALS AND METHOD

In the wall tile composition, A-1, A-2, A-3 clays, Kütahya region kaolin, F coded feldspar, K coded calcite raw materials were obtained from NG Kütahya Seramik, the chemical, physical, and firing properties of all raw materials used were prepared according to standard test procedures, and all results are shown in Tables 1-2.

Table 1. Body recipe compositions of standard tile [D-1] and new tile [D-2 ,-D-3, D- 4, and D-5] [% wt,]

Raw materials	D-1	D-2	D-3	D-4	D-5
A-1	13	11	9	7	5
A-2+A-3	56,5	56,5	56,5	56,5	56,5
F	18	18	18	18	18
K	12,5	12,5	12,5	12,5	12,5
Kütahya region kaolin	/	2	4	6	8

The chemical analysis results of the clays used in the study are shown in Table 2. Figures 1 and 2 show the TG-DTA analyses of these clays, while Figure 3 shows the XRD analyses. The raw materials were weighed according to the recipe proportions, taking into account the moisture values, and ground in ball mills with the addition of water and electrolyte to a density of approximately 1680 g/lt and 63 μm sieve balance of 6-6.5%.

Density [g/lt], viscosity [pour sec], and sieve balance [+63 μm] values of the prepared slurries were measured. The sludge obtained was dried in a laboratory oven at 150 ℃ and crushed in a mortar and pestle. After sieving through a 650 μm sieve, it was humidified to 5.5-6% moisture content and kept for one day to ensure homogeneity.

Samples were prepared by forming granules with dimensions of 110x55mm under a pressure of 320kg/cm^2 using a laboratory-type press. These samples were then dried in a laboratory oven at 150 ℃ for 2 hours to dry. The prepared samples was carried out in NG Kütahya Seramik Tile Factory at 1120-1140 ℃ for 29 minutes, following the firing conditions for wall tiles.

The sintering behaviour of the concretes after firing can be explained by water absorption [%] and firing shrinkage [%]. In addition, the firing strength of the samples, fired in an industrial roller kiln, was measured using a triaxial compression gauge [Gabbrielli SRL, Italy]. Colour measurements (L, a*, and b*) of the baked specimens were also conducted using a Minolta 3600d device. XRD analyses

[Rigaku brand model Miniflex, Japan] were carried out to determine the crystalline phases formed in both the unfired samples.

Measurements were performed with a step size of 0.02° to cover the 2θ angle range of 5°-70°. The results of the phase analysis for all samples examined are available in the JADE analysis programme, including qualitative phase values and PDF cards.

3. RESULTS AND DISCUSSION

For the recipe compositions are shown in Table 1, clay was formulated as a recipe composition with increasing proportions of kaolin from the Kütahya region, while keeping the proportions of feldspar, calcite and A-2+A-3 clay constant compared to the standard D-1 recipe composition, prepared with the standard blending recipe. Accordingly, the increasing proportions of the D-1 formula are coded as D-2, D-3, D-4 and D-5.

In the study, Kütahya region kaolin and A-1 clay obtained from NG Kütahya Seramik were used to prepare a standard wall tile recipe. The X-Ray Fluorescence [XRF] analyses of the clays used in the study are provided in Table 2.

Table 2: Chemical compositions [% wt] determined by X-Ray Fluorescence [XRF] in the standard body recipe.

Element	A-1	Kütahya region kaolin
L.O.I	2,39	5,10
Na_2O	0,43	0,08
MgO	0,17	0,19
Al_2O_3	14,80	13,95
SiO_2	75,14	78,66
SO_3	0,12	0,25
K_2O	5,51	0,05
CaO	0,26	0,18
TiO_2	0,07	0,73
Fe_2O_3	1,16	0,50

Clays initiate viscous flow with melting in their structure at lower temperatures due to impurities such as Na_2O, K_2O, CaO and MgO in their content.

The wall tile recipe in Table 1 is designed to maintain a fixed ratio of A-2 and A-3 clays with a total clay content of 56.5%, determined by correlating X-ray fluorescence [XRF], X-ray diffraction [XRD], DTA, SEM analyses of A-1 clay, and Kütahya region kaolin with the physical and mechanical properties in all recipes. The usage rate and effect of Kütahya region kaolin in the recipe are observed.

According to the results of the chemical analyses shown in Table 1, the total alkali content of the Kütahya Region kaolin is about 0.5%, and the sum of Fe_2O_3 and TiO_2 is 1.28%. In contrast, the total alkali content of A-1 clay is about 6.37%, and the sum of Fe_2O_3 and TiO_2 is 1.23%. There are significant differences between kaolin from Kütahya region and A-1 clay, including a difference of about 53.13% in fire loss rates. Kütahya region kaolin has a much higher fire loss value, a difference also evident in the TG-DTA analysis shown in Figure 1.

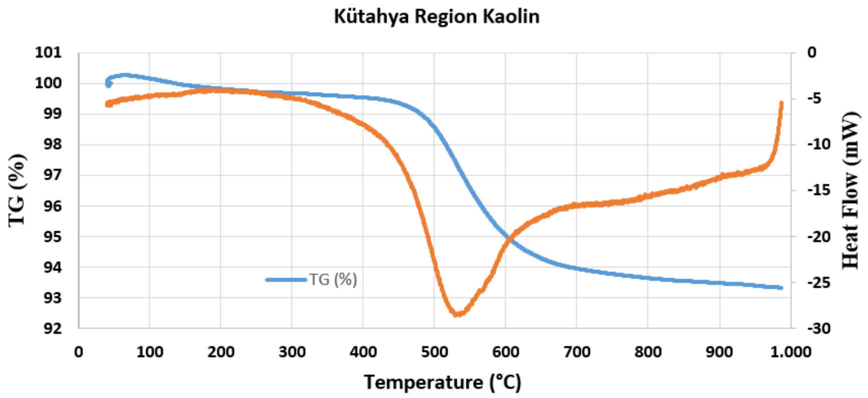


Figure 1. Kütahya Region kaolin TG-DTA Curve

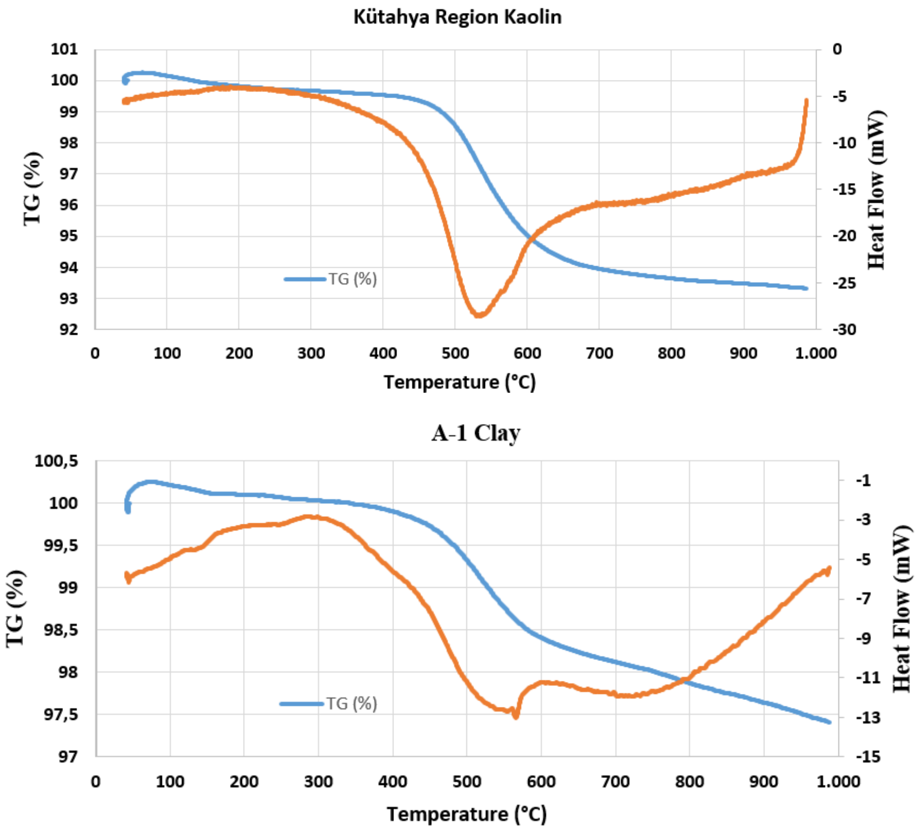


Figure 2. TG-DTA Curve of Standard A-1 clay

When the TG analysis of kaolin from the Kütahya region is analysed, a total mass loss of 6,93%, physical water loss of 0,39% between about 62-181℃, removal of 0,17% organic matter between 181,68-265,05℃, and formation of meta-kaolin between 434,91-698,8℃ are observed.

Figure 2 shows a total mass loss of 2.838% in A-1 clay. The kaolin from Kütahya region contains 1.98% melter addition, and A-1 clay contains 7.6% melter addition. This proportional difference is shown in the DTA analysis in Figure 2 as a temperature-dependent DTA analysis. The endothermic peak observed at 572 ºC is attributed to quartz transformation, and a gradual weight loss is observed in the sample. The total weight loss was determined to be 1.32%. It is suggested that this weight loss is due to the loss of crystal water from the micaceous compounds in the sample.

When the DTA analyses of the kaolin and A-1 clay from the Kütahya region used in the study were examined in detail, the structural changes clays with temperature were compared. Changes occur in the clay mineral structure depending on the Al_2O_3: SiO_2 ratio, the grain size of the clays, and the presence of alkali oxides in the layered structure.

Figure 2 shows 1 sharp peak at A-1 and a small peak at 1 point. While the small endothermic peak around 200 ºC indicates the removal of water adsorbed on the surface, the second sharp endothermic broad peak at 470,13 ºC indicates the dehydration of kaolinite, The fact that the temperature and intensity of the dehydration peak in this sample are lower compared to the kaolin of Kütahya region indicates that the amount of kaolinite in this sample is lower and the amount of smelter is higher, While Al_2O_3 / SiO_2: 0,19 in A-1 clay, Al_2O_3 / SiO_2 : 0,17 in Kütahya region kaolin, the similarity of this value caused no significant peak at 700 and 900 ℃ in both clays.

The results of the physical property tests applied to all recipes are summarised in Table 2. All prepared recipes were dried prior to fast firing, and the strength values after drying are shown in Table 2. When the measurement results are analysed, there is a change in the dry strength value in the form of both increase and decrease, which is due to the decrease in the proportion of core raw material and the increase in the proportion of glassy phase with the incorporation of Kütahya region kaolin into the recipe.

After 29 min, roll sintering at a temperature of about 1130-1150 ℃, an increase and decrease in strength value is observed with the increase of Kütahya region kaolin in D-1 standard recipe and D-4 recipe in the recipe composition with the addition of Kütahya region kaolin, a loss in strength value about 5% is observed between D-1 recipe and the D-4 recipe, water absorption values increase with the increase of Kütahya region kaolin compared to D-1 recipe and the lowest strength and highest water absorption values are observed in D-2.

The ratio of Al_2O_3 /SiO_2 in Kütahya region kaolin is [0,17] and the ratio of Al_2O_3 / SiO_2 in A-1 clay structure is [0,19]. The kaolin from the Kütahya region has a total ratio of NaO_2, MgO, K_2
O, and CaO that is about 92% lower than that of A-1 clay. This discrepancy leads to a reduction in strength and an increase in water absorption."

A fixed amount of electrolyte was used in the prepared recipes. In addition, grinding times varied between 11 and 12.5 minutes. Changes in grinding times were made to achieve standard density and sieve balance in the recipes. The proportion of coreless raw materials in the kaolin from Kütahya region was reflected in the grinding time. The sieve balance values show a decreasing trend between D-1 and D-2 as the grinding time is adjusted, ranging from 1700 to 1704 gr/cm³. In addition, the flow times increase by 17.85% with the increase in the incorporation of kaolin from Kütahya region

Table 3 summarises the physical analysis results obtained after firing at 1130-1150 ℃ of the prepared recipes.

Table 3. Standard **D-1** Physical properties of recipes **coded D-2, D-3, D-4, D-5** with the addition of Kütahya Region kaolin.

Parametre	D- 1	D-2	D-3	D-4	D- 5
Size (mm)	110,33	110,27	110,25	110,18	110,19
Total Shrinkage (%)	0,33	0,38	0,4	0,46	0,46
Water Absorption (%)	18,26	17,79	18,34	18,88	19,14
Dry Strength (N/mm^2)	14,13	17,4	15,51	15,54	14,95
Baking Strength (N/mm^2)	182,3	204,6	208,1	214	192,3
Firing Breaking Strength (N)	876	611	660	846	820
L	75,9	74,61	75,64	75,19	75,64
a	7,83	7,88	7,92	8,53	7,84
b	19,06	19,5	19,87	20,52	19,87

An increase in the total shrinkage amount, an increase in the water absorption amount, an increase and decrease in the dry strength value, an increase and decrease in the dry strength value, and an increase and decrease in the firing strength value were observed with the increase of Kütahya region kaolin in the formulation. In addition, a decrease in the L* value, an increase and decrease in the a* value and an increase and decrease in the b* values are observed after firing compared to the D-1 standard formulation.

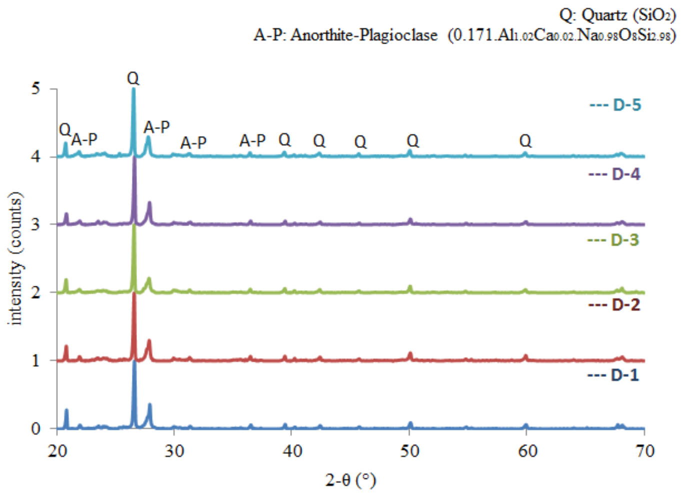


Figure 3. XRD analysis results taken after firing of recipes coded D-1, D-2, D-3, D-4 and D-5 [Q: Quartz, AP: Anorthite-Plagioclase]

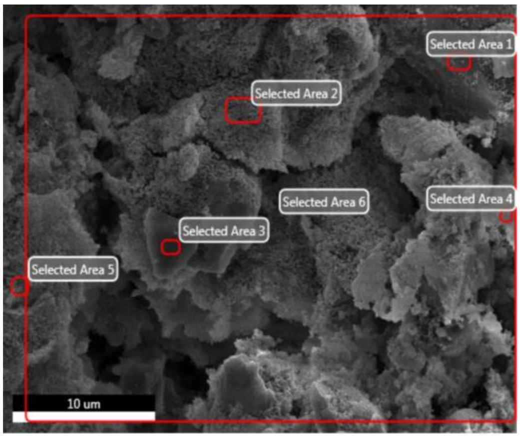
When the X-ray diffraction [XRD] analysis of the D-1, D-2, D-3, D-4, and D-5 coded formulations is examined after firing, it is observed that similar phases are formed within the mineralogical structures of the formulations. The intensity of the quartz and plagioclase phases in the structure is very low, and the peak sharpness is significantly reduced in the D-1 formula where the A-1 clay content is minimal. The X-ray diffraction [XRD] analysis results indicate that the intensity of anorthite plagioclase phase does not change significantly with the addition of kaolin from the Kütahya region and the decrease in A-1 clay. (PDF card no for anorthite-plagioclase: 98-009-0142; 0.171. Al1.02 Ca0.02. Na0.98 O8 Si2.98) .

It can be observed that the higher sieve balance and grinding time in recipe D-4 results in a larger quartz grain size and a lower quartz anorthite-plagioclase value compared to the recipe D-1. While a sharp and intense peak was observed in the quartz phase in the D-4 formulation, the peak intensity decreased slightly in the D-5 formulation and the sharp peak changed to a slightly broadened peak. Figures 4-7 show the SEM and EDX analyses of the standard body and the standard body with technical specifications. The microstructure of both bodies is similar, containing irregularly shaped quartz grains, anorthite-plagioclase phase, and pores found in a typical wall tile body.

According to the results of the EDX analysis carried out on the indicated points, the crystals formed in the region contain aluminium, silica, and calcium, in agreement with existing literature. In the backscattered electron images, heavy elements appear whiter than other elements. In the microstructural images, the dark grey tones represent quartz grains, while the black tones indicate porosity.

Figures 4-7 show the SEM images and EDX analysis results of formulations D-1, D-2, D-3, D-4, and D-5 at x10,000 magnification. These samples were sintered at the standard firing temperature. Observation of the SEM images shows vitrification in the structures, with grains coalescing to form a sinter. This phenomenon is indicative of high temperature sintering, which creates an environment where clay grains react with other minerals to form a vitreous phase.

While the firing strength decreases with the addition of A-1, the water absorption rate increases according to the physical properties table. SEM and EDX analysis show that the glassy phase ratio is the highest in the D-4 recipe, which exhibits the highest strength.



Element	Weight %	Atomic %
Na_2O	0,00	0,00
MgO	1,34	2,57
Al_2O_3	57,54	43,67
SiO_2	26,19	33,72
K_2O	1,01	0,83
CaO	13,92	19,21

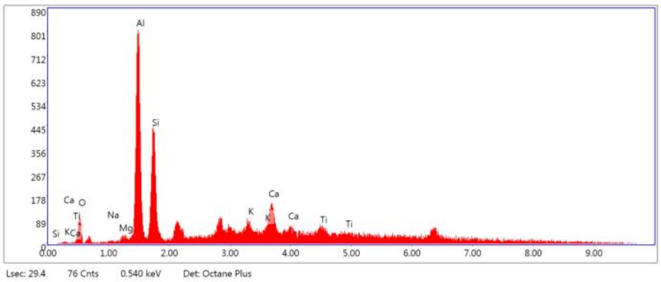
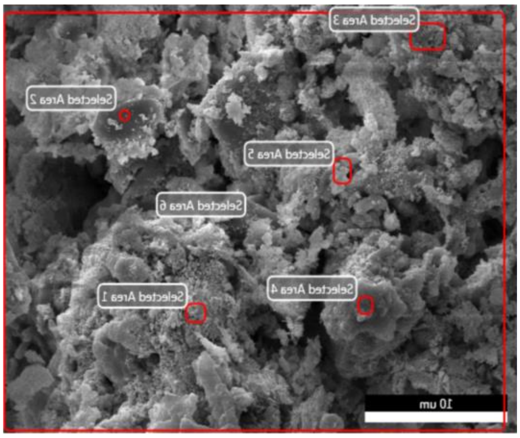


Figure 4b. SEM-EDX analysis result of the D-1 coded sample (Selected area 2 for EDX) sintered in the wall tile regime [x10000 magnification]



Element	Weight %	Atomic %
Na_2O	0,00	0,00
MgO	0,26	0,43
Al_2O_3	35,78	23,75
SiO_2	9,94	11,19
K_2O	1,16	0,83
CaO	52,87	63,80

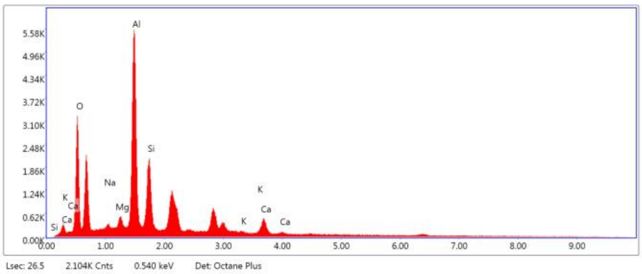
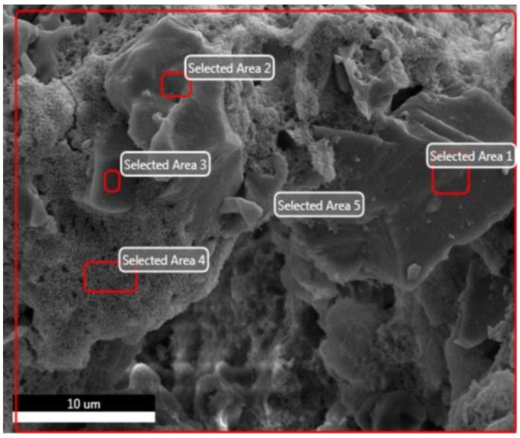


Figure 5. SEM-EDX analysis result of the D-2 coded sample (Selected area 4 for EDX) sintered in the wall tile regime [x10000 magnification]



Element	Weight %	Atomic %
Na_2O	0,00	0,00
MgO	0,26	0,43
Al_2O_3	35,78	23,75
SiO_2	9,94	11,19
K_2O	1,16	0,83
CaO	52,87	63,80

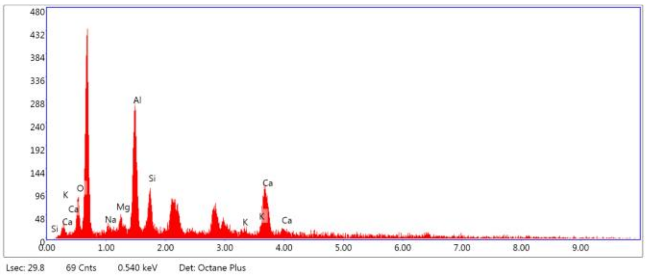
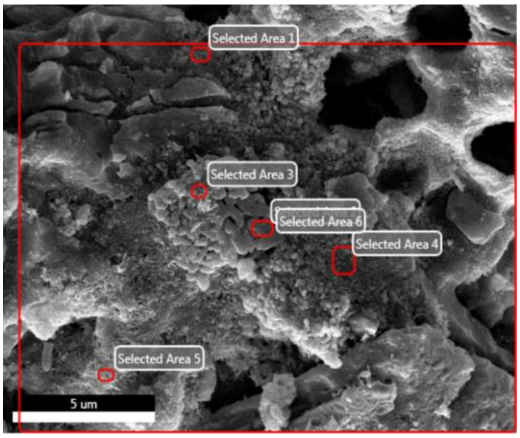


Figure 6. SEM-EDX analysis result of the D-3 coded sample (Selected area 4 for EDX) sintered in the wall tile regime [x10000 magnification]



Element	Weight %	Atomic %
Na_2O	2,67	4,54
Al_2O_3	27,03	18,17
SiO_2	48,59	55,45
K_2O	9,52	6,93
CaO	12,20	14,91

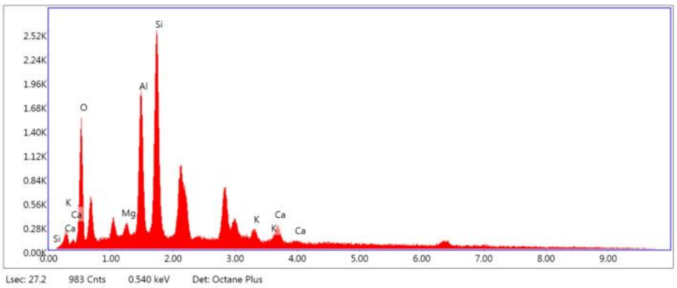


Figure 7. SEM-EDX analysis result of the D-4 coded sample (Selected area 6 for EDX) sintered in the wall tile regime [x10000 magnification]

4. CONCLUSIONS

The study investigated, the properties of Kütahya region kaolin and A-1 coded clay raw materials. The results show that the inclusion of Kütahya region coded kaolin in the recipe composition, increased the structure, grinding time and the sieve balance. In addition, the raw strength value decreased. However, its low deformation value suggests that its use will darken the final product (L* whiteness and a* redness values are high). Investigations into its use rate in the formulation are ongoing.

The use of this kaolin in excess of the established standard formulation ratio has a significant effect on the deformation and colour values of the structure. However, the dry strength value remains very close to the dry strength value of the standard formulation.

In all recipes, the amount of electrolyte was kept constant for a constant litre weight due to the reduction in particle size. The raw strength and raw density values increased, indicating that the packing density of the moulded sample increased. Water absorption and firing shrinkage increased. Microstructure images showed that the number of pores decreased slightly, and their size decreased with the increase of raw material A-1 kaolin. These results were supported by SEM and XRD analyses.

CONFLICT OF INTEREST

The authors stated that there are no conflicts of interest regarding the publication of this article.

CRediT AUTHOR STATEMENT

Senem Algan: Formal analysis, Investigation, Conceptualization. **Fatih Öztürk:** Formal analysis, Investigation, Conceptualization. **Büşra Yay:** Formal analysis, Investigation, Conceptualization. **Zehra Emel Oytaç:** Formal analysis, Writing - original draft, Visualization, Conceptualization, Supervision. **Eda Taşçı:** Formal analysis, Writing - original draft, Visualization, Conceptualization, Supervision.

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