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Investigation of Mineralogy and Ceramic Technology Properties of Sivrihisar Kurtşeyh Sepiolites (Türkiye)

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Abstract − In this study, the geology, mineralogy, and potential uses of the Sivrihisar sepiolite and dolomitic sepiolites of the Eskişehir region (Türkiye) for ceramic wall tiles were investigated. A geological map of the open pits in the village of Kurtşeyh and generalized stratigraphic sections are presented. Samples taken from the pit sequences were characterized in terms of their mineralogy by XRD (X-ray diffraction). Chemical and Thermal analysis of the raw materials was determined by XRF (X-ray fluorescence), Thermal analysis (TA), and Differential Thermal analysis (DTA). Besides these studies, the thermal properties and shrinkage (%), water absorption (%), and color (L, a, b) of the fired wall tile bodies were investigated to determine their technological properties. In light of all the analysis results, alternative body formulations were created, and these recipes were fired under wall tile conditions. The fired phases were then mineralogically characterized, and the physical and technological properties of the different bodies were studied. As a sum, 10% dolomitic sepiolite usage instead of İstanbul clays is suitable for manufacturing wall tiles.

Keywords − *Sepiolite, whitewares, geology, ceramic, mineralogy*

1. Introduction

Our country's ceramic sector supplies 90% of the plastic clays it uses in production from the Şile (Istanbul) region. In addition, it imports high-strength and colored fired clays from Ukraine, which it also uses in its recipes. Recently, there has been news that the area permitted for mining in the Şile region by the Ministry of Forestry and the General Directorate of Forestry has been reduced to 1/3 of the current amount. If this restriction is realized, the clay reserve, estimated to be approximately 250 million tons, will have decreased to 80-90 million tons. Only 30% of this is suitable clay for ceramics production.

Due to this limited situation and the state of war in Ukraine, ceramic producers have difficulty obtaining clay and are resorting to alternative solutions. For this purpose, the use of carbonated sepiolitic clays, known to exist in the area extending from Sivrihisar and its vicinity in the south of Eskişehir in inner NW (Northwest) Anatolia and towards Polatlı, Ankara, has been brought to the agenda. The main objectives of this study are to investigate the geology of the region in question, to reveal the geology of different types of rock assemblages that form the basis of sepiolitic clays and the geological features of the Neogene basin that developed on this

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basis and include clays, to determine the sedimentary deposits in the basin, to investigate which zones are suitable for ceramic production, to reveal the clay mineralogy, and to compare whether the clays of the Eskişehir region can be an alternative with the clays of the Şile region in Istanbul.

Sepiolite occurs in two different polymorphic structures in nature. The first of these is α-sepiolite, which is amorphous, compact, and in the form of massive nodules and is known as meerschaum in German because its external appearance resembles sea foam; the second is β-sepiolite, which occurs in the form of small, flat, and round particles or amorphous aggregates [1]. Sepiolite is formed due to the decomposition of serpentine as a secondary mineral. It has high sorption power by holding water up to 200-250 times its weight. The pH value suitable for its formation is 8-8.5, and when it is heated above 300ºC, its structure changes and pores deteriorate [2]. Sepiolite, formed in layers due to sediment deposition, mostly has an earthy, fine-grained, and slippery appearance. This type of sepiolite contains 90% sepiolite mineral, and the remaining percentage is generally dolomite and smectite group clays, magnesite, palygorskite, and detrital minerals [2].

The Sivrihisar region lies within the tectonic unit known in Turkish geology as the Tavşanlı zone. This zone, which represents the northern part of the Anatolid-Torid block, underwent high-pressure, low-temperature metamorphism in the Late Cretaceous [3-5]. The Tavşanlı Zone consists of three main tectonic units. These are the ophiolite, the Cretaceous-aged accretionary complex, and the Orhaneli passive continental margin metamorphic stack [6]. This complex basement is cut by Late Paleocene-Eocene aged granitic rocks, and on top of this composite basement are the Neogene-aged sedimentary rocks known as the Sakarya and Porsuk formations, which are equivalent to each other and separated by an uplift [7-9]. These sedimentary rocks, known by two different names, consist of dolomitic carbonates at the bottom, gypsiferous clays at the top, milky-brown sepiolites, dolomitic sepiolites, sepiolitic dolomites, montmorillonitic clays, and finally fossiliferous micritic limestones and porcelain-like dolomites at the top (Figure 1).

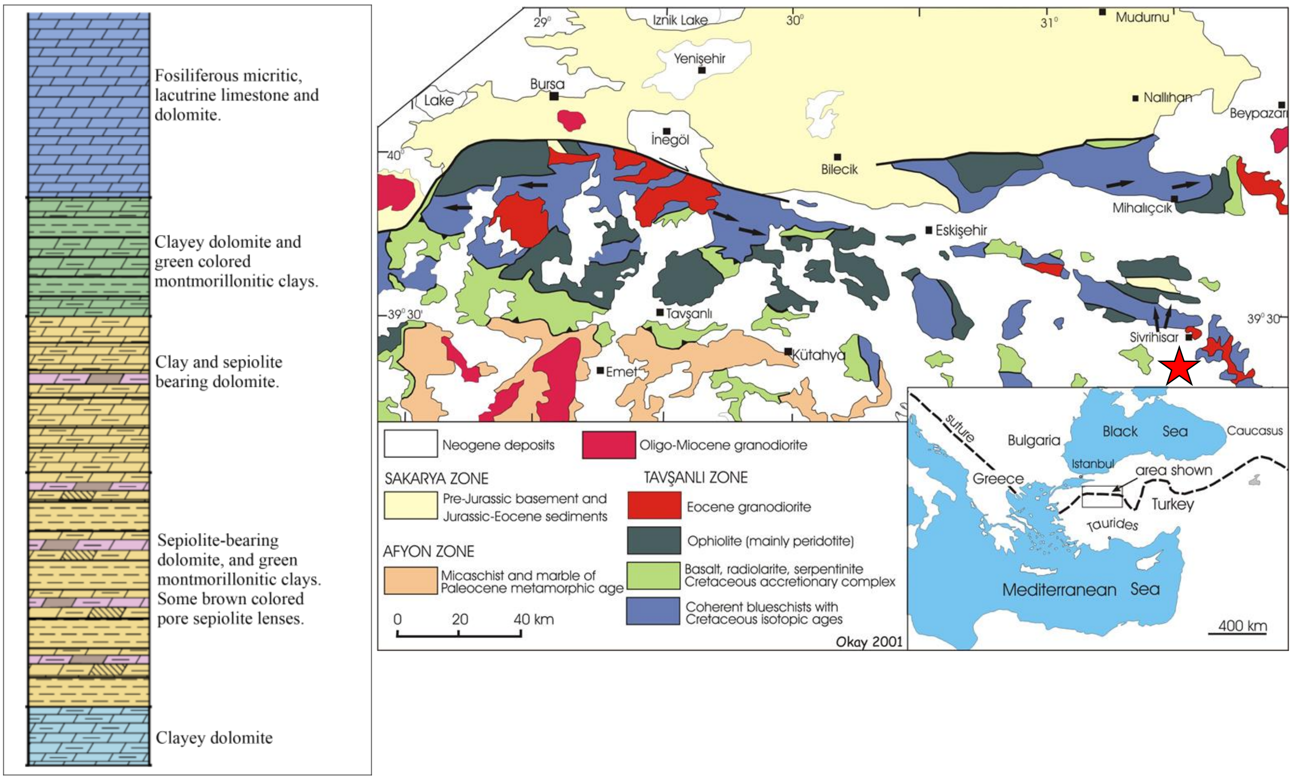

Figure 1. Generalized geology map of the Bursa, Eskişehir, and Kütahya provinces [5] and generalized stratigraphic section of the Sakarya-Porsuk formations around the Kurtşeyh area (red star indicates the study area)

Sepiolites also developed within these lacustrine beds. The age of the Sakarya Formation was determined to be Miocene in studies by Karakaş [10] based on the gastropod and ostracod fossils in the unit. However, later studies by Kadir et al. [7, 8] indicated that the age of the unit is Early Pliocene.

The Neogene deposits in the Kurtşeyh region of the Sakarya Formation, terrestrial deposits formed in a shallow lacustrine environment within the Central Anatolian Neogene Basin, were formed in a playa (alkaline) lake environment and are therefore rich in sepiolite (Figure 1). The brown sepiolite unit, which is rich in organic matter, was formed in anoxic marshes in the center of the playa lake, while the dolomite sepiolite was formed in small ponds of the playa lake, Kadir et al. [8]. Sepiolite, derived from Mg-silicate and dolomitic precursors, is formed in shallow-lacustrine, alkaline (pH: 8.0-9.5), low-salinity, and anaerobic areas [11].

Wall tiles are a mixture of clay, Kaolin, and calcite, which are known as internal decoration materials. The Wall tiles, which may have very different dimensions and thickness, are fired between 1120 C and 1160 C. In general, the wall tile body formulation is used min. 45-40% clay materials to obtain eligible strength values [12]. Sümer [13] obtained the density of sepiolite as 2.08 g/cm3 and its porosity as 37.5% due to some experimental studies on Eskişehir sepiolite used in ceramic production, dimension determination, and chemical analysis. In light of this information, it was observed that up to 40% of sepiolite material could be used in tile ceramics; with sepiolite addition, the density of the ceramic changed from 1.9 g/cm^3 to 2.3 g/cm^3, the water absorption rate changed from 9% to 7.8%, the bending strength changed from 170 kg/cm^2 to 196 kg/cm^2, the heat expansion coefficient changed from 8×10^6 to 5×10^6; the experimental tiles produced had good shock resistance and were mainly in compliance with the standard TSE-4037.

There are many studies on the region in question due to the economic importance of sepiolite and the complex geological structure of the Sivrihisar region. Kayacı and Genç [14] investigated using clay minerals and granites in ceramic bodies and examined this issue with physical-chemical experiments and analyses. Various studies have been conducted for the phases required in ceramic production.

Işık [15] examined the sepiolite formations in the region between Türkmentokat and Karatepe in Eskişehir, performed geochemical analyses of meerschaum (nodular sepiolite), and determined that the meerschaum found in the Sarısu region was genuine. Whitney et al. [16] defined the Sivrihisar Massif, defined the metamorphic rocks in the region, and suggested that it underwent Barrovian-type metamorphism. The formation of metamorphics and their relationship with granites were modeled, and it was determined that metamorphism occurred during the Eocene period. The large-scale study by Okay et al. [17] also included the Günyüzü Pluton. As a result of new datings from granitoids, they determined that the granitoids were Late Paleocene-Early Eocene. It was suggested that the formation of granites was related to subduction and occurred simultaneously with the collision. This study investigated the geology, mineralogy, and ceramic technological properties of sepiolite in the Eskişehir region, and their usability in ceramic bodies was discussed.

2. Materials and Methods

XRF is an analysis method in which the main element oxides are determined from the samples. The sample preparation processes were carried out in the Kaleseramik R&D Laboratory. First, the samples were broken one by one using a tungsten carbide Fritsch pulverized 1 model jaw crusher. In order to ensure homogeneity of the broken samples, sampling was done using the quadrupling method. To remove the samples' moisture, they were dried in a laboratory-type oven at 105°C for 1 day. The dried samples were passed through a 2 mm sieve and ground in a laboratory-type electric mortar. Pellets were prepared from the samples passed through a 63-micron sieve, and primary oxide element analyses were carried out with a Panalytical Axios brand-type XRF device.

Some samples passed through a 63-micron sieve during XRF studies were separated for XRD analysis. Powder samples were taken with a metal spoon and placed in a Pyrex container; a few drops of water were added, mixed, and turned into a paste. The prepared pastes were applied to glass coverslip sections. After drying, they were placed in the PANalytical X'Pert Pro MPD Diffractometer CuKα radiation brand type device in R&D for XRD shootings. The peaks obtained were evaluated with HighScore Plus software.

In order to determine the thermal properties of the samples determined to be dolomitic sepiolite as a result of XRF and XRD studies, Differential Thermal Analysis (DTA), Thermogravimetric Analysis (TG), and dilatometer methods were used in Kaleseramik R&D. The samples, which were dried at 25°C and turned into powder, were analyzed with the Seteram brand Labysy EVO model DTA-TG-DSC device.

Thermal and optical experiments were conducted to determine the physical-mechanical properties of clay samples from the field. 300 grams of the crushed and quartered samples were taken and placed one by one into ceramic ball containers with a spoon, then 700 grams of water, 1.5 grams of glass water, and 0.6 grams of sodium tripolyphosphate (STPP) were added. The reason for adding these materials is to facilitate the separation of grains in acidic environments. Ceramic containers were placed in a laboratory-type mill device and left to rotate for 10 minutes.

The clay samples passing through the 63-micron sieve were placed on trays by spreading them homogeneously and put in the oven for 24 hours to dry. The samples from the oven were ground by rotating in an agate electric mortar. To gain plasticity, the powdered sample was given 15% of its weight in homogeneous moisture; a 25 g sample was taken and placed in a laboratory-type hydraulic press. Tablets were pressed by applying 36 bar pressure, and the lengths of the pressed tablets were measured using tape.

The prepared tablets were fired at 1125°C for 40 minutes, the wall tile condition in a Sacmi model roller kiln of the Kaleseramik Factory. The length (%), shrinkage (%), and water absorption tests of the samples brought from the furnace were performed according to ISO-EN 10545-3. Color values (L, a, b) were measured with a Minolta brand 3600d Colorimeter device.

3. Results and Discussion

3.1. Mineralogical and Chemical Analysis of Clays in the Kurtşeyh Region

32 samples were collected from the sepiolite-bearing succession of the Kurtşeyh region. Their XRF analysis was carried out to determine the chemical characteristics. The compositional ranges, average chemical compositions of 30 samples, and the two typical samples (SEP-A and SEP-B) are shown in Table 1. In dolomitic sepiolite materials, there is an increase in CaO, MgO, and a decrease in SiO_2. The SiO_2 amount is low for dolomite samples, and CaO and MgO are high and equal in proportion. The other chemical feature is the high LOI, as expected from the carbonaceous rocks. The XRF results combined with the XRD results predicted that the samples studied were sepiolite, dolomitic sepiolite (SEP–B), sepiolitic dolomite (SEP–A), clayey (and siliceous), and common dolomite.

Table 1. XRF results for the samples

SAMPLE	LOI	SiO_2	Al_2O_3	TiO_2	Fe_2O_3*	CaO	MgO	Na_2O	K_2O	MnO	P_2O_5	SO_3
Min-Max	13.50-43.29	3.31-43.12	0.31-12.4	0.04-0.72	0.09-4.98	7.77-37.36	3.12-24.26	0.03-0.47	0.08-2.94	Nd	0.08-0.19	0.12-0.48
Average	34.97	19.22	2.98	0.22	1.13	22.14	18.15	0.19	0.59	Nd	0.12	0.16
SEP-A	21.48	43.12	7.90	0.62	2.61	8	14.22	0.26	1.43	Nd	0.11	0.15
SEP-B	33.71	24.68	1.74	0.20	0.44	15.6	23.04	0.12	0.18	Nd	0.13	0.21

Compositional ranges of the 30 samples, averages, and XRF analysis for the SEP-A and SEP-B samples. LOI: loss on ignition. Here, * denotes total iron. Nd: below detection limits. SEP-A and SEP-B are from [18]

This classification was made for predictive purposes only and does not fully reflect the real character of the material. To more clearly estimate the character of 32 samples that were thought to have a composition that could be used for ceramic production due to XRF analysis, powdered samples were subjected to XRD analysis.

As a result of the examinations, the maximum peak of the sepiolite mineral was determined at 12.5 Å. In addition, sepiolite mineral peaks at 4.4 Å and 4.2 Å. The maximum peak was determined at 2.88 Å for dolomite. Calcite was encountered at 3.03 Å peaks. For quartz, 4.26 Å and 3.34 Å peaks were used for identification purposes.

Table 2 shows some of the rheological properties of the SEP-A, SEP-B, and İstanbul clays. Slip viscosity, flow rate, and thixotropic aspects of SEP-A and SEP-B increase due to their complex structure with needle-shaped fiber agglomerates [19]. The anisotropic structure of sepiolite provides an advantage in the presence of its layered and fibrous structures.

Table 2. The rheological properties of the SEP-A, SEP-B, and İstanbul clays.

Material	Weight volume (gr/lt)	Flow rate (sn)	Viscosity (cp)	Tixotrophic aspect (sn)
İstanbul clay	1650	25	400	32
SEP-A	1660	98	1800	127
SEP-B	1650	89	1700	120

3.2. DTA and TG Tests

As a result of XRF and XRD studies, DTA and TG experiments were performed on a sample suitable for ceramic production. The purpose of this experiment is to characterize the material with thermal processes. The temperature of the sample is gradually increased during the experiment, and the mass change in response to heat is measured. The purpose of the DTA and TGA experiments is to find the temperature at which the physical and chemical water in the raw material decomposes and obtain information about phase or crystal transformations and structure decompositions.

The DTA-TG curves of the sepiolitic dolomite samples (SEP-A, SEP-B) are illustrated in Figures 2a and 2b, respectively. Four weight loss events (peak) are seen in the curves, which occur at around 105°C, 300°C, 650°C, and 800°C. The first endothermic peaks concern the evaporation of residual water. The second weight loss occurs probably due to aluminum and iron hydroxyl hydroxylation. The third and final weight loss is related to the decomposition of $CaCO_3$ and $MgCO_3$. The loss of water and dehydration of clay minerals involve the mass loss of samples.

According to the study, it was concluded that the material is suitable for use in real ceramic recipes. This is because, after 830°C, the material starts to remain stable; that is, the material begins to behave precisely like clay. Before 830°C, the deformation in the crystal structure was visible. The abundance of sepiolite shows that it is in accordance with the prescription.

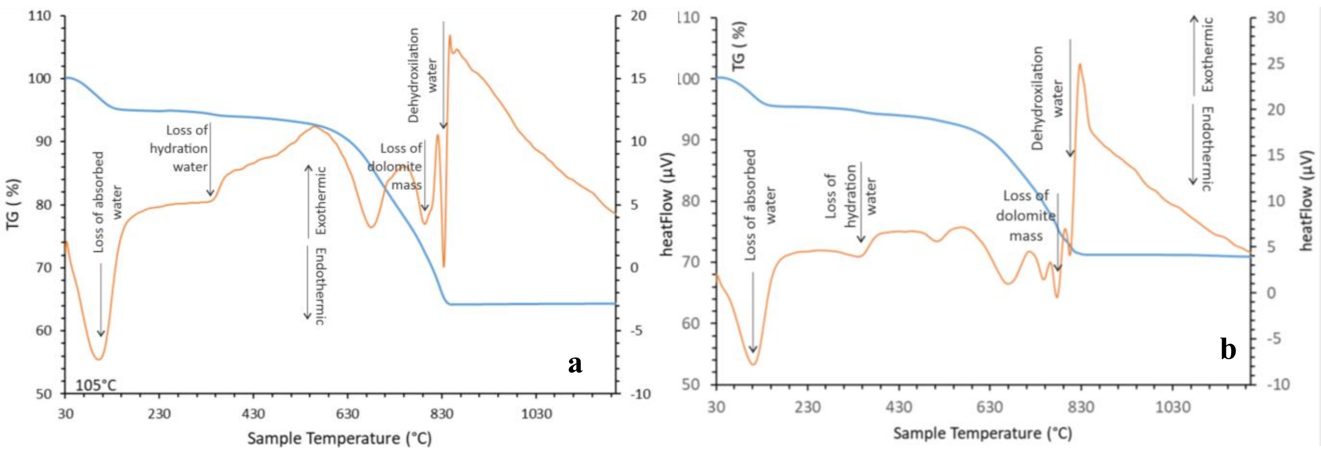

Figure 2. DTA-TG analysis graph of Kurtşeyh dolomitic sepiolite (SEP-A and SEP-B) ground for 60 min

3.3. Thermal and Optical Behaviors

In order to test the suitability of different samples taken from the Kurtşeyh region for use in ceramic production, the changes that occur after firing were determined. The firing temperature of the tablet samples was determined as °C, firing time (min), shrinkage (%), water absorption (%), and color change values L (whiteness), a (yellowness), b (redness) (not shown here). The Sepiolite A sample sintered later than the Sepiolite B sample due to its high SiO_2 and low MgO content. In Figure 3, the % shrinkage and % water absorption values of Sepiolite A and B at different % utilization rates support this conclusion. The results of these tests, samples SEP-A and SEP-B, were chosen to appear very suitable for ceramic tile production and are shown in Figures 3a and 3b. As can be seen, SEP-A and SEP-B for wall tiles shrank by nearly 1% compared to their initial state after being fired at 1125°C for 40 minutes. Usually, the shrinkage value is expected to be between 4-6% for clays, but it is pretty low for this material. This shrinkage value indicates that the raw material is suitable for high amounts in the recipe.

The water absorption value was determined to be 25-32%, which is relatively high compared to normal clays. This value indicates that the material creates visible porosity. The L value was measured between 77 to 92. This shows that the material is highly white, a desired color in ceramic production.

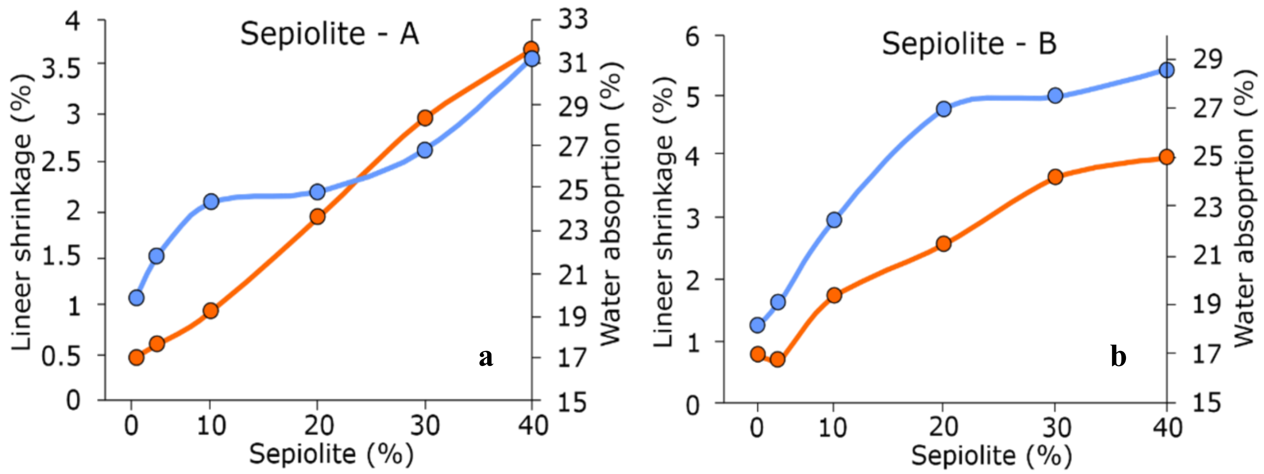

Figure 3. Thermal and water absorption test results of the SEP-A and SEP-B samples taken from the Kurtşeyh region (the blue lines show the linear shrinkage, and the orange ones are water absorption curves).

3.4. The Use of Kurtşeyh Sepiolite in Ceramic Recipes

As a result of the XRF, XRD, thermal, mechanical, and optical experiments, samples that could be used as alternatives to clay minerals in ceramic recipes were determined. Two (SEP-A and SEP-B) were selected and used for the real ceramic recipes. First, the question of what ratio of dolomitic sepiolite should be added instead of clay minerals in the recipe should be understood to obtain more efficiency. Tables 3 and 4 show the chemical and proportional aspects of the components of newly prepared recipes. As can be seen in Table 4, 40% dolomitic sepiolite was used instead of clay minerals.

Table 3. XRF results of the recipe components

	L.O.I.	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	CaO	MgO	Na_2O	K_2O
Marble	44.02	0.65	0.34	0.01	0.05	55.02	1.06	0.05	0.04
Kaolin	9.26	65.38	23.23	0.78	0.32	0.24	0.03	0.02	0.37
Clay	8.78	56.70	24.23	1.05	2.34	0.69	1.04	1.19	2.04

Marble comes from Çanakkale marble/calcites, and Kaolin and clay are from Şile and Ukraine. For SEP-A and SEP-B, see Table 1. Partly after [17]

Table 4. Body formulations by adding dolomitic sepiolites (SEP-A and SEP-B) in varying proportions instead of clay (after [17])

Raw Material	STD	A-3	B-3	A-5	B-5	A-10	B-10	A-20	B-20	A-30	B-30	A-40	B-40
SEP-A		3		5		10		20		30		40	
SEP-B			3		5		10		20		30		40
Marble/Calcite	12	12	12	12	12	12	12	12	12	12	12	12	12
Kaoline	38	38	38	38	38	38	38	38	38	38	38	38	38
Clay	50	47	47	45	45	40	40	30	30	20	20	10	10

Later, these different recipes were again subjected to optical and thermal experiments, and the extent to which the structures changed was examined. Results are shown in Tables 5, 6. We have compared the linear thermal expansion values of the Standard wall tile with the formulations of A-40 and B-40 (Tables 5 and 6). The Linear thermal expansion (20-400°C) value for the Standard wall tile decreased from 66.45 to 63.34 and 62.17 for A-40 and B-40 formulations, respectively. At the same time, moisture expansion values decreased from 0.034% to 0.016% and 0.014%. From non-contact optical dilatometer curves, it was determined that the fastest temperature values at which sintering occurs are 1006°C in the standard body, 970 and 861°C in the bodies with sepiolite addition (Figures 4a and 4b). As the amount of dolomitic sepiolite increases, improvements in thermal properties are observed, and the degree of whiteness also increases.

Table 5. Thermal and optical behaviors of the SEP-A added bodies at various rates

SEP-A Recipe	STD	A-3	A-5	A-10	A-20	A-30	A-40
Linear thermal expansion (20-400°C)	66.45	66.22	65.45	64.98	63.43	62.45	62.34
Moisture expansion (%)	00.34	0.031	0.026	0.022	0.024	0.018	0.016
L	75.23	77.50	77.29	78.80	83.46	88.92	92.56
a	6.16	4.83	3.91	3.98	2.26	1.07	0.65
b	18.41	16.70	17.54	16.14	13.36	9.36	6.17

Table 6. Thermal and optical behaviors of the SEP-B added bodies at various rates

SEP-B Recipe	STD	B-3	B-5	B-10	B-20	B-30	B-40
Linear thermal expansion (20-400°C)	66.45	66.10	65.22	64.67	63.33	62.21	62.17
Moisture expansion (%)	0.034	0.028	0.022	0.019	0.017	0.016	0.014
L	75.23	75.56	75.05	77.49	82.09	83.91	84.99
a	6.16	4.99	4.91	3.63	1.71	1.45	1.25
b	18.41	18.07	20.35	15.31	13.17	11.73	10.86

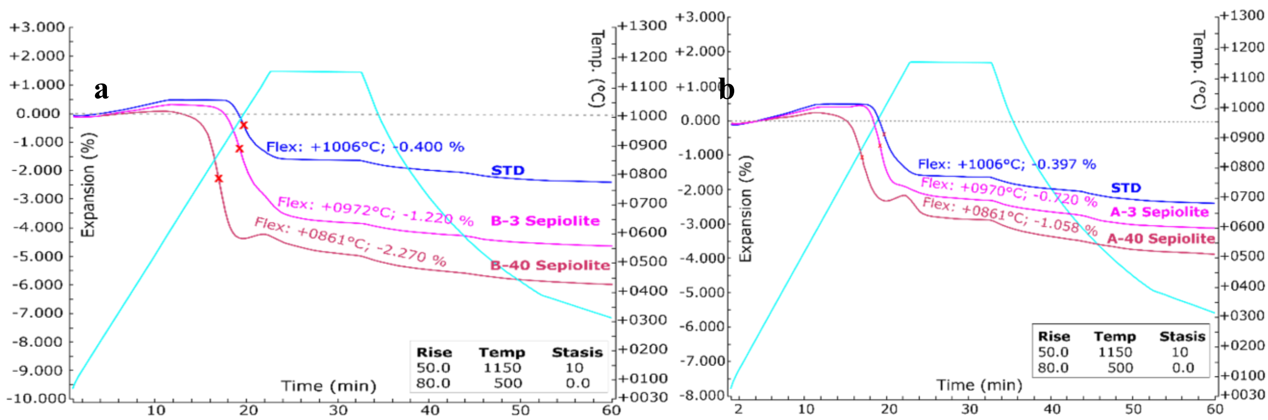

Figure 4. Non-contact optical dilatometer curves for the standard (STD), Recipe-A (A-3, A-40), and Recipe-B (B-3, B-40)

XRD analysis was done on the different fired bodies to understand the new mineral phases. Figures 5a and 5b display the quartz (COD: 96-101-1173), anorthite (COD: 96-901-1632), and enstatite (COD 96-900-1179) phases on the XRD graph. As a result of the increase in dolomitic sepiolite instead of clay, quartz phases decrease, and anorthite and enstatite phases are formed [18]. The decrease in quartz means an increase in crystallization, which is a positive result of using dolomitic sepiolite instead of clay. The enstatite phase indicates the importance of the usability of dolomitic sepiolite compared to clay as an element that increases strength and durability. Anorthite is an indispensable phase for wall tiles regarding porosity and dimensional stability. Sivrihisar dolomite-bearing sepiolites possibly transformed to enstatite at 845°C and amorphous silica at 862°C. This transformation can be formulated as follows [20, 21]:

$$Si_{12}O_{30}Mg_8(OH)_4 \dashrightarrow 4Mg_2Si_2O_6 + 4SiO_2 + 2H_2O$$

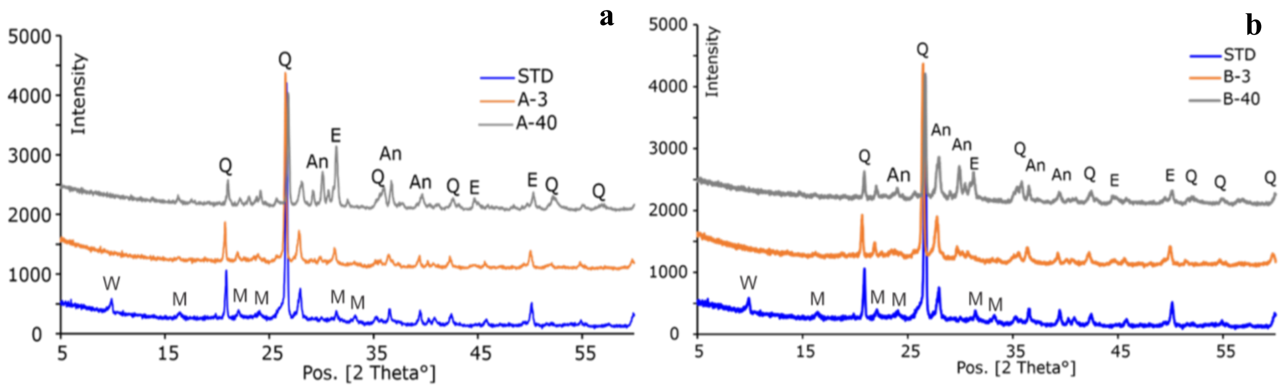

Figure 5. Mineral phases of the fired bodies by XRD (Q: Quartz, An: Anorthite, E: Enstatite, W: Wollastonite, M: Mullite, after [18])

4. Conclusion

In the literature, sepiolite minerals are not commonly used as alternatives to clay minerals used in ceramic recipes. The main reasons for this are that, because of the physical properties of sepiolite, appropriate results cannot be obtained in ceramic recipes (especially since it creates rheological problems), and its reserve is quite low compared to other clay minerals in the world and our country. These physical properties' shrinkage and water absorption values are not considered suitable for recipes. The water absorption value and shrinkage were quite high compared to the clays. This study observed that dolomitic sepiolites processed from the Kurtşeyh region are suitable for use in ceramic production based on XRF, XRD, and other physical and optical tests. This is because dolomite accompanying the sepiolite mineral changes the material's physical, optical, and thermal properties. Sepiolite was transformed into enstatite through dolomite, which positively affected the test results.

Author Contributions

All the authors equally contributed to this work. They all read and approved the final version of the paper.

Conflict of Interest

The author declares no conflict of interest.

Ethical Review and Approval

No approval from the Board of Ethics is required.

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