

Use and characterization of sugar beet molasses as a strength enhancer in glazed porcelain ceramic floor tile bodies

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ARTICLE INFO

Dates:

Received: 03.02.2026

Accepted: 05.06.2026

Doi:

10.65206/pajes.1880880

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ABSTRACT

Context—Sugar factories produce large quantities of sugar beet molasses as a byproduct. Although molasses is used in some industrial sectors, our research has shown that it has not been used as a strength enhancer in glazed porcelain ceramic floor tile body sludge granules for ceramic press machines.

Objective—The aim of this article is to investigate the use of sugar beet molasses as a strength enhancer in ceramic floor tile body sludge granules for ceramic press machines in ceramic factories, and to fill this gap in the literature.

Method—Press machines are used in the shaping process of ceramic floor tile production. Due to the lower-than-required moisture content and dry strength values of the granules, approximately 10% losses occur in ceramic tile production during and after glazing, and at the firing stage. To minimize these losses and achieve this at the lowest possible cost, sugar beet molasses, a byproduct of sugar factories, were added to standard floor tile body sludge at weight percentages of 0.1%, 0.2%, 0.3%, 0.4%, and 0.5% to increase its strength.

Subsequently, the changes occurring before and after the addition were analyzed using X-ray fluorescence (XRF) spectroscopy, SEM analysis, thermogravimetric analysis (DTA-TG), and physical properties (strength, color measurement, density, flow time, sieve residue, particle size, dry strength, fired strength, shrinkage, water absorption, etc.) and incorporated into the process. The analysis showed that standard floor tile body sludge containing 0.3% sugar beet molasses had higher strength than standard body floor tile sludge without molasses.

Results—In a ceramic factory that consumes approximately 250 tons of granules per day with ceramic press machines, an average of 7500 tons of glazed porcelain ceramic floor tile body granules are used monthly. Considering that the 10% loss due to strength loss in ceramic press machine production can be recovered, the waste of 750 tons of granules per month will be prevented. The studies have shown that using waste sugar beet molasses as a strength enhancer in glazed porcelain ceramic floor tile body slurry granules in ceramic press machines will provide significant economic benefits to the company as well as support sustainability in the sector.

Conclusion—The use of sugar beet molasses will contribute to the sustainable development of the ceramics and sugar industries, prevent environmental pollution, and simultaneously reduce the cost of ceramic tiles. By reusing waste molasses, the company will achieve significant economic gains and will pioneer environmentally friendly production practices.

Key Words—Glazed porcelain ceramic floor tile, Glazed porcelain ceramic floor tile body sludge, Molasses, Sugar beet.



I. INTRODUCTION

The ceramics industry is one of Türkiye's oldest and fastest-growing sectors. The sector improves its products and increases their variety every year. The Turkish ceramics sector, with a production value of approximately 4.6 billion USD and exports exceeding 1.8 billion USD, is among Türkiye's important industries. The ceramics sector provides 68,000 direct and 500,000 indirect jobs in the Turkish economy. Companies producing ceramic tiling materials export their products to 148 countries, while companies producing sanitary ware export to 140 countries. Thanks to its growing production capacity, modern technology investments, and high-quality advantages, the competitiveness of the Turkish ceramics sector in world markets is also increasing. Ceramic tile production, one of the fastest-growing sectors thanks to advancements in technology and scientific research, continues to increase market share for manufacturers and offer consumers unlimited options. The Turkish ceramic tiling materials sector, especially with the investments it has made since 1990, has managed to become a major player in world ceramic tile production. According to 2022 data, Türkiye, with a production of 385 million m², is the 3rd largest producer of ceramic tiles in Europe and the 9th largest in the world, and the 3rd largest exporter of ceramic tiles in Europe and the 5th largest in the world [1], [2]. Ceramic tiles are widely used in construction and interior design, as well as various industrial applications, due to their high hardness, stability, ionizing radiation shielding capabilities, and excellent wear resistance [3]-[6]. Besides the raw materials used in their production—quartz, feldspar, and clay—the components that enhance the structural integrity and visual appeal of the final product play a significant role [7]-[9]. Of these raw materials, quartz provides thermal and dimensional stability, hardness and abrasion resistance, while feldspar contributes to the overall strength and density of ceramic tiles. Clay provides flexibility, formability and dry mechanical strength [10]. As is known, wall, floor, and glazed porcelain tile bodies are quite different from each other in composition. Floor and glazed porcelain tile bodies are relatively dense and non-porous structures, whereas wall tile bodies are porous. In floor and glazed porcelain tile bodies, clay, quartz, and alkaline raw materials are used, while in wall tile bodies, unlike these, marble, wollastonite, and small amounts of calcite and dolomite are used [11]. The high cost of these raw materials and the possibility of their depletion over time make the production process difficult in terms of economics and sustainability [12]. Under the conditions of an increasingly globalized world and growing competition, producing without compromising quality, at low cost, and in line with customer satisfaction has become the goal of every company. It is necessary to continuously research and develop alternative sources for high-quality raw material resources that are depleting day by day, discover new fields, and use existing resources more efficiently. Due to shrinking reserves in recent years, the supply of high-quality raw materials in the ceramic industry has become increasingly difficult. In this context, research into alternative raw material sources and studies on the usability of waste materials resulting from production or process waste of other industrial sectors is steadily increasing. Finding alternative materials will provide economic gains in return for rising raw material prices and, considering the limited nature of these resources, environmental sustainability [13]. Recently, efforts to reduce the cost of raw materials used in ceramic tile production include incorporating materials such as fly ash [14], glass shards [15], filter press tile cakes [13], slag [16], [17], ceramic sanitary ware waste [18] and cement raw mix waste [19] into ceramic tile compositions.

In this article, the use of sugar beet molasses as a strength enhancer in the glazed porcelain ceramic body granules of ceramic press machines in ceramic factories was investigated. Sugar produced in many countries around the world is of great

importance in human nutrition. Beet sugar production, which was 38.3 million tons in the 2023/24 period, is expected to increase by 1 million tons in the 2024/25 period, reaching 39.3 million tons [20]. Molasses constitutes approximately 4% of the total beet mass and generally comprise 75-86% dry matter and 14-25% water [21]. Türkiye, a major global producer of beet sugar, ranks fifth in the world and fourth in Europe. Sugar production in Türkiye is estimated to reach 2.76 million tons in the 2024/25 period [20]. Sugar beet molasses is a low-value byproduct of the sugar industry [22] and was used mostly as animal feed in the past [23]-[25]. It has also been used in various industrial areas, one of which is the concrete industry as a water-reducing and retarding additive [26]-[28]. Molasses is also preferred by the cement industry because of its lack of toxic substances and its economic and logistical advantages [29]-[31]. In addition, detailed studies have been conducted on the effect of molasses on the properties of clay soils [32]-[36]. However, it was mainly used as a raw material for ethyl alcohol production and as a growth medium for yeast production [37]. Nevertheless, the wastewater remaining from these processes contains high concentrations of organic matter and brown pigment [38]. Molasses causes pollution of groundwater and increases disposal costs. Wastewater discharged directly into the environment without adequate treatment pollutes groundwater and causes significant environmental problems [39], [40].

Considering the dwindling resources resulting from ever-advancing technology, the importance of waste recycling becomes even more apparent. Nowadays, the use of waste in various sectors not only reduces the cost but also reduces environmental pollution caused by waste accumulation [41], [42]. Since the use of waste materials will reduce natural ingredients, energy consumption and environmental pollution will be prevented and economic gains will be achieved [43]. Finding a suitable recycling method for molasses, which is produced in large quantities as a byproduct in sugar factories in addition to sugar production from sugar beets, would be significantly beneficial for both the sugar industry and the environment. Although molasses is used in some industrial branches as mentioned above, according to our research, it has not been used as a strength enhancer in glazed porcelain ceramic body clay granules. The purpose of this study is to examine the use of sugar beet molasses in the ceramics industry and fill this gap in literature. The use of sugar beet molasses will contribute to the sustainable development of the glazed porcelain ceramic tiles and sugar industries, preventing environmental pollution and simultaneously reducing the cost of ceramics.

II. EXPERIMENTAL SECTION

A. Materials

A Spectro Xepos device was used for XRF analysis, a Netzsch Proteus STA 449 F3 Jupiter device for thermal analysis, a Netzsch DIL 402 Expedit Classic brand Dilatometer for thermal expansion, a Nannetti CC-96-2006 for strength, a Nannetti Press EA for shaping, a Malvern Panalytical Mastersizer 3000E for size distributions, and a Mitutoyo CD-20APX for size measurement. A Sacmi Forno brand factory industrial kiln was used for firing (single firing), a Xrite CI6X device for color measurement, a Ceramic Ins. BC 4100 device for density measurement analysis, and a Ceramic Ins. VSVD as a water absorption device. Tescan Vega and Quorum Q 150R ES Plus devices were used for SEM analysis and carbon coating.

B. Method

Experimental studies were carried out according to the physical and chemical properties of the original glazed porcelain body composition. The body composition of the glazed porcelain ceramic tiles contains approximately %50 feldspar, %35 clay and %15 kaolin. Grain size and sieve residue measurements were

made first since they were similar to each other in terms of chemical properties. In these experimental studies, firstly, waste sugar beet molasses supplied from the Kütahya (Türkiye) sugar factory was added to the glazed porcelain ceramic floor tile body sludge at certain rates, and density and viscosity measurements were made, respectively, on the slurries added at the determined rates. After these analyses, the plant body sludges taken as standard samples and added with sugar beet molasses were dried and granulated at 550 °C in spraydryer machine, and granule grain distributions were measured. The granulated slurries were formed into dimensions of 55 x 110 mm primarily in a laboratory press with an average humidity of 5.7% and a pressure of 400 kg/cm². All shaped samples were dried by keeping them in a laboratory oven at 150 °C for 24 hours. While some of the dried samples were separated for dry strength analysis, some were fired for firing. In our factory (industrial kiln), since the products are single-fired, all products in the tests were single-fired. Physical and chemical analyses were performed on all the dried and fired samples. Optimum operating conditions have been taken into consideration in all the experiments conducted.

1. Determination of molasses ratios to be added

Sugar beet molasses was added to the operating body sludge in the determined proportions. These rates are determined according to the sugar beet molasses addition amount rates in Table 1.

2. Calculation of the amounts of molasses to be added

When calculating the amount of molasses to be added to the operating body sludge, the addition was calculated according to the dry amount contained in the sludge with a density of 1700 g/L (1000 g dry amount), as in (1):

$$(DA) = (AS)(AD), \quad (1)$$

where *DA*, *AS*, and *AD* imply the amount of dry matter, the amount of the slurry to be taken, and the amount of dry matter of the sludge of the determined density, respectively.

Dry matter content in sludge with a density of 1700 g/L (%) = 64.09% and total slurry mixture to be taken to obtain 1000 g of dry matter. Amount of the slurry = $1000 \times 100/64.09 = 1561$ g; Since the additions are made on 1000 g dry matter, for an additional 0.1% = 1 g; for an additional 0.2% = 2 g; for an additional 0.3% = 3 g; for an additional 0.4% = 4 g and for an additional 0.5% = 5 g sugar beet molasses additions were made.

3. Results of the density measurement experiment

The density measurement of standard sludges prepared as mixtures by mass with 0.1%, 0.2%, 0.3%, 0.4%, 0.5% sugar beet molasses addition and without addition was made using a metal pycnometer with a volume of 100 mL and a mass of 200 g, calibrated by TSE (Turkish Standards Institute). The dry and clean pycnometer was first placed on the scale and then filled with sludge and its perforated lid was closed. The sludge overflowing from the hole on the lid was cleaned and pycnometer weighing was performed. The read value was multiplied by 10 and the result was calculated as g/L. The density measurement results of standard sludges prepared as mixtures by mass with

0.1%, 0.2%, 0.3%, 0.4%, 0.5% sugar beet molasses addition and without addition are shown in Table 1.

Density and flow time measurements are performed to ensure control of the conditions required in the process. For the granulation of the sludge, under operational conditions, the density should be 1680–1720 g/L, and the flow time should be a maximum of 35 seconds [44].

According to Table 1, the densities of the samples (ML-1-ML-5) with molasses addition were found to be in accordance with the desired values and slightly lower than the value of ML-0 without molasses addition. Density changes of clay related to the use of some industrial waste such as sanitary plumbing waste products [18] and cement raw mix waste powders [19] in porcelain tiles have been reported.

4. Flow time analysis

The flow time measurement experiments of standard sludges that mass mixtures are prepared 0.1%, 0.2%, 0.3%, 0.4%, 0.5% with and without molasses addition were performed using a viscosity container (Ford-cup) with a diameter opening of 4 mm calibrated by TSE. A thoroughly mixed sludge was filled into the viscosity container, the opening of which was kept closed. The stopwatch was started as soon as the hole of the viscosity cup opened. The stopwatch was stopped as soon as the sludge in the container was completely drained and the flow stopped. The value read on the stopwatch shows the flow time of the sludge in seconds. The second flow time measurement was made as described below.

The sludge was mixed thoroughly and filled into the viscosity container, and it was left to stand for 10 minutes without being subjected to any stirring or shaking. Then, the hole of the viscosity cup was opened and the stopwatch was started at the same time. As soon as the sludge in the container completely ran out and the flow stopped, the stopwatch was stopped and the value read on the stopwatch gave the second time of sludge flow in seconds. The flow times of the prepared sludges are shown in Table 1.

According to Table 1, it was found that the flow times of ML-1, ML-2, and ML-3 with added molasses were shorter than the maximum flow time of 35 seconds, while the flow times of ML-4 and ML-5 were longer than the maximum flow time. In a study where sugarcane molasses was used as a cement additive, it was reported that cement mortars containing molasses exhibited higher fluidity compared to those without [45].

5. Dry strength measurement

The strength measurements of sludge samples with and without molasses additives, prepared in mass mixtures of 0.1%, 0.2%, 0.3%, 0.4%, and 0.5% molasses, were performed using the three-point bending method. In the experiment, pieces shaped by pressing at a pressure of 400 kg/cm² and dried, with dimensions of 55 × 110 × 8.5 mm, were used. The shaped samples were dried at 150 °C for 24 hours before measurement. The strength values of the mixtures were calculated using (2):

$$MOR(DS) = (3 \times p \times l)/(2 \times b \times h^2 \times 9.81), \quad (2)$$

where *MOR(DS)*, *p*, *l*, *b*, and *h* denote the dry strength (kg/cm²)(N/mm²), average breaking strength (N)(kgm/s²), support distance (cm), width (cm), and thickness (cm), respectively. The constant 9.81 is in (m/s²) and states the gravitational acceleration (in the three-point bending test, two points are fixed, and the middle point is pulled downward).

The strength measurements of sludge samples with and without molasses additives, prepared in mass mixtures of 0.1%, 0.2%, 0.3%, 0.4%, and 0.5% molasses, are shown in Table 1. Dry strength measurement is controlled to ensure that the green products can move along the production line (150 meters)

Table 1. Proportions of the additional amount of planned sugar beet molasses and some characteristics of samples without molasses addition (ML-0) and with molasses addition (ML-1-ML-5).

Samp. code	Percent. (%)	Density (g/L)	Flow time (s)	MOR(DS) (N/mm ²)	MOR(FS) (N/mm ²)
ML-0	0.0	1700	23	4.26	42.88
ML-1	0.1	1698	23	4.69	42.85
ML-2	0.2	1695	24	5.23	42.80
ML-3	0.3	1695	32	5.47	42.87
ML-4	0.4	1693	37	5.33	42.60
ML-5	0.5	1692	43	5.13	42.66

without deformation. Due to operational requirements, it must be at least 3.5 N/mm².

As shown in Table 1, adding molasses to the operating body sludge has been shown to increase dry strength. The dry strength measurements of the ML-1-ML-5 samples with molasses addition have the desired values and are greater than the dry strength value of the ML-0 sample without molasses addition. A study on the compressive strength of cements containing sugarcane molasses at different hydration ages showed that when less than 0.04% of sugarcane molasses was added, the compressive strength gradually increased with increasing sugarcane molasses content [31]. These increases can be attributed to the water-reducing effect of sugarcane molasses [28]. It has been stated that the use of molasses in cement production has positive effects on concrete compressive strength [21].

6. The fired strength measurement results

Fired strength measurements of standard sludge bodies, both with and without molasses additions at 0.1%, 0.2%, 0.3%, 0.4%, and 0.5% by weight, were performed using the three-point bending method. The mixtures shaped to dimensions of 55 × 110 × 8.5 mm were fired (single firing) at 1200 °C/49 min (industrial kiln), after which the strength tests were conducted. The strength values of the mixtures were calculated using (3):

$$MOR(FS) = (3 \times p \times l) / (2 \times b \times h^2 \times 9.81), \quad (3)$$

where $MOR(FS)$ denotes the fired strength (kg/cm²)(N/mm²).

The strength measurements of standard sludge bodies prepared with and without molasses additions at 0.1%, 0.2%, 0.3%, 0.4%, and 0.5% by weight are presented in Table 1. Fired strength measurement is measured and controlled in accordance with TSE Double Star criteria. The firing strength measurement value must be at least 40 N/mm². As seen in Table 1, the firing strength measurement values of the samples with molasses addition (ML-1-ML-5) were found to be in accordance with the TSE standards and slightly lower than the firing strength measurement value (ML-0) before the addition of molasses. It has been observed that the amount of pumice added up to 7% instead of clay in porcelain tile production increases the firing strength values, but after 7%, the firing strength values decrease [10].

7. The firing color analysis

Firing color measurements of standard bodies prepared with and without molasses additions at 0.1%, 0.2%, 0.3%, 0.4%, and 0.5% by weight were performed using Lab spectrophotometer. In the experiment, mixtures shaped to dimensions of 55 × 110 × 8.5 mm were fired (single firing) at 1200 °C/49 min (industrial kiln), after which color measurement tests were conducted. The results of the color measurement tests conducted on the samples are presented in Table 2.

The firing color analysis was carried out to investigate the effect of the added molasses on the body color. The firing color of ceramic tiles is affected by factors such as the substances in the raw material composition, added additives, surface roughness, and firing conditions [46]. When the firing colors of the samples were compared with the reference material (ML-0), it was determined that the body whiteness value and parameters a and b generally decreased with increasing amount of added molasses.

Table 2. Results of the firing color analysis.

Sample code	L (whiteness)	a (+ red, - green)	b (+ yellow, - blue)
ML-0	66.49	4.06	14.36
ML-1	66.22	3.92	14.14
ML-2	66.03	3.90	14.10
ML-3	66.33	4.01	14.20
ML-4	65.66	3.86	14.05
ML-5	65.32	3.86	14.08

However, it was found that the body whiteness and parameters a and b in the sample with 0.3% molasses addition (ML-3) were closer to the values of the reference material (ML-0). Even if the firing conditions and additives were the same, the composition ratio affected the color parameters of the tiles [18], [47].

8. The densities and the particle size distributions

Firstly, the glazed porcelain ceramic body sludges that mass mixtures are prepared 0.1%, 0.2%, 0.3%, 0.4%, 0.5% with and without molasses addition were measured the density with the density cup of 100 ml calibrated by TSE. Then the glazed porcelain ceramic body sludges that mass mixtures are prepared 0.1%, 0.2%, 0.3%, 0.4%, 0.5% with and without molasses addition were measured the size distribution with Malvern Panalytical Mastersizer 3000E.

The densities of the glazed porcelain body sludges (with and without molasses addition) and the particle size distributions for these processes were shown in Table 3. The particle size distribution analysis is controlled in accordance with operational requirements. The specifications are d97; 55-60 µm, d50; 8-12 µm, and min. 97 % passing 63 microns.

The addition of 0.01-0.05% molasses based on cement weight is useful in increasing the grindability of blended cement, especially in reducing the particle size of granulated blast furnace slag. Therefore, the addition of molasses to cement plays an important role in improving the hydration reactivity of granulated blast furnace slag and increasing the strength of blended cements [48]. Although it was determined that the samples were of appropriate size and density, their density decreased depending on the increasing molasses ratio (Table 3). The decrease in intensities is within the desired range, 1680–1720 g/L.

9. Granule particle size analysis

The spray dryer unit produces granules in accordance with the special operating conditions, with a particle size distribution of min. 25 % retained on the 500 µm sieve and max. 2% passing through the 90 µm sieves. Therefore, the added molasses does not have any effect on the granule particle size distribution [44]. The granulations of the operating body sludge (with and without molasses addition) by granulating, i.e. the granular grain size distribution, are shown in Table 4.

10. The results of chemical analysis (XRF)

Since the added molasses are organic compounds, they have only affected the loss of ignition (LoI). They have not significantly affected the other oxides. The molasses we added, being organic material, had only a limited effect on the LoI. It did not cause any appreciable change in the oxide composition. The chemical (XRF) analysis results of the plant body sludge (with and without molasses additives) are shown in Table 5.

11. The results of total shrinkage measurements

In the total shrinkage test, the initial measurement of the granule samples shaped in the press was taken as the size of the press mold (I_1). After the shaped samples were dried at 150°C for 24 hours, they were fired (single firing) at 1200°C/49 min (industrial kiln). The fired length of the samples (I_2) was measured after firing. The results were calculated as the total shrinkage percentage using (4). The data of the mixtures with calculated total shrinkage are shown in Table 6.

Table 3. The densities and the particle size distributions.

Sample code	Density (g/L)	10 µm (%)	Particle size		
			63 µm (%)	d50 µm	d97 µm
ML-0	1700	48.11	97.80	10.35	56.70
ML-1	1698	47.98	97.85	10.33	56.66
ML-2	1695	48.00	97.86	10.30	56.53
ML-3	1695	47.88	97.90	10.37	56.35
ML-4	1693	48.15	97.88	10.40	56.40
ML-5	1692	48.00	97.91	10.41	56.38

Table 4. Humidity and particle size and distribution of granule.

Sample code	Humidity (%)	> 500 μ	> 400 μ	> 300 μ	> 200 μ	> 90 μ	< 90 μ
ML-0	5.70	26.37	25.64	24.70	14.32	7.87	1.10
ML-1	5.70	26.14	25.87	24.51	14.62	7.79	1.17
ML-2	5.70	26.42	25.21	24.33	14.77	8.11	1.16
ML-3	5.70	26.63	25.41	24.30	14.85	7.61	1.20
ML-4	5.70	26.08	25.67	24.59	14.48	7.93	1.25
ML-5	5.70	26.44	25.62	24.17	14.35	8.28	1.14

Table 5. The results of chemical analysis (XRF).

Sample code	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	TiO ₂	SO ₃	LoI
ML-0	67.52	18.92	2.24	0.41	0.44	3.11	2.89	0.58	0.02	3.85
ML-1	67.36	18.62	2.27	0.41	0.45	3.09	2.92	0.59	0.03	3.87
ML-2	67.45	18.88	2.29	0.42	0.42	3.08	2.90	0.59	0.02	3.89
ML-3	67.50	18.80	2.25	0.42	0.43	3.12	2.88	0.60	0.03	3.90
ML-4	67.43	18.53	2.37	0.44	0.45	3.10	2.90	0.60	0.03	3.92
ML-5	67.46	18.73	2.26	0.43	0.43	3.14	2.87	0.57	0.01	3.95

Table 6. The result of total shrinkage measurements.

Sample code	1st measurement (I_1) (mm)	2nd measurement (I_2) (mm)	TS (%)
ML-0	110	101.20	8.00
ML-1	110	101.15	8.04
ML-2	110	101.17	8.03
ML-3	110	101.21	8.00
ML-4	110	101.20	7.99
ML-5	110	101.18	8.01

$$TS (\%) = [(I_1 - I_2)/I_1] \times 100, \quad (4)$$

where TS implies the total shrinkage as in percentage. Total shrinkage measurement is carried out because the press mold is designed according to the firing shrinkage of the product. When comparing the shrinkage of the reference material (ML-0) with the molasses-added samples (ML-1-ML-5), it was observed that the shrinkage increased with increasing molasses addition in samples ML-1, ML-2, and ML-5, the total shrinkage did not change in the sample with 0.3% molasses addition (ML-3), but the total shrinkage was lower in the sample with 0.4% molasses addition (ML-4) compared to the others. On the other hand, ceramic mixtures containing raw material additives such as calcium oxide and magnesium oxide have been reported to be resistant to shrinkage during the firing process. This is because in coarse crystalline fused clinkers, calcium compounds with low melting points are found in small clusters among larger calcium oxide and magnesium oxide crystals, thus hardening the structure [49]. In our study, the sample containing 0.3% molasses was also found to be more resistant to shrinkage.

12. The results of water absorption analysis

In the water absorption tests, the samples shaped in the press and fired (single firing) at 1200 °C for 45 minutes in the industrial kiln are kept in an oven at 100 °C for 24 hours to reach a constant weight before being immersed in water for the test. The samples, after reaching a constant weight, are weighed to obtain their initial weight (M_1) before being immersed in water. The weighed samples are kept under a vacuum of 10 kPa for 30 minutes, then immersed in water for 15 minutes. At the end of the process, the samples are lightly dried, and their second weight measurements (M_2) are taken. The water absorption results were calculated using the weight values obtained, as in (5):

$$WA (\%) = [(M_2 - M_1)/M_1] \times 100, \quad (5)$$

where WA implies the water absorption. Total water absorption and the related total open porosity are important indicators for determining the properties of sintered materials, and these data can be used to determine the suitability of ceramic tiles for outdoor or indoor use. It was reported that as the molasses content increased in the lightweight roof tile samples, the water absorption values of the samples decreased [50]. According to TSE Double Star certification and process requirements, water

absorption must be a max. 0.3 %. In our work, the calculated water absorption values were observed to be 0.09% for all samples. This means that the addition of molasses did not change the total water absorption, and the obtained values were found to be in accordance with TSE standards.

13. Thermal behavior analysis

Standard body samples prepared with and without the addition of molasses at weight percentages of 0.1%, 0.2%, 0.3%, 0.4%, and 0.5% were first dried in a laboratory furnace at 100 °C for 24 hours, then ground into powder, weighed with 0.1 g precision, and subsequently analyzed for thermal behavior using a DTA-TG device. The analysis was conducted at 1000 °C, and the effect of heat on the sample was observed. The DTA-TG graphs of standard clay bodies prepared with and without the addition of molasses at weight percentages of 0.1%, 0.2%, 0.3%, 0.4%, and 0.5% are presented in Fig. 1.

Fig. 1a shows the DTA-TG graph of the standard sludge body without molasses addition. As a result, the mass loss is observed to be 4.23%. It is a body with low organic content and controlled moisture. Between 0–200 °C, free moisture is removed. Between 200–500 °C, hydroxyl groups in clay minerals are eliminated. A low amount of organic matter is present. Between 500–650 °C (at 526 °C), dehydroxylation occurs; in particular, kaolin transforms into metakaolin. A small amount of carbonate decomposition takes place. At 784 °C, sintering begins, and the formation of a glassy phase starts.

Figs. 1b-1f show the DTA-TG graphs of the standard sludge mass with 0.1%-0.5% molasses added, respectively. As a result of these analyses, a mass loss of 4.23% was observed in the sample with 0.1% molasses added; 4.49% in the sample with 0.2% molasses added; 4.37% in the sample with 0.3% molasses added; 4.86% in the sample with 0.4% molasses added; and 4.84% in the sample with 0.5% molasses added. Kaolin transformations occurred between 523–527 °C, indicating that the body contains clay-kaolin. El-Maghraby et al. reported that the peak at 506 °C was the dehydroxylation of Kalabsha clay mineral (kaolinite) and the peak at 671 °C was the formation of metakaolinite [9]. The onset of sintering occurred between 780–784 °C, suggesting that major gas release in the body ended early and that the risk of glaze defects is low. Considering all these results, it is seen that the bodies have clean and well-balanced compositions.

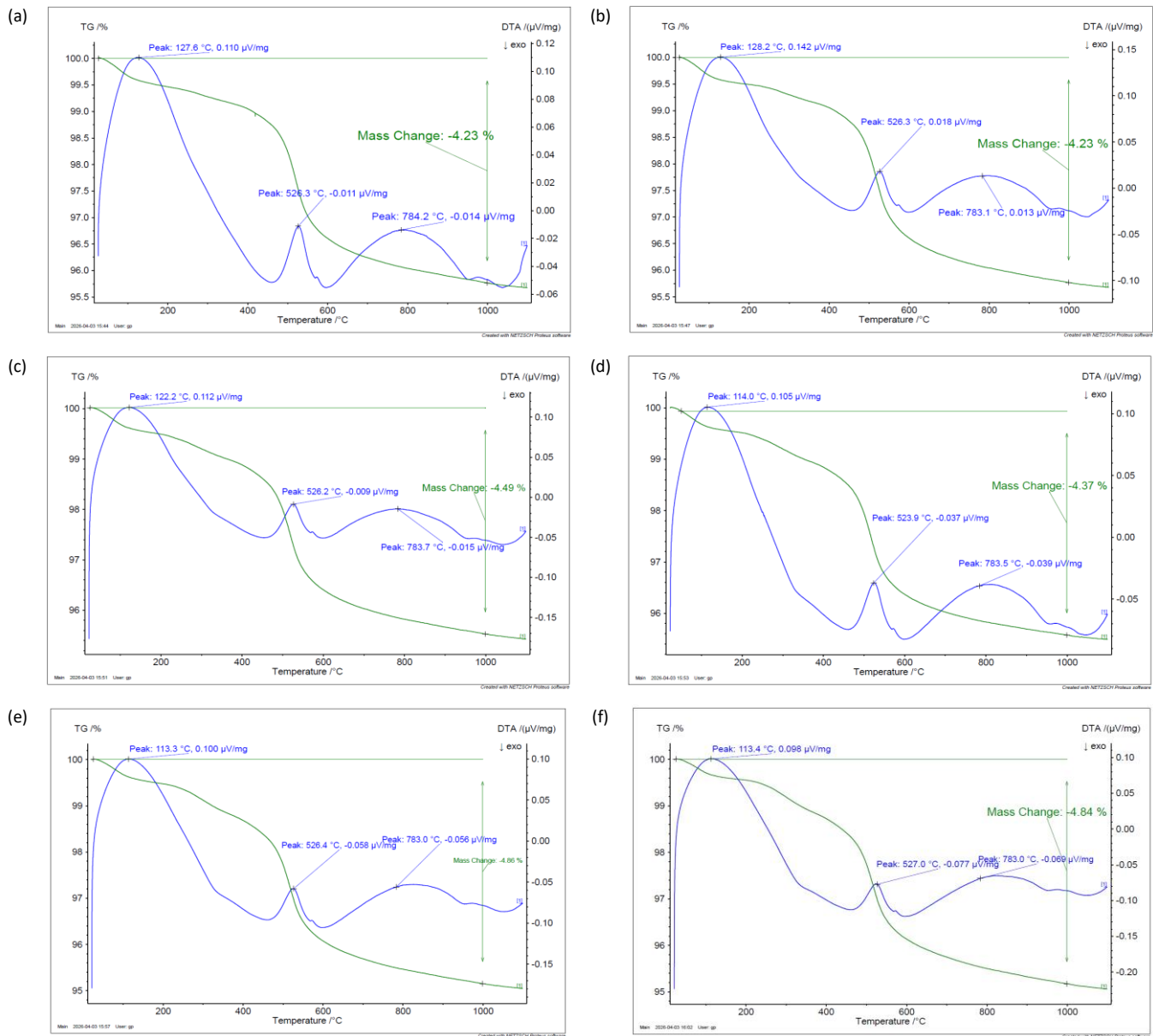


Fig. 1. DTA-TG analysis of standard dried body: (a) without molasses addition, (b) with 0.1% molasses addition by weight, (c) with 0.2% molasses addition by weight, (d) with 0.3% molasses addition by weight, (e) with 0.4% molasses addition by weight, (f) with 0.5% molasses addition by weight.

14. Thermal results of the linear thermal expansion test

Standard sludge bodies prepared without and with molasses additions of 0.1%, 0.2%, 0.3%, 0.4%, and 0.5% by weight were shaped into $55 \times 110 \times 8.5$ mm specimens using a laboratory press. After firing (single-fired) at 1200°C in an industrial kiln, the specimens were cut to dimensions of $3 \times 3 \times 50$ mm for linear thermal expansion analysis and tested using a dilatometer. The linear thermal expansion results of the standard sludge bodies prepared without and with molasses additions of 0.1%, 0.2%, 0.3%, 0.4%, and 0.5% by weight are presented in Fig. 2 and Table 7 respectively. The linear thermal expansion coefficients of the samples between 50°C and 400°C were calculated as follows: $68.242 \times 10^{-7} \text{ 1/K}$ for the standard sludge mass without molasses addition; $68.711 \times 10^{-7} \text{ 1/K}$ for the standard sludge mass with 0.1 wt% molasses; $68.401 \times 10^{-7} \text{ 1/K}$ for the standard sludge mass with 0.2 wt% molasses; $68.545 \times 10^{-7} \text{ 1/K}$ for the standard sludge mass with 0.3 wt% molasses; $68.909 \times 10^{-7} \text{ 1/K}$ for the standard sludge mass with 0.4 wt% molasses; and $68.319 \times 10^{-7} \text{ 1/K}$ for the standard sludge mass with 0.5 wt% molasses (Fig. 2).

According to the results in Table 7 when the thermal expansion results, water absorption results, and total shrinkage results are evaluated together, it is observed that the thermal expansions are very close to each other, the water absorption values are the

same, and the total shrinkage values are almost the same. This strongly indicates that (i) the microstructures are similar, (ii) the formation of the glass phases is similar, and (iii) densifications and pore structures are similar. This also shows that the added molasses acts only as a temporary organic binder and is removed during firing. It does not have a significant effect on water absorption, structure, or thermal properties. From a ceramic perspective, this means that it is suitable in terms of firing behavior, dimensional stability, and glaze-body compatibility.

The risk of cracks, bloating, or abnormal shrinkage is very low. Therefore, the addition of molasses does not disrupt the standard recipe; since it is organic, it completely burns out during firing and leaves the system without leaving any residue that would alter the structure. Since production in our factory is single-firing, the samples in our experiment were also fired using single-firing process in the industrial kiln. Therefore, the necessary explanations were provided together with the water absorption, total shrinkage and thermal expansion values.

15. SEM analysis results

Standard sludge bodies prepared without and with molasses additions of 0.1%, 0.2%, 0.3%, 0.4%, and 0.5% by weight were shaped into $55 \times 110 \times 8.5$ mm specimens using a laboratory

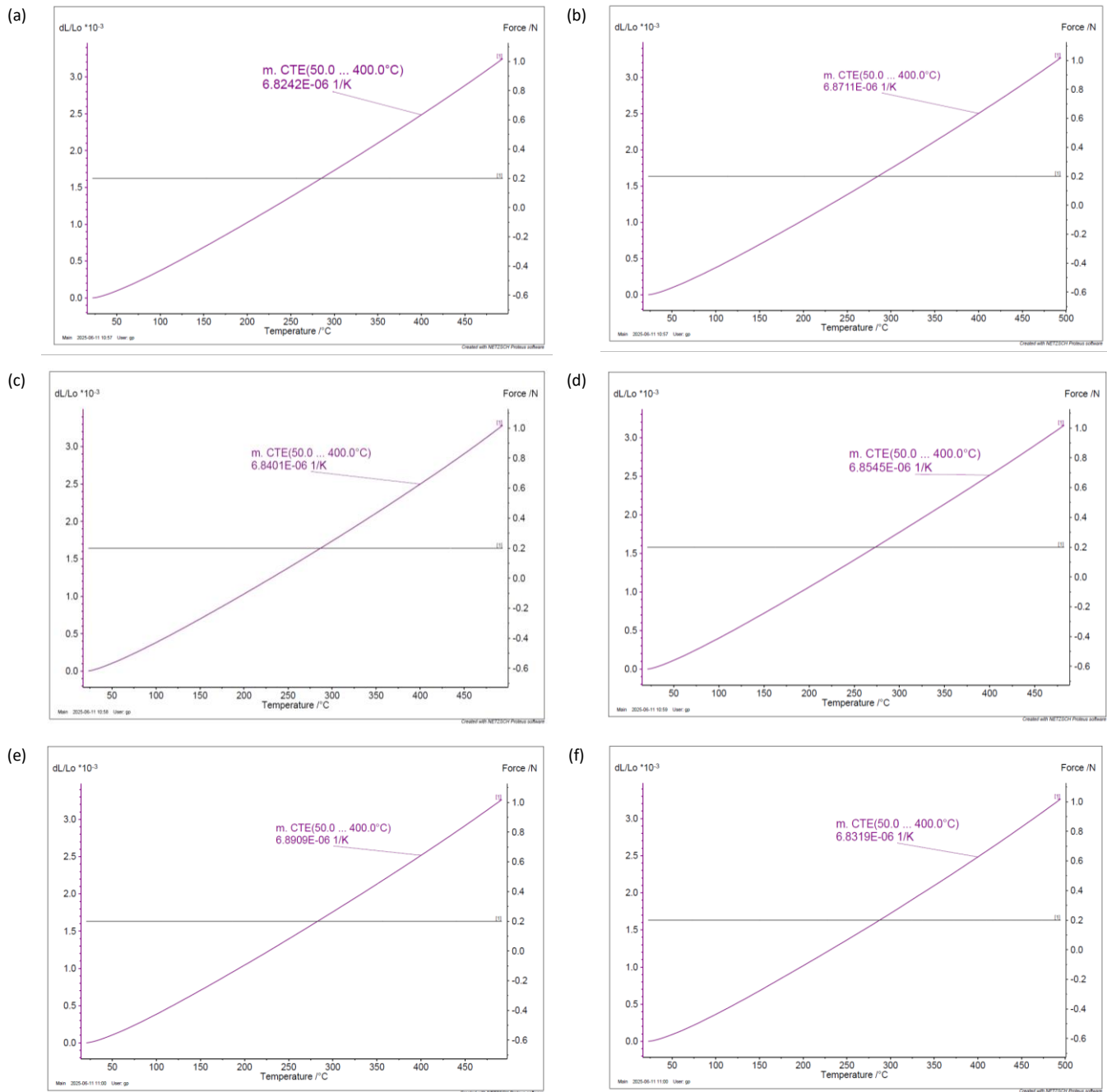


Fig. 2. Linear thermal expansion analysis of the standard fired body: (a) without molasses addition, (b) with 0.1% molasses addition by weight, (c) with 0.2% molasses addition by weight, (d) with 0.3% molasses addition by weight, (e) with 0.4% molasses addition by weight, (f) with 0.5% molasses addition by weight.

Table 7. Results of total shrinkage, water absorption, and T.E.C measurements.

Sample code	TS (%)	WA (%)	T.E.C ($\times 10^{-7}$ 1/K) (50-400°C)
ML-0	8.00	0.09	68.24
ML-1	8.04	0.09	68.71
ML-2	8.03	0.09	68.40
ML-3	8.00	0.09	68.45
ML-4	7.99	0.09	68.90
ML-5	8.01	0.09	68.31

press. After firing (single-fired) at 1200 °C in an industrial kiln, the specimens were cut to dimensions of 10 × 10 × 8,5 mm for SEM analysis and coated with carbon and the analysis was performed using a Tescan Vega SEM analysis device in Kale Seramik R&D Center. The SEM images of the standard sludge bodies prepared without and with molasses additions of 0.1%, 0.2%, 0.3%, 0.4%, and 0.5% by weight are presented in Fig. 3.

SEM analyses revealed that all examined porcelain body series generally exhibited a similar microstructural character. In all samples, it was observed that, as is typical for porcelain systems,

crystalline phases dispersed within a glassy phase matrix coexisted with open and closed pores of different sizes. However, significant differences were identified between the series in terms of degree of vitrification, amount of porosity, morphology of crystalline phases, and microstructural homogeneity. It is thought that this may be caused by the initial sample preparation process (body preparation, granulation, pressing and packaging etc.).

In low-magnification SEM images (200x), a fine porosity structure distributed across the surface was observed in all

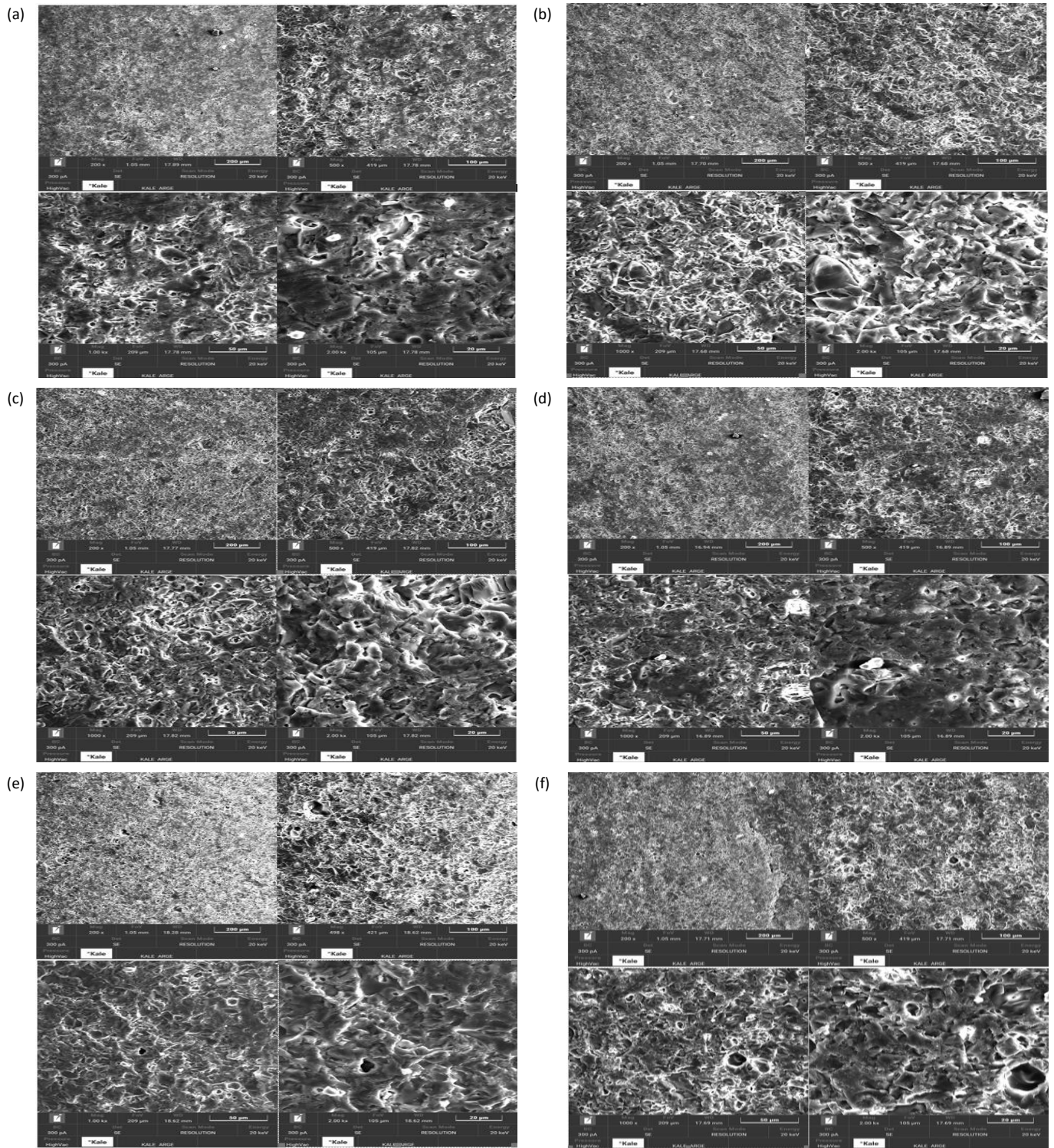


Fig. 3. SEM analysis of the standard fired body: (a) without molasses addition, (b) with 0.1% molasses addition by weight, (c) with 0.2% molasses addition by weight, (d) with 0.3% molasses addition by weight, (e) with 0.4% molasses addition by weight, (f) with 0.5% molasses addition by weight.

series. In some samples, the pores were finer and more homogeneously distributed, while in others, larger and more irregular voids were formed. Particularly in the first series, pore distribution was more heterogeneous, and large voids were occasionally present. This suggests that densification during sintering was not fully completed and that pore formation due to local gas release continued. In contrast, in later series, it was observed that the pores became smaller, more rounded, and more uniformly distributed.

At 500x and 1000x magnifications, more detailed examination of the microstructure revealed that crystalline phases became more pronounced. In particular, lamellar and angular crystalline structures drew attention in some series. These structures are

likely to be either undissolved quartz phases or mullite crystals formed during sintering. In series where crystalline phases were observed with dense and sharp boundaries, the structure was evaluated as having a more crystalline character. In contrast, in some samples, crystalline phases were observed to be more dissolved within the glassy matrix and to have a more rounded morphology. This indicates an increase in the amount of liquid phase and a higher degree of vitrification [9].

At high magnification (2000x), differences in densification between the series became much more evident. In more compact samples, it was observed that grain boundaries were largely fused, the glassy phase became continuous, and pores predominantly transformed into closed, rounded morphologies.

Such structures are considered to indicate that liquid-phase sintering occurred effectively. Additionally, the reduction in open porosity in these series suggests that densification has progressed and a structure with potentially lower water absorption behavior has formed.

In contrast, in some series, it was observed that voids persisted around crystalline phases, grain boundaries were not fully merged, and relatively large pores remained in certain areas. Particularly in samples with pronounced crystalline phases, the continuity of the glassy matrix was lower, and the microstructure exhibited a more heterogeneous character. This may be associated with a relatively low degree of vitrification, insufficient sintering time, or heterogeneity in raw material distribution.

When all series are evaluated together, it is concluded that the fundamental microstructural constituents are similar, but the structures exist at different levels of densification depending on the degree of sintering. As the sintering degree increases, the amount of glassy phase increases, pores become smaller and more rounded, grain boundaries fuse significantly, and a more homogeneous microstructure is formed. In addition, in series with a higher amount of crystalline phases, the structure is considered to exhibit a more brittle and less dense character.

Overall, the most successful microstructure can be described as one with a homogeneously distributed fine pore structure, well-developed glassy phase continuity, crystalline phases well bonded to the matrix, and a minimal amount of large open pores. Such structures are thought to provide advantages in terms of mechanical strength, density, and low water absorption. However, it should also be considered that in excessively vitrified structures, the increase in glassy phase content may lead to an increased tendency toward brittle fracture. Therefore, the optimum microstructure is considered to be one with controlled vitrification and a balanced porosity distribution.

III. CONCLUSIONS

This study presents research on the use of sugar beet molasses as a strength enhancer for ceramic body granules in ceramic press machines in ceramic factory. Press machines are used in shaping during ceramic tile production. In these presses, losses of approximately 10% can occur during and after the shaping process. The causes of these losses are the granule moisture content and the dry strength values of the composition being lower than required. In addition, losses also occur during glazing, after glazing, and at the kiln entry stages. To minimize these losses and achieve this at the lowest possible cost, sugar beet molasses, a byproduct from sugar factories, was added to the standard body sludge to enhance its strength. In this study, following the addition of sugar beet molasses an industrial waste at 0.1%, 0.2%, 0.3%, 0.4%, and 0.5% by weight to the standard body sludge physical and chemical analyses were conducted. Standard body sludge with sugar beet molasses additions were shaped under the same conditions and fired simultaneously in the same kiln. All physical and chemical tests were conducted under identical conditions. Our study has demonstrated the usability of sugar beet molasses as a viable and sustainable raw material source in ceramic tile production. The results of the research findings are summarized below:

- **Shrinkage management:** The total shrinkage measurement is performed because the press mold is designed according to the shrinkage of the product during firing. In samples ML-1, ML-2, and ML-5, shrinkage increased with increasing molasses addition. In the sample containing 0.3% molasses (ML-3), the total shrinkage did not change, but in the sample containing 0.4% molasses (ML-4), the total shrinkage was lower than the others. As a result, the sample containing 0.3% molasses was found to be more resistant to shrinkage.

- **Improved strength:** It was observed that adding molasses to the operating body sludge increased the dry strength, with the greatest increase found in the ML-3 sample containing 0.3% molasses.
- **Water absorption:** Total water absorption and the related total open porosity are important indicators for determining the suitability of ceramic tiles for outdoor or indoor use, and for defining the properties of sintered materials. The addition of molasses did not change the total water absorption rate in any sample, and the same value (0.09%) found for all samples was found to be following TSE standards (maximum 0.3%).
- **Firing color:** Even if the firing conditions and additives were the same, the composition ratio affected the color parameters of the tiles. When the firing colors of the samples were compared with the reference material (ML-0), it was determined that the body whiteness value and parameters a and b generally decreased with increasing amount of added molasses. However, in the sample containing 0.3% molasses (ML-3), the body whiteness and parameters a and b were found to be closer to the reference material (ML-0) values.
- **Densities:** Although it was determined that the samples were of appropriate size and density, their density decreased depending on the increasing molasses ratio. The decrease in intensities is within the desired range.
- **Flow times:** The flow time of the standard body sludge increased depending on the amount of sugar beet molasses it contained. The flow times of the sample without sugar beet molasses (ML-0) and standard stem slurry containing 0.1% (ML-1), 0.2% (ML-2), and 0.3% (ML-3) sugar beet molasses were shorter than the maximum flow time of 35 seconds, while the flow times of samples containing 0.4% (ML-4) and 0.5% (ML-5) sugar beet molasses were longer than the maximum flow time.
- **Thermal behaviors:** The thermal behaviors of the samples subjected to testing were similar to each other.
- **SEM analysis:** In SEM analyses, it has been generally observed that the fundamental microstructural components show similarity.

According to these findings, among the mixture ratios examined, the standard body sludge containing 0.3% sugar beet molasses was found suitable, considering that it had higher strength than the standard body sludge without molasses, the similarity rate in the test results was high, and for the granulation of the sludge under operational conditions, the density should be 1680–1700 g/L and the flow time a maximum of 35 seconds.

As a result of these studies, the addition of sugar beet molasses, an industrial waste, to the standard body sludge has been successfully implemented without any issues, providing positive contributions to the operation through waste recovery. In a ceramic factory producing with an average of 250 tons of standard body sludge granules using ceramic pressing machines, the average monthly consumption is approximately 7.500 tons of granules. In production using ceramic pressing machines, a 10% loss due to strength reduction can be recovered, preventing 750 tons of granules from becoming waste. The cost of 1 ton of glazed porcelain ceramic body clay for the company is €47 euros. From this, the return to the company, in its simplest form, will be over €35000. By evaluating the waste molasses as specified, the company will both recycle and achieve economic gains. This utilization of waste molasses will not only provide significant economic benefits to the company but also support sustainability in the industry. The company will also pioneer environmentally friendly production practices by repurposing waste materials.

AUTHOR STATEMENT

Plagiarism Check—The article has been scanned with iThenticate and found to be compliant with the journal's plagiarism policy.

Conflict of Interest—There is no conflict of interest with any person/organization in the article prepared.

Ethics Committee Approval—Ethics committee approval is not required for this article.

Use of Artificial Intelligence Tools—In this study, no artificial intelligence tools were used in article writing. All content reflects the original contribution of the author.

Funding—This study was supported by the Scientific and Research Council of Türkiye (TÜBİTAK) with grant number 119C129. The authors also thank Kütahya Dumlupınar University for their support in the Project.

Data availability—The data supporting the findings of this study were generated by the author(s) and may be obtained from the author(s) upon request.

CRedit Author Contribution—Methodology, Validation, Research, Writing - Review and Editing, Data Collection (**First Author**); Writing - Original Draft, Validation, References, Data Collection, Visualization (**Second Author**).

Acknowledgment—The authors thank the Scientific and Research Council of Türkiye (TÜBİTAK) with grant number 119C129.

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