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Original Research

Investigation of wet ground limestones on porous ceramic body properties

Yaş öğütülmüş kireçtaşlarının gözenekli seramik bünye özelliklerine etkisinin araştırılması

Emrah Durgut[a,*]

a Çanakkale Onsekiz Mart University, Çan Vocational School, Mining and Extraction Department, Çanakkale, TÜRKİYE

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A B S T R A C T

Wall tiles must have a 10-20% water absorption depending on the EN 14411 standard. In order to meet this water absorption criterion, calcite and dolomite minerals, which are in carbonated structure, are used in wall tile body composition. Manufacturers supply these minerals from limestone sources close to the enterprises, which are abundant in nature. The size of raw materials must be reduced to finer sizes (-45 µm) in aqueous medium in order to produce qualified wall tile body composition. In this context, this study aimed to investigate the effects of ground limestone samples obtained from the Derenti, Nevruz, and Terzialan regions of Çanakkale province (Türkiye), on ceramic wall tiles based on laboratory and industry-scale experiments/analyses. The limestone samples were firstly ground in a batch mill, and the grinding performances were compared in terms of time-dependent particle size, energy consumption, and capacity. Then, wall tile bodies were analyzed in terms of sintering properties and TS EN 14411 standards. The results from this study showed that Bond Work Indexes of Derenti, Nevruz, and Terzialan limestone samples were determined as 12.8, 11.7, and 13.0 kWh/t, respectively. As a result of industrial-based batch grinding studies, the energy consumption increased from 67.4 to 73.4 kW/t and the capacity decreased from 1.34 t/h to 1.23 t/h with quartz content increment of limestone samples from 0.8% to 2.1% and a ground limestone sample was gained with d90: 16.97 µm, d50: 1.85 µm, and d10: 0.37 µm values. Additionally, sintering studies showed that the shrinkage value decreased from 0.43% to 0.31% with CaO increment from 53.9% to 54.6% and wall tile with TS EN 14411 water absorption standard was obtained by using 13% limestone. In conclusion, the mineral content in limestone showed significant effects on particle size distribution, energy consumption, and capacity in the fine grinding process as well as on sintering properties of ceramic wall tile body.

***Keywords:** Limestone, Wet grinding, Sintering, Wall tile ceramic properties.*

Introduction

Ceramic tiles are produced by sintering plastic and non-plastic raw materials such as clay, quartz, calcite, and feldspar minerals at high temperatures (1150-1200°C). These coating materials are widely used on interior and exterior spaces such as walls, floors, kitchens, bathrooms, etc. The usage areas of ceramic tiles are determined by abrasion, scratch/break resistance, water resistance, easy cleaning, frost resistance, hygiene, and aesthetics properties. And, ceramic tiles are divided into different classes according to measurable properties, and, the raw materials used in the structures differ depending on the ceramic tile classes.

Wall tiles consist of three layers; body, engobe, and glaze from bottom to top. The body is at the bottom of the wall tile where the physical properties are given. Nowadays, the producers want to use local resources due to economic reasons, so the color of the body of ceramic tile becomes darker. Therefore, engobe is applied as a lining between the body and the glaze to provide decorative effects on the top surface more visible. At the top of the wall tile, the glaze is applied and the product is presented to the customer aesthetically. The water absorption value is considerably high (10-20%) compared to the floor tile due to the porous structure (TS EN 14411). Because of this feature, it adheres well to the wall and creates less vertical load due to its low density. They are not paved on the floor because they do not have as much load-bearing capacity as floor tiles.

Ceramic tile production consists of different processes from the preparation of raw materials to the packaging of the final product (Sacmi, 2002). The process starts with the preparation of the raw materials that make up the ceramic tile body, engobe, and glaze compositions. Raw materials taken from nature in line with the criteria determined by the producers are first brought to very fine sizes by size reduction and classification processes. At this stage jaw, cone, and impact crushers are used to subject the

* Corresponding author: emrahdurgut@comu.edu.tr • https://orcid.org/0000-0002-4637-7087

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raw materials individually to primary size reduction and sieved to the size to be fed to the grinding or clay mixing system. Wall tile bodies generally consist of raw materials such as clay minerals, siliceous kaolin, and limestone (Niall and Evitt, 2000). Plastic clay group raw materials are fed into a clay mixer with high-speed agitators and dispersed to reach the size of natural clays from an agglomerated state. Non-plastic hard raw materials such as siliceous kaolin and limestone are ground in separate batches in mills. If the calcite mineral in the wall tile body has not reached a fine enough particle size, it will not form a crystalline or glassy phase and remain free in the sintering stage. This will lead to thermal shock resistance problems due to moisture expansion in the product and the limestone needs to be ground to very fine sizes, separate from the siliceous kaolin (Dvoáková et al., 2021). Raw material character, mill type (batch/continuous mill), lining material (alumina, silex, rubber), grinding media, mill rotation speed, and regime are affecting parameters in the grinding process (Bazin and Lavoie, 2000; Altun, 2014; Deniz, 2021).

Limestone is composed mostly of calcite that has a glassy luster, transparent, easily breakable, coarse crystalline properties, and is chemically composed of calcium carbonate. Besides, its hardness is 3 in terms of the Mohs scale and has a specific gravity of 2.6-2.8 g/cm^3. Calcite is also used in glass, paint, paper, and plastic industries as well as the ceramic industry and needs to be ground into fine particle size in order to be used effectively (El-Sherbiny, 2015; Çayırlı et al., 2023).

In ceramic wall tile production, firstly, the particle size reduction process is applied to raw materials. Then, clay, siliceous kaolin, and limestone groups brought to fine particle size are sieved and dosed in a wet medium. Afterward, wall tile granules are obtained from the body slip, which is mixed according to the recipe ratios determined in the laboratory and sent to spray dryers. The granules are shaped in press to form green tiles and the green tiles are then dried for gaining strength. The dried tile is sintered in the kiln after decorative effects are applied on the surface and the final product is obtained. The final product is first visually inspected for quality control purposes and then subjected to tests for compliance with quality control standards to be sent to the customer. For this reason, the compliance of each process step with the production conditions is very important (Framinan et al., 2014; TS EN 14411).

In this study, the structural properties of three different limestone samples from the Çanakkale region were first characterized by chemical, mineralogical, and physical methods. Then, the grinding characteristics of limestones were identified by laboratory-scale grinding tests, and, the effects of the grinding process in separate industrial-based mills on particle size, energy consumption, and capacity were investigated. Finally, the ground limestones were mixed with clay and siliceous kaolin/fired ceramic waste to produce porous wall tile ceramic bodies, and their sintering properties were determined. The effects of limestone grinding on particle size, energy consumption, capacity, and the use of ground limestone on ceramic wall tile bodies were novel aspects of the research.

1. Materials and Methods

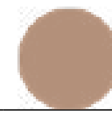
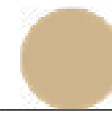
In this study, the effects of wet grinding performances of several limestone samples obtained from different regions (Derenti, Terzialan, and Nevruz) in Çanakkale province (Türkiye) on wall tile sintering properties were investigated with the combination of laboratory scale and industrial approach. In this context, firstly, chemical and mineralogical analyses of the limestone samples along with local siliceous kaolin+fired waste (SK+FW) and İstanbul clay samples were performed, within the scope of characterization studies. The chemical analyses of the samples were carried out with PANALYTICAL Brand AXIOS MAX model X-Ray Spectrophotometer (XRF) device and the mineralogical analyses (XRD) were carried out with PANALYTICAL Brand X'PERT PRO MPD diffractometer with an angular range of 3-70° between 2θ, step size 0.02, deviation slit ¼, anti-reflection slit ½. The results of the chemical and mineralogical analyses of the samples used in the experimental studies are presented in Table 1.

Table 1. Results for chemical and mineralogical analysis of the samples used in this study

Raw materials	Chemical composition									Mineralogical composition
	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	CaO	MgO	Na_2O	K_2O	LOI	
Derenti	1.4	0.3	0.0	0.1	54.3	0.8	0.0	0.0	43.1	
Nevruz	0.8	0.3	0.0	0.1	54.6	0.8	0.0	0.0	43.4	Calcite, quartz
Terzialan	2.1	0.3	0.0	0.1	53.9	0.6	0.0	0.0	42.9	
Siliceous kaolin + Fired waste	64.7	18.8	0.7	2.4	1.6	1.8	1.2	2.8	5.8	Kaolin, quartz, gehlenite, anorthite
Clay	63.2	21.6	1.2	2.8	0.6	0.6	0.5	2.1	7.2	Illite, quartz, kaolinite

LOI: Loss on ignition As seen in Table 1, the presence of calcite and quartz minerals in the mineralogical analyses of all limestone samples are the main sources of CaO and SiO_2 contents in the chemical analyses. Theoretically, calcite mineral consists of 56.0% CaO and 44.0% CO_2, which gives an idea about the CaO differences in the content of limestone samples (Rodriguez-Navarro et al., 2009). In this context, Derenti, Nevruz, and Terzialan limestones are composed of approximately 96.9%, 97.5%, and 96.3% calcite minerals, respectively. Such compounds were not seen in the mineralogical analyses due to the particularly strong calcite peak and trace amount of other minerals. SK+FW group minerals were mainly composed of kaolin+quartz and gehlenite+anorthite originating from raw materials and fired waste, respectively. The main mineral of clay was illite, and other minerals were quartz and kaolinite. The Al_2O_3, Fe_2O_3, and MgO contents are thought to be due to clay minerals, and alkaline and ferrous compounds in raw materials. The loss on ignition (LOI) value was due to the chemically bounded water of clay minerals in SK+FW and clay group raw materials (Kagonbe et al., 2021). In order to determine the sintering characteristics, the limestone, and SK+FW samples were ground to reach 2% for +63 µm, and the clay sample was mixed and sieved to -90 µm in a wet medium. Then, the ground slurries were dried in an oven at 105°C. Next, the dried raw materials were ground with agate and mortar and sieved to -250 µm and 6% humidified by spraying water to reach granules which were shaped to 5×5 cm by a laboratory press with 325 kg/cm^2 specific pressure and sintered in the wall tiles conditions. The application conditions and sintering properties as shrinkage, water adsorption, and color values of the sintered raw material groups are given in Table 2.

Table 2. Sintering properties of raw materials used in the study

Raw material group		Limestone Derenti	Nevruz	Terzialan	SK+FW	Clay
Specific pressure (kg/cm^2)		325				
Max. sintering temperature (˚C)		1150				
Sintering duration (min)		38				
Shrinkage (%)					5.0	5.4
Water absorption (%)					11.8	7.2
	L	88.0	88.8	87.2	62.7	74.9
Color	a	0.1	0.1	0.2	11.4	4.9
	b	4.2	3.8	4.9	17.1	23.5
Sintered specimen						

*Shrinkage and water adsorption analysis could not be done due to the crumbled samples.

The wall tile body subject to the study currently consists of plastic clay minerals, SK+FW, and limestone raw materials. All of the raw material groups are prepared in aqueous media in the presence of dispersants in certain proportions. The clay minerals are dispersed in a mechanical mixer and then sieved into a clay stock pool. SK+FW sample was ground, sieved, and taken to the SK+FW stock pool. Fine-sized quartz as a silica source causes rheological problems in the ceramic slip as well as high shrinkage value in the sintering process (Dana and Das, 2002). On the other hand, quartz content in the ceramic body can remain as residual quartz without reacting with calcium and aluminum during the sintering process and cannot form crystal and glass phases (Tarhan and Tarhan, 2018). Therefore, the quartz amount and particle size are important parameters for ceramic wall tile recipes. Limestone was ground to a fine particle size compared to the other raw material groups and then sieved to the limestone stock pool. Afterward, 34% of clay, 53% of SK+FW, and 13% of limestone were mixed in the dosing pool by weight and sent to the spray dryer to obtain granules with 6% moisture content. The production process used in the wall tile body preparation is shown in Figure 1.

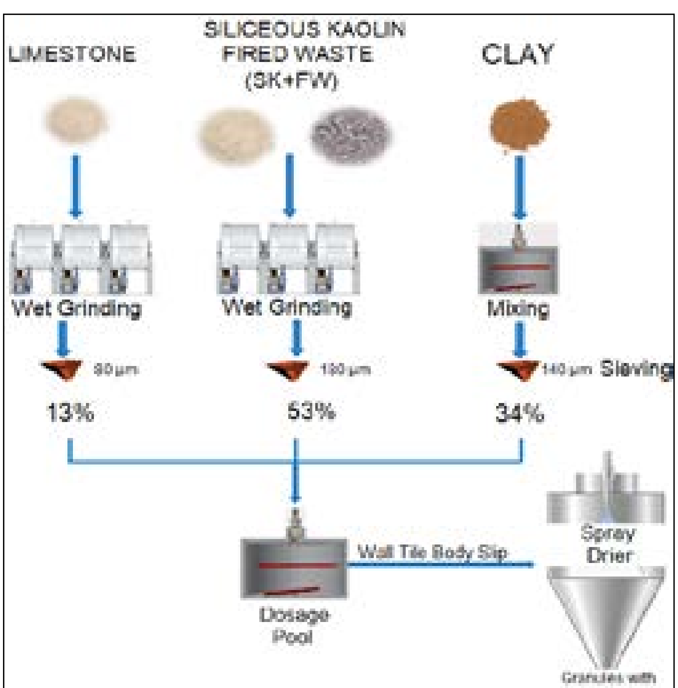


Figure 1. Wall tile body preparation process flowsheet

A laboratory-type jaw crusher was used for the primary particle size reduction of raw material. Within the scope of pre-grinding laboratory studies, 500 g of -5 mm crushed limestone samples were ground in a porcelain vibrating jet mill with a volume of 1100 mL until reaching 0.2% sieve residue for 45 µm in the presence of municipal water at 1800 g/L density and compared in terms of grinding duration. 300 g alumina grinding media were used with 50% of 15 mm, 35% of 20 mm, and 15% of 25 mm ball ratios in a porcelain vibrating jet mill. On the other hand, the grinding work indexes were also compared with a standard method, namely the Bond Work Index. The limestone samples were firstly crushed and then sieved to a size of -3.36 mm for feeding to the Bond Work Index experiments. A 30.5 cm diameter and 30.5 cm long stainless-steel standard Bond mill was used in the experiments. The mill was loaded with 22.648 kg of stainless steel balls with diameters of 38.1, 31.75, 25.40, 19.05, and 12.70 mm corresponding to 22% of the clearance volume, and operated at 70 rpm, 86% of the critical speed. In each experiment, 100% of the ball cavity volume (700 cm^3) was filled with -3.36 mm size feedstock. The experiments were performed according to TS 7700 for the standard bond work index determination.

-5 mm crushed limestone samples were fed to a batch mill with 310 cm diameter and 460 cm length and with an inside alumina liner to determine the industrial grinding characteristics. After 30% of the mill volume was filled with alumina balls, the effects of the grinding media on the yield were also investigated at a constant rotational speed of 16 rpm. The used batch mill and applied grinding parameters are given in Figure 2. The energy analysis was obtained by reading the electricity meter connected to the mill. The capacity was calculated by dividing the total grinding time by the amount of ground raw material. The particle size distribution analysis of the ground materials was performed using a Malvern Mastersizer Micro Plus laser scattering device with a measurement range of 0.03 to 555 µm.

Figure 2. Representative demonstration of batch mill and wet grinding parameters

Finally, the clay and SK+FW slips prepared according to the flow chart in Figure 1 were mixed with the limestone sample obtained from the grinding studies to obtain wall tile body granules. The granules were shaped in 5×5 cm size at 325 kg/cm^2 specific pressure and sintered in the conditions of maximum 1150ºC temperature and 38 min sintering duration and compared in terms of compliance with wall tile standards (EN 14411). Netzch brand DIL 402 CD model dilatometer was used to determine the thermal expansion coefficients. PCE XXM 30 color measuring device that had a 400-700 nm wavelength range was used for analyzing the color values (L, a, b) of sintered specimen by scanning the surface, and three analyses were applied for each specimen with 0.08 average color measurement accuracy (ΔE). Delta E (ΔE) is a calculation of the change in color as measured in three-dimensional axes L, a, and b color space. ΔE calculated from the color difference data of the colorimeter is the color difference between the product and the sample, where L_2: standard color, L_1: measuring color, a_2: standard color, a_1: measuring color, b_2: standard color, b_1: measuring color as given in Eq. 1:

$$\Delta E{:}\sqrt{[(L_2\text{-}L_1)^2 + (a_2\text{-}a_1)^2 + (b_2\text{-}b_1)^2]}$$

$$\sqrt{[(L_2\text{-}L_1)^2 + (a_2\text{-}a_1)^2 + (b_2\text{-}b_1)^2]} \quad (1)$$

2. Results and discussion

The grinding times until reaching 0.2% sieve residue for +45 µm at a constant 1800 g/L density in the laboratory porcelain vibrating jet mill and Bond work index values of Derenti, Nevruz, and Terzialan limestone raw materials are shown in Figure 3.

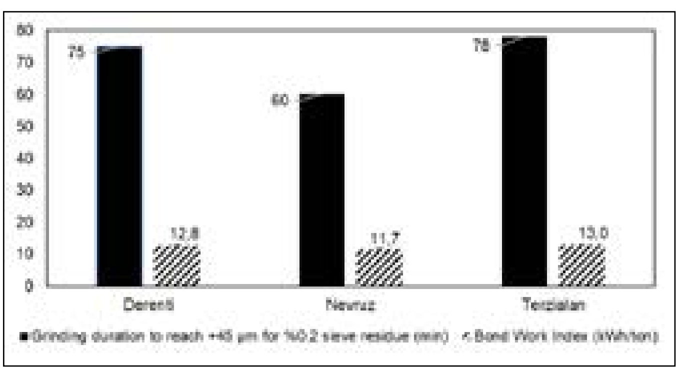


Figure 3. Results for Porcelain vibrating jet mill and Bond Work Index grinding experiments

As seen in Figure 3, there are grinding differences between Derenti, Nevruz, and Terzialan limestone samples in the laboratory-based vibrating jet porcelain mill and Bond mill. The grinding duration until reaching 0.2% sieve residue for +45 µm in the vibrating jet porcelain mill - Bond Work Index values were determined as 75.0 min - 12.8 kWh/ton, 60 min - 11.7 kWh/ton and 78 min - 13.0 kWh/ton for Derenti, Nevruz, and Terzialan samples, respectively. Limestones are composed of mainly calcite and small amounts of quartz minerals. The specific crushing energy values required for particle liberation were found to be considerably higher in quartz than in calcite (Ma et al., 2022). Quartz mineral is hard to grind compared to other raw materials in the particle size reduction of the ceramic body, so quartz behaves as a grinding medium and causes abrasion of other minerals (Haner, 2021). Besides, the Bond Work Index became higher with quartz mineral increment in the marl (Karakaş, 2006). It is also known that quartzite rock has higher specific fracture energy than limestone (Zhang and Ouchterlony, 2022). Therefore, the grinding duration in the vibrating jet mill and the Bond Work Index of the Terzialan sample are higher than the other samples due to the high quartz content and the structure due to the formed geological environment. Figure 4 shows the industrial-based grinding experiment results of Derenti, Nevruz, and Terzialan limestones in the alumina ball mill in terms of particle size change with grinding time.

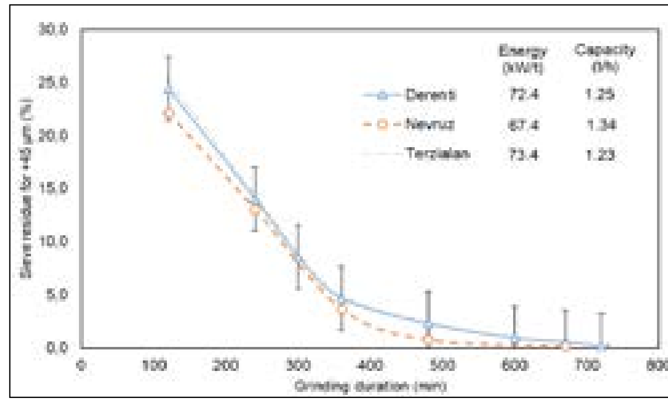


Figure 4. Results for industrial batch grinding experiments

As seen from Figure 4, 0.2% sieve residue for +45 µm was reached as a result of grinding Derenti, Nevruz, and Terzialan limestones at 720, 670, and 730 min, which resulted in 72.4, 67.4, 73.4 kW/t of energy consumption and 1.25, 1.34, 1.23 t/h of capacity, respectively.

Additionally, the ground limestone samples were conducted laser particle size distribution measurement. According to the results of laser particle size measurement performed after the grinding, the d_{90} values of Derenti, Nevruz, and Terzialan limestones were measured as 18.34 µm, 16.97 µm, and 18.79 µm, respectively (Figure 5). Laboratory results confirmed the industrial results and the Nevruz sample, which had a lower content of hard to grind quartz compared to the other samples, was ground in less time and obtained a finer size distribution.

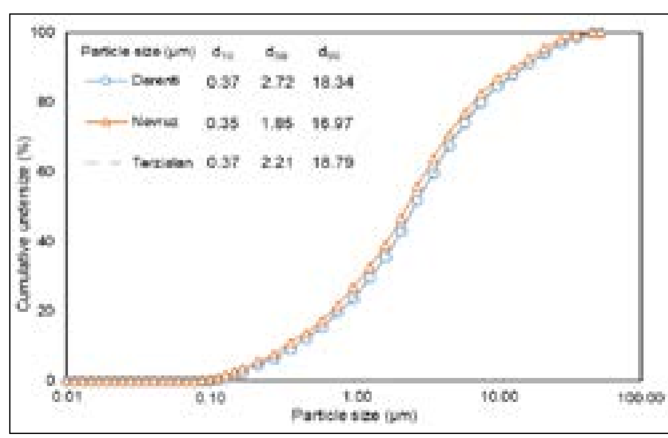
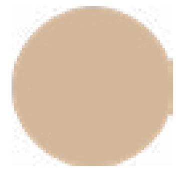
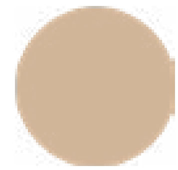


Figure 5. Particle size distribution of ground limestone samples to 0.2 sieve residue for +45 µm

Limestone samples ground to +45 µm with 0.2% sieve residue were mixed separately based on the ratios in Figure 1 and wall tile composition slurries were formed. The wall tile composition slurries were sent to a spray dryer to obtain granules with a 6% moisture content. The granules were firstly shaped in a laboratory-based press at a specific pressure of 325 kg/cm^2 in 5×5 cm size and sintered at a maximum temperature of 1150°C and 38 min (Table 3).

Table 3. Sintering conditions and results of wall tile body compositions obtained with different limestones

Wall tile composition with		Derenti	Nevruz	Terzialan
Specific pressure (kg/cm^2)		325		
Max. sintering temperature (˚C)		1150		
Sintering duration (min)		38		
Shrinkage (%)		0.36	0.31	0.43
Water absorption (%)		18.2	18.2	18.1
	L	75.6	76.1	75.7
Color	a	6.9	6.9	7.0
	b	18.2	17.7	18.3
Sintered specimen				

In the sintering studies carried out under wall tile conditions, the shrinkage-water absorption values were found to be 0.36-18.2%, 0.31-18.2%, and 0.43-18.1% for the bodies prepared with Derenti, Nevruz and Terzialan limestones, respectively. L-a-b values after sintering of Derenti, Nevruz, and Terzialan samples containing bodies were measured as 75.6-6.9-18.2, 76.1-6.9-17.7, and 75.7-7.0-18.3 (Table 3).

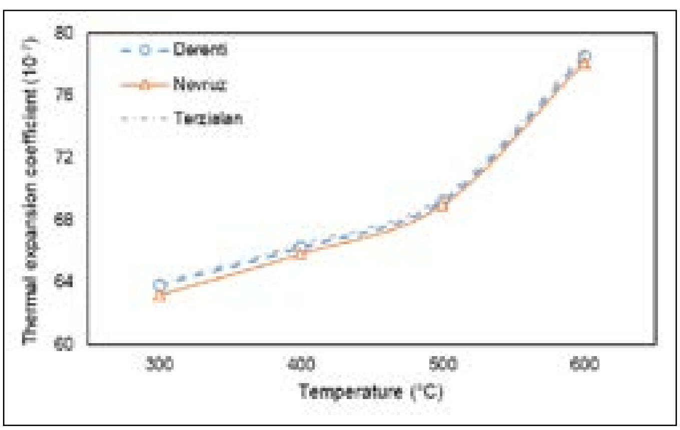


Figure 6. Thermal expansion coefficients of wall tile bodies

Thermal behavior of the wall tile body compositions prepared with Derenti, Nevruz, and Terzialan limestones are given in Figure 6 as dilatometric values. The volume change in ceramic bodies after α - β quartz transformation between 573°C significantly affects the coefficient of thermal expansion values (Johnson et al., 2020). Such change may cause crack formation during ceramic firing and is tried to be solved by passing the cooling phase slowly (Şan and Koç, 2011). While there is no important difference in the thermal expansion coefficient results of Derenti and Terzialan samples containing bodies, the thermal expansion coefficient was lower in the Nevruz sample containing body due to the low amount of quartz that was seen in chemical composition (Table 1).

3. Conclusion

In this study, the properties of different limestone samples were determined by mineralogical and chemical analysis. Then, the effects of their structural differences on grinding and porous wall tile composition were investigated by experimental studies.

As a result of mineralogical analysis, calcite and quartz minerals were observed in limestone samples. In chemical analyses, calcite components were reflected as CaO and loss on ignition, while quartz was reflected as SiO_2 value, and mineral quantity differences were observed accordingly.

The effects of mineral content in limestone on grinding were first investigated in a laboratory-based environment, and the results obtained from laboratory experiments were confirmed by industrial-based studies. It was determined that with an increase in the amount of quartz in limestone from 0.8% to 2.1%, the industrial grinding time will increase from 670 min to 730 min, the energy consumption will increase from 67.4 kW/t to 73.4 kW/t, and the capacity will decrease from 1.34 t/h to 1.23 t/h. As a result of industrial limestone grinding carried out in an alumina ball environment under the specified conditions, d_{10}; d_{50}; and d_{90} values were analyzed as 0.37; 2.72; 18.34 µm, 0.35; 1.85; 16.97 µm and 0.27; 2.21; 18.79 µm for ground Derenti, Nevruz and Terzialan samples, respectively.

As a result of the sintering studies, the increase in the CaO content in the limestone (from 53.9% to 54.6%) and the decrease in the SiO_2 content (from 2.1% to 0.8%) caused a decrease in the wall tile shrinkage value (from 0.43% to 0.31%) and thermal expansion coefficients. It has been determined that wall tile structures complying with EN 14411 water absorption standard could be obtained by using 13% ground limestone.

Quartz increment in limestone thought to be an increase in grinding time and energy consumption while capacity decreases. The increase in CaO content resulted in shrinkage decrement of the wall tile body after sintering. On the other hand, the increase in quartz in the wall tile body caused the thermal expansion coefficient increment, while the fine particle size of limestone was one of the reasons for to decrease. In conclusion, chemical and mineralogical differences in limestone structure have important effects on the grinding and sintering properties of wall tile composition.

Acknowledgments

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