Physicochem. Probl. Miner. Process., 61(3), 2025, 203420

http://www.journalssystem.com/ppmp

Physicochemical Problems of Mineral Processing

ISSN 1643-1049

Development of alternative wall tile compositions by using different industrial wastes

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Abstract: Effective utilization of industrial wastes plays an important role in reducing environmental impacts and efficient use of natural resources. In this context, it is important to investigate the usability of industrial waste materials in different application areas. In Türkiye, there are a huge amount of boron resources, and its mining wastes, together with the cutting wastes of the marble and/or limestones cutting factories. Beside these, fly ashes from the coal-burning thermic power plants, silica-rich raw material wastes used by the glass industry are in high amounts in some companies. Additionally, ceramic tile scraps of the ceramic tile manufacturers were also an important industrial waste. In this study, the potential of using boron mineral wastes from NW Anatolian boron deposits, Marmara Island limestone/dolomitic marble wastes, fly ashes obtained from Karabiga and sand wastes of the glass industry from Bilecik region in wall tile production was analysed. The waste materials were sampled on site and firstly their characterizations were carried out in the laboratory. Mineralogical properties (XRD), chemical composition (XRF), % water absorption, moisture adsorption analyses and firing behaviors in wall tile regime were investigated. Considering the results obtained in the later stages of the study, alternative wall tile compositions in which boron, fly ash, sand and marble wastes were used at a ratio of 50% were realized. The different body compositions were fired separately and their firing behaviors, moisture adsorption, water absorption characteristics were tested and the most suitable recipe for wall tiles was revealed. The production of wall tiles using different industrial wastes will provide sustainable raw material management by providing less use of natural raw materials on the one hand, and it will reduce raw material costs and ultimately reduce CO_2 emissions.

Keywords: mining wastes, boron mineral wastes, marble wastes, sand wastes, fly ashes, CO_2 emission

1. Introduction

The fact that the ceramic raw material resources in Türkiye are diminishing and the energy input is expensive is pushing technologists to search for alternative raw material resources. Instead of the standard raw materials used in the production of ceramic tiles, there is an increasing number of studies on the reuse of waste materials resulting from the production process or raw materials from other industries, the so-called secondary raw materials (Kayacı et al, 2014; Kayacı et al, 2019; Torres et al, 2007; Tarhan et al, 2017). Among the most studied waste materials in tile compositions are fly ash, chamottes from different ceramic products such as sanitaryware and bricks, waste from the construction and demolition of buildings, mining waste, foundry sands, blast furnace slag and waste glass (Rahmannan et al, 2013; Tucci et al, 2004; Zanelli et al, 2021). Some research show that it is possible to achieve percentages above 50 wt% of residual material in the final ceramic compositions by means of an appropriate selection and combination of them (Castellano et al, 2022; Junkes et al, 2012).

Efficient reuse of the wastes in the economy and reduction of their quantities are very important in terms of both economic and environmental problems. However, when evaluating wastes, not only their disposal should be considered, but also their use in areas with high added value. This is the case in the

DOI: 10.37190/ppmp/203420

ceramic tile sector, where Türkiye stands out as one of the top 10 manufacturers in the world. In fact, annual production exceeds 370 million m^2 of tiles according to the latest literature data, with more than 20% of this production being exported (Baraldi, 2024). This underlines the sector's continued focus on quality products at reasonable costs through the rational management of resources and the use of cutting-edge technology.

Tiles are classified according to their porosity into wall tile or earthenware, floor tile or stoneware and porcelain tile. Each of these types has different properties and differs in the use for which it is designed. Concretely, the present work deals with the incorporation of wastes in wall tile compositions. These tiles are characterized by high porosity, above 10% according to ISO standards, limited mechanical strength and low linear shrinkage. This is achieved by using raw materials that promote the formation of pores during firing and delay sintering of the material.

Standard wall tile compositions consist of a mixture of clay-kaolin, calcite and quartz raw materials in certain proportions. Each of these raw materials plays a different role in the composition (Sousa and Hollanda, 2005). Therefore, in the production of ceramic wall tiles, the design of the composition, the selection and the ratio of the raw materials that make up the composition must be adjusted with great precision (Kayacı et al, 2010).

Clays are used to increase plasticity and raw strength in the shaping stages of wall tile compositions. These clays play a key role in the processability of the material and have different qualities depending on their plasticity, whiteness and fluxing properties. Another important group of raw materials used in the production of wall tiles are carbonates, in particular calcium carbonate. The decomposition of carbonates in the composition formulation results in open pores and water absorption. The reactions of the resulting oxides with other materials to form new crystalline phases (such as anorthite and gehlenite) affect important properties of the final product such as dimensional stability and moisture adsorption (Garcia et al, 2008; Traore et al, 2007). Meanwhile, quartz raw materials and sands increase refractoriness and are used as filler material.

In this study, the potential of using boron mineral wastes from NW Anatolian boron deposits, Marmara Island limestone/dolomitic marble wastes, fly ashes obtained from Karabiga and sand wastes of the glass industry from Bilecik region in wall tile production was analysed. The potential for using secondary industrial wastes in different proportions in wall tile compositions has been demonstrated. In particular, it has been shown that wall tile products made from such different industrial wastes are potential raw materials for the ceramic industry.

2. Materials and methods

2.1. Raw materials and wastes

The wastes of the raw materials include boron mineral wastes from NW Anatolian boron deposits, limestone/dolomitic marble wastes from Marmara Island, fly ash obtained from Karabiga, and sand wastes from the glass industry in the Bilecik region. Fig. 1 details the locations of these wastes.

As far as mineral boron is concerned, Türkiye has 73.4% of the world's boron reserves (https://boren.tenmak.gov.tr/tr/calisma-alanlari/rezervler.html). These are located in Kırka (Eskişehir), Emet (Kütahya), Bigadiç (Balıkesir), Kestelek (Bursa) (Fig 1). Boron mining here was started by the French in 1865 and still continues recently. The main minerals are ulexite and tincalconite. In 1969, boron was recognized as a "strategic mineral" in Türkiye. There are huge wastes from mining operations and boron processing plants. Some of these wastes were sampled and used in this study.

On the other hand, Marmara Island is a location that gave its name to marble (Marmor) and the marbles here have been used since ancient times (Fig 1). Gray banded and white dolomitic marbles are famous. Approximately 1 billion m^3 of marble resources have been identified on the island. Millions of m^3 of marble waste from many years of production and marble processing activities constitute one of the biggest environmental problems of the island. In the last few years, a small micronized grinding plant has been established to utilize the waste.

Sand waste, which is one of the main materials of this study, is provided from Camiş factories operating in Bilecik region (Fig 1). The natural material is Lower Jurassic aged feldspathic sandstones (Altınlı, 1973). This material is used for glass production and the unwanted (in this study the desired) parts are disposed of as filter press waste.

The other component used in the study is fly ash from Karabiga İçdaş thermal power plant (Fig 1). This material has been the subject of various studies and is used as secondary waste material in different industries. In addition to heavy metals, glassy/amorphous phases, quartz, CaO, anhydrite, hematite and mullite were detected in fly ash (Algül and Yoncalı, 2021).

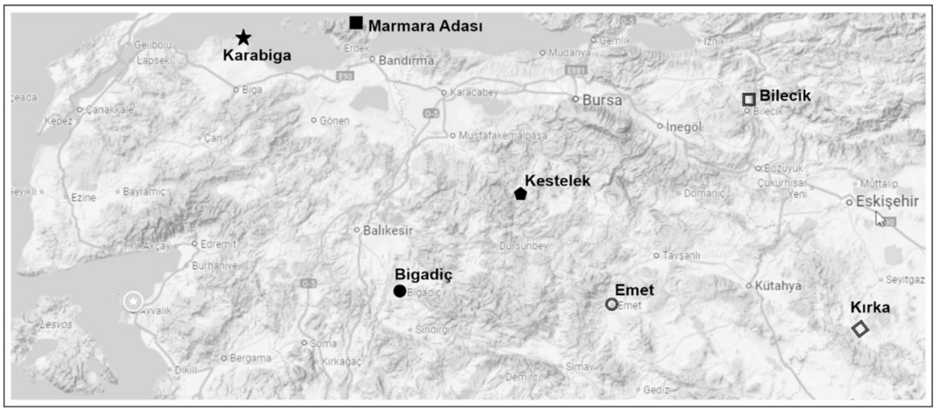

Fig. 1. Locations of the waste materials used in this study were obtained (filled and open circles, pentagon, diamond: Boron wastes, open rectangle: glass industry waste, star: fly ash waste, filled rectangle: marble waste. Base map after www.maps.google.com)

The chemical and mineralogical analyses of these secondary wastes are presented in Tables 1 and 2. For raw material characterization, chemical and mineralogical analyses of the sample were carried out with Axios Maxmodel X-ray Spectrophotometer device (XRF, PANalytical) and X'pert Pro MPD X-ray Diffractometer (XRD, PANalytical). Duplicate analysis was done for each sample, and the average values were taken into account.

As can be seen, marble dust is practically a pure calcium carbonate with a small percentage of quartz and dolomite. Fly ash is a residue from a combustion process with a chemical and mineralogical composition in accordance with the above. Tile scraps have a typical composition for wall tiles with SiO_2, Al_2O_3 and CaO. Sand waste is rich in quartz with some presence of kaolinite. Finally, boric waste shows ulexite [$NaCaB_5O_6(OH)_6\ 5(H_2O)$] according to XRD analysis and more than 7% B_2O_3 according to XRF.

Together with waste, a ball clay with high plasticity was used, whose chemical analysis is detailed in Table 3. It is an illitic-kaolinitic clay with a high percentage of Al_2O_3, over 25%, and a low percentage of chromophores (TiO_2 and Fe_2O_3).

Table 1. Chemical analysis results of the waste materials (XRF)

	L.O.I.	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	CaO	MgO	Na_2O	K_2O	B_2O_3	Total
Marble Dust	42.6	2.5	0.8	-	0.3	53.0	0.3	-	0.1	-	99.6
Fly Ash	8.2	54.7	21.5	0.8	5.5	3.7	1.6	0.8	1.9	-	98.7
Tile Scraps	0.5	66.7	17.6	0.7	1.5	7.2	1.1	1.1	2.1	-	98.5
Sand Waste	5.0	76.4	14.9	0.4	1.2	0.3	-	-	1.3	-	99.5
Boric Waste	31.1	18.5	1.3	0.2	0.3	15.6	19.7	4.0	0.7	7.3	98.7

Table 2. Mineralogical compositions of the waste materials (XRD)

Material	Mineral			
Marble Dust	Calcite	Quartz	Dolomite	
Fly Ash	Amorphous phase	Mullite	Quartz	Hematite
Tile Scraps	Anorthite	Quartz	Amorphous phase	
Sand Waste	Quartz	Kaolinite		
Boric Waste	Dolomite	Ulexite		

Table 3. Chemical analysis results of the ball clay (XRF)

L.O.I.	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	CaO	MgO	Na_2O	K_2O	Total
7.9	59.0	26.7	1.3	1.0	0.4	0.5	0.5	2.2	99.5

2.1.1. Design, preparation and characterization of proposed compositions

To maximize the use of waste raw materials, a development study for wall tile formulations was carried out. The new formulations are shown in Table 4 as WWT1, WWT2 and WWT3. The formulations are mixtures of the different waste materials with the plastic ball clay.

The compositions were prepared on a laboratory scale using the standard processing method for ceramics, which included wet milling, drying, pressing, and firing in an electric kiln. Grinding was performed in planetary mills with ball loading for 10 minutes. The slurry was dried using infrared lamps and then milled dry to produce a fine powder. This powder was mixed with water to achieve a moisture content of 5.5 wt% and pressed at a pressure of 300 kg/cm². Cylindrical samples, measuring 40 mm in diameter and approximately 5 mm in thickness, were fired using a cycle that involved a fast heating ramp up to 500 ºC, a temperature increase of 25 ºC/min from 500 ºC to the maximum temperature, and a dwell time of 6 minutes at the peak temperature. The firing temperatures were set at 1100 ºC, 1120 ºC, 1140 ºC and 1160 ºC. Additionally, a spray-dried powder composition corresponding to wall tile STD was prepared for comparative purposes.

The characterisation of the samples included both the determination of typical properties (bulk density, linear shrinkage and water absorption) and moisture adsorption.

On the one hand, the laboratory-fired samples were analysed by measuring their linear shrinkage and water absorption. Linear shrinkage was computed by comparing the diameters of the specimens before and after firing, using a caliper to measure these dimensions, and reporting this parameter on a dry basis. Water absorption was assessed using the vacuum method in accordance with the ISO 10545-3:2018 standard.

On the other hand, moisture adsorption is a property that should be minimized in fired wall tiles (Sanchez et al, 1990). Typically, this characteristic is assessed by measuring dimensional changes directly. However, due to the very small magnitude of adsorption, measuring it by weight gain is preferred for greater precision. Therefore, moisture adsorption (MAd) was determined by the increase in weight (mf–mi) of the specimens after subjecting them to an autoclave cycle at a pressure of 10 kg/cm² for 5 hours, as described in Equation (1). Prior to measuring the final weight (mf), the specimens were dried in an oven at 110 °C for 20 minutes in order to remove the water that is not actually adsorbed (Sanchez et al, 2024).

$$MAd\ (‰) = (mf - mi)/mi * 1000 \quad (1)$$

Table 4. Alternative body formulations

Material	Formulations			
	STD	WWT1	WWT2	WWT3
Ball Clay	45	45	45	45
Kaolin	35			
Carbonates	15			
Marble Dust		15	15	15
Fly Ash		20	20	10
Tile Scraps	5	20	10	10
Sand Waste		-	10	15
Boric Waste		-	-	5

3. Results and discussion

In order to investigate the mineral phases of the fired bodies of the standard and WWT1 To WWT3, we have carried out the XRD analysis. The XRD results showed the main mineral phases of the standard body was quartz (Q) + anorthite (An) + diopside (Di) + hematite (He) + gehlenite (Ghl) + amorphous phases for the WWT1 to WWT3 (Figs. 2a, d). The gehlenite was probably crystallized from the high alumina contents of the fly ash and the tile scraps. It is crystallized from the metakaolinite and calcium by the reaction of $Al_2Si_2O_7$ + (2+n) CaO → $Ca_2Al_2SiO_7$ + n$CaOSiO_2$. Later the anorthite formed from gehlenite, aluminium and silicon remaining from the metakaolinite and the quartz via the equation as follows; $Ca_2Al_2SiO_7$ + 3SiO_2 + Al_2O_3 → 2$CaAl_2Si_2O_8$ (Traore et al, 2003, and the references therein). Absence of the mullite phase in the fired bodies is possibly due to our body compositions are near to the anorthite field (Fig. 3).

Anyway, anorthite is formed in all cases, with a peak that is clearly appreciably. The presence of this crystalline phase in wall tile bodies is beneficial since it delays the sintering process at higher temperatures. This improves the ability of the tile to adsorb water, i.e. it reduces the moisture adsorption (Dvořáková et al, 2021). It also enhances dimensional stability by hindering shrinkage of the material and displacing it at higher temperatures (Yekta & Alizadeh, 1996).

Table 5 represents, the technical properties of fired bodies. The interplay between waste material types, their proportions, and firing temperature significantly influences the technical properties of the

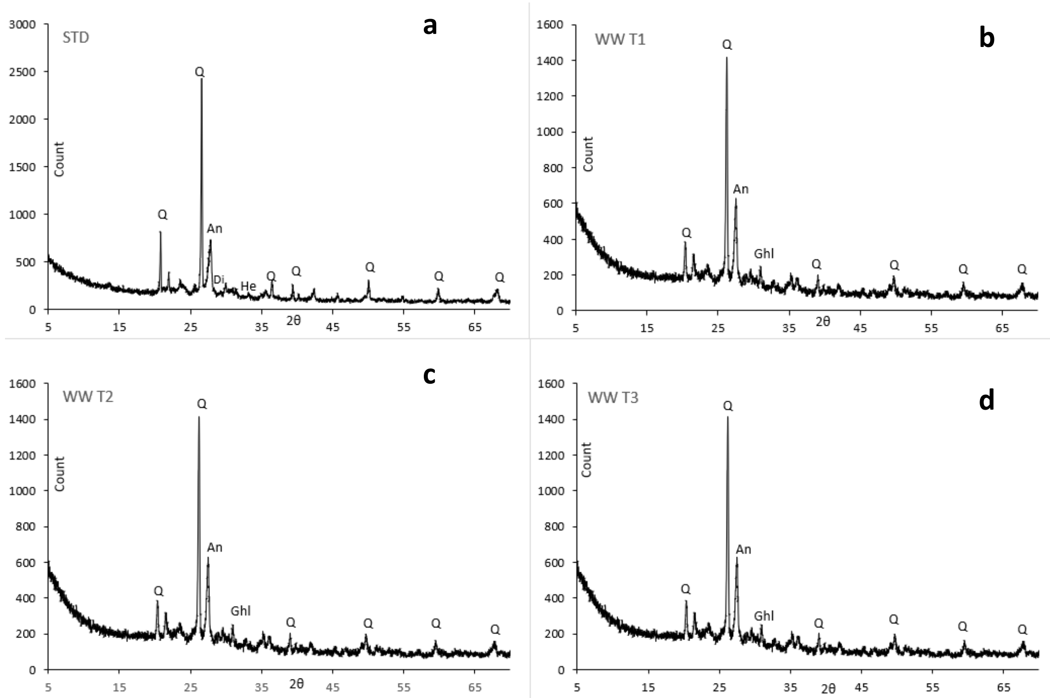

Fig. 2a-d. Mineral phases of the STD and WWT1 to WWT3 (XRD Cu Kα)

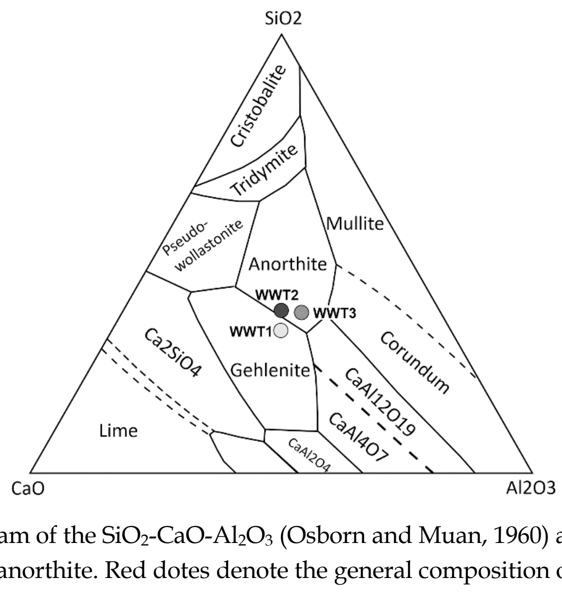

Fig. 3. Ternary phase diagram of the SiO_2-CaO-Al_2O_3 (Osborn and Muan, 1960) and the stability fields of the gehlenite and the anorthite. Red dotes denote the general composition of the body recipes.

ceramic bodies. Increasing the firing temperature raises the amount of liquid phase, which implies an increment in shrinkage and bulk density and a reduction in porosity. Moisture adsorption, a critical factor in the durability of ceramics which is related to the presence of hydratable phases after firing, decreases with temperature and amount of glassy phase. This hydration is related to expansion problems and can lead to wall detachment after installation or even breakage of the pieces.

According to Table 5, linear shrinkage (LS) and bulk density (ρ) generally rises across all samples as the firing temperature increase. The increase with temperature is more or less pronounced depending on the composition and there is always an agreement between shrinkage and density. Likewise, water absorption (WA) decreases consistently with rising temperature, reflecting improved vitrification and reduced porosity across all samples. The values vary from 21.8% for STD fired at 1100 ºC to 15.6% for WWT3 when the firing temperature is 1160 ºC.

Meanwhile, moisture adsorption (MA) is most pronounced at lower firing temperatures and in compositions with higher fly ash content, as seen in WWT1 and WWT2. This suggests that while fly ash aids in densification, it may also increase moisture sensitivity unless adequately vitrified at higher temperatures. In contrast, WWT3, which incorporates boric waste and a reduced amount of fly ash, shows significantly lower moisture adsorption, particularly at higher firing temperatures. This indicates that boric waste effectively enhances moisture resistance, likely due to its fluxing properties that promote early vitrification and reduce porosity.

Table 5. Technical Properties of Fired Bodies (Temp: Temperature, L.S.: Linear Shrinkage, W.A.: Water Absorption, M.A.: Moisture Adsorption, D: Density)

Body	Temp. (°C)	L.S. (%)	W.A. (%)	M.A. (%)	**ρ** (g/cm³)
STD	1100	0.5	21.8	5.1	1.68
	1120	0.7	21.3	4.6	1.69
	1140	1.1	20.1	2.7	1.71
	1160	2.2	17.6	0.0	1.77
WWT1	1100	0.7	20.1	8.1	1.71
	1120	0.8	19.9	6.1	1.71
	1140	0.9	19.6	4.0	1.71
	1160	1.2	18.4	1.0	1.73
WWT2	1100	0.9	18.7	9.3	1.72
	1120	0.9	18.8	6.0	1.72
	1140	0.9	18.4	4.3	1.73
	1160	1.1	17.3	0.9	1.74
WWT3	1100	1.0	19.3	1.8	1.72
	1120	1.2	19.1	0.9	1.73
	1140	1.5	18.2	0.0	1.75
	1160	2.3	15.6	0.0	1.80

For better comparison, the results are plotted in Fig. 4. On the one hand, Fig. 4a represents the moisture adsorption behaviour of formulations prepared with different waste materials. Analysing this Fig., it is clear that the type of waste material used is a crucial factor in determining how the material behaves across different temperature ranges. As discussed, WWT1 and WWT2 show high moisture adsorption at lower temperatures, while it is minimised for comparison WWT3. These findings emphasize the importance of carefully selecting waste materials based on the desired moisture adsorption properties for specific applications.

On the other hand, Fig. 4b represents the relationship between temperature and two key properties, linear shrinkage and water absorption, for the different ceramic formulations: STD, WWT1, WWT2 andWWT3. As can be seen, all formulations exhibit an increase in linear shrinkage as the temperature increases from 1100°C to 1160°C. This is expected as higher temperatures typically cause more significant densification in ceramics, leading to greater shrinkage.

Regarding compositions with waste material, WWT3 formulation shows the similar linear shrinkage with the STD, particularly at 1160 °C. This implies that both compositions start to shrink noticeably at similar temperatures, although the WWT3 formulation shows a more stable shrinkage over a wide temperature range, from 1100 ºC to approximately 1130 ºC. This is even more evident in WWT1 and WWT2. These formulations show a very stable shrinkage over the whole temperature range and similar values to STD in the central zone (1120-1140 °C). Therefore, even better behaviour and higher dimensional stability can be expected for the pieces of the compositions with waste than for the standard wall tile pieces. The firing diagram confirms the fluxing character of boric waste in relation to the other residues. As described in previous works (Zanelli et al, 2019), boron has a very strong fluxing effect on ceramic bodies and small variations in its percentage can lead to appreciable changes in porosity and linear shrinkage. The amount of boron in the used waste is not too high (around 7%), but it also has a large amount of Na_2O and other oxides that also contribute to fluxing behaviour, as shown in Table 1.

Additionally, the water absorption percentage generally decreases as the temperature increases for all formulations. This is due to the reduction in porosity as the material densifies, reducing the capacity to absorb water. The standard formulation (STD) has the highest water absorption across all temperatures, indicating that it remains more porous than the formulations containing waste materials. Anyway, all the compositions have a water absorption higher than that specified for porous wall tiles (10%) and within the usual range for this type of product (15-20%) (Dana & Das, 2002).

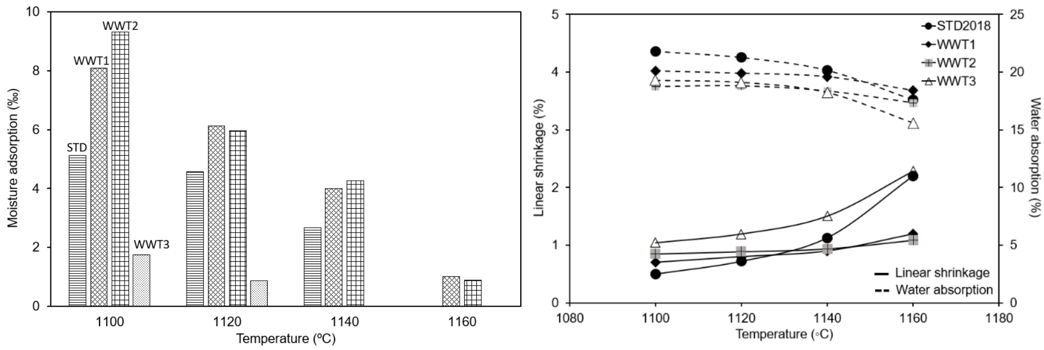

Fig. 4a, b. Moisture Adsorption of Fired Bodies (a), Water Absorption and Linear Shrinkage of Fired Bodies (b)

4. Conclusions

Model wall tile formulations were developed by simply using different industrial wastes and appropriate number of clays. Marble dust, fly ash, tile scraps, sand waste and boron mineral waste from different areas of Türkiye were selected as residues. These new formulations show similar firing behaviour and consequently technological properties to the traditionally industrial compositions. Concretely, linear shrinkage, water absorption, bulk density and moisture adsorption of laboratory test pieces have been characterized as a function of the maximum firing temperature.

The results show that it is possible to obtain wall tile bodies with suitable properties, in some cases even better than the standard starting composition, by introducing a percentage of waste higher than 50% in the formulation. This is achieved by an appropriate combination of the selected residues, based on their chemical and mineralogical composition and their fluxing or refractory behaviour. Compositions with waste could be industrially processed under the same milling, pressing and firing conditions as standard wall tile compositions.

New recipes are expected to provide ease of operation, reduce company cost and contribute to environmental sustainability of the manufacturing process. However, further modifications on shaping and sintering conditions and also trials are required in order to confirm the suitability of new recipes for industrial applications.

Acknowledgments

This research has been performed at Kaleseramik Tile Factories and Kaleseramik Research and Development Center that was supported by Ministry of Science, Industry and Technology, Republic of Türkiye.

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