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Evaluation of Basalt Cutting Waste in Coloring of Different Ceramic Glaze Compositions

Zahide Bayer Öztürk [a], Semra Kurama [b, #], Ayşe Eser [a]

[a] Nevsehir Hacı Bektas Veli University, Department of Metallurgy and Materials Engineering, Nevşehir, Turkey
[b] Eskisehir Technic University, Department of Material Science Engineering, Eskişehir, Turkey

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ABSTRACT

Color effects in ceramic glazes play a decisive role in the aesthetic and attractiveness of ceramic pieces. The colorants used in glazes interact with the ceramic materials during the firing process, resulting in a wide range of fascinating colors and effects. Due to the natural properties of basalt such as high compressive strength and wear resistance, this natural volcanic rock is attracting a lot of attention when used in ceramic production processes. The aim of this study is to evaluate the effects of basalt cutting waste (BCW) obtained from a stone company in Kayseri, Turkey, on the color effect of ceramic glazes in combination with different frits. For this purpose, glazes were prepared in which BCW was incorporated in different weight percentages of 0-20 % together with different frits. The glazes were characterized by analyzing chemical, phase, and thermal behavior of BCW using X-ray fluorescence (XRF), X-ray diffraction (XRD), and thermal microscopy, respectively. The prepared glazes were then applied to engobed wall tiles and fired at a temperature of 1,200 °C in a laboratory furnace. After firing, the glazed surfaces were subjected to color analysis, while the phase composition was analyzed by XRD. The microstructure analysis was carried out using electron microscopy (SEM/EDS). Increasing the proportion of BCW in the glaze compositions resulted in a change of the color of the finished surfaces from cream to yellow-beige tones. This study sheds light on the potential use of BCW as a coloring agent in the production of ceramic glazes.

1. Introduction

Industrial advances have the potential to improve people's health and living conditions. However, it is important to recognize that industrial development can also have negative consequences such as pollution and resource scarcity. The ceramics production sector, which plays a crucial role in the daily living environment, often generates significant amounts of waste without an effective recovery process. As technology advances in the manufacture of ceramic products, the demand for raw materials increase. However, the sourcing of such materials can lead to air and environmental pollution, depending on the energy sources used. In recent years, various factors, including conflicts and the ongoing pandemic, have led to challenges in raw material supply, subsequently impacting prices in the economy. These circumstances have highlighted the importance of sustainable practices and the development of efficient processes that minimize waste generation and reduce pollution in the ceramics industry.

The universal problems experienced in recent years

[#] Corresponding author: skurama@eskisehir.edu.tr

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are limiting the raw material resources and thus increasing cost. Improving the sintering process or replacing the initial raw materials with waste materials are used as two different methods of reducing production costs. For this purpose, studies on the use of waste materials have been increasingly carried out in recent years. To this purpose, cullet (Garbonchi et al., 1999; Matteucci et al., 2002; Gennaro et al., 2003), various industrial wastes (Andreola et al., 2002; Karamanov et al., 2006), and also some outcrops with an acidic component such as volcanic ash and zeolite (Abadir et al., 2002; Dana et al., 2004; Torres et al., 2004) are used. These materials find restricted application at various stages within the traditional ceramic industry.

Basalt is a preferred choice for various applications because of its durability and resistance to stains. Nevertheless, when shaping and cutting this natural stone product, a substantial amount of dust and crumbs can be generated as waste. The waste powders of basalt are stored by business owners in an area that is not operational (Koçyiğit and Çay, 2019).

Basalt, which has a fine-grained structure in different colors from grey to black, is in the group of volcanic rocks. It occupies a very large area (2.5 million square meters) on the earth surface and is a cheap and easily available raw material (Ercenk et al., 2018).

The chemical composition of basalt is SiO_2 and Al_2O_3 as major oxides with about 40-55 % and 10-20 % SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO and there are other oxides such as K_2O and TiO_2 (Ercenk et al., 2018).

The widespread use of this material in glass-ceramic production is due to the high proportion of silica in its chemical composition. Basalt-based glass-ceramics have good resistance to wear, abrasion, and chemicals. In addition, this material is also known for its good heat, sound, and fire resistance (Ergul et al., 2009; Binici, 2007; Ercenk et al., 2018).

The glaze applied to the surface of ceramic materials plays an important role in gaining the necessary properties (such as hygiene, visual effect, and strength) in the use of the product. Coloring of glazes, features such as glossy/matt, and surface smoothness are provided by the harmony of the colorants and additives used together with the glaze raw materials.

In recent years, the use of waste materials in the development of glazes has attracted considerable attention, both in terms of reducing running cost and environmental impact. When the studies in the literature in this area are examined, it can be seen that the waste materials are used in different areas as a source of pigment, glaze, and additive to the body. In the field of glaze application, there are studies in the literature on the use of marble waste (Yesilay et al., 2017), borax solid waste (Karasu et al., 2011), vitrified waste (Tarhan et al., 2017), fly ash (Bayer Öztürk, 2010), and zinc ore waste (Bayer Öztürk et al., 2017).

The previous studies demonstrate the potential of basalt-based glazes for ceramics, offering benefits such as lower firing temperatures, cost-effectiveness, stability, and attractive color options. Notably, the basalt from different locations shows promise in producing high-quality, pressure-resistant, and economically efficient glazes (Yilmaz et al., 2006; Andric et al., 2012).

The aim of this study was to investigate the potential of using basalt-cutting waste powder in ceramic tiles to improve the properties of the glaze, including color, surface properties, and sintering behavior. To achieve this, the waste powder obtained from BCW was incorporated into three different glaze compositions. The study focused on investigating the sintering behavior and physical properties of the final ceramic product resulting from these glaze formulations. By investigating these aspects, the effectiveness of using basalt-cutting waste powder in improving the glaze quality of ceramic tiles was to be evaluated.

2. Experimental Procedure

The chemical analysis of BCW, which was supplied from Emre Taş Mining (Kayseri, Turkey), was given in Table 1. The chemical analysis was conducted with XRF (Rigaku ZSX Primus model). The X-ray analysis (XRD, Rigaku Miniflex 600) of the BCW showed that Andesine ($Al_{0.735}$ $Ca_{0.24}$ $Na_{0.26}$ O_4 $Si_{1.265}$), Anorthite ($Al_2Ca(SiO_4)_2$), and Quartz (SiO_2) are main phases in its composition (Fig.1). The termogravimetric/differential thermal analysis (TGA/DTA- STA 409PG LUXX) were carried out to investigate the thermal behavior of BCW.

Additionally, thermal behavior of frits was examined using the thermal analysis microscopy (MISURA). The opaque and matt frits used in experimental researches were supplied from Gizem Firit Company and their chemical compositions ranges are given in Table 3. The BCW percentage in the glaze compositions was varied between 0-20 wt. %, while the percentage of dry frit (92 wt. %), kaolin (8 wt. %), sodium tripolyphosphate (STPP 0.2 g) was kept constant.

The wet milling system was used to prepare the frits with addition of 60 cc of water (residue <2 % at 63 µm). The glazes at 1,500 g/L were applied on 5×5×0.7 mm engobed tiles and fired in a laboratory kiln (at 1,200 °C for 6 h). The codes of glazes are presented in Table 2. Phase analysis of opaque and matt glazes prepared with BCW (0-20 % waste addition) was performed with an X-ray diffraction.

The color parameters (L*, a*, b* values) were determined using a Minolta CM-3600d color measuring device in the color analysis of glazed samples. The development of the phases in the microstructure was determined by examining the glazed samples containing 20 wt. % basalt-cutting waste by scanning electron microscope (SEM, Zeiss EVO 50 EP, an accelerating voltage of 20 kV to ensure accurate elemental composition and microstructure determination).

Table 1
Chemical composition (wt. %) of basalt cutting waste (BCW)

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	TiO_2	K_2O	MnO	P_2O_5	SO_3	Loss of ignition (LOI)
49.47	17.24	10.26	10.35	6.24	3.42	1.33	0.61	0.16	0.22	0.07	0.53

Table 2
Glaze mixture proportions and their codes

Glaze code	Frits (Opaque (O)-Matt (M)-Transparent (T)) (wt. %)	Kaolin (wt. %)	Basalt cutting waste (wt. %)
M0	92 M	8	0
M5	92 M	8	5
M10	92 M	8	10
M15	92 M	8	15
M20	92 M	8	20
O0	92 O	8	0
O5	92 O	8	5
O10	92 O	8	10
O15	92 O	8	15
O20	92 O	8	20
T0	92 T	8	0
T5	92 T	8	5
T10	92 T	8	10
T15	92 T	8	15
T20	92 T	8	20

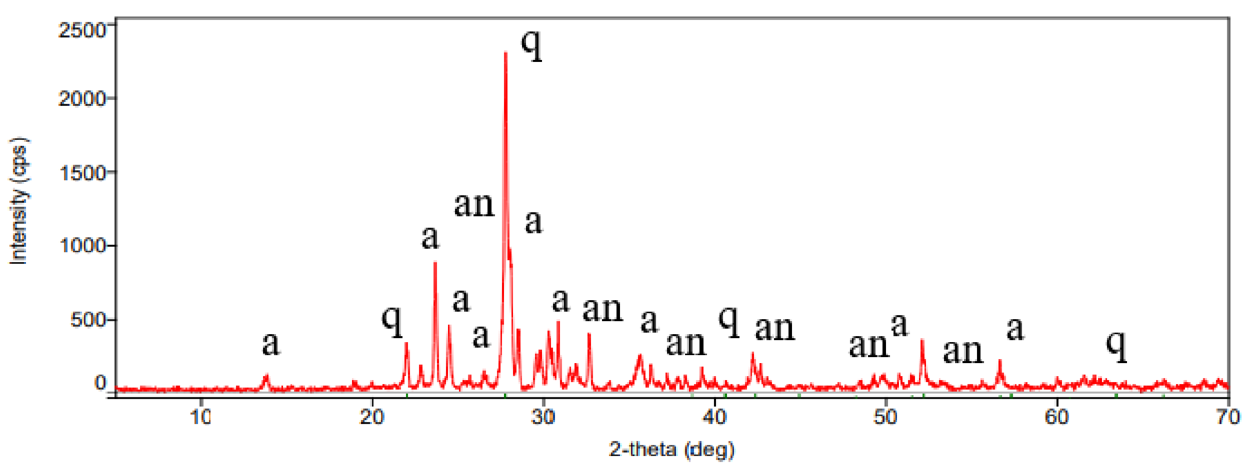

Figure 1. Phase analyse pattern of BCW
(**a**: Andesine, $Al_{0.735}$ $Ca_{0.24}$ $Na_{0.26}$ O_4 $Si_{1.265}$, **an**: Anorthite, Al_2 Ca $(SiO_4)_2$, **q**: Quartz SiO_2)

3. Results and Discussions

3.1. Characterization of basalt cutting waste

The DTA/DTG analysis results of BCW are given in Figure 2. The mass loss is indicated by four exothermic peaks which are 0.097 µV/mg mass loss at 670.9 °C, 0.247 µV/mg mass loss at 953 °C, 0.311 µV/mg mass loss at 1,036 °C, and 0.413 µV/mg mass loss at 1,188 °C, respectively. The phase analysis of the BCW (Figure 1) revealed the presence of large amount of feldspar (anorthite and andesine) and quartz. In the available literature, it is suggested that the presence of albite and pyroxene phases may be responsible for the observed endothermic peaks (Mahmood, 2014). Additionally, no crystallization peak was detected in the DTA analysis of the BCW. The melting behavior of the BCW was analyzed by a heat microscopy between 1,166 and 1,214 °C and the results are given in Figure 3. As can be seen in the analysis, the temperature at which it is subjected to a 5 % change in size gives the sintering temperature if the sample image is as assumed to be 100 %.

The softening point of the sample is examined at 1,188 ºC, at which the liquid phase appears on the surface of the sample. The spherical shape was observed at 1,198 ºC. In this step, the sample consists entirely of the liquid phase, and the shape of the sample is controlled by the surface tension.

After that sample reached the shape of hemisphere at 1,206 ºC and the height of the sample decreased by half the width at this temperature. The last step is called the melting point at which the sample fell below a third of its original height (Paganelli and Sighinolfi, 2008; Bayer Öztürk et al., 2020).

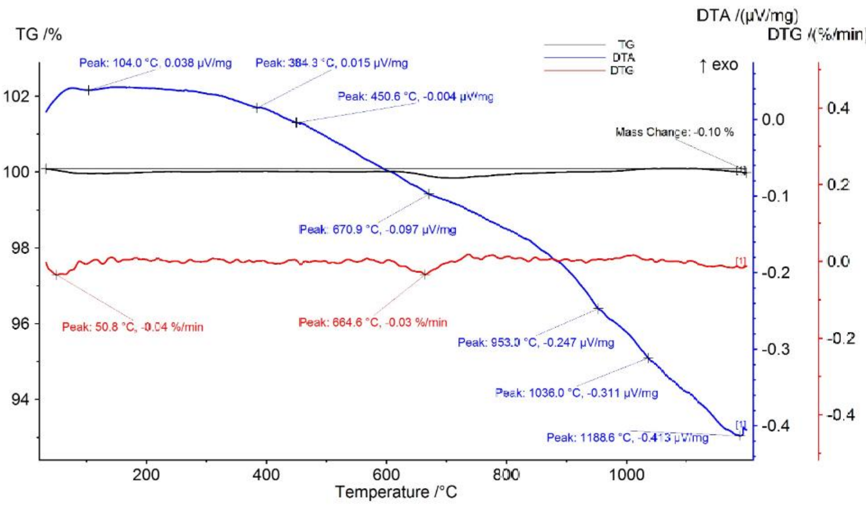

Figure 2. The thermal behavior characterisation result of BCW (DTA-DTG graph)

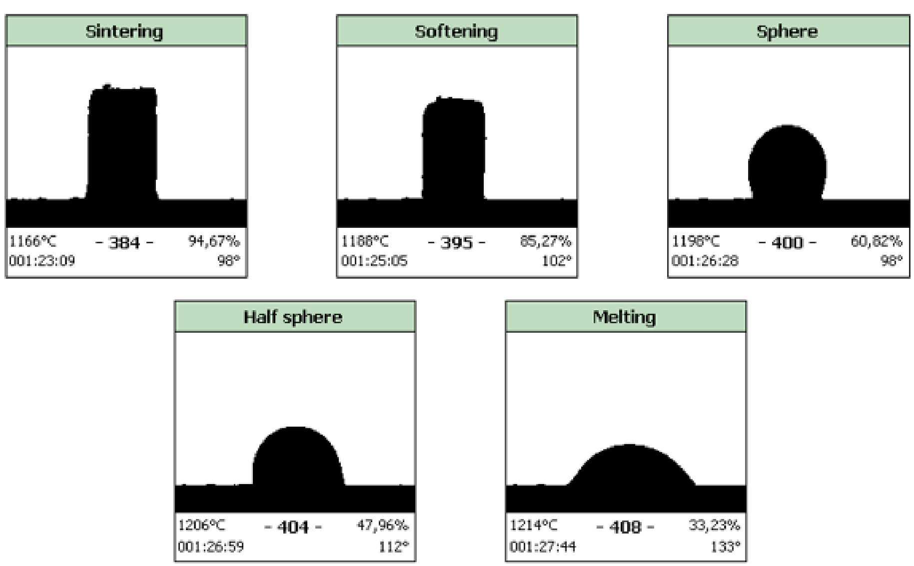

Figure 3. Heat microscopy results of basalt cutting waste

3.2. Characterization of the obtained glazes

The chemical compositions of studied frits supplied from Gizem Frit are presented in Table 3. As it is seen in chemical analysis, the percentage of SiO_2, B_2O_3, K_2O oxides is higher in opaque and transparent frits than in the matt frit. It is also noteworthy that the opaque frit has a high ZrO_2 content compared to the matte and transparent frit.

The characteristic temperatures were determined between 740 and 1,102 °C for matt frit, 840 and 1,264 °C for opaque frit, 816 and 1,208 °C for transparent frit in Table 4. The color of obtained glazes based on BCW were changed from a light yellow to beige color and the firing colors of glazed tiles fired at 1,200 °C can be seen in Figure 4. The increasing amount of BCW (from 5 to 20 wt. %) in the glaze composition led to a change from beige to yellow shades. The color parameters (L*-a*-b* values) of the glazed tiles are shown in Table 5.

Compared to other glaze samples, low L-values were achieved for matte (L* 58.19) and opaque (L* 61.73) glaze applications with 20 wt. % BCW. As the proportion of BCW in the glaze composition was increased, it was observed that the a* and b* values also increased. In this point, the effective parameter to change the color of glazes is the amount of iron oxide (10.26 wt. %) derived from BCW.

For the transparent glaze with 20 % BCW, the L* value was 49.11, the values for a* and b* were 2.76 and 33.71 respectively.

Table 3
The chemical compositions of matt, opaque and transparent frits

Frit	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**CaO**	**MgO**	**Na_2O**	**K_2O**	**ZnO**	**B_2O_3**	**TiO_2**	**ZrO_2**
Matt	47-49	13-15	0.05-0.1	12-14	0.25-1	6-8	0.05-0.5	17-19	0.5-2	0.05	-
Opaque	54-57	5-7	0.05-0.1	8-10	2-4	1-3	3-5	5-7	9-11	0.05	6-8
Transparent	54-57	8-10	0.05-0.1	12-14	0-1	1-2	5-7	4-6	9-11	0.05	-

Table 4
Typical points result of frits obtained by hot-stage microscopy (°C)

Sample	**$T_{sintering}$ (°C)**	**$T_{softening}$ (°C)**	**T_{sphere} (°C)**	**$T_{hemisphere}$ (°C)**	**$T_{melting}$ (°C)**
Matt (MT)	740	1,082	-	1,092	1,102
Opaque (OP)	840	1,012	1,076	1,202	1,264
Transparent (TR)	816	962	1,014	1,108	1,208

Table 5
The color parameters of glazed tiles

Sample	**L***	**a***	**b***
M0	89.73	-0.29	2.82
M5	88.37	-1.62	10.25
M10	80.65	-1.15	22.91
M15	64.20	3.47	34.62
M20	58.19	7.96	36.78
O0	90.17	-0.52	1.69
O5	85.17	-1.22	8.74
O10	76.48	-0.16	12.44
O15	68.43	1.35	16.88
O20	61.73	3.01	25.67
T0	86.86	-0.35	1.30
T5	81.65	-1.75	13.41
T10	67.82	-0.25	23.48
T15	68.93	0.11	27.14
T20	49.11	2.76	33.21

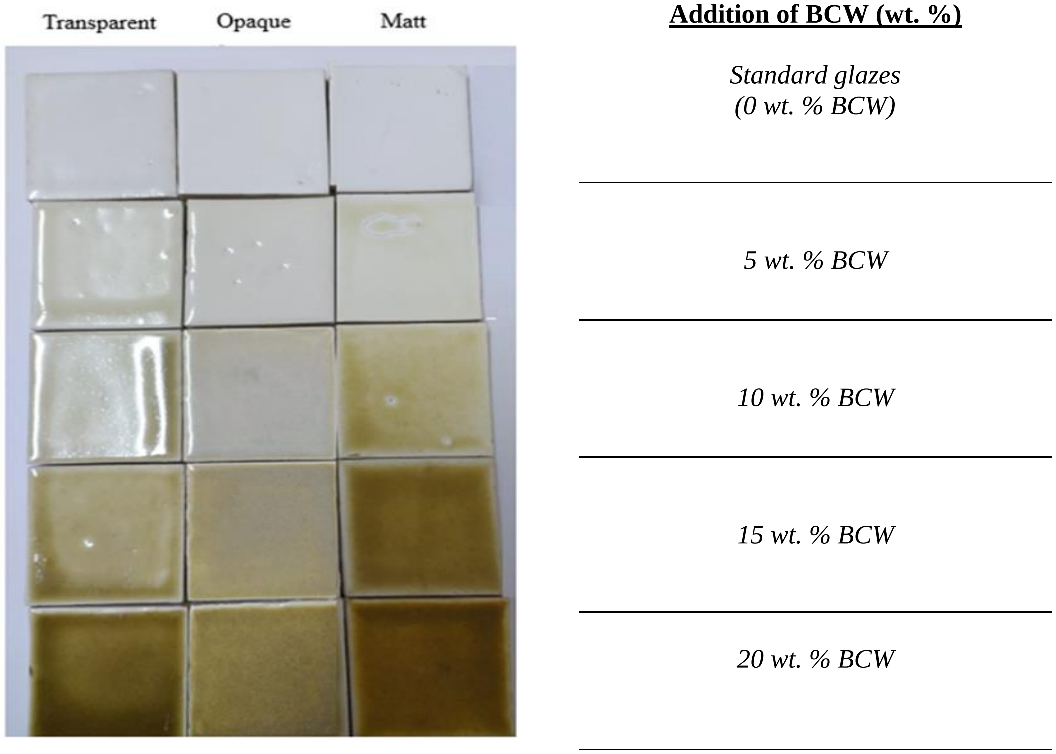

Figure 4. The effect of BCW addition on glaze applications

The higher color change was observed when the basalt-cutting waste was added at a proportion of 20 wt. %.

XRD and SEM analyses were conducted on both standard samples (without basalt-cutting waste) and glazed samples containing 20 wt. % BCW. The results are depicted in Figures 5-7, illustrating the phases present in the glazed samples. In the case of opaque glazes (from O0 to O20), the major crystalline phases identified were quartz, zircon, and anorthite. Meanwhile, for matt glazes (from M0 to M20), the phases observed included quartz, anorthite, diopside, and aluminum silicate.

These analyses provided insight into the crystalline structures and composition of the glazes, offering a better understanding of the materials' behavior and the impact of incorporating basalt-cutting waste on the phase composition of the glazed samples. No crystalline phases were observed in transparent glazes (from T0 to T20). In XRD analysis, it was determined that the addition of BCW to the glazes resulted in an increase in the intensities of the anorthite and diopside phases.

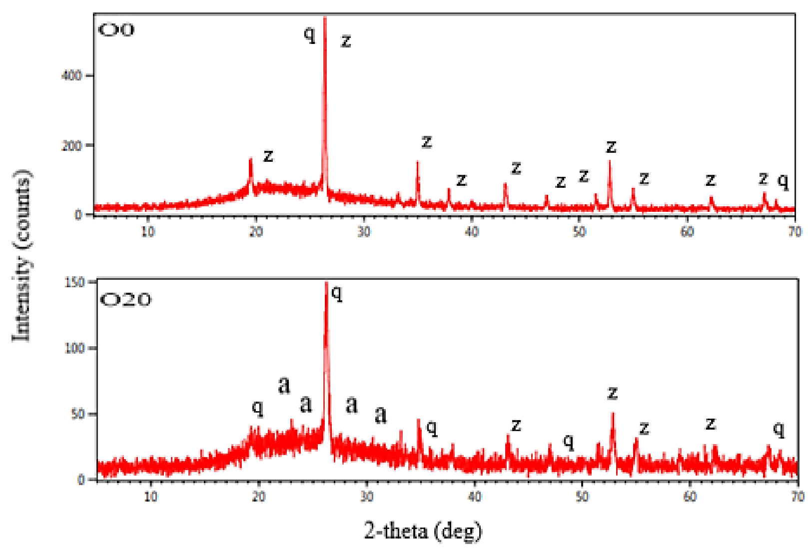

Figure 5. XRD patterns of O0 (standard) and O20 glazes (**q**:quartz, **z**:zircon, **a**:anorthite)

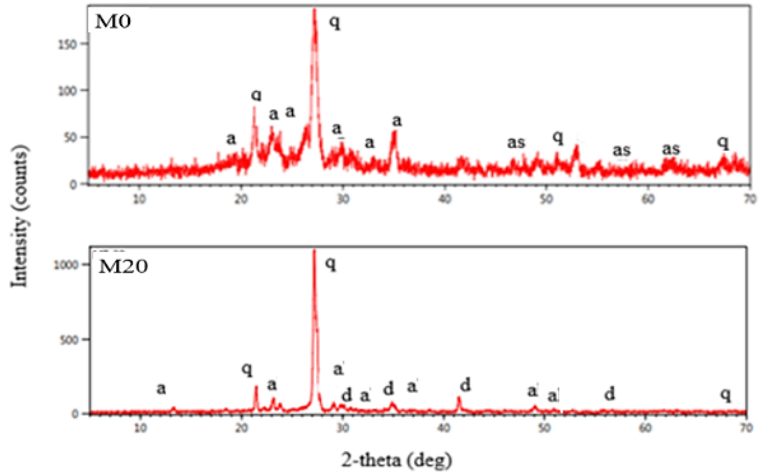

Figure 6. XRD pattern of M0 (standard) and M20 glazes (**q:**quartz, **as:** aluminum silicate, **a**:anorthite, **d:**diopside)

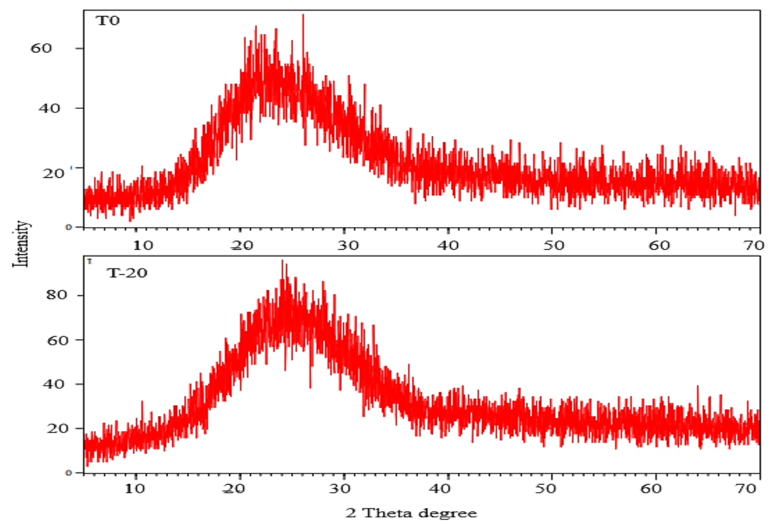

Figure 7. XRD pattern of T0 (standard) and T20 glazes (amorphous phase)

According to the SEM-EDS results of the opaque glazed tiles, while zircon crystal was present at 60.92 wt. % Zr, 20.49 wt. % Si, 18.59 wt. % O in standard opaque glazed tiles, other zircon region for O20 sample contained 22.46 wt. % O, 33.66 wt. %Si, 4.85 wt. % Al, 23.88 wt. % Zr, 3.30 wt. % Zn, 5.01 wt. % Ca, 2.25 wt. % Fe, 1.26 wt. % Mg, 1.18 wt. % Na as can be seen in Figure 8. It shows that the zircon contained in the opaque frit was detected in the phase and microstructure analyses of the opaque glazed samples.

Anorthite containing phase region involved 47.78 wt. % O, 30.46 wt. % Si, 4.03 wt. % Na, 10.33 wt. % Al, 4.67 wt. % Ca, 0.98 wt. % K, and 1.74 wt. % Zn for standard matt glazed tile in Fig. 10. On the other hand, anorthite region was detected as 42.26 wt. % O, 25.51 wt. % Si, 6.11 wt. % Al, 6.94 wt. % Ca, 2.37 wt. % K, 2.80 wt. % Na, 11.38 wt. % Zn and 2.63 wt. % Fe in M20 (Figure 9).

It was determined that the addition of BCW formed a needle-like anorthite crystal, especially in the matt-glazed sample. It has been reported in the literature that anorthite crystallizes in the glazing as a needle-like shape (Tunali and Selli, 2014; Tunali et al., 2015). As detected in the EDS analysis, samples containing basalt-cutting waste in the glassy phase contain iron. This proves that there are iron oxide crystals in the glassy phase, although they cannot be detected in XRD, and color development occurs depending on iron oxide (Gultekin, 2020).

According to the SEM-EDS results of the transparent glazed tiles, the amorphous phase region contained 32.69 wt. % Si, 46.58 wt. % O, 6.98 wt. % Ca, 4.88 wt. % Al, 4.26 wt. % Zn, 3.01 wt. % K, 1.60 wt. % Na for standard transparent glaze. Additionally, other amorphous phase region contained 32.21 wt. % Si, 47.25 wt. % O, 5.14 wt. % Ca, 7.03 wt. % Al, 1.78 wt. % Zn, 2.31 wt. % K, 1.86 wt. % Na and 2.44 wt. % Fe for T20 as can be observed in Figure 10. The presence of iron content was more prominent due to the basalt waste in the glassy phase than in the standard transparent glaze.

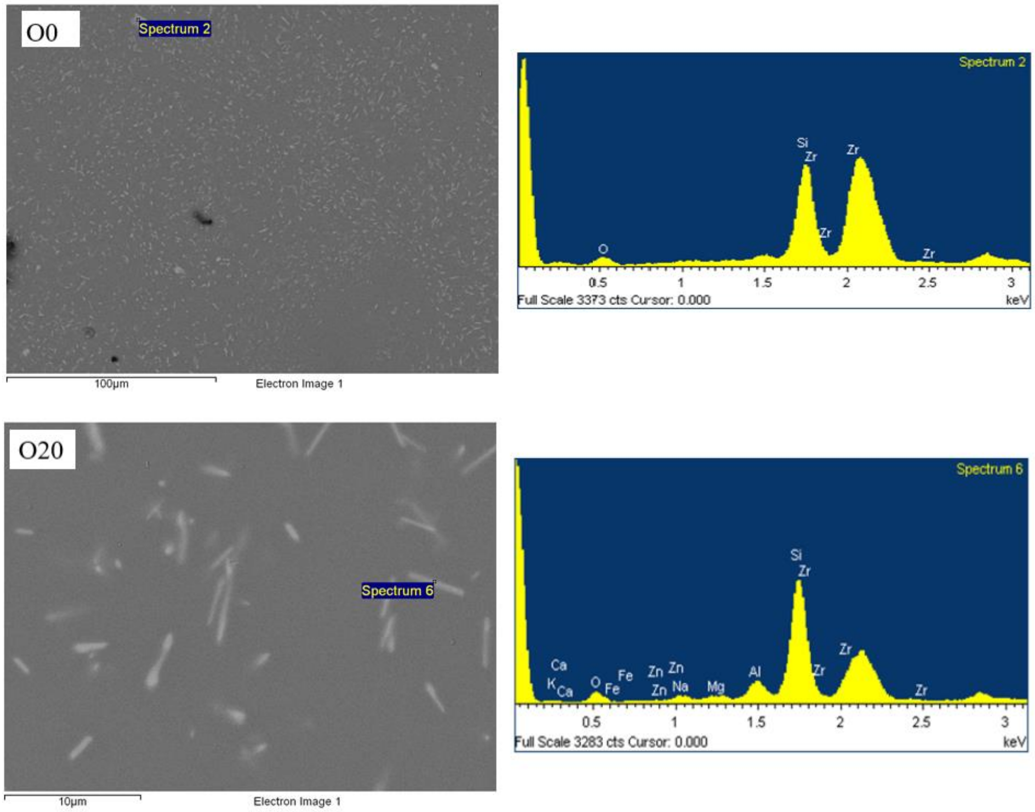

Figure 8. EDS spectra and spot analysis of glazed samples (O0 and O20)

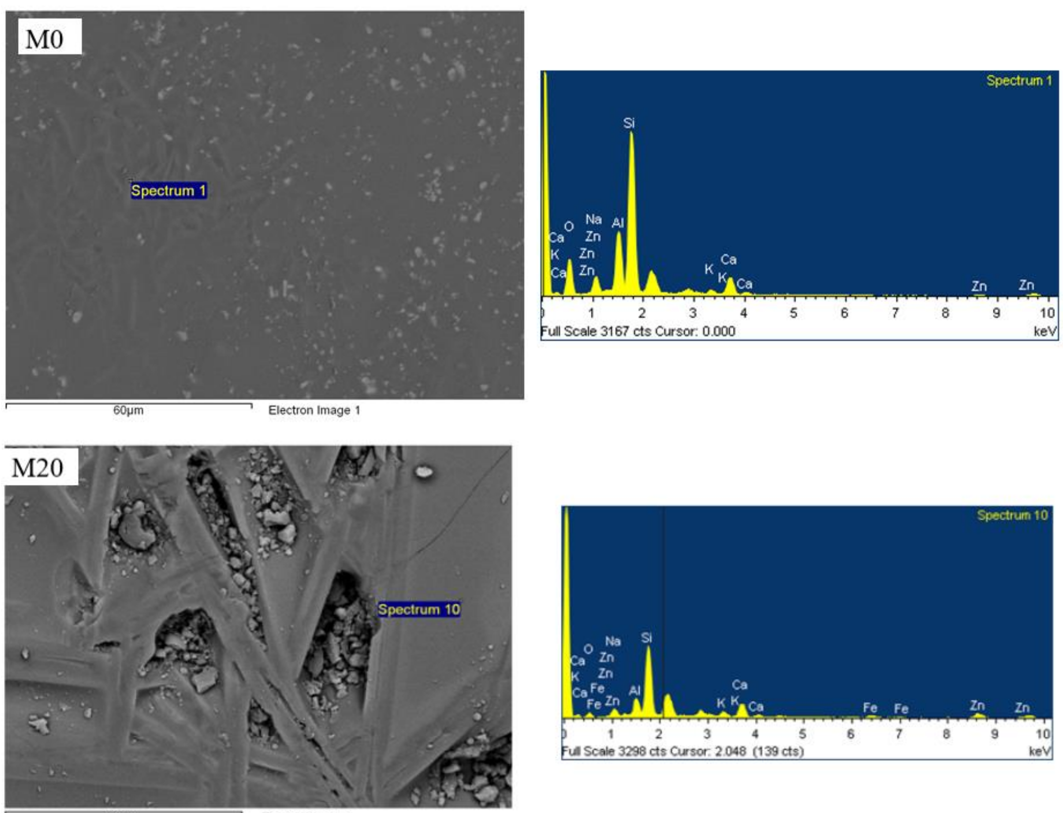

Figure 9. EDS spectra and spot analysis of glazed samples (M0 and M20)

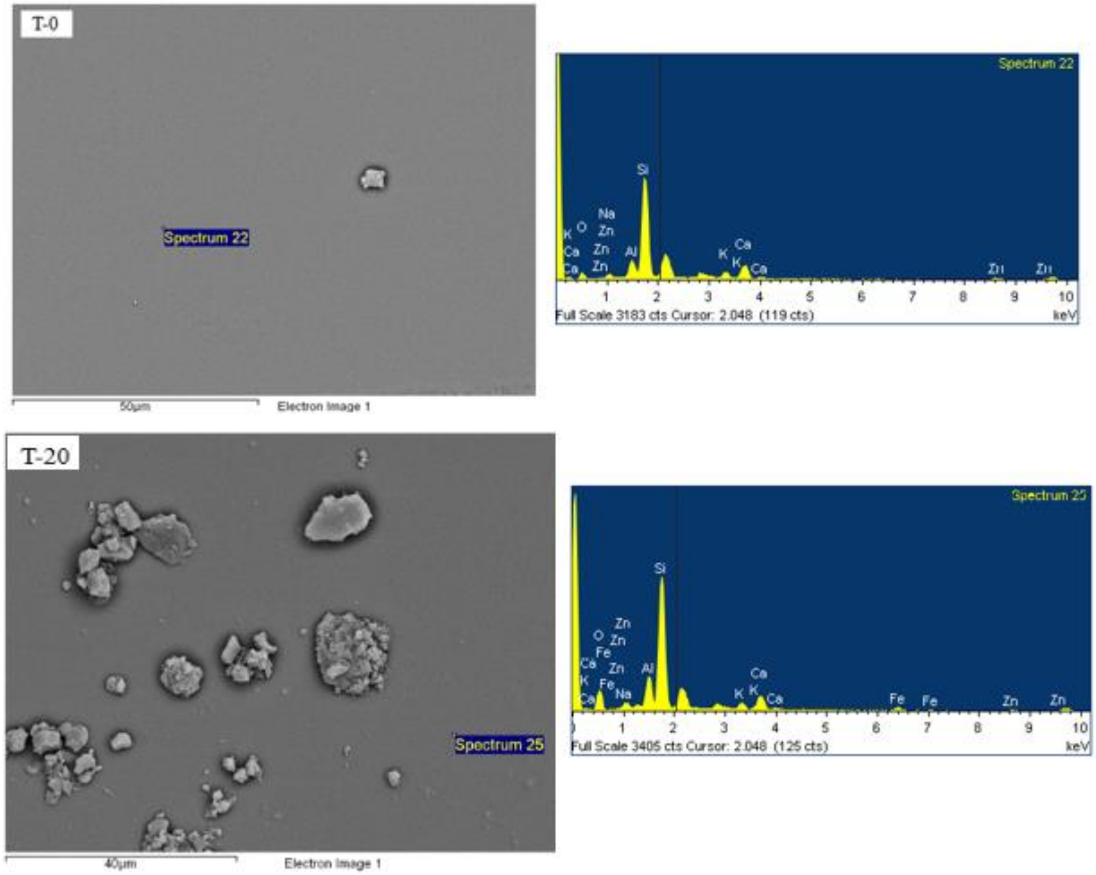

Figure 10. EDS spectra and spot analysis of glazed samples (T0 and T20)

4. Conclusions

The study aimed to investigate the impact of incorporating basalt-cutting waste into opaque, matt, and transparent glazes at various proportions. The addition of basalt-cutting waste resulted in the observation of yellow and beige shades in the glazed tiles. Due to the presence of iron oxide in the basalt-cutting waste, the L* values decreased as the waste content increased in all glaze types compared to standard opaque, matt, and transparent glazes. Conversely, the a* and b* values increased generally with higher proportions of basalt-cutting waste in the glazes. The presence of the coloring agent iron oxide in the basalt allowed the use of this material as a glaze coloring agent. The rest of the components of the basalt would contribute to the glaze matrix and with some frits the formation of anorthite was promoted.

Analysis of the phases formed in the glazed samples revealed that the opaque glazes contained zircon and quartz crystals, while the introduction of basalt-cutting waste led to the formation of anorthite crystals. The matt glazes exhibited anorthite and quartz crystals, with an increase in anorthite intensity when combined with basalt-cutting waste, accompanied by the formation of the diopside phase. The intensity of the amorphous phase increased in transparent glazes.

The SEM-EDS analysis of both opaque and transparent glazed tiles unveiled notable variations in their elemental composition. In the case of opaque glaze tiles, the presence of zircon crystals and anorthite phases showed marked distinctions between the standard and O20 samples. The inclusion of basalt-cutting waste led to the formation of needle-like anorthite crystals, a phenomenon previously documented in research. Furthermore, EDS analysis confirmed the existence of iron in the glassy phase of samples containing basalt waste, influencing color development, despite its absence in XRD analysis.

In transparent glaze tiles, differences in Si, O, Ca, Al, Zn, K, Na, and Fe content within the amorphous phase were evident when comparing standard and T20 samples. The higher iron content observed in the T20 was attributed to the incorporation of basalt waste. Based on the findings, it can be concluded that basalt-cutting waste can be economically utilized to produce yellow-beige pigments for different industrial ceramic glazes, depending on the glaze composition. The study sheds light on the potential of recycling the basalt-cutting waste in ceramic glaze production and expanding the range of color options available. This study provides valuable insights into the impact of basalt-cutting waste on the composition and microstructure of glazed tiles, offering significant implications for the ceramics industry.

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Procena upotrebe otpada pri sečenju bazalta u bojenju različitih kompozicija keramičkih glazura

Zahide Bayer Öztürk [a], Semra Kurama [b, #] Ayşe Eser [a]

[a] Univerzitet Nevsehir Haci Bektas Veli, Odsek za metalurgiju i materijale, Nevsehir, Turska
[b] Tehnički Univerzitet Eskisehir, Odsek za nauku o materijalima, Eskisehir, Turska

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I Z V O D

Efekat bojenja kod keramičkih glazura ima presudnu ulogu u estetici i privlačnosti keramičkih predmeta. Pigmenti korišćeni u glazurama reaguju sa keramičkim materijalima tokom procesa pečenja, što kao rezultat daje spektar fascinantnih boja i efekata. Zahvaljujući prirodnim svojstvima bazalta kao što su čvrstoća na pritisak i otpornost na habanje, ovaj prirodni vulkanski kamen privlači mnogo pažnje kada se koristi u procesima proizvodnje keramike. Cilj ovog istraživanja je procena efekta otpada pri sečenju bazalta (BCW) iz Turske na efekat bojenja keramičkih glazura u kombinaciji sa različitim fritovima. U tu svrhu, pripremljene su glazure u koje je uključen BCW u različitim procentima težine od 0-20 % zajedno sa različitim fritovima. Kod glazura su ispitani hemijsko i termalno ponašanje, kao i faze. Za ispitivanje su korišćene sledeće tehnike: rendgenska fluorescencija (XRF), termalna mikroskopija i rendgenska difrakcija (XRD). Pripremljene glazure su zatim nanešene na engobirane zidne pločice i pečene na temperaturi od 1,200 °C u laboratorijskoj peći. Nakon pečenja, glazirane površine su podvrgnute analizi boja, dok je faza kompozicije analizirana XRD-om. Analiza mikrostrukture je izvršena korišćenjem elektronske mikroskopije (SEM/EDS). Povećanje udela BCW u kompozicijama glazura rezultiralo je promenom boje završenih površina sa krem do žućkasto-bež tonova. Ovo istraživanje pruža uvid u potencijalnu upotrebu BCW kao pigmenta u proizvodnji keramičkih glazura.