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Authors: Muhterem KOÇ, Eda TAŞÇI

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Investigation of the Usage Properties of Steel Slag as Pigment in Low-Temperature Glaze

Muhterem KOÇ[1], Eda TAŞÇI[*2]

Abstract

The evaluation of industrial wastes in the ceramics industry has been the subject of research studies in recent years. This study investigated the use of steel slag as a colouring pigment in ceramic glazes. The colour effects of 45 compositions created with a triple-phase diagram with steel slag, CoO, and Cr_2O_3 on low-temperature glaze composition were determined. The microstructural evaluation of the selected colours by SEM and EDX analysis were carried out.The prepared glaze compositions were applied by pouring on the high silica ceramic body. Glazed tiles were sintered in a muffle furnace at 950 ℃ and then L*a*b colour and gloss brightness values were measured. By using steel slags instead of Fe_2O_3, black glazes were obtained in the triple diagram.

Keywords: Low-temperature coloured glaze, steel slag, high silica-containing body, L*a*b

1. INTRODUCTION

Glazes are called glassy surfaces that adhere to the ceramic material and provide basic properties such as permeability, semi-permeability, opacity, matte appearance, necessary thermal expansion and chemical resistance. They can be prepared as a powder or liquid suspension. They are applied to ceramic body surfaces by dipping, spraying or sprinkling methods. Glazes can be applied on the ceramic body as raw or fritized, wet or dry. They may have contained different compositions depending on the purpose of use [1]. In addition, they provide a shiny and smooth surface to the body on which they are applied, they do not have gas permeability, their mechanical strength is high, they are resistant to abrasion, scratching and chemicals, and they are easy to clean. They create a decorative effect by reflecting matte, glossy and crystalline properties on the surface of the products [2].

The most important feature of ceramic glazes, which are formed by mixing chemically alkali and alkaline earth oxides with inappropriate recipe ratios and sintering at the appropriate temperature, is that they can form physical and chemical bonds with the ceramic body on which they are applied, under normal conditions [3]. In the formation of glaze types, classification can be made according to the structure of the body in which the glaze is

* Corresponding author: eda.tasci@dpu.edu.tr (E.TAŞÇI)
[1] Kütahya Dumlupınar University, Department of Industrial Design, Kütahya, Türkiye
[2] Kütahya Dumlupınar University, Department of Metallurgical and Materials Engineering, Kütahya. Türkiye
E-mail: muhterem.koc@dpu.edu.tr
ORCID: https://orcid.org/0000-0003-3661-4410, https://orcid.org/0000-0003-3346-8833

applied, the variety of industrial or artistic applications, and most importantly, according to the firing temperatures [4]. Natural and synthetic oxides and pigments are used in the colouring of ceramic glazes. Natural pigments exist in nature as simple oxides. Among natural pigments, iron oxides, which can impart different colours, are widely used especially in the ceramic industry [4].

Natural and synthetic inorganic-based pigments, glazes containing homogeneous mixtures that disperse without dissolution and chemical reaction at high temperatures; They are widely used for the bulk colouring of various ceramic products, including glass, ceramic body and porcelain enamel. The use of blending systems for raw materials or colourants in the development of a new glaze composition is important for a systematic study.

Ceramic production is increasing rapidly in our country and throughout the world. In the 21st century, in line with the developing technology and industrialization and sustainable development goals, the correct use of by-products and wastes generated during production processes all over the world gains importance. Along with this increase in production, the world reserves decreasing over time not only restrict the use of high-quality raw material stocks in the ceramic industry, but also cause an increase in costs. Therefore, the importance of R&D studies on the use of industrial waste materials, as an alternative to the use of natural raw material reserves, is increasing day by day. The recycling of industrial wastes in this direction is widely used in both sectoral and academic studies. In addition, considering the economic effects of the reuse of wastes, it provides significant gains to the ceramics industry.

The studies carried out cover the subjects of recycling, conservation of natural resources, reducing the demand for landfills and waste management [4-5]. In the ceramics industry, there are studies on the use of industrial wastes as alternative raw materials in ceramic glazes [6-10], ceramic tile bodies [6-9], ceramic pigments [8-12] and concrete building materials [13-14].

In addition, studies are carried out on the use of solid wastes generated as a result of industrial production as starting raw materials for use in ceramic pigment, glaze or body colouring. In this regard, colourants, which are quite expensive in terms of production cost and in which heavy metal oxides and mixtures containing heavy metal oxides are used, take the first place [15-16]. Apart from these, On the other hand, many studies are carried out to reduce the damage caused by industrial wastes to nature by recycling and to make them reusable in the development of ceramic glazes. With the use of industrial wastes in the ceramic sector, raw material costs can also be reduced. When the studies on the recycling of wastes to be used in the ceramic industry are examined, it is seen that positive results have been obtained [17-18]. In addition, natural industrial wastes are used in ceramic industry recipes, especially marbel wastes, boren wastes etc. [19].

The concept of colour, which we express as a physical perception, is a chemical process. Different quality colours are obtained by mixing ceramic paints and pigments produced as a result of the chemical process. Obtaining the desired colour in the ceramic industry is a very important and difficult process. The use of ceramic colours both industrially and artistically depends on the content of the raw materials used first. Secondly, the production process is an effective parameter [20-21]. Colouring salts, oxides and compounds or pigments containing them are used in ceramics. Although colourants have their own unique colours, different colours can be obtained by controlling parameters such as production process, firing conditions, grain size and application thickness. The effects of different oxides and oxides in the form of pigments on ceramic glazes differ.

The aim of this study is to examine the properties of steel slag, which is iron and steel factory waste, as a colouring agent in ceramic glazes, which is an important component of ceramic products. Within the scope of the study, the effects of 45 recipes formed as a triple phase diagram of steel slag with CoO and Cr_2O_3 on the glaze composition selected as low temperature glaze were investigated. L*a*b color and gloss values of the compositions were measured after firing under laboratory conditions at 950 ^oC. Microstructural colour effects of black, green and blue tone colours on low temperature glaze, the effect of waste, CoO and Cr_2O_3 on colour performance were evaluated by SEM and EDX analyzes in the 3-phase diagram recipes, which are considered as paint application, in the recipes containing the most appropriate slag ratio affecting the colour density. In this study, the usability of steel slags in high silica bodies and leaded low temperature glazes as iron-based colouring oxide was investigated. Compositions formed with steel slag, CoO and Cr_2O_3 were used as colourants at low temperatures.

2. MATERIALS AND METHODS

Within the scope of this study, the chemical analysis of the steel mill waste obtained from Kardemir Karabük Iron and Steel Industry is given in Table 1. Pigment mixture compositions were prepared based on the triple phase diagram given in Figure 1, consisting of steel slag, CoO and Cr_2O_3 components. In Figure 2, recipe compositions are given.

45 pigment-dye recipes prepared in the form of a triple phase diagram were weighed separately as shown in Table 2 and ground as aqueous for 30 minutes. After the drying process, the pigment recipes were prepared as glaze compositions by adding 3% to the low temperature (950°C) glaze recipe and then grinding for 15 minutes separately. The prepared pigment-containing glaze recipes were applied on the high silica bodies produced within the scope of the T.C. Strategy and Budget Directorate project, by the pouring method. High silica bodies contain % 91.54 SiO_2, % 5.42 Na_2O, % 1.01 Al_2O_3, % 1.21 CaO, % 0.42 Fe_2O_3, % 0.13 K_2O, % 0.18 MgO, %0.04 TiO_2. In the XRD analysis of the body given in Figure 1, high quartz and low cristobalite phases were detected.

The tiles were glazed with colored glazes and fired in a Protherm brand HLF60 model oven at 950ºC for 30 minutes in a laboratory environment. According to the results of the triple-phase diagram, the colour scale obtained visually is given in Figure 3.

L*a*b* colour values of glazed coloured bodies were measured with a PCE CSM-2 brand model device. The effect of waste on the colour distribution of the recipes selected from high silica bodies was examined with the Nova NanoSEM 650 brand model SEM EDX device and the results are given in Figures 4 and 5 and Tables 2,3,4 and 5.

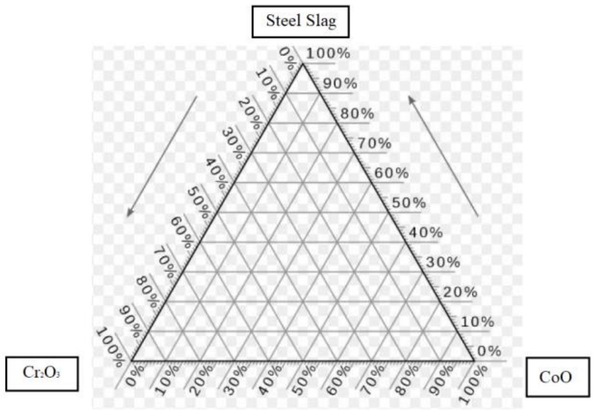

Figure 1 Triple Phase Diagram.

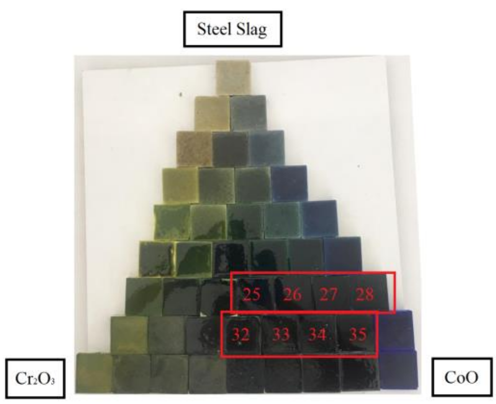

Figure 2 Colour values obtained in the triple phase diagram

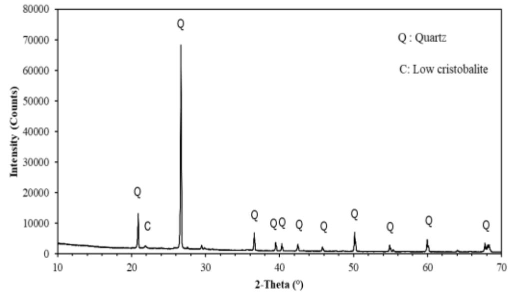

Figure 3 Phase analysis of high silica body.

Table 1 Chemical analysis results of steel slag

Oxides	Steel slag (%)	Glaze (%)
PbO	-	55.88
CaO	48.02	0.85
Fe_2O_3	21.09	0.10
Na_2O	0.1	5.42
SiO_2	12.34	34.14
MgO	3.86	0.30
Al_2O_3	2.92	3.17
MnO	5.11	0.05
SO_3	1.08	-
P_2O_5	0.76	-
TiO2	0.48	0.01
Cr_2O_3	0.24	-
LIO	4.10	-

LIO: Loss on igniton

3. RESULT AND DISCUSSION

The gloss values of glazed samples are given in Table 2. Gloss can be defined as an optical property that indicates how well a completely impermeable surface reflects light in a specular (mirroring) direction. Another definition; It can be expressed as the ratio of the light intensity reflected from the surface of an object to the light intensity incident on the surface. In gloss meter measurements, the illumination from the light source in the gloss meter reflects from the surface at a certain angle (such as 20°, 60° and 85°) and reaches the sensors on the other side of the gloss meter. In the meantime, the ratio of the amount of light reflected on the surface and the amount of light coming from the device source is measured.

The resulting numerical value is expressed by converting to Gloss Unit (G.U.), a unit of gloss [22]. It is important to choose the correct angle to measure the gloss, and to choose the surface gloss angle. This method is used on smooth, refined or unrefined surfaces such as polished stone, wood, laminate, flooring and ceramics. With the gloss meter, it can be determined whether the material analyzed is a high gloss, semi-gloss and matte. Gloss is measured by directing a constant-intensity light beam at a fixed angle to the test surface and then watching the light reflect off from the same angle. This visual reflection is measured using a gauge. Different surfaces have different reflection angles. The gloss measurement is based on the amount of light reflected on the surface relative to the polished glass reference standard measured in luminous units (G.U.).

By examining the color, surface quality and composition characteristics of the samples in the triple phase diagram, 8 samples with a visually black effect were selected for further examination. The recipe compositions of the selected samples are given in Table 3.

Table 2 Recipe and L*a*b* and gloss values obtained according to the triple phase diagram.

Samples	L^*	a^*	b^*	İmages	Gloss (GU)
25	6.17	-1.10	3.85	Semi glossy	32.00
26	2.84	0.55	-0.11	Mat	10.90
27	30.40	0.83	-0.01	Mat	9.60
28	3.36	0.80	-1,39	Glossy	95.60
32	8.96	-2.52	-0.14	Glossy	94.00
33	8.21	-1.89	-0.52	Glossy	24.90
34	12.27	-2.29	-0.10	Glossy	46.60
35	4.75	0.53	-0.16	Semi glossy	53.70

Although various colour systems have been developed to characterize glaze colours in ceramics, CIE (Commission Internationale de l'Eclairage) systems are most commonly used today. In the CIE system, the colour detection phenomenon is based on experimental observations. In colour measurement, the light source, the observer and the surface are parameters that should always be considered. Colour measurements are needed for the estimation of the new colours that will be formed as a result of the determination of the colour according to the reference values, its continuity and mixing with different colours. Colour is also a basic parameter that determines the glaze surface's visuality.

Colours are expressed with a three-dimensional coordinate system and this system is called colour space. Colour parameters were determined by measuring the colour distribution of the dye-added glaze trials, the recipe of which was determined according to the triple diagram, which was visually checked after firing (Table 2). The colour values of the glaze surfaces are determined by making L*a*b* measurements of the 3 main colours for each glaze recipe. By comparing the data obtained as a result of this test, it was examined how the changes in waste, CoO and Cr_2O_3 additives affected the changes in colors. The result obtained is green if it has a negative sign, and red if it has a positive sign. The b value is the yellow-blue value. The result obtained is blue if it has a negative sign, and yellow if it has a positive sign [23-24].

Table 3 Recipe compositions of glazed tiles.

Samples	Steel Slag (%)	CoO (%)	Cr_2O_3 (%)
25	25	37.5	37.5
26	25	25	50
27	25	12.5	62.5
28	25	0	75
32	12.5	50	37.5
33	12.5	37.5	50
34	12.5	25	62.5
35	12.5	12.5	75

While there is fixed iron ore waste in the selected 25, 26, 27 and 28 recipes, it is seen that the CoO ratio decreases and the Cr_2O_3 ratio increases in the recipe. In recipe no. 25, a semi-matt 32° gloss value was obtained. While the presence of waste CoO and Cr_2O_3 created matte and semi-gloss in the recipe, the presence of waste and Cr_2O_3 resulted in high gloss and low L*a*b* colour values. When recipe 26 and recipe 28 were compared, the darkening rate of the colour was the highest. The presence of CoO in the glaze recipe created a matt effect on the surface.

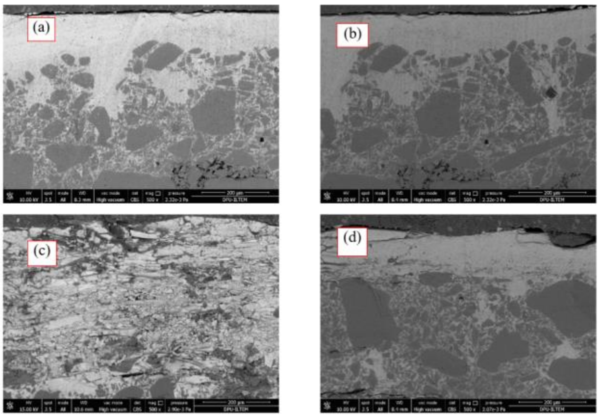

Figure 4 Microstructure images of coloured glazed samples; a) 25, b) 26, c) 27, d) 28.

In the SEM images of approximately 200-micron magnification given in Figure 4, it was determined that the oxides in the recipe compositions used as colouring dye in the glaze recipe were homogeneously distributed. As a result of the oxide-based EDX analyses given in Table 4; The presence of oxides varies proportionally in the direction of increase and decrease in the point analyzes at constant increasing CoO and decreasing Cr_2O_3 ratios.

Table 4 EDX analyzes of the samples (recipes 25,26,27 and 28)

Oxides	Samples			
	25	26	27	28
Fe_2O_3	0.20	0.00	1.43	0.00
CoO	0.12	0.00	3.66	0.00
Na_2O	4.42	7.67	5.80	2.94
MgO	1.04	0.00	0.05	0.98
Al_2O_3	1.58	0.36	0.58	2.50
SiO_2	41.02	79.57	46.24	79.64
PbO_2	43.17	11.31	38.84	11.91
K_2O	0.84	0.01	-	0.26
CaO	2.13	0.41	1.70	1.21
Cr_2O_3	-	0.66	1.70	0.56

When the SEM images in Figure 5 were examined, it was determined that there was a homogeneous microstructure in the colored glazed samples. However, it was determined that the glazes were damaged during the polishing process. While there is fixed iron ore waste in these recipes, it is seen that the CoO ratio decreases and the Cr_2O_3 ratio increases in the recipe. In recipe 32, the high gloss value was 94 degrees. While the presence of waste, CoO and Cr_2O_3 created matte and semi-gloss in the recipe, the presence of waste and Cr_2O_3 brought high gloss and low L*a*b* values. When recipe 33 and recipe 28 were compared, the darkening rate of the colour was the highest. The presence of CoO caused some opacity on the surface.

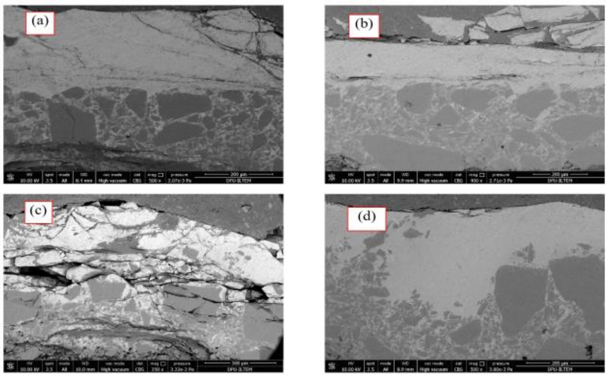

Figure 5 Microstructure images of coloured glazed samples; a) 32, b) 33, c) 34, d) 35

Table 5 EDX analysis of the samples (recipes 32,33,34 and 35)

Oxides	Samples			
	32	33	34	35
Fe_2O_3	-	-	0.01	-
CoO	-	-	-	0.34
Na O	3.08	4.14	3.47	2.71
MgO	0.18	-	-	0.24
Al_2O_3	0.60	-	0.70	0.80
SiO_2	39.09	43.08	52.50	40.28
PbO_2	48.44	48.49	39.00	49.51
CaO	2.01	-	1.74	3.00
Cr_2O_3	6.59	3.30	2.58	3.12

When the EDX analysis results and gloss values of the colour-glazed recipes 32, 33, 34 and 35 are examined, the presence of PbO_2 shows the gloss value in the recipes (Table 5). Among the samples examined, prescriptions 32 and 33 showed high gloss, while recipes 34 and 35 showed semi-gloss properties. It can be said that the presence of alkali and alkaline earth oxides causes the semi-gloss feature [25].

In Figure 6, the use of some of the compositions in which coloured glaze experiments were carried out, are given. While a much more colourful image was obtained in glazes, it was determined that the colours remained lighter in dyeing studies. It is thought that this situation causes the colour to be darker due to a thicker application in paint works. It has been seen that the real image of the colour is obtained in the painting studies.

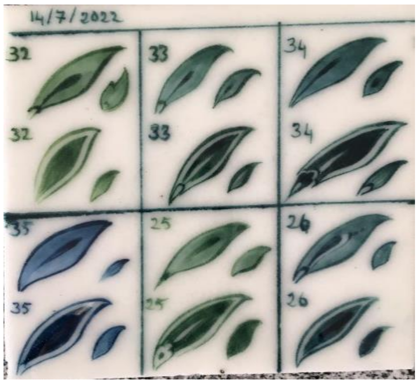

Figure 6 The image of dyeing studies of some compositions as colourant.

4. CONCLUSIONS

In the study, the usability of waste steel slag as paint was examined by making recipe development studies over the triple phase diagram. The use of waste in glaze recipe has been evaluated by using it in low temperature glazes on high silica bodies. Glaze surfaces varied in the range of high gloss, semi-gloss and matte. With the change of oxides in the prescriptions, the change in L*a*b* values and the rate of change in gloss values show differences. In the SEM-EDX examinations of the samples with high dark colour ratio, the effect of fixed waste ratio and varying oxide ratios on gloss, L*a*b* was tried to be explained. Pigment application examples in underglaze pattern design are very diverse. Among the pigment recipes as 33, 34, 35, 25 and 26 the pattern design apllications in Figure 6 are presented, and they show that they can be used as under-glaze paint applications and can be used as an alternative to under-glaze paint applications, especially for low-temperature glazes. It has been seen that a wide colour scale can be obtained when this study is carried out in more.

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