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Exploring the Chemical Components of Porcelain Tiles Commercially available in Benghazi City, Libya

Maysson M. Yaghi [1], Khaled M. Elsherif [2,3] and Majdi A. Abdulhadi [1]

[1]*Chemistry Department, Science Faculty Al-Abyar, Benghazi University, Benghazi, Libya.*
[2]*Libyan Authority for Scientific Research, Tripoli, Libya.*
[3]*Chemistry Department, Science Faculty, Benghazi University, Benghazi, Libya.*

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ABSTRACT

The objective of this study was to analyze the chemical composition of porcelain tiles by determining their mass percentages. Porcelain tile samples were collected from various markets in Benghazi, Libya, including those from Egypt, India, Spain, and Turkey. The chemical components of the tiles were identified using an X-Ray Fluorescence Spectrometer. The assessment criteria for high-quality raw mineral materials consisted of high representative oxide content, low impurity oxides, and low loss on ignition (LOI). Notably, significant variations were observed in the chemical composition of porcelain tiles. In general, two main formulations were identified: one group characterized by high silica content (45.65% to 48.24%), elevated levels of alumina (11.65% to 11.83%) and alkaline oxides, and low magnesium oxide (MgO); the other group consisted of tiles with low silica content (39.26% to 34.96%), high MgO (1.00%) and alumina content (5.89% to 9.88%), and relatively lower alkaline oxides. The results provided the average mass percentages of different components present in the porcelain tiles. Silica (SiO_2) exhibited a resistant property to melting and shrinkage, accounting for 44.37% of the mass. Alumina (Al_2O_3) played a role in polishing and grinding the tiles, representing 9.85% of the mass. Lime (CaO) contributed to enhancing the tiles' resistance against heat and abrasion, constituting 7.26% of the mass. MgO served as a sintering aid, with a mass percentage of 0.40%. Potassium oxide (K_2O) improved heat resistance, abrasion resistance, and overall appearance of the tiles, accounting for 0.77% of the mass. Sodium oxide (Na_2O) was present at 0.61%. Iron oxide (Fe_2O_3) and titanium oxide (Ti_2O) acted as colored impurities, comprising 2.84% and 0.82% of the mass, respectively. Additionally, calcite ($CaCO_3$) was identified at 11.68%, aiding in the melting and shrinkage process during heating by releasing CO_2.

1 Introduction

Porcelain tile is a highly resistant material that can withstand compact forces and frost, while also exhibiting good durability and low porosity (Amorós et al., 2022). It must adhere to ISO 10545-3 (Alonso et al., 2022), which stipulates a maximum water absorption of 0.5%. For nearly four decades, porcelain tiles have served as exceptional construction materials for walls, floors, pavements, and urban squares (Demarch et al., 2021). The market offers four types of porcelain tile, including glazed and unglazed varieties, as well as polished and unpolished finishes. In addition to their technical performance, aesthetic features play a crucial role in the

Corresponding author: E-mail: khaled.elsherif@uob.edu.ly

decision-making process for end-users. The visual appeal of unglazed porcelain tiles relies heavily on the body colour, while glazed tiles, although the glaze layer covers the body colour, still consider it important, albeit to a lesser extent than unglazed products (Li et al., 2023).

Porcelain tile, which consists of different clay minerals, feldspars, and quartz, is classified as a silicate ceramic material (Berto, 2007). It belongs to the triaxial ceramics category because of the presence of these components. With the rapid progress of the porcelain industry, there is an increasing worry regarding the depletion of clay minerals, which are essential for tile manufacturing. The primary oxide components in porcelain tile are SiO_2 and Al_2O_3, followed by Na_2O, K_2O, CaO, and MgO. Impurities like Fe_2O_3 and TiO_2 are commonly found, and their high concentrations during firing can lead to undesired coloration in an oxidizing atmosphere (Esposito et al., 2005). Therefore, it is crucial to carefully manage the levels of Fe_2O_3 and TiO_2. Traditionally, small quantities of $ZrSiO_4$ have been used to enhance the whiteness of unglazed porcelain tiles (Selli, 2015).

The porcelain tiles exhibit a noticeable range of colors, transitioning from a glossy, vibrant appearance to a more subdued, matte finish. The variation in color can be attributed to several factors. Firstly, it is influenced by the minerals present in the composition of the tiles. These minerals, including ferric oxide (Fe_2O_3) and $CaCO_3$, combine in different proportions to create distinct hues. Additionally, the presence of impurities interacts with the minerals and affects the overall color. Lastly, the atomic bonds within the mineral structure play a significant role in determining how light is absorbed and reflected by the tiles, which in turn impacts the wavelengths perceived by our eyes (Leonelli et al., 2001).

The production process employed for manufacturing porcelain tiles is a well-established and widely used method in the market. It shares similarities with the manufacturing processes of other ceramic tiles. This process comprises three primary stages: (1) the wet milling and homogenization of raw materials, followed by the spray-drying of the resulting suspension; (2) the uniaxial pressing of the spray-dried powder at a pressure of 40-50 MPa, with a moisture content ranging from 5 to 7%; (3) the rapid firing process, lasting 40-60 minutes at temperatures of 1180-1220 °C, to achieve maximum density (De Noni et al., 2010; Njindam et al., 2018). Porcelain tiles are renowned for their exceptional flexural strength, exceeding 35±2 MPa, as well as their low water absorption (<0.5%) and minimal abraded volume (< 175 mm^3). These characteristics make them highly suitable for various applications, both indoors and outdoors, whether as floor or wall tiles (Njoya et al., 2017).

X-ray fluorescence spectrometry (XRF) is an established atomic analytical technique widely used for qualitative and quantitative chemical analysis of environmental samples with diverse compositions. It covers a broad range of elements, spanning from B to U, in atomic number order. XRF provides rapid and non-destructive results, offering sensitivity in the range of 10^{-8} g (depending on the specific element of interest). This makes it highly suitable for various environmental research studies. Its key attributes include its ability to analyze multiple elements simultaneously, its acceptable speed and cost-effectiveness, ease of automation, portability, and the capability to directly analyze solid samples without prior acid digestion. These features have contributed to its maturity as an analytical tool used for routine control in various scenarios, not just limited to traditional industrial applications. XRF can be effectively employed for direct field analysis in agronomy research, on-line analysis of atmospheric particulate matter, remote acquisition of XRF spectral data, and as analytical support in environmental research laboratories (Beckhoff et al., 2006).

This study aims to present a comprehensive analysis of the chemical compositions of porcelain tiles, with the goal of exploring the utilization of untapped mineral resources from Libya. Additionally, it seeks to establish an effective mixture formulation for the successful production of porcelain tiles. The evaluation of these tiles will be conducted in accordance with the ISO 13006 standard. Currently, Libya imports porcelain tiles from Egypt, India, Spain, and Turkey. However, the country possesses abundant raw materials required for manufacturing high-quality porcelain tiles. To assess the chemical properties of porcelain tiles, samples were collected from various locations in Benghazi.

2 Materials and Methods

2.1 Sample collection

A total of 12 porcelain tile products from Egypt, India, Spain, and Turkey were selected to represent the production of these countries. Chemical analyses of the tiles were performed using X-ray fluorescence (XRF) techniques. The major components found in the composition of porcelain tiles, expressed as mass percentages, include SiO_2, CaO, Al_2O_3, Fe_2O_3, Ti_2O, MgO, and K_2O. These samples were carefully chosen to be representative, and they were obtained from various marketing and selling points. Each sample was labeled with a type designation (P for porcelain) and the corresponding country code (E, I, S, and T) for Egypt, India, Spain, and Turkey, respectively.

2.2 Sample preparation

From each type of porcelain tile, random samples were taken to ensure representativeness. The sample preparation method utilized in this study involved a simple and direct approach using pressed pellets. In general, the samples required minimal preparation; however, it was important to present the materials to the spectrometer in a consistent and uniform manner. For the porcelain tiles, a grinding process was employed to achieve a flat surface, while powders were carefully reduced to a controlled particle size before being pressed into pellets for ease of handling. The porcelain tile samples underwent a drying process in an oven at 100°C for 24 hours to eliminate moisture, after which they were finely crushed to obtain a powder with an average particle diameter of 100 μm. The average particle diameter of 100 μm was measured using a RETSCH woven wire mesh sieve. Subsequently, 10.00 g of the porcelain samples were ground and mixed with 1.00 g of binder before being prepared as pressed pellets. It is important to note that the elemental compositions of the binder used in the study were well known and previously characterized, given the established nature of the experimental setup and the use of the binder in the preparation of pressed pellets. These pellets were placed in suitable sample cups. The straightforward sample preparation method allowed for quick availability of analytical results shortly after the sample was taken. To ensure optimal count rates for various elements, the tube current was optimized and fixed (Sverchkov et al., 2023).

2.3 X-ray fluorescence (XRF) analysis

The S2 PUMA, a compact energy-dispersive X-ray fluorescence (EDXRF) instrument, offers fast and dependable quality control capabilities. It utilizes HighSense™ technology, optimizing beam path geometry to achieve high count rates, reduced measurement times, and improved precision. The measurements were conducted using the Bruker AXS GmbH advice, specifically S2 version 5.0.0, spectraEDX v2.4.2, X-ray fluorescence - Serial-No S2-204420, 220V, 3-50/60 Hz. This instrument is equipped with a 50 W X-ray tube-K230C80 and an XFlash® LE silicon drift detector-A20D800, enabling the determination of chemical components in the porcelain samples. Within a few minutes of sample collection, a single measurement provides analytical results for major components like $CaCO_3$, as well as minor and trace compounds such as Al_2O_3, SiO_2, and Fe_2O_3. The technique offers advantages including programmable measurement conditions for each element, high sensitivity, and low detection limits. The conversion of X-ray intensities into element concentrations is achieved through calibration using reference standards of known compositions.

3 Results and discussion

The primary chemical components found in porcelain tiles are silica (SiO_2), alumina (Al_2O_3), lime (CaO), magnesia (MgO), and sodium and potassium oxides (Na_2O and K_2O). Minor constituents or impurities such as Fe_2O_3 and TiO_2 are also present, and their high concentrations can lead to undesired colors during firing, particularly in an oxidizing atmosphere (Tripati et al., 2017). Therefore, it is important to carefully consider the levels of Fe_2O_3 and TiO_2. Traditionally, a small amount of $ZrSiO_4$ has been used to enhance the whiteness of unglazed porcelain tiles (Hamidalddin, 2020).

The studied oxide ratios exhibited variation within the study samples, with the average concentration order as follows: $SiO_2 > CaCO_3 > CaO \sim Al_2O_3 > Fe_2O_3 > TiO_2 > MgO \sim Na_2O \sim K_2O > Cl > MnO \sim P_2O_3 > SO_3 > MgCO_3$. The levels of oxides in different porcelain samples are presented in Table 1. The SiO_2 percents ranged from 48.28% (Egyptian porcelain) to 34.96% (Indian porcelain), with an average of 44.37±4.05%. Silicon dioxide (SiO_2) was the predominant oxide in all types of porcelain under study, but its concentrations were lower than those reported in previous studies (Sánchez et al., 2010; Gultekin et al., 2017; Montanari et al., 2022). The second most common oxide was $CaCO_3$, followed closely by Al_2O_3 and CaO, with average perecents of 11.68±3.16%, 9.86±1.61%, and 7.26±2.06%, respectively. The remaining oxides were present in proportions below 1% each, while the levels of $MgCO_3$ were below the detection limit.

Table (1) Chemical composition of tested porcelain samples

Parameters (%)	**Sample Code**											
	PE1	**PE2**	**PE3**	**PI1**	**PI2**	**PI3**	**PS1**	**PS2**	**PS3**	**PT1**	**PT2**	**PT3**
SiO_2	45.65	47.87	48.24	39.26	42.48	34.96	41.92	47.73	47.28	46.67	44.55	45.87
Al_2O_3	9.68	11.65	11.83	9.77	10.28	5.89	8.60	10.83	10.28	11,43	9.72	9.88
Fe_2O_3	2.03	4.95	3.21	2.45	2.90	2.01	2.23	2.23	2.41	3.56	2.51	3.55
CaO	5.07	5.75	5.67	5.71	5.48	8.94	10.24	6.80	5.13	8.86	9.34	10.12
MgO	0.47	0.00	0.00	0.00	0.70	0.90	1.00	0.00	0.00	0.63	0.45	0.68
Na_2O	0.66	0.62	0.65	0.55	0.62	0.63	0.58	0.65	0.66	0.61	0.59	0.55
K_2O	0.20	0.75	0.65	0.87	0.75	0.93	1.18	0.64	0.40	1.26	0.99	0.56
TiO_2	0.86	0.85	0.88	0.83	0.85	0.76	0.70	0.81	0.82	0.86	0.79	0.88
MnO	0.02	0.02	0.02	0.02	0.02	0.02	0.01	0.02	0.01	0.03	0.02	0.02
P_2O_5	0.08	0.06	0.10	0.07	0.06	0.09	0.08	0.08	0.07	0.05	0.08	0.08
SO_3	0.00	0.003	0.005	0.00	0.00	0.01	0.01	0.01	0.00	0.01	0.01	0.01
$CaCO_3$	9.05	10.27	10.98	10.19	9.79	15.97	18.28	12.17	9.15	7.64	14.78	11.92
$MgCO_3$	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cl	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15

P: Porcelain, E: Egypt, I: India, S: Spanish, T: Turkey

The composition of porcelain tiles reveals silicon oxide (SiO_2) as the predominant component, accounting for a significant portion of the tile's mass, ranging from 34.96% to 48.20%. This information is visually depicted in Figure 1. Additionally, small quantities of aluminum oxide (Al_2O_3) are present, comprising approximately 5.89% to 11.83% of the tile's mass as shown in Figure 2. The provided data reveals clear differences in the ratio of Al_2O_3 (alumina) compared to SiO_2 (silica) among the samples. These variations indicate distinct disparities in the composition of the two elements throughout the samples. The higher proportions of Al_2O_3 suggest a stronger presence of alumina, while the range of SiO_2 percentages indicates a wider distribution of silica content. These findings highlight the contrasting nature of the two elements and emphasize the importance of considering their individual ratios in further analyses or investigations. The Figure 3 provide a graphical representation of the calcium oxide (CaO) values, which range from 5.07% to 10.24%. The prominence of silicon oxide as the major component in porcelain tiles is crucial to their structural integrity and durability. SiO_2, commonly known as silica, is a key building block in ceramics due to its high melting point and its ability to form strong bonds with other elements. Its presence ensures that the tiles possess the necessary strength and resistance to withstand various stresses and impacts.

The inclusion of aluminum oxide within the composition of the tiles contributes to their aesthetic appeal and performance characteristics. Al_2O_3 acts as a fluxing agent during the firing process, enabling the tiles to achieve their desired shape and preventing excessive shrinkage. Moreover, aluminum oxide imparts certain desirable properties to the tiles, such as enhanced hardness and resistance to wear and abrasion (Dondi et al., 2005).

The calcium oxide content, as indicated in Figure 3, plays a crucial role in the final properties of the porcelain tiles. CaO acts as a flux in the ceramic composition, lowering the melting point and facilitating the verification process during firing. This results in a denser and more impervious tile surface, enhancing its resistance to moisture, staining, and chemical damage. Understanding the composition and distribution of these components within the porcelain tiles provides valuable insights into their physical characteristics, performance, and potential applications. By carefully controlling the proportions of silicon oxide, aluminum oxide, and calcium oxide, manufacturers can tailor the properties of the tiles to meet specific requirements, such as strength, durability, and aesthetic preferences (Cheng et al., 2012).

Figure (1) silicon oxide SiO_2 (mass%) of the porcelain tile samples

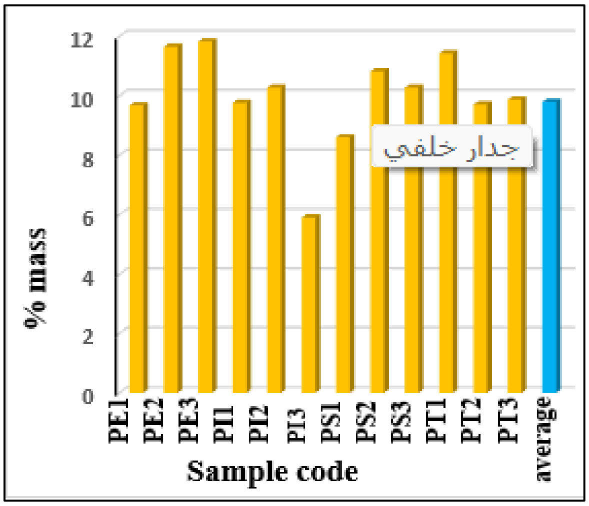

Figure (2) Aluminum oxide Al_2O_3 (mass%) of the porcelain tile samples

Figure 3 depicts the presence of minute quantities of certain constituents in porcelain tiles. These include traces of magnesium oxide (MgO), with concentrations ranging from 0.00% to 1.00% of the tile's mass. It is worth noting that the maximum concentration of MgO detected does not exceed 1.00% of the total mass. The presence of MgO in porcelain tiles can have several implications for their properties and performance. Magnesium oxide acts as a flux during the firing process, aiding in the vitrification and densification of the tiles. It can contribute to the overall strength and durability of the tiles, enhancing their resistance to cracking and structural failure. Additionally, the presence of MgO can influence the thermal expansion characteristics of the tiles, allowing them to withstand thermal stresses and temperature fluctuations without compromising their integrity. Furthermore, Figure 4 also demonstrates the presence of sodium oxide (Na_2O) and potassium oxide (K_2O) in the porcelain tiles. The concentrations of Na_2O range from 0.55% to 0.66% of the tile's mass, while K_2O concentrations range from 0.20% to 1.26%. The inclusion of Na_2O and K_2O in the tile composition can serve various purposes. These oxides act as fluxes and can lower the melting point of the ceramic mixture. This facilitates the sintering process during firing, allowing for the formation of a dense and well-consolidated tile structure. Additionally, Na_2O and K_2O can influence the viscosity of the liquid phase during firing, affecting the flow and leveling behavior of the glazes used on the tiles. This can result in improved surface quality and better adhesion of the glaze. The careful control and understanding of these minor constituents, such as MgO, Na_2O, and K_2O, in the composition of porcelain tiles are essential for achieving desired properties and ensuring consistent quality. Manufacturers can fine-tune the levels of these constituents to optimize various aspects of the tiles, such as strength, thermal stability, and glaze performance, ultimately delivering high-quality products to meet the diverse needs of architects, designers, and end-users (Martín et al., 2010; Pérez et al., 2012).

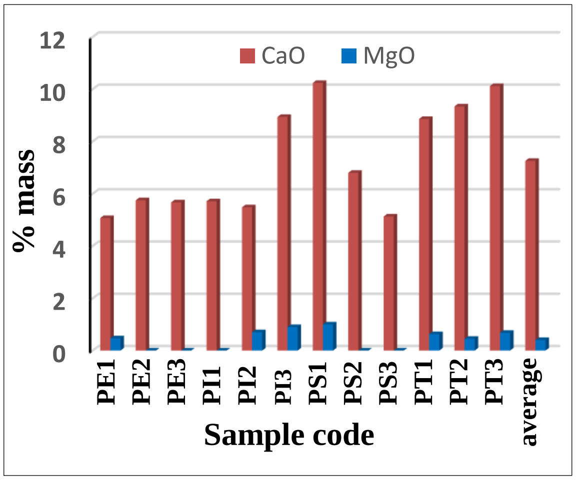

Figure (3) Calcium oxide CaO + Magnesium oxide MgO (mass%) of the porcelain tile samples

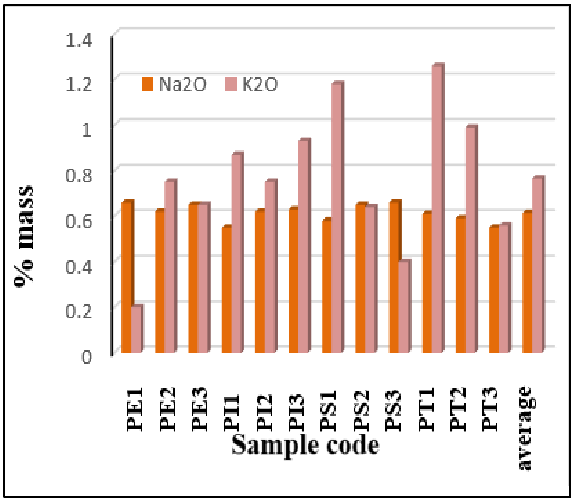

Figure (4) Potassium oxide K_2O + Sodium oxide NaO_2 (mass%) of the porcelain tile samples

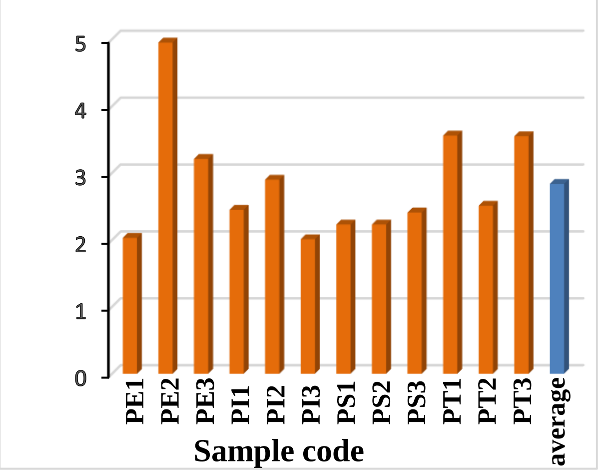

Figure (5) (Fe_2O_3 mass%) of the porcelain tile samples

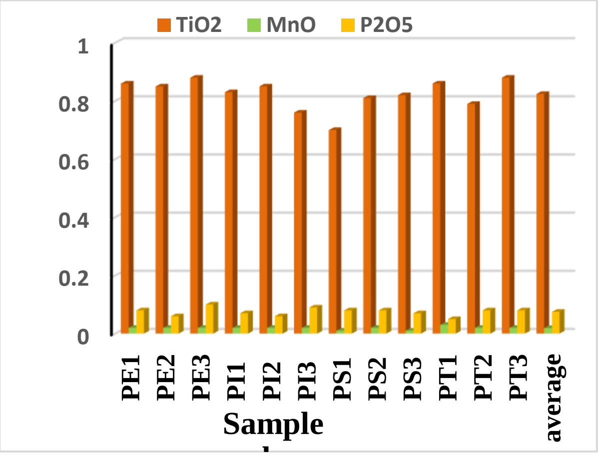

Figure (6) (P_2O_3 + TiO_2 + MnO - mass%) for all the porcelain tile samples

Figures 5 and 6 reveal the presence of minor constituents or impurities in the samples of porcelain tiles. These constituents include iron oxide (Fe_2O_3), titanium dioxide (TiO_2), and phosphorus trioxide (P_2O_3), detected in small amounts within the tiles. The measured values for Fe_2O_3 range from 2.01% to 4.95% of the tile's mass, while TiO2 concentrations fall between 0.70% and 0.88%. Additionally, P_2O_3 concentrations range from 0.05% to 0.10%. The detection of these minor constituents or impurities in porcelain tiles can have significant implications for their physical and chemical properties. Fe_2O_3, also known as iron(III) oxide or ferric oxide, is responsible for the reddish or brownish hues observed in some tiles. Its presence can add warmth and depth to the color palette of the tiles, offering a range of earthy tones. However, excessive amounts of Fe_2O_3 can affect the overall color consistency and may lead to undesirable variations in the final appearance of the tiles. TiO_2, or titanium dioxide, is a versatile compound that plays a crucial role in the ceramic industry. It acts as an opacifier, imparting brightness and opacity to the glazes used on porcelain tiles. TiO_2 can enhance the whiteness and brilliance of the glaze, improving the overall aesthetic appeal of the tiles. Additionally, it contributes to the tiles' resistance to UV radiation and provides durability against fading or discoloration over time. P_2O_3, or phosphorus trioxide, is a less commonly encountered constituent in porcelain tiles. Its presence may be attributed to impurities in the raw materials or as a deliberate addition for specific purposes. Phosphorus compounds can exhibit unique properties; such as flame retardancy or the ability to enhance certain glaze effects. However, its concentration is typically low in porcelain tiles, and its influence on the overall properties is relatively minor (Kamseu et al., 2007; Ferrari et al., 2006).

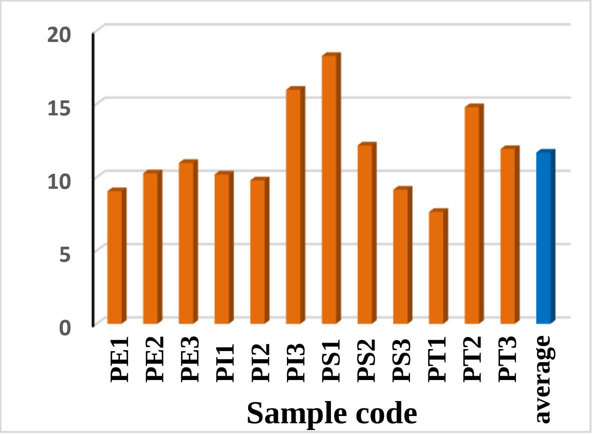

Figure (7) Calcite ($CaCO_3$ mass%) of the porcelain tile samples

Calcium carbonate is an affordable building material known for its primary physical characteristic: a white, odorless powder composed of colorless crystals that are nearly insoluble in water. In today's industrial and commercial applications, calcium carbonate is widely utilized as a mineral material. Its presence significantly enhances the stability of various building materials such as cement, bricks, and adhesives. The analysis of the samples revealed substantial levels of calcite as shown in Figure 7, which is the crystalline form of calcium carbonate ($CaCO_3$). In porcelain tiles with high silica content, the calcite content ranged from 7.64% to 12.17% of the tile's mass. Conversely, in porcelain tiles with low silica content, the calcite content varied from 9.05% to 18.28%. Examining the data presented in Table 1, we observe a consistent $CaCO_3$/CaO ratio across most samples, approximately 1.79. However, there were a few exceptions. Samples PE3, PT1, PT2, and PT3 exhibited ratios of 1.94, 0.89, 1.58, and 1.18, respectively. The $CaCO_3$/CaO ratio is of particular interest as it provides insights into the composition and behavior of the porcelain tiles. This ratio reflects the proportion of calcium carbonate to calcium oxide, both of which play vital roles in the properties and performance of the tiles. A consistent ratio suggests a balanced and controlled manufacturing process, ensuring the desired characteristics of the tiles. However, the deviations observed in certain samples indicate potential variations in the production process or the presence of additional factors that influenced the composition (Esposito et al., 2005; Bragança et al., 2011).

Based on the recorded results in Table 1, the samples can be categorized into two groups based on their silica content and other oxides:

1. Tiles with high silica content (>46% by mass), ranging from 46.67% to 48.20%. These tiles are rich in Al_2O_3 (>10% by mass) and have low concentrations of alkaline oxides (<1.4% by mass), except for sample PT1, which had a concentration of 1.87% by mass. The alkaline-earth oxides (CaO) ranged from 5.67% to 8.86% by mass, and MgO was absent except for sample PT1, which had a concentration of 0.63% by mass. The Al_2O_3/SiO_2 ratio falls within the range of 0.22-0.24, with most samples in this group having a ratio of 0.24.
2. Tiles with low silica content (<46% by mass), ranging from 34.96% to 45.86%. These tiles have higher concentrations of alkaline oxides (>1.4% by mass), except for samples PE1 and PT3, which had concentrations of 0.86% and 1.11% by mass, respectively. They also have lower amounts of Al_2O_3 (<10% by mass) and alkaline-earth oxides (CaO ranging from 5.07% to 10.24% by mass, and MgO ranging from 0.47% to 1.00% by mass). The Al_2O_3/SiO_2 ratio consistently remains equal to or below 0.22 in this group.

4 Conclusion

X-ray fluorescence (XRF) techniques were employed to determine the mass percentages of chemical components in twelve different samples of porcelain tiles collected from various markets in Benghazi city. The results revealed that percaline samples exhibited high mass percentages of quartz (SiO_2) and trace amounts of various metal oxides, including Al_2O_3, CaO, MgO, K_2O, Na_2O, Fe_2O_3, Ti_2O, P_2O_5, and MnO. Overall, there were no significant variations observed in the content of these metal oxides. The average mass percentages of silica (SiO_2), alumina (Al_2O_3), CaO, MgO, K_2O, Na_2O, Fe_2O_3, and Ti_2O were determined as 44.37%, 9.85%, 7.26%, 0.40%, 0.77%, 0.61%, 2.84%, and 0.82%, respectively. The average content of calcite ($CaCO_3$) was found to be 11.68%. From a scientific and economic perspective, it is more advantageous to use a smaller variety of raw minerals in the composition of porcelain tiles. This approach simplifies the analysis process and reduces the complexity associated with a larger number of components. However, from an engineering application standpoint, a diverse range of raw minerals in the formulation of porcelain tiles is preferable. By utilizing multiple types of minerals, the content of each mineral can be reduced. This allows for potential substitutions of similar minerals, while maintaining almost unchanged product properties. Consequently, this approach significantly mitigates the limitations associated with the availability of specific mineral resources.

Conflict of Interest: The authors declare that there are no conflicts of interest.

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