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Evaluating pumice as a sustainable raw material in porcelain tile production: impact on technical properties

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Abstract

Pumice, a porous rock resulting from the rapid cooling of tuff fragments during volcanic activity, exhibits a spongy texture and light color due to its low density. Found predominantly in Central Anatolia and Eastern Anatolia, it has drawn interest for industrial applications. This study delved into utilizing micronized pumice within the porcelain tile manufacturing process. Comparative analyses were conducted between formulations incorporating micronized pumice and the standard ceramic tile recipe. In place of feldspar, micronized pumice was introduced at concentrations of 3%, 5%, and 7%, while clay was substituted with micronized pumice at concentrations of 3%, 5%, 7%, and 10% by weight. The prepared bodies were fired in an industrial kiln at 1210 °C for 54 min, and various physical and mechanical properties were evaluated. These included viscosity, sieve residue, green strength pre-firing, firing shrinkage, water absorption, firing strength, and firing color after-firing. The results indicated that the samples incorporating micronized pumice closely matched the physical and mechanical properties of the standard porcelain tile. Phase and microstructural analyses revealed the presence of mullite and quartz phases. Notably, micronized pumice demonstrated promise as a substitute for clay or feldspar, with the optimal usage rate determined to be 7% in the porcelain tile recipe. This indicates that pumice has the potential to be an alternative raw material in the production of porcelain tiles.

Keywords Pumice · Porcelain tile · Strength · Alternative raw material · Physical properties

Introduction

Porcelain tiles occupy a prominent position among ceramic materials extensively utilized in construction and interior design, owing to their outstanding aesthetic characteristics, durability, and versatile applications. The production of

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these tiles hinges on the thoughtful selection of raw materials, with a primary emphasis on components that play crucial roles in enhancing the end product's structural integrity and visual allure [1-2]. The production of ceramic tiles relies on using natural raw materials, primarily clay, quartz, and feldspar [3]. These raw materials, extracted from the Earth, form the foundation for the manufacturing process, contributing essential properties to the final products. Clay provides plasticity, formability, and dry mechanical strength; quartz adds hardness, thermal, dimensional stability, and resistance to wear, while feldspar contributes to the overall strength, durability, and promotes the densification of ceramic tiles. This natural composition ensures that the resulting items, such as porcelain tiles or ceramic wares, exhibit desirable aesthetic appeal, durability, and versatility [4–8]. However, the sustainability and continuity of this manufacturing process face challenges due to the high cost of these raw materials and their gradual depletion over time [9]. The escalating prices of clay, quartz, and feldspar and concerns about their diminishing availability pose significant challenges to the production process's economic viability and

environmental sustainability [10]. The high price of these essential raw materials impacts the overall cost of manufacturing and raises questions about the long-term feasibility of relying solely on these finite resources. As a result, there is a growing recognition within the industry of the need to explore and develop alternative materials. The identification and adoption of alternative substances would not only mitigate the economic challenges associated with escalating raw material costs but also foster environmental sustainability by decreasing reliance on limited resources [5].

Recently, the significance of studies has heightened in utilizing alternative, cost-effective raw material resources for ceramic tile production. These studies also contribute significantly to enhancing production and firing rates, as well as improving overall quality [11]. Certain studies directed their attention towards incorporating slag [12, 13], fly ash [14], waste from ceramic sanitaryware [15], filter press tile cakes [5], cement raw mix waste [16], and glass shards [17] into ceramic tile compositions. Conversely, other studies conducted assessments on the impact of alternative raw materials like nepheline syenite [18], perlite [19], zeolite [20], talc [21], and boron resources [22, 23].

The objective of this study is to assess the influence of pumice, an alumina-silicate material rich in alkali, on the composition of porcelain tiles as a substitute for feldspar and clay. Pumice originates in the Earth's crust through volcanic processes, holding significant significance among natural resources and experiencing widespread usage. It is described as a glassy mineral with numerous pores formed as high-temperature magma releases gases abruptly and solidifies. Pumice deposits are globally abundant, notably in the USA, Turkey, and Italy. Turkey is a significant contributor, providing approximately 15-16% of the world's pumice resources. The host country, with reserves totaling around 3 billion tons, plays a crucial role in distributing this valuable material [24]. Pumice is widely used in various domains, including construction [25], composite [26], chemistry [27], textile [28], agriculture [29], and insulation materials [30, 31], reflecting its extensive utilization across diverse fields and sectors.

Studies on the utilization of pumice in the ceramic industry are scarce. Lardizabal-G et al. studied cost-effective glass-ceramic foams created by combining glass with waste materials from pumice and calcite [32]. Another study identified the potential of pumice as a substitute for feldspar in the glaze formulation of porcelain and wall tiles [33]. Furthermore, a separate examination focusing on its application in ceramic glazes found that micronized pumice, instead of kaolin, feldspar, and clay, exhibited a significant impact on the flow length and whiteness of glazes in ceramic sanitaryware [24]. Töre and Civan investigated using of pumice as a fluxing agent instead of feldspar in the glaze composition of porcelain and wall tiles. They examined hardness, firing color, dilatometry, phase, and microstructure analyses, and determined that pumice could be used in ceramic glazes [33]. In another study on ceramic glazes of roofing tile, the feasibility of using pumice as an alternative fluxing agent instead of Na-feldspar has been investigated. Additionally, compositions prepared using statistical mixture design have shown that pumice can be used in place of Na-feldspar in terms of color, brightness, hardness, and glaze melting behavior [34]. Pumice and volcanic lapillus scraps were utilized in varying proportions as alternative raw materials to imported feldspars in porcelain stoneware mixtures in another study. The work aimed to develop naturally colored substrates to reduce reliance on artificial while maintaining the standard product's technical properties [10]. In previous studies examining the usability of pumice in the ceramic sector, it has generally been noted as a potential glaze coating material. While research on the use of pumice in ceramic body compositions is limited, there is a significant need to explore the potential of alternative raw materials within the body composition. This study aims to evaluate the feasibility of using pumice instead of feldspar and clay typically used in porcelain tiles and to examine its impact on technological properties during industrial production processes. The results obtained have been compared with those of standard porcelain tiles.

Materials and methods

In this study, clays (three types), feldspar, kaolin, and micronized pumice were used in tile body composition. Pumice was supplied by TeknoBims company (Nevşehir/ Turkey). The X-ray fluorescence analysis (performed using a Rigaku ZSX Primus instrument) was employed to characterize the composition of the raw materials used in porcelain tile bodies, and the chemical analysis of the changing raw materials used in tile recipes is presented in Table 1. The raw materials to be used were wet-ground to a residue on a 63 µm sieve of 2.5-3% and then dried in a drier until a constant weight was reached. According to the chemical analysis results, the alumina-silica ratios of pumice are close to clay 1, and the total alkali oxide content is close to feldspar. The total alumina silica content of clay 1 is 89.6%, whereas for pumice, this value is 85.67%. In the feldspar used, the total alkali ratio $(Na_2O + K_2O)$ is 10.58, while for pumice, this value is 8.22. Therefore, pumice was incorporated into porcelain tile formulations as substitutes for clay1 and feldspar at concentrations of 3%, 5%, 7%, and 10% by weight. A standard industrial ceramic porcelain tile recipe was designated as the reference (std) composition. In the formulation recipes, the total proportion of clay 2-3 and kaolin was

Loi 3.33 0.55 3.63

maintained constant at 50% by weight, while the quantities of other raw materials varied as detailed in Table 2. Each prepared recipe was divided into 1 kg portions, and 500 mL of water was added. The resulting slip was ground in a ball mill for 35 min, and the viscosity, density, and sieve residue of the slurries were measured and their results are shown in Table 3. Subsequently, the slip was dried in a drier at 100 °C for 2 hours. Following the drying process, the mixtures were ground and compacted into plates measuring 55*110*8 mm using a Gabrielli press at a pressure of 152.95 kg/cm². The raw plates were fired in an industrial kiln with an industrial rapid firing regime, reaching a peak temperature of 1210°C for 54 min. The bending strength, firing shrinkage, firing color parameters, and water absorption were analyzed on the fired tiles in Table 3. The crystal phases of the samples after firing were examined using XRD (Rigaku Rint 2000), and their microstructure was analyzed using SEM (Zeiss Evo).

Results and discussions

Technical properties results

The viscosity, density, and sieve residue of the slurry samples were evaluated, and their analysis results are presented in Table 3. Two tiles were pressed: one for green strength testing and the other for after-firing tests. The densities of all slurry recipes fell within the range of 1740-1750 gr/lt, with viscosities (Ford-cup viscometer) ranging from 87 to 116 s, and sieve residues above 63 microns ranging from 0.23 to 0.28%. Additionally, green bending strength, firing shrinkage, and firing characteristics (shrinkage, strength, water absorption, color) were analyzed. The viscosity of the mixtures has a higher viscosity value compared to the standard. It can be observed that the sieve residue values are close to each other for all mixtures. When comparing the green strength results of tiles, it is observed that micronized pumice with additions of 7% and 10% instead of clay 1 and additions of 5% and 7% instead of feldspar reduces the green strength values compared to the standard. It is believed that the alteration observed may be attributed to the substitution of the plastic clay raw material with pumice. When examining the firing shrinkage values, it is observed that as the amount of pumice added instead of clay increases, the shrinkage values tend to increase. This is because pumice has a similar alumina-silicate content to clay, but its alkali content is higher than clay 1. It has also been observed that the shrinkage is high in samples P6 and P7 where pumice is added instead of feldspar. The elevated alkali content inherent in pumice, coupled with its porous structural characteristics, can be posited as a contributing

Table 1 Chemical	analysis of var	ying raw materia	als in compositi	ions (wt%)							
Raw materials	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	P_2O_5	MnC
Clay 1	74.8	14.6	0.12	1.16	0.38	0.10	0.73	4.57	0.12	0.03	0.04
Feldspar	69.4	17.6	0.30	0.23	0.77	0.30	10.2	0.38	0	0.27	0
Pumice	74.0	11.67	0	1.75	0.55	0.15	4.12	4.10	ı	ı	'

Table 2 The m	1xture proportions	s for porcelain til	es					
Recipes	Std	P1	P2	P3	P4	P5	P6	P7
Clay 1	17	17	17	17	17	14	12	10
Feldspar	33	30	28	26	23	33	33	33
Pumice	-	3	5	7	10	3	5	7

1

Table ? Tasknissi properties regults of properties

Regulta	Std	D1	D2	D2	D4	D5	D6	D7
Kesuits	Siu	<u>rı</u>	r2	r3	<u>r4</u>	<u>r</u> 3	<u>F0</u>	F /
Viscosity (sec)	87	98	105	115	89	103	105	116
Sieve residue (above 63 µm)	0.23	0.27	0.21	0.24	0.27	0.28	0.22	0.23
Green strength (N/mm ²)	1.90	2.06	1.92	1.72	0.96	2.26	1.66	1.63
Firing shrinkage (%)	8.38	8.18	8.45	8.81	9.63	7.94	8.52	9.06
Firing strength (N/mm ²)	65.14	66.11	65.31	63.25	56.27	67.26	70.4	66.74
Water absorption (%)	0	0	0	0	0.02	0.01	0	0
L*	54.06	55.34	53.54	50.21	56.35	54.95	53.7	48.2
a*	5.29	5.25	6.90	6.12	4.53	4.92	4.92	5.79
b*	12.31	12.36	12.78	11.99	14.27	12.93	12.68	11.27
ΔΕ	-	0.71	1.60	3.95	4.25	1.22	0.59	5.95

factor to the observed heightened levels of shrinkage [35]. Pumice shares a similar alumina-silicate composition with clay but surpasses it in alkali content, which plays a pivotal role in altering the behavior of the ceramic matrix. Notably, in samples where pumice substitutes feldspar, a notable increase in shrinkage is observed. The augmentation in shrinkage is predominantly ascribed to the intrinsic high alkali content and porous characteristics of pumice. Moreover, the presence of iron oxide further affects the sintering process, notably reducing the required maturation temperature [36]. This slightly higher shrinkage can attribute to the potential weight loss resulting from the chemical decomposition of pumice around 1000 °C [37]. According to the firing strength values, it is observed that as the amount of pumice added instead of clay 1 increases up to 7%, the strength values increase, but after 7%, the values decrease. The highest strength values are obtained in samples where pumice added instead of feldspar is used, with the highest resistance achieved in sample P6 at 70.40 N/mm². When evaluating the impact of pumice on the firing strength of tiles, it is evident that, despite containing the same phases as determined in phase analysis, higher strength results, up to 7% addition, are achieved compared to the standard. This is due to the pumice exhibiting a more homogeneous structure and less porosity in the microstructure [38]. When more than 7% pumice is used in tile composition, the decrease in viscosity during firing can be attributed to the increase in alkali content, specifically Na₂O and K₂O, which exhibit fluxing properties. Consequently, it is believed that the decrease in firing strength results from the resulting microstructural changes (such as porosity, crystal size, etc.) [39]. When comparing water absorption values, generally, porcelain tiles should have a water absorption value of less than 0.5% [38], and the obtained values are within the standards.

The firing color technique enables the determination of tile color by measuring three parameters: L* (brightness) ranging from absolute white (L=100) to absolute black (L=0), a* (red-green), and b* (yellow-blue), derived from the visible spectra [10]. When comparing the firing colors of the samples, it can be observed that as the amount of pumice added instead of clay and feldspar increases, the body whiteness value decreases, the a* parameter (red) increases, and the b* parameter decreases. The parameters affecting the firing color in ceramic tiles are raw material composition, firing conditions, additives, and surface roughness [40]. Even when firing conditions and additives are the same, the presence of metal oxides such as iron oxide in the content of pumice added instead of clay and feldspar in the composition, and microstructural changes resulting from the firing process due to the alkaline content in the composition (crystal size, porosity), have influenced the color parameters in tiles. The reason for this is thought to be the relatively high iron oxide content of 1.75% in pumice and the densification caused by the high alkali oxide content lowering the maturation temperature [35].

Phase analysis

The phase analyses of samples P4 and P7, which used a high amount of pumice instead of clay 1 and feldspar, along with the standard sample, resulted in obtaining mullite and quartz phases. The observed graphs are shown in Figs. 1, 2 and 3. Quartz remained as a residual phase in the fired tile bodies, while mullite formed during firing [15]. This formation of crystalline phases is an expected outcome in porcelain tiles. The interplay among crystalline phases, such as quartz and mullite, identified in the phase analysis of tiles, influences the tiles' behavior throughout the sintering process and



Fig. 1 XRD graph of the standard tile



Fig. 2 XRD phase analysis of the P4 sample



Fig. 3 XRD phase analysis of the P7 sample

subsequently impacts their mechanical and microstructural characteristics. Comprehending these interactions can aid in devising an optimized tile production process by leveraging insights from microstructural investigations into crystal distribution and grain size.

Microstructure analysis

The secondary electron images obtained from the polished fractured surfaces of the standard body, P4 and P7 coded bodies, where pumice is highly added instead of clay 1 and feldspar, are presented in Fig. 4. The EDX analyses conducted on quartz and mullite crystals are presented in



Fig. 4 Microstructures of the selected tile samples (Std, P4, P7)





Fig. 5 SEM photograph and EDS spectra of standard sample

Figs. 5, 6 and 7. The microstructure of all three bodies is similar, containing a vitreous phase, irregularly shaped quartz grains, and aligned mullite crystals typically found in a general porcelain tile body. It is noteworthy that pores are not observed in the P7 sample compared to the other samples. When there is a decrease in porosity and pore diameter associated with smaller quartz particle size, the probability of cracks surrounding quartz particles transforming into long continuous cracks is lower. Conversely, with larger quartz particle sizes, it can be observed that cracks expand and porosity increases [38, 41]. Consequently, it has been

observed that cracks and pores are present in areas where the residual quartz grain size is larger than 3 μ m in both standard and P4 samples. The absence of cracks or pores in the P7 sample is attributed to the residual quartz grain size below 2 μ m. As a result of this reduction in defects, the fracture strength is higher than the other samples.

The EDX analysis conducted on acicular and sharpedged crystals in the microstructure of the selected porcelain bodies revealed mullite crystals rich in Al, Si, and O, and quartz crystals rich in Si and O. According to the EDX analysis results performed on the indicated points, the





2 2.5 3 0.5 1.5 3.5 1 Full Scale 2473 cts Cursor: 4.157 (21 cts) ke\

Fig. 6 SEM photograph and EDS spectra of the P4 sample





compositions of the crystals formed in the mentioned two regions are presented in Table 4, exhibiting parallelism with the existing literature [42, 43]. In the EDS analysis conducted on quartz crystals, Si and O were detected, while on mullite crystals, Al and Si percentages were higher due to the characteristic X-rays obtaining data from a wider and deeper area, enabling the detection of other elements such as Na, K, Fe [44].

Conclusions

0.5

0.5

1.5

2

This study investigated the feasibility of using micronized pumice instead of clay and feldspar in porcelain tile bodies and its effects on body technical properties. The addition of pumice, instead of clay and feldspar, in porcelain tile bodies generally increased the slump viscosity during preparation and reduced the dry strength values. As the amount



2.5

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3.5

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Element	Std	Std			P7	P7	
	Spectrum 1	Spectrum 2	Spectrum 20	Spectrum 22	Spectrum 11	Spectrum 12	
0	33.66	36.32	41.98	38.91	49.96	29.95	
Na	-	2.12	-	1.96	2.26	1.10	
Al	-	23.71	-	17.90	17.88	14.13	
Si	66.34	31.33	38.02	36.80	28.33	41.25	
K	-	1.86	-	2.27	1.57	3.93	
Fe	-	4.66	-	1.53	-	6.64	
Ca	-	-	-	0.63	-	2.24	
Mg	-	-	-	-	-	0.76	

 Table 4 EDS results were obtained from selected regions

of addition increased, the firing shrinkage also increased, while there was no significant change in water absorption values. Additionally, adding pumice to the body instead of 7% clay and 5% feldspar increased the firing strength of the body. The firing colors of porcelain tile bodies were within standards. As the amount of addition increased, the body's whiteness decreased. It has been found that it is possible to develop porcelain tile recipes with technical properties suitable for standard body characteristics by reducing the proportion of clay by 7% and feldspar by 5% and using pumice in the developed recipes. Mullite and quartz phases were identified through phase and microstructural examinations. Considering all these physical and microstructural properties, it was concluded that reducing the clay and feldspar ratio and using micronized pumice up to 7% in porcelain tile body compositions was suitable. The residual quartz grain size is less than 2 µm in the P7 microstructure, which suggests that the addition of micronized pumice up to 7% resulted in a finer microstructure with smaller quartz particles, contributing to improved properties such as reduced porosity and increased fracture strength. The successful integration of pumice as an alternative raw material in porcelain tile production not only demonstrates its sustainability but also highlights its potential to have a positive impact on the industry. This can be seen through the enhancement of environmental practices and the contribution to the development of eco-friendly ceramic materials.

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Declarations

Competing interests The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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