Development ceramic floor tiles with increased shear and pressure strengths

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Abstract
In the present research work is studied and described the influence of alumina and zirconia powders on physical and mechanical properties of the conventional ceramic floor tiles. After testing several different raw material compositions the authors have found that adding 5 m% commercial ZrO₂ powders the shear strengths and pressure strengths of these floor tiles can be considerably increased. These new floor tiles with increased mechanical strengths may be suitable for use in dairy, meat, food and other industries.

Keywords: alumina, ceramics, compacting, density, floor tiles, mixtures, powders, zirconia

1. Introduction

The ceramic floor tiles are widely used as building materials both for interior architectural purposes in the living and public buildings and for hygiene purposes in the dairy, meat, food and other industries [1-2]. Because of this circumstance there are several research works which are studying, analyzing and describing the physical, chemical, thermal, acoustic and morphological properties both of the used raw materials and the sintered glazed or unglazed floor tiles [3-12]. Generally the production lines of wall and floor tiles industry are used fast drying and sintering technology with roller furnaces of less than 50 minutes cold to cold firing cycle. Because of this fast heating and cooling the green bodies of tiles are very sensitive to the diffusion of water vapor and heating curve in the beginning part of the fast firing roller kilns.

The beautifully designed wall and floor tiles which are excellent in the living and public buildings usually cannot be used in the industrial workshops, storage pools, silos and reservoirs because of the shortages of their durability and mechanical strengths. It can be possible to increase the mechanical strengths through increment of sintering temperature [13] but this is not so easy because of the low melting temperature of some of the components of the used pulverized powder-mix raw materials. Generally it is possible to get ceramic products with increased mechanical strengths trough composite material structures [14-18]. Our aims are to increase the mechanical properties and durability of the ceramic floor tiles used in food and chemical industries by creating a new ceramic composite structure adding ceramic powders with increased mechanical properties.
strengths, hardness and durability to the conventional raw materials as pulverized floor tile powders.

2. Materials and experiments

In the last 10-15 years ceramics and ceramic matrix composite materials (CMC) are widely used because of their low density and excellent wear resistance, hardness, toughness and mechanical strength [19-24]. Really the most widely used and popular technical ceramics [25-33] have relatively low density and very competitive mechanical properties (Table 1).

| Type of ceramics | Density g/cm³ | Tensile strength MPa | Bending strength MPa | Compressive strength MPa | Young Modulus GPa | Fracture toughness MPam¹/² |
|------------------|---------------|----------------------|----------------------|--------------------------|-------------------|--------------------------|
| Al₂O₃             | 3.98          | 210                  | 560                  | 2800                     | 392               | 5.5                      |
| SiC sintered      | 3.1           | 175                  | 560                  | 3920                     | 420               | 4.4                      |
| Si₃N₄ reactively bound | 2.5         | 140                  | 245                  | 1050                     | 210               | 3.3                      |
| Si₃N₄ hot pressed | 3.2           | 560                  | 910                  | 3500                     | 315               | 5.5                      |
| SiAlON            | 3.24          | 420                  | 980                  | 3500                     | 315               | 9.9                      |
| ZrO₂ partially stabilized zirconia (PSZ) | 5.8  | 455                  | 700                  | 1890                     | 210               | 11.0                     |
| ZrO₂ transformation toughened zirconia (TZT) | 5.8  | 350                  | 806                  | 1750                     | 203               | 12.1                     |

Table 1. The mechanical properties of the most widely used technical ceramics
1. táblázat A leggyakrabban alkalmazott műszaki kerámiák mechanikai tulajdonságai

In the laboratory experiments were used conventional pulverized powder mixtures of floor tiles which were prepared from the following raw materials: clay Petény I and II, clay Teplicsány, rhyolite, conventional clay Kisős, GVZ and phonolite. The mineralogical composition of these powder mixtures is described in the Table 2 and the typical microstructure and chemical components are shown in Figure 1.

| Minerals            | Chemical composition | Quantity, m% |
|---------------------|----------------------|--------------|
| β-quartz            | SiO₂                 | 22.51        |
| α-quartz            | SiO₂                 | 37.31        |
| tridimite           |                     | 7.35         |
| orthoclase          | K(Al, Fe)Si₂O₆       | 0.3          |
| nepheline           | 3Na₂O K₂O 4Al₂O₅ 9SiO₂ | 5.37        |
| albite              | NaAl₅Si₄O₁₄          | 4.55         |
| microcline          | K Al₅Si₄O₁₄          | 5            |
| illite              | 2K₂O 3MgO Al₂O₅ 12H₂O | 3.69        |
| sanidine            | (Na, K)Si₂Al₄O₁₀     | 2.89         |
| glauconit-1         | K(Fe, Al₂)Si₂O₁₀(OH)₂ | 3.32        |
| sodium aluminum silicate | Na₂O SiO₂ Al₂O₃ | 6.83         |
| sodium iron oxide   | NaFeO₂               | 0.93         |

Table 2. The mineralogical composition of the used floor tile powder mixtures
2. táblázat A hagyományos padlólap porkeverék ásványi összetétele

Fig. 1. Micrograph and chemical composition of the conventional pulverized powder of traditional floor tiles
1. ábra A hagyományos padlólap atomizer por mikroszerekete és kémiai összetétele

On the basis of the earlier experiments with alumina [34-37] and zirconia [38-40] of the authors in this research to the conventional floor tiles powder Martoxid KMS-94 alumina (Figure 2) and TZP zircon-dioxide (Figure 3) powders were added in different ratios.

Fig. 2. Micrograph and chemical composition of the Martoxid KMS-94 alumina
2. ábra Martoxid KMS-94 alumínium-oxid mikroszerekete és kémiai összetétele

Fig. 3. Micrograph and chemical composition of the TZP zircon-dioxide
3. ábra TZP cirkon-dioxid mikroszerekete és kémiai összetétele

To increase the mechanical properties to the conventional pulverized floor tiles powders Al₂O₃ (A) and commercial ZrO₂ (Z) powders were added in portion of 5m%, 10m%, 15m% and 20m% as they are shown in Table 3.

| Mixture sign | Pulverized powder | Martoxid KMS-94 alumina | TZP zircon-dioxide |
|--------------|-------------------|-------------------------|-------------------|
| A5           | 95                | 5                       | -                 |
| A10          | 90                | 10                      | -                 |
| A15          | 85                | 15                      | -                 |
| A20          | 80                | 20                      | -                 |
| Z5           | 95                | -                       | 5                 |
| Z10          | 90                | -                       | 10                |
| Z15          | 85                | -                       | 15                |
| Z20          | 80                | -                       | 20                |
| ZA5          | 95                | 2.5                     | 2.5               |
| ZA10         | 90                | 5                       | 5                 |
| ZA15         | 85                | 7.5                     | 7.5               |
| ZA20         | 80                | 10                      | 10                |

Table 3. The composition of prepared new mixtures by m%
3. táblázat A készített keverékek összetétele tömegszázalékból [m%]
From each mixture of the powders were taken out by 200g weights and milled and mixed in a Retch PM 400 planetary ball mill at 200rpm trough 5, 10 and 15 minutes. The so prepared powder mixtures were divided by 10g and compacted through one-sided pressing to discs in a cylindrical die cavity with 20mm diameter and filling depth of 50mm on uniaxial mechanical press at compressing pressures of 158MPa, 196MPa, 234MPa, 274MPa and 312MPa. After compacting the geometrical parameters of the discs were measured and determined the “green” density of the specimens. Further these discs were sintered in an electrical laboratory kiln heating up to 1250°C. When the kiln achieved this temperature the current was switched off and the kiln cooled down freely. The influence of the material composition on the color of the sintered specimens is very well seen in the Figure 4.

3. Result and discussions

During the experiments the green density of the pressed specimens were determined depending on the quantity (portion) of the zirconia and on milling times as function of the compaction pressure. It is obvious from Table 1 that density of zirconia is 5.8 g/cm³ which is much higher than the density of conventional floor tiles after sintering. So with increasing the portion of TZP powders in the mixtures will increase the green density of the pressed specimens. At the same time the densities of the pressed specimens are increased also with increments the values of compression pressures (Figure 5).

The influence of milling times also was studied at different compacting pressures (Figure 6). For these 2 different mixtures were prepared with 5m% of zirconia (a) and 5m% of alumina (b) and milled and mixed in a Retch PM 400 planetary ball mill at 200rpm trough 5, 10 and 15 minutes. In both cases there is no remarkable different between the pressed green densities after 5 min and 10 min milling but after 15 minutes of milling the densities of the pressed green specimens have decreased considerable. This phenomenon can be explained with intensive mechanochemical processes taking place during fine comminution of the ceramic powders. Thanking to this mechanochemical processes the powder mixtures have very fine grain and pore structures and the submicron particles are electro statically repel each others in the compacting die cavity during and after pressing.

While the green densities of the pressed specimens are followed the densities of the used high-tech ceramics powders after sintering the specimens with TZP zirconia additives had the smallest densities, meanwhile the ZrO₂+Al₂O₃ mixed additives have generated the highest sintered densities (Figure 7).
After the sintering the cylindrical disc specimens were tested on shear strength and pressure strength depending on the containments of zirconia (ZrO₂) and alumina (Al₂O₃) powders in the raw material mixtures. The typical destruction and fracture surfaces of the cracked specimens are shown in Figure 8 and the average values of the shear strengths and pressure strengths are shown in Figure 9.

![Fig. 8. Typical fracture surfaces of specimens after shear test (a) and pressure test (b)](image)

It is obvious from the Figure 9 that the volumes of Al₂O₃ and ZrO₂, raw materials very strong influence both on the shear strengths (a) and pressure strengths (b) of the floor tile specimens sintered at 1250°C. The highest values of shear strengths were achieved when 5m% zirconia powder was mixed into raw materials and the smallest when 15m% of (Al₂O₃+ZrO₂) mixed powders were added. The pressure strengths are also minimum when 15m% of Al₂O₃ itself and 15m% of (Al₂O₃+ZrO₂) additives are mixed into the conventional floor tile pulverized powders.

![Fig. 9. The shear strengths (a) and pressure strengths (b) of the sintered specimens made from new raw material compositions](image)

In cases when only alumina powder additives are used the shear strengths of the sintered specimens are higher in each case than their pressure strengths at 10m%, 15m% and 20m%. This phenomenon can be explained that the used 1250°C sintering temperature is not high enough when the volume of Al₂O₃ is higher than 5 m%. At the same time the largest pressure strengths of the floor tile specimens made from the new powder mixtures were achieved at volume of 20m% ZrO₂ but at 5m% ZrO₂ also very good pressure strengths were achieved with average values about 45MPa.

4. Conclusion

It is obvious from the realizes experiments that the traditional raw material composition can be used to produce ceramic floor tiles with increased mechanical shear and pressure strengths just adding to its 5m% commercial ZrO₂ powders and sintering it at 1250°C.

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Növelt nyíró- és nyomósúlyállású kerámia padlólapok fejlesztése

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Próba kiemelten vitatott padlólapokat, amelyekhez a fenti anyagokat használtak.

5. A padlólapok kiemelten vitatott anyagokat használtak.