REDUCING THE DENSITY OF FIRED CERAMIC PRODUCTS

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Abstract

Fired brick is one of the most widespread building materials, in the manufacture of which, in addition to strength, great emphasis is placed on the thermal insulation capacity of products. Reduction in bulk density is essential to further improve thermal insulation performance. During our work, in addition to adding sawdust of different fractions, we reduced the bulk density of the fired ceramic samples by mixing 10, 20, and 30 vol% expanded perlite. For the experiments, clay masses were prepared with a laboratory pan mill with a moisture content of 22 wt%. The formed and dried samples were fired at 870 °C for 4 hours. The body density of the samples decreased from 1708 to 1295 kg/m³ as the mixing rate of expanded perlite increased. The coefficient of thermal conductivity decreased significantly, the lowest value was 0.16 W/m·K. Thermal insulation capacity can be improved without significant reduction in compressive strength, by mixing expanded perlite.

Keywords: brick production, bulk density, clay, compressive strength, expanded perlite

1. Introduction

Nowadays, there is an increasing focus on improving the thermal insulation capacity of building materials. This is due to the increasingly stringent energy regulations on the one hand and the increased demand for sustainable residential buildings on the other (Maafa et al., 2023; Leite da Chuna et al., 2020). Fired bricks are one of the oldest building materials, the properties of which depend on the composition of the clay used as raw material, the additives used, the type of brick, and the manufacturing process (Cuce et al., 2022) and (M. Ibrahim et al., 2020). In brick production, pore-forming additives are often used, which are added to the plastic clay mass to improve the thermal insulation capacity of products (Kocserha et al., 2013). Pore-forming additives are typically of organic origin, which burn out of the formed product during brick burning, leaving pores behind, thereby reducing bulk density and increasing thermal insulation capacity (Ozturk, 2023). Sawdust is mostly used as a pore-forming additive, which is a widely available by-product of the production of fuel firewood, sawwood, veneer, cellulose and paper (Cultrone et al., 2020). However, the grain size and morphology of additives affect the physical and mechanical properties of bricks (Muñoz et al., 2016) and (Ruparathna et al., 2016). The application of organic additives is limited, as it significantly reduces mechanical strength when used in

large quantities (Taurino et al., 2019). Another negative aspect of the use of these additives is that due to their organic nature, they increase the CO_2 emission generated during production (González et al., 2011). It is quite a challenge to improve the thermal insulation capacity of burnt brick products while maintaining compressive strength. For this reason, research is ongoing on additives that remain thermally stable at the firing temperature of brick production. This temperature typically ranges between 840–900 °C. These additives are being studied for their structure, as they have a positive effect on the thermal insulation capacity. Several researchers studied the use of expanded perlite as a pore-forming additive, in each case the aim was to reduce bulk density and increase porosity. (Sadik et al., 2013) made porous burnt bricks using clay, recycled additives, and expanded perlite at a firing temperature of 1600 °C, resulting in samples of 1550 kg/m³. In another study (Topçu and Işikdağ, 2007), samples with a density of 255 kg/m³ were prepared by incorporating 50 wt% expanded perlite with a firing temperature of 950 °C; however, in this case, the compressive strength was 2 MPa. A. Georgiev used expanded vermiculite and expanded perlite mixed in 0-8 wt% and investigated their effects on thermal conductivity (Georgiev et al., 2018). The firing temperature was in the case also above 900 °C. Most of the literature-reviewed studies were used temperature ranges above 900 °C and they have not been tested study the use of expanded perlite in combination with other pore-forming additives. Perlite mainly contains silica (70-75 wt%), alumina (12-15 wt%), and 2-3 wt% potassium, sodium, iron and magnesium oxides (Orhan, 2004). In the manufacture of expanded perlite, perlite rock is crushed into grains of 1–3 mm, fractionated, and dried. The finely ground perlite granules are sent to an oven, where they are heated abruptly to 800 to 1200 °C. In this case, due to the high temperature, the outer surface of the granules softens, the 2–5 wt% crystalline water contained in them evaporates and they swell up to 5-20 times their original volume (Topçu and Işikdağ, 2007) and (Celik, 2015). Its thermal insulation properties are due to the many small pores formed by the swelling process. Expanded perlite is noncombustible (up to 900 °C) and has a low coefficient of thermal conductivity (0.045–0.059 W/m·K) (Pichor, 2009).

In this study, the effects of expanded perlite on the drying and firing shrinkage, water absorption, compressive strength, and thermal conductivity of fired ceramics were investigated.

2. Material and methods

2.1. Raw materials

For the studies, a Hungarian brick clay mixture was used, which was prepared with a mixture of two clays mixed together with yellow (YC) and gray (GC) clay in a 3 : 1 ratio. The mineral composition of the clays was analyzed by X-ray powder diffraction measurement (Rigaku Miniflex II, Cu K α , in the range of $3-90^{\circ} 2 \Theta$). The quantitative results were calculated using Rietveld's full profile fitting analysis. The mineral composition of yellow and grey clays is presented in *Table 1*. The clay mineral content (plastic components) in yellow clay was 45 wt% (illite, muscovite, smectite, kaolinite, illite-smectite, chlorite), while in grey clay it was 41 wt%. As a result, yellow clay exhibited more plastic properties. The amount of non-plastic components in the yellow clay was 53 wt% (quartz, albite, microcline, calcite, dolomite, rutile) while the grey clay contained 51 wt%. In addition to plastic and non-plastic minerals, the yellow clay contained 2 wt% and the grey clay 8 wt% X-ray amorphous material.

Sawdust with two different grain size fractions (d < 1 mm and d < 4 mm) and expanded perlite with grain sizes between 0-1 mm were added to the clay mix as additives.

Raw mat.	Q	Ι	Μ	А	SM	K	MI	С	D	ISM	R	СН	AM
[wt%]													
YC	34.07	28.32	7.93	8.98	2.88	3.43	5.72	2.70	1.16	0.53	0.37	1.92	2
GC	37.08	24.93	7.99	8.76	2.90	2.61	2.13	1.74	0.86	0.22	0.46	2.34	8

Table 1. Mineral composition of raw materials

Q – quartz; I – illite; M – muscovite; A – albite; SM – smectite; K – kaolinite; MI – microcline; C – calcite; D – dolomite; ISM – illite-smectite, R – rutile; CH – chlorite; AM – amorphous content

2.2. Mixing and sample preparation

For the tests, plastic clay masses with different recipes were prepared by pan mill (*Figure 1*), the compositions of the mixes were summarized in *Table 2*. The two different grain size fractions of the saw dust (d < 1 mm and d < 4 mm) can be seen in *Figure 2*. During the application of the expanded perlite, it was necessary to pre-wet the perlite to reduce the dusting off. The moisture content of all clay masses was 22 wt% per dry matter. The masses homogenized with the pan mill were rested in an airtight container for 48 hours, then cylindrical specimens with a diameter of 33 mm and a height of 50 mm were prepared with a KEMA PVP 5/s laboratory vacuum extruder (*Figure 3*), 8 pieces per set.

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I wor	<i>u</i> " .	110	purcu	1111111111111		icounts

Set	S	Р	Sot	S	Р	Set	S	Р	Sat	S	Р
Sel.	[vol%]	[vol%]	Sel.	[vol%]	[vol%]	Sel.	[vol%]	[vol%]	Sel.	[vol%]	[vol%]
1S23P0	23*	0	4S23P0	23**	0	1S33P0	33*	0	4S33P0	33**	0
1S23P10	23*	10	4S23P10	23**	10	1S33P10	33 [*]	10	4S33P10	33**	10
1S23P20	23*	20	4S23P20	23**	20	1S33P20	33 [*]	20	4S33P20	33**	20
1S23P20	23*	30	4S23P30	23**	30	1S33P30	33*	30	4S33P30	33**	30

S: Sawdust, P: Perlite (70 kg/m³), * sawdust grain size $d > 1 \text{ mm} (130.25 \text{ kg/m}^3)$, ** sawdust grain size $d > 4 \text{ mm} (117.95 \text{ kg/m}^3)$



Figure 1. Pan mill



Figure 2. The sawdust fraction used as pore forming additive (left: d < 1 mm; right: d < 4 mm)



Figure 3. KEMA PVP 5/s laboratory vacuum extruder

After forming, the specimens were dried for 48 hours in the open air and then at 105 °C in a drying chamber. The dried samples were placed in a laboratory furnace of type HŐKER 2/3 1200 (*Figure 4.*), and fired at 870 °C. The heating rate was 95 °C/h and the samples remained at the maximum temperature for 4 hours. The fired samples are shown in *Figure 5*.



Figure 4. Dried test specimens



Figure 5. Samples fired at 870°*C*

3. Results and discussion

3.1. Drying and firing shrinkage

The height and diameter of the green and fired specimens were measured after raw production. The drying and firing volume shrinkage of the test specimens was calculated from the measured data. For each mixture, the average value of 10 samples was taken. *Figures 6.* and *Figure 7.* show the drying shrinkage values.

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Figure 6. Drying shrinkage of samples containing 23 vol% sawdust

Figure 7. Drying shrinkage of samples containing 33 vol% sawdust

The results showed that the drying volume shrinkage of specimens prepared with d < 1 mm sawdust was reduced by the addition of expanded perlite. Drying shrinkage was reduced from 13.91% to 11.37% with 10 vol% expanded perlite at 23 vol% sawdust The combination of 33 vol% sawdust and 10 vol% expanded perlite has caused a 2% reduction in drying shrinkage of the samples. In the case of d < 4 mm, which contains a larger grain size, the opposite trend was observed, as the change in drying volume compared to the reference increased. An exception is made for samples made with 23 vol% sawdust and 10 vol% expanded perlite, as the volume change compared to the reference was reduced from 10.85% to 10.50%.



Figure 8. Firing shrinkage of samples containing 23 vol% sawdust

Figure 9. Firing shrinkage of samples containing 33 vol% sawdust

Figure 8 and *Figure 9* show the change in firing shrinkage as a function of the amount of expanded perlite. Results showed very hectic changes. In the case of the clay used, it was observed that it sometimes does not shrink during firing, but swells slightly, and the shrinkage values are also quite small. Therefore, the firing shrinkage in some cases shows negative values. The addition of 23 vol% of sawdust with a particle size of less than 1 mm without expanded perlite showed a shrinkage of 0.21%, whereas the addition of expanded perlite showed a swelling of the specimens. For sawdust below 4 mm and up to 20 vol% of perlite, swelling was observed in samples, but the addition of 30 vol% of expanded perlite caused volume shrinkage. Different results were observed at 33 vol% sawdust. Swelling of samples was observed by adding sawdust d < 1 mm and 10 and 30 vol% perlite, while 20 vol% perlite caused 0.08% shrinkage. For sawdust with a diameter of less than 4 mm, the shrinkage of samples without perlite was 0.69%. In contrast, shrinkage was almost negligible at 10 vol% perlite but increased at 20 and 30 vol%.

3.2. Bulk density and water absorption

The bulk density of the samples was calculated based on the measured geometric data and the mass after firing. For the results, an average of 8 samples was taken for each setting. In addition to determining bulk density, a 24-hour water absorption test was also performed, which shows the ratio of open pores in samples. *Figure 10* shows the change in bulk density of fired specimens prepared with 23 vol% sawdust as a function of the amount of expanded perlite added. In this case, the density decreased from 1500.76 kg/m³ to 1431.63 kg/m³ at d < 1 mm sawdust and from 1708.16 kg/m³ to 1455.80 kg/m³ at the wider fraction of sawdust.



Figure 10. Bulk density of samples containing 23 vol% sawdust



Figure 11. Bulk density of samples containing 33 vol% sawdust

Figure 11 summarises the change in body density of samples containing 33 vol% sawdust. It showed a similar trend as in the case of 23 vol% sawdust. By increasing the amount of expanded perlite, the density of the specimens decreased.





Figure 12. 24-hour water absorption test results of samples containing 23 vol% sawdust

Figure 13. 24-hour water absorption test results of samples containing 23 vol% sawdust

The results of the water absorption tests are shown in *Figure 12* and *Figure 13*. In this case of 23 vol% sawdust, water absorption of the smaller fraction increased from 16.50% to 22.32% by mixing 30 vol% expanded perlite, while for d < 4 mm sawdust this figure increased from 13.40% to 21.66%. The rate of water absorption of samples continued to increase, by increasing the amount of sawdust. The average water absorption of samples made with d < 1 mm sawdust showed an increase of 60% compared to the results of specimens made without expanded perlite, while at d < 4 mm sawdust the difference was already 80%. Results show that by increasing the grain size of sawdust and the amount of expanded perlite, the proportion of open pores increases.

3.3. Compressive strength test

The compressive strength test was carried out with 10 T material tester on 8 samples per setting. The results of the study are shown in *Figures 14* and *15*. *Figure 14* shows that when 23 vol% d < 4 mm sawdust was applied, the strength increased from 19.89 MPa to 21.43 and 21.21 MPa by mixing 10 and 20 vol% of expanded perlite, and then decreased to 17.53 MPa for 30 vol%. In the addition of d < 1 mm of sawdust, there was a clear decreasing trend: compressive strength gradually decreased from 22.94 MPa to 18.06 MPa. By increasing the amount of sawdust to 33 vol%, a decrease was observed for both sawdust fractions: strength decreased from 19.15 MPa to 11.25 MPa for d < 1 mm, and from 19.89 MPa to 9.51 MPa for d < 4 mm.



Figure 14. Compressive strength of samples (23 vol% sawdust)



Figure 15. Compressive strength of samples (33 vol% sawdust)

3.4. Thermal conductivity

The coefficient of thermal conductivity of the samples was determined by C-Therm TCi thermal conductivity measuring device. In this case, 1 sample per set was measured 3 times, then the average of these was given as the result. Numerical values are presented in *Figure 16* and *17*. *Figure 16* clearly shows that at 23 vol% sawdust, the thermal conductivity decreased with increasing amount of expanded perlite, independent of the sawdust particle size. 35% reduction was obtained by adding 30 vol% expanded perlite for d < 1mm sawdust fraction. For samples prepared with a larger sawdust particle size fraction, the reduction was 52%. Increasing the amount of sawdust to 33% by volume resulted in 50% and 48% decrease in thermal conductivity of samples, respectively.



Figure 16. Thermal conductivity of samples containing 23 vol% sawdust



Figure 17. Thermal conductivity of samples containing 23 vol% sawdust

4. Conclusion

The thermal insulation ability of building materials is closely related to bulk density. The addition of expanded perlite showed a decrease in bulk density, therefore the thermal conductivity also decreased. The average bulk density (23 vol% d < 4 mm fraction sawdust) of samples was 1710.07 kg/m³ and the related coefficient of thermal conductivity was 0.56 W/m·K. Samples prepared with sawdust with smaller particle sizes had a bulk density of 1612.58 kg/m³ with a thermal conductivity coefficient of 0.43 W/m·K. The addition of expanded perlite further reduced the body density of the samples. The d < 4 mm sawdust fraction reduced density by 14.87%. Adding 30 vol% perlite to the clay mix, the coefficient of thermal conductivity reduces by 50% from 0.56 W/m·K to 0.26 W/m·K. The water absorption capacity of samples prepared with a smaller sawdust fraction increased by increasing the amount of expanded perlite. The greatest increase (45%) was observed at samples containing d < 4 mm sawdust fraction and 30 vol% expanded perlite, compared to samples made without expanded perlite. The reduction in compressive strength of 12–21% was observed for samples containing 23% by volume of sawdust mixed with expanded perlite. The compressive strength was observed to remain above 10 MPa, even when 30% by volume of expanded perlite was added. This indicates that the tested samples can be used for load-bearing masonry (MSZ EN 771-1). Based on the results, the bulk density can be reduced by mixing expanded perlite. Masonry with a lower heat transmission coefficient can be produced with appropriate compressive strength values resulting in energy saving in buildings.

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