

INFLUENCE OF THE ADDITION OF PORE FOAMING AGENT ON MECHANICAL AND THERMAL PROPERTIES OF PORCELAIN TILES

PANIDA WIMUKTIWAN, MANA RODCHOM, KANIT SOONGPRASIT,
DUANGDUEN ATONG, [#]SUPAWAN VICHAPHUND

*Ceramics and Construction Materials Research Group, National Metal and Materials Technology Center (MTEC),
National Science and Technology Development Agency (NSTDA),
Khlong Nueng, Khlong Luang, Pathumthani, 12120, Thailand*

[#]E-mail: supawank@mtec.or.th

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In this work, the effect of pore foaming agents (polymethyl methacrylate, PMMA) on the densification characteristics as well as mechanical and thermal properties of sintered porcelain samples was investigated. The influence of the PMMA content (0 - 20 wt. %) and sintering temperature (1000 - 1300 °C) were also studied. The result showed that the incorporation of PMMA in porcelain bodies led to a decrease in the density, while the porosity increased with an increase in sintering temperature. At 1300 °C, the bulk density and porosity of porous porcelain samples with PMMA additions (5 - 20 wt. %) were in the range of 1.91 - 2.23 g·cm⁻³ and 1.3 - 17.2 %, respectively. The flexural strength of the porous porcelain samples prepared by adding 5 - 20 wt. % PMMA and sintering at 1300 °C was in a range of 36 - 56 MPa, which is higher than the minimum strength values (35 MPa) of ISO 13006 standard for ceramic tiles. The thermal conductivity of porous porcelain is a function of the sintering temperatures. The increase in sintering temperature had a positive effect on the thermal conductivity of ceramic samples, because the thermal conductivity decreased significantly with increasing porosity levels. The addition of 20 wt. % PMMA in porcelain samples sintered at 1300 °C reduced the thermal conductivity up to 34 % while maintaining acceptable flexural strength values (~ 36 MPa). Thus, it is possible to use these porcelain samples as ceramic tiles in order to enhance the thermal resistance of building materials.

INTRODUCTION

Nowadays, porcelain stoneware tiles have become very popular as these products have high physical and mechanical properties. The ceramic tiles manufactured in the industrial sector are commonly classified according to ISO 13006 [1], which is mainly divided based on water absorption and different strength levels. The classification of floor tiles or wall tiles depends on their usage and their properties. Floor and wall tiles are widely used in buildings and houses, bathrooms, kitchens, swimming pools, laboratory rooms, hospitals and industries.

Several compositions of porcelain stoneware have been reported [2–17]. They commonly consisted of clay 40 - 75 %, feldspar 0 - 18 %, and quartz 27 - 50 % [3]. A lot of works have focused on the feasibility of replacing ceramic components by applying alternative raw materials [4, 18], functional ceramic tiles with cleanability or photocatalytic properties [19] and the improvement of thermal property in order to increase the efficiency of construction materials in terms of reducing energy consumption for environmental friendliness [5, 7, 20]. For this purpose, pore forming agents including polymer particles [5, 7], starch [21], carbon black

[22], and biomass [23, 24] have been widely studied as they can easily control the pore size as well as create the various porosity levels, leading to a reduction of thermal conductivity. However, only a few works focused on both the mechanical and thermal properties of clay-based materials [5, 7, 24]. Among of pore forming agents, starch seems to be an appropriate pore foaming agent because it is a natural biopolymer, with easy handling and processing, good commercial availability relatively low cost. Unfortunately, the pore size range is limited, typically between 5 and 50 micrometers. This limits the application when large pores are desirable [5]. In the case of biomass, irregular and larger pores are usually found in the porous ceramics owing to the agglomeration and nonuniformity of biomass as a pore-forming agent, resulting in lower mechanical strength. In addition, some impurities such as K₂O, Na₂O and SiO₂ may also reduce the mechanical properties of ceramics [22]. Polymer particles are an effective alternative to starch and biomass. They exhibit defect-free burn out at relatively low temperature. Furthermore, they are usually environmentally friendly and also relatively cheap. Additionally, they allow very efficient pore size control and are easily mixed with ceramic powders. These advantages make them economically attractive for industrial applications [5].

In this research, polymethyl methacrylate (PMMA) was selected as a pore forming agent to generate porosity in porcelain tiles (which may be considered as a special type of porcelain stoneware tiles), as PMMA is widely used in ceramics since it presents a good balance of thermal properties suitable for the proposed application. PMMA decomposes almost exclusively to its monomer and at a very high and steady state [5]. The aim of this research was to study the influence of pore foaming agents (polymethyl methacrylate, PMMA) on the physical, mechanical and thermal properties of sintered porcelain samples. The contents of PMMA (0 - 20 wt. %) and sintering temperature (1000 - 1300 °C) were also studied.

EXPERIMENTAL

The raw materials used in this investigation were kaolin and potassium feldspar (both from Amarin Ceramics Co., Ltd, Thailand) and quartz (Cernic International Co., Ltd, Thailand). All raw materials had an industrial grain size less than 45 µm (mesh 325). The chemical compositions of ceramic raw materials were determined by X-ray fluorescence (XRF; Rigaku ZSX Primus) analysis. Polymethyl methacrylate (PMMA with a density of 1.19 g·cm⁻³, obtained from Liack Seng Trading Co., Ltd., Thailand) was used as a pore foaming agent. Prior to the experiment, PMMA particles were ground with a high speed rotor mill (Ultra centrifugal Mill ZM200, Retsch) in order to reduce the particle size from 3 - 4 mm to 45 - 250 µm.

Table 1. Batch compositions of porcelain samples.

Compositions	Raw materials (wt. %)			Pore forming agents (wt. %)
	kaolin	Quartz	K-feldspar	PMMA
P-Blank	50	25	25	0
P-PMMA 5	50	25	25	5
P-PMMA 10	50	25	25	10
P-PMMA 20	50	25	25	20

Initially, a porcelain composition was prepared by mixing 50 wt. % kaolin, 25 wt. % quartz and 25 wt. % potassium feldspar by conventional ball milling with distilled water for 5 h. The slurry was dried at 100 °C for 24 h and ground in a porcelain mortar. The mixed powder then passed through a 60-mesh screen (250 µm). In order to fabricate the porous porcelain, body mixes were formulated by adding various amounts of pore foaming agent (polymethyl methacrylate, PMMA) with a size of 45 - 250 microns on the proportion to 0 (P-Blank), 5 (P-PMMA 5), 10 (P-PMMA 10), and 20 wt. % (P-PMMA 20), respectively. The batch compositions of porcelain samples are given in Table 1. Then, the mixed powders were uniaxially pressed to form bar-shaped specimens (3 × 4 × 30 mm) at 92 MPa and disc specimens with a diameter of 35 mm at 82 MPa. After that, the specimens were sintered in an electric furnace at four different temperatures (1000, 1100, 1200 and 1300 °C) with a heating rate of 5 °C·min⁻¹ and a dwell time of 1 h at maximum temperature. A schematic diagram of the experimental procedure is shown in Figure 1.

The Archimedes method was employed to determine the bulk density, water absorption and porosity (apparent, i.e. open, porosity) of the sintered samples. The three-point bending strength at room temperature was determined using a universal testing machine (Instron model UTM 55R4502) with a span of 20 mm and a crosshead speed of 0.5 mm·min⁻¹. Phase analysis of samples was conducted through X-ray diffraction (XRD; PANalytical X'pert pro) with 40 kV, 30 mA, CuKα radiation. The sample was scanned in a diffraction angle range of 10° to 90° with a step size of 0.04 and time per step 1 s. The microstructure of samples was observed using a scanning electron microscope (FESEM; HITACHI, SU5000). Mercury intrusion porosimetry (Autopore V9600, Micromeritics) was used to study the pore size and pore size distribution. Thermal conductivity was measured using a thermal conductivity analyser (TPS2500S, Hot Disk) at room temperature. Samples were prepared in the disc shapes with 35 mm diameter and thickness of approximately 5 mm.

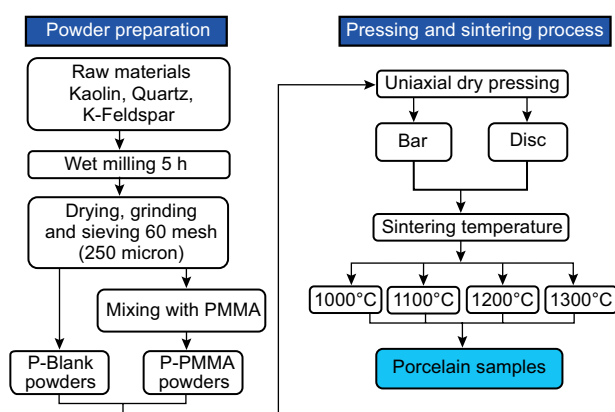


Figure 1. Schematic diagram of experimental procedure.

RESULTS AND DISCUSSION

Raw material characterization

The chemical analysis of raw materials used for this study is given in Table 2. From the results, it was found that kaolin mainly consisted of SiO₂ (47.5 %) and Al₂O₃ (35.9 %) with a small amount of Fe₂O₃ (1.2 %), K₂O (1.8 %), MgO (0.1 %), etc. The quartz contained pure SiO₂, 99.4 %. Generally, quartz is an inert filler providing high strength to sintered specimens [9, 25]. The main components of potassium feldspar were SiO₂ (64.7 %), Al₂O₃ (16.1 %) and K₂O (15.6 %). Due to its high alkali contents, potassium feldspar acted as a fluxing agent, promoting a liquid phase at low temperature [3].

Table 2. Chemical composition of raw materials used in this study.

Component (Oxide, wt. %)	Raw materials (wt. %)		
	Kaolin	Quartz	K-feldspar
SiO ₂	47.49	99.40	64.65
Al ₂ O ₃	35.85	0.05	16.11
Na ₂ O	0.03	0.02	1.15
MgO	0.10	0.05	0.06
Fe ₂ O ₃	1.18	0.02	0.26
K ₂ O	1.78	0.02	15.55
P ₂ O ₅	0.04	–	0.34
SO ₃	0.04	0.01	0.13
CaO	0.02	0.13	0.79
TiO ₂	0.06	–	0.04
MnO	0.05	–	0.01
L.O.I (%)	12.86	0.22	0.53

Green porcelain samples

Figure 2 shows the variation of green density values as a function of a pore foaming agent content. The low density of green samples was due to the lower density of PMMA (1.19 g·cm⁻³). The green densities of the porcelain samples were in the range of 1.75 - 1.54 g·cm⁻³.

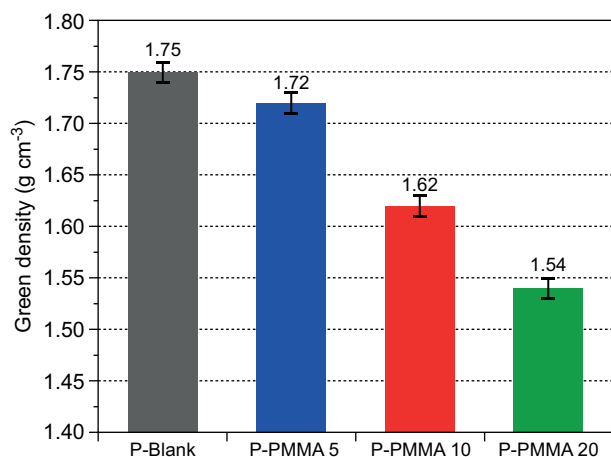
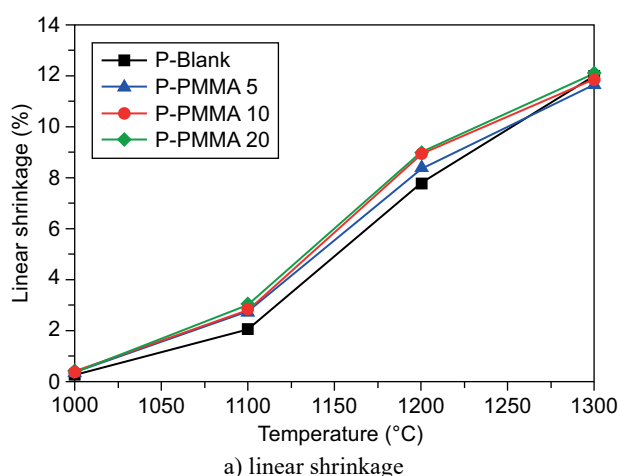


Figure 2. Green density of samples.

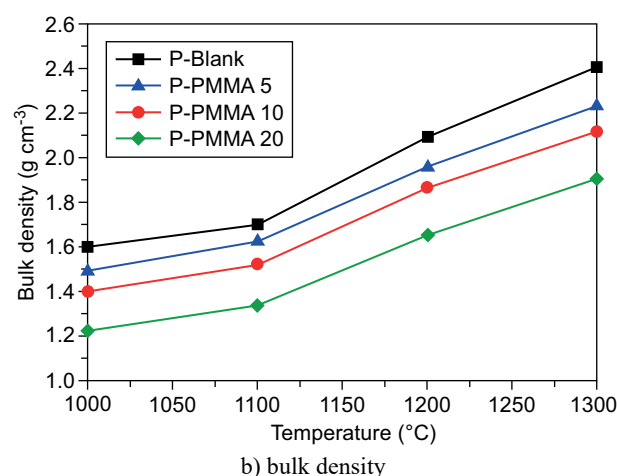


a) linear shrinkage

Considering a standard porcelain sample without PMMA addition (P-Blank), the green density value (1.75 g·cm⁻³) is comparable to that obtained from other porcelain compositions in the range of 1.8 - 2.0 g·cm⁻³ [26].

Effect of PMMA addition on the densification characteristics and mechanical and thermal properties of sintered porcelain samples

The porosity of the porcelain samples can be controlled by adding PMMA with contents in the range 0 - 20 wt. %. Therefore, variations of linear shrinkage, bulk density, porosity and water absorption can be observed after firing to different temperatures. The linear shrinkage of the porcelain sample without (P-Blank) and with PMMA additions (P-PMMA) had a similar tendency as shown in Figure 3a. The linear shrinkage of samples continuously increased with an increase in temperature and reached the maximum value (11.7 - 12.2 %) at 1300 °C. The variation of bulk density for all porcelain samples is similar to their linear shrinkage as exhibited in Figure 3b. The bulk density for P-Blank sample significantly increased with a progressive rise in temperature and achieved a value of 2.41 g·cm⁻³ at 1300 °C, which is consistent with that of typical porcelain-like bodies, 2.3 - 2.5 g·cm⁻³ [4, 18, 20, 27]. In addition, the incorporation of PMMA into porcelain bodies (P-PMMA) led to a decrease in the bulk density values due to the high level of porosity generation after the burnout of the organic particles during the firing process [5]. As can be seen in Figure 3c, the apparent porosity increased with an increment of PMMA. At 1300 °C, the sample without PMMA (P-Blank) had the lowest apparent porosity of 0.2 %. In contrast, the porosity increased up to 17.2 % when adding 20 wt. % of PMMA (P-PMMA 20). The results indicate that this pore foaming agent can generate open porosity in the fired porcelain bodies [5]. Considering the water absorption (Figure 3d), these values tended to decrease with



b) bulk density

Figure 3. Densification characteristics of porcelain samples after sintering at temperatures in the range 1000 to 1300 °C: a) linear shrinkage, b) bulk density. (Continue on next page)

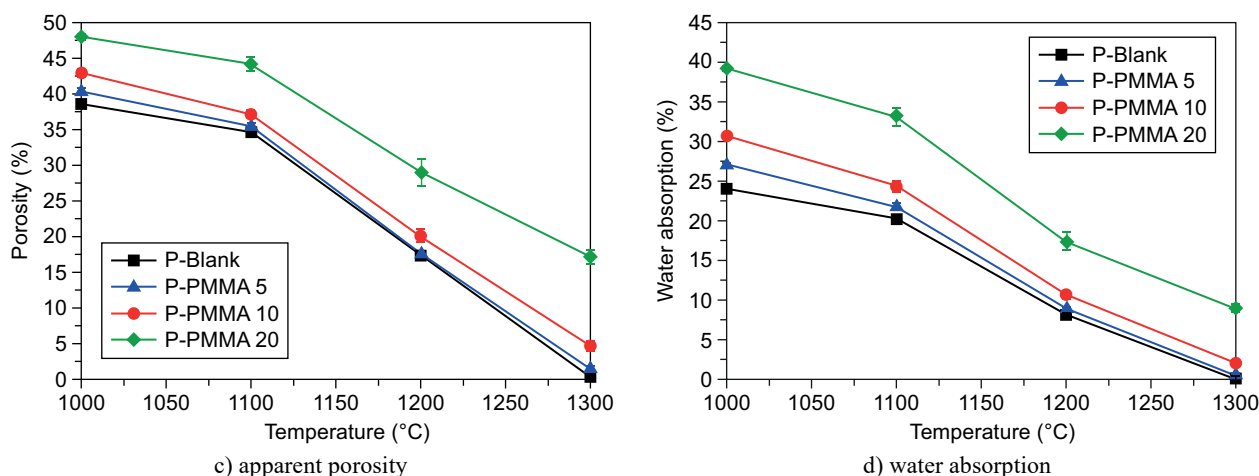


Figure 3. Densification characteristics of porcelain samples after sintering at temperatures in the range 1000 to 1300 °C: c) apparent porosity, d) water absorption.

increasing temperatures due to the reduction of apparent porosity through a liquid phase formation during the firing process. At the highest temperature, the water absorption of P-Blank and P-PMMA 5 samples was close to zero (i.e., 0.1 - 0.6 %), while the addition of PMMA 10 - 20 wt. % increased the water absorption values to more than 2 %.

Figure 4 displays the flexural strength of sintered porcelain samples with and without PMMA addition. The sample without PMMA (P-Blank) showed an increase in strength with firing temperature and reached the highest value (77 MPa) at 1300 °C. The addition of PMMA caused a lower strength of porcelain samples, 36 - 56 MPa. It is well known that a high level of porosity resulted in the reduction of mechanical properties [5]. In this case, the densification of porcelain consisted of two steps including PMMA decomposition and liquid phase sintering process as shown in Figure 5. Initially, the PMMA particles were completely decomposed above 500 °C [5], creating porosity in the ceramic bodies. Then, the densification of porcelain involving a liquid phase sintering process initially occurred at around 900 - 1000 °C. A liquid phase began to form, surrounding

the particles and generating a process-driving capillary underpressure (capillary suction) at the contact points, leading to shrinkage and densification (porosity reduction). Towards the end of the intermediate stage of sintering, at around 1180 °C, pores began to close

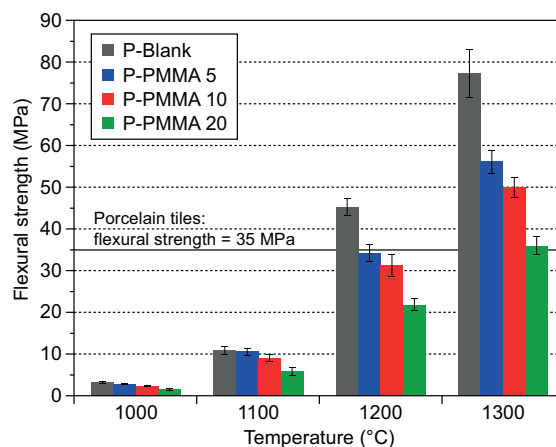


Figure 4. Flexural strength of sintered porcelain samples with and without PMMA addition sintered at temperatures in the range 1000 to 1300 °C.

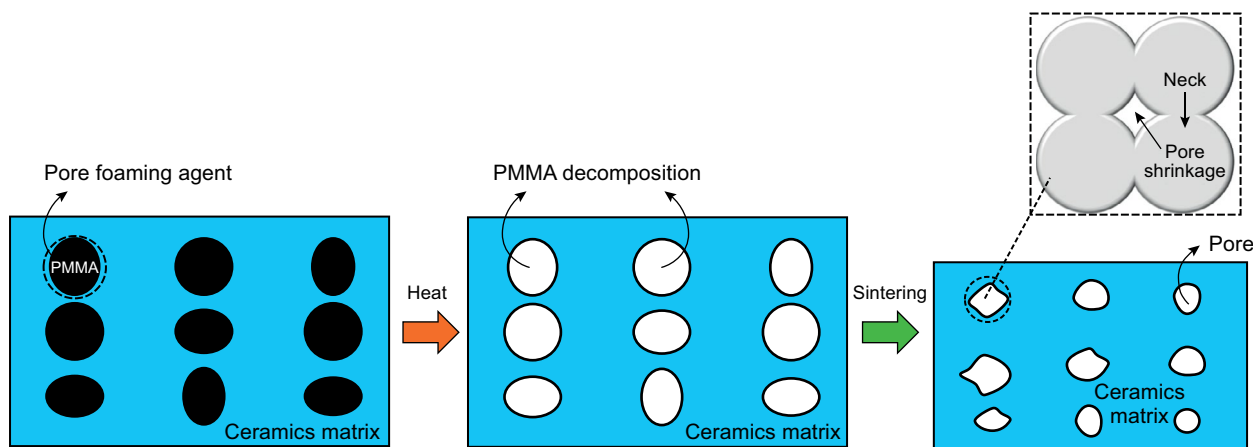


Figure 5. Schematic of porous porcelain fabrication using pore foaming agent.

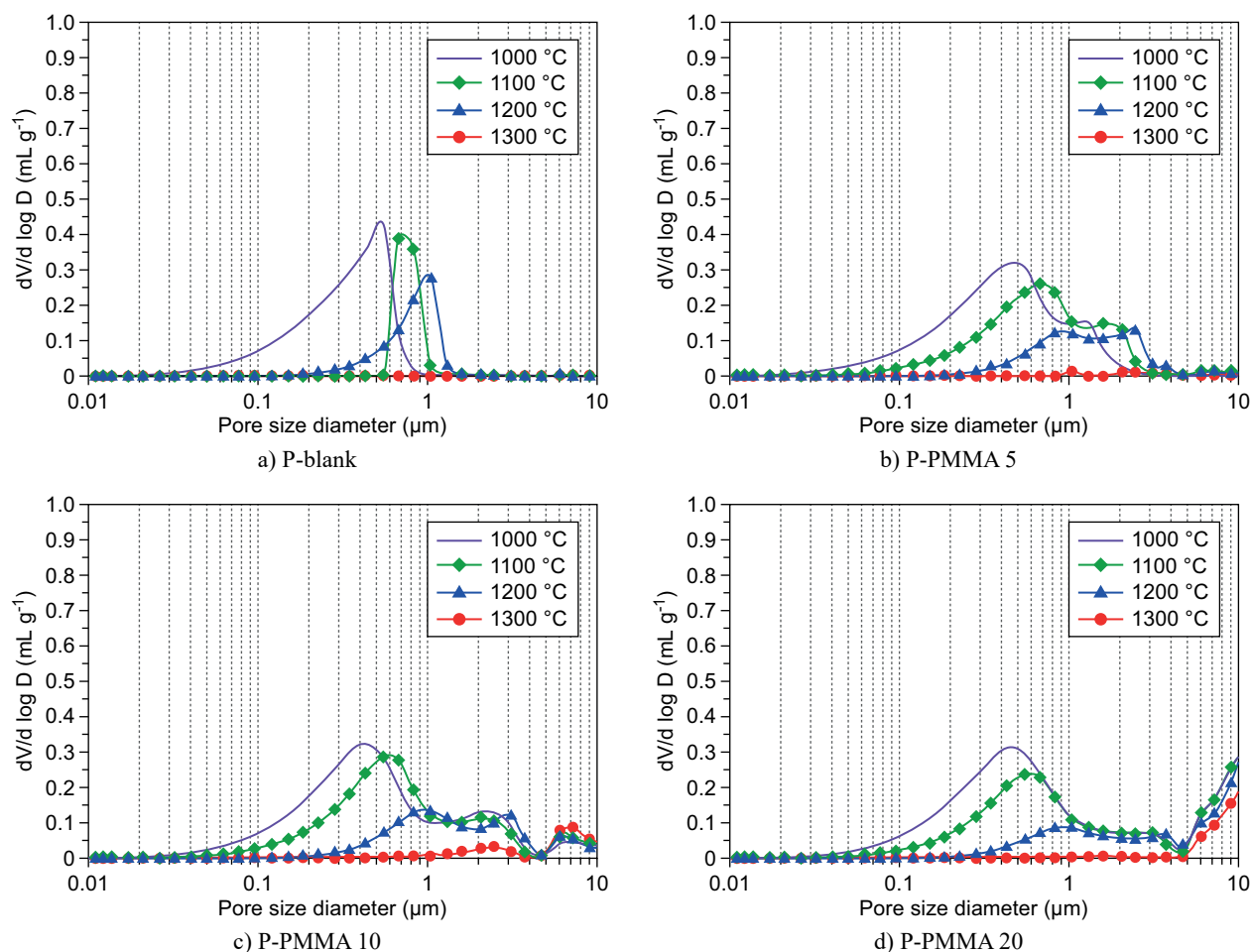


Figure 6. Pore size distribution (Mercury Intrusion Porosimetry) of sintered samples at 1000 to 1300 °C: a) P-blank, b) P-PMMA 5, c) P-PMMA 10 and d) P-PMMA 20.

because inter pore connections were eliminated. In the final stage (above 1200 °C), the liquid phase flow was terminated, and closed gas-filled pore remained in the ceramic bodies [28, 29]. The complete densification of porcelain samples during the three sintering stages was hindered by the porosity generated from PMMA decomposition. This caused the decrease in bulk density and mechanical properties.

From the results, it can be seen that the strength values of porous porcelain samples prepared by adding PMMA 5 - 20 wt. % sintered at 1300 °C is higher than the minimum strength values (35 MPa) of the ISO 13006 standard for ceramic tiles [4, 27]. Thus, it is possible to use this type of porcelain for ceramic tiles in order to enhance the thermal resistance of building materials.

In order to investigate the pore evolution created by pore foaming agent (PMMA), the pore size and its distribution as well as the microstructure are considered. The pore size, pore size distribution and open porosity of sintered porcelain samples prepared with and without PMMA determined by mercury intrusion porosity (MIP) are presented in Table 3 and Figure 6. In Figure 6, the amount of mercury intrusion into the pores of the

Table 3. Pore characterization obtained from mercury intrusion porosimetry of sintered ceramics prepared with PMMA (45 - 250 μm).

Samples	T (°C)	Average pore diameter (μm)	Open porosity (%)
P-Blank	1000	0.25	35.4
	1100	1.00	14.1
	1200	0.84	18.4
	1300	16.08	1.0
P-PMMA 5	1000	0.29	37.7
	1100	0.63	38.2
	1200	1.27	23.1
	1300	5.80	7.4
P-PMMA 10	1000	0.38	44.8
	1100	0.55	37.8
	1200	1.42	25.5
	1300	15.90	31.0
P-PMMA 20	1000	0.59	54.2
	1100	0.89	46.0
	1200	2.98	38.2
	1300	12.70	23.2

porcelain samples reduced with an increase in firing temperature. Consequently, a decrease in the open porosity was obtained. The results of the open porosity determined by mercury intrusion (Table 3) were in good

agreement with the apparent porosity evaluated by the Archimedes method as reported in Figure 2. Gultekin et al. suggested that the reduction of open porosity during the initial and intermediate stage of sintering contributed

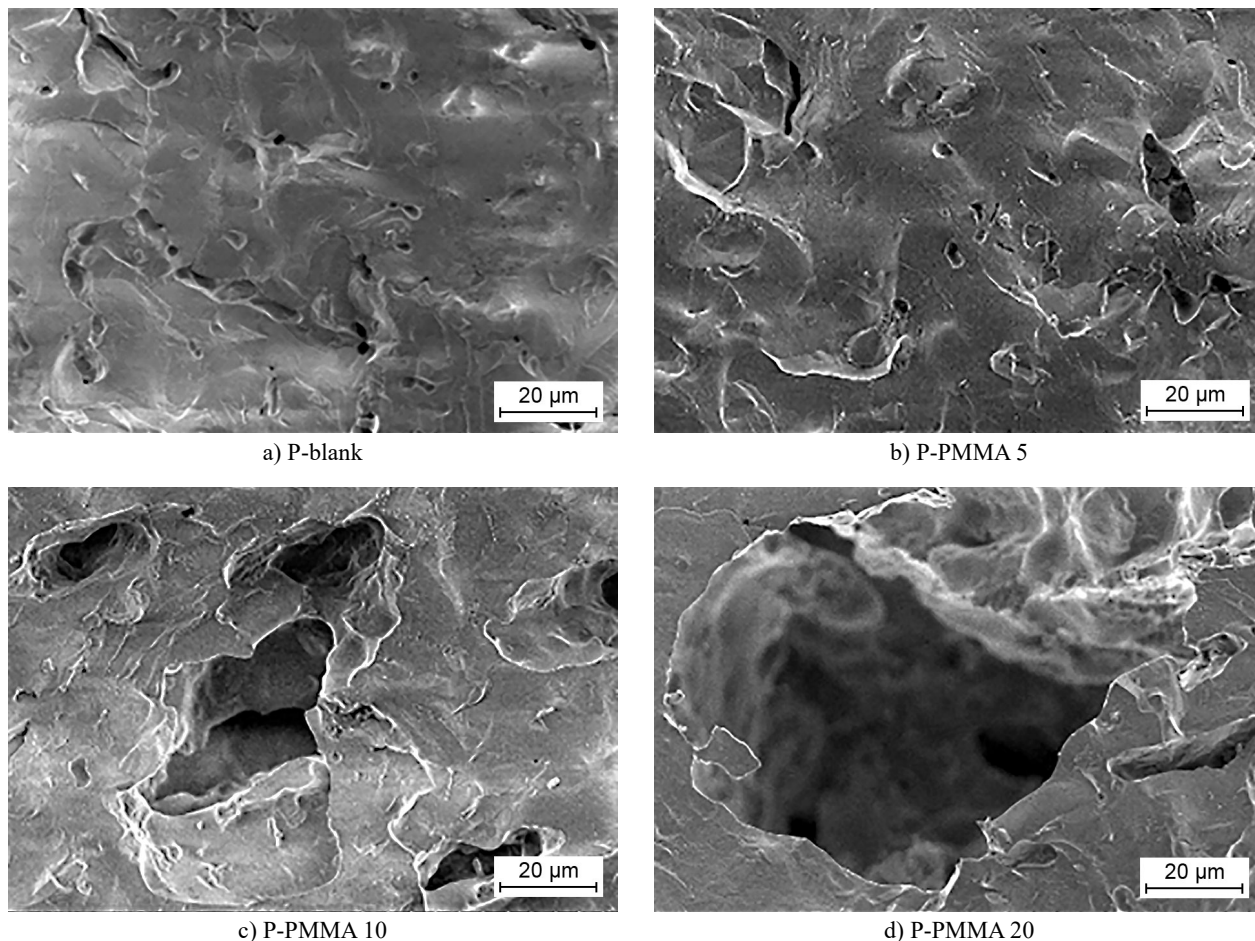


Figure 7. SEM micrographs of porcelain samples (fracture surfaces) after sintering at 1300 °C: a) P-blank, b) P-PMMA 5, c) P-PMMA 10 and d) P-PMMA 20.

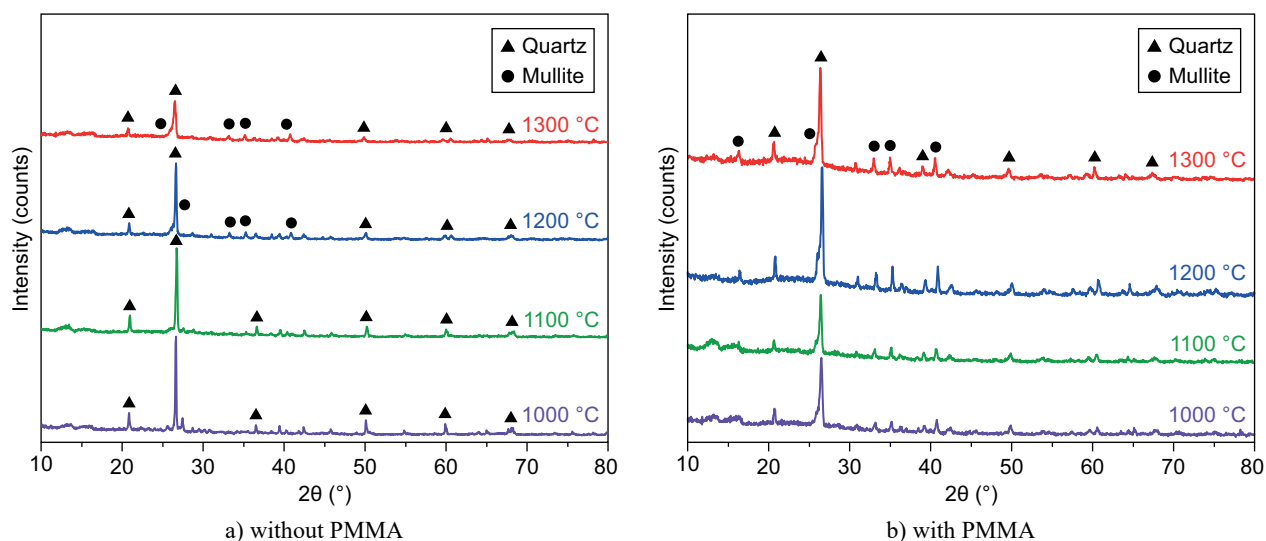


Figure 8. XRD patterns of sintered porcelain samples: a) without PMMA addition (P-blank) sintered at different temperatures, b) with PMMA contents of 0 - 20 wt. % sintered at 1300 °C.

to the elimination of smaller pores, and, consequently, the development of larger pores could have happened due to particle rearrangement [28]. Thus, the larger average pore diameter was observed with the porcelain samples fired at the highest temperature of 1300 °C. Furthermore, the introduction of PMMA led to the growth of the pore diameter. As can be seen in Figure 6, the equivalent pore diameter curves shifts towards higher values when more PMMA was added to the porcelain bodies. In addition, a broadening of the peaks can be observed in the porcelain samples by adding PMMA (P-PMMA). The reason is that mercury intrusion porosimetry probes the pore throats (not the pore cavities) [21] and these are naturally larger when for a higher content of pore forming agent is used.

Figure 7 shows the SEM micrographs of porcelain samples with various porosity levels as a result of the addition of PMMA in the range 0 - 20 wt. % sintered at 1300 °C. A dense structure with a low level of porosity and uniform micro-pore distribution was observed in the sample P-Blank (without PMMA, Figure 7a), while the formation of larger pores started with the addition 5 wt. % and 10 wt. % of PMMA (Figures 7b and c). The largest pores (> 50 µm) can be detected with the addition of 20 wt. % of PMMA.

Figure 8 shows the XRD patterns of the porcelain samples with different PMMA content (0 - 20 wt. %). At a low sintering temperature (1000 - 1100 °C), the only crystalline phase is quartz (JCPDS, number 46-1045), whereas the development of mullite (Mullite, JCPDS No. 73-1389) was found at a higher sintering temperature (1200-1300 °C) as shown in Figure 8a. Indeed, mullite typically occurs at a temperature of around 1200 °C [2]. In addition, the incorporation of pore former (PMMA) did not hinder the formation of mullite at a high temperature. As a result, the porous porcelain samples prepared in this study can maintain the suitable mechanical strength that was higher than the minimum strength values (35 MPa) of the ISO 13006 standard for

porcelain tiles, as demonstrated in Figure 4.

Figure 9 shows the thermal conductivity of porous porcelain as a function of the expected, an increase in the sintering temperature increases the thermal conductivity of the ceramic samples. Moreover, the thermal conductivity significantly decreased with increasing porosity levels. Even at 1300 °C, the reduction of thermal conductivity can be clearly observed. Thermal conductivity decreased from 0.82 W·m⁻¹·K⁻¹ for dense porcelain sample (P-Blank) to 0.72, 0.65 and 0.54 W·m⁻¹·K⁻¹ for the porous porcelain samples made by adding 5, 10 and 20 wt. % PMMA, respectively. A similar behaviour in the reduction of thermal conductivity was observed by Novais et al. [5]. They created the porosity using polymer particles (polypropylene and PMMA) in porcelain specimens and found that the specimens with both pore formers added had a lower thermal conductivity as compared to the original porcelain. In the present work, the addition of 20 wt. % PMMA in porcelain samples sintered at 1300 °C reduced the thermal conductivity by up to 34 % while maintaining acceptable flexural strength values (~ 36 MPa).

It can be concluded that porous porcelain tiles with high mechanical strength and low thermal conductivity can be prepared using PMMA as a performing agent in order to improve the thermal resistance of building materials.

CONCLUSIONS

Porous porcelain tiles were successfully prepared by adding PMMA with a size of 45 - 250 µm in the proportion of 5 - 20 wt. % for improving the thermal resistance. The incorporation of PMMA to generate pore structures in porcelain bodies resulted in a decrease of the density values, while the apparent porosity and water absorption increased with increasing PMMA content. The increase in the porosity into the porcelain bodies promoted the reduction of thermal conductivity while maintaining acceptable mechanical strength (> 35 MPa) according to the ISO 13006 standard. The addition of 5 - 20 wt. % PMMA in porcelain samples sintered at 1300 °C reduced the thermal conductivity by 12 - 34 %, while the flexural strength values are in the range of 36 - 56 MPa. Obviously, the thermal conductivity reduction is tantamount with an increase in the thermal resistance of ceramic building materials, i.e. a reduction of heat transfer from the outside into buildings and vice versa, which may generally contribute to energy saving.

Acknowledgements

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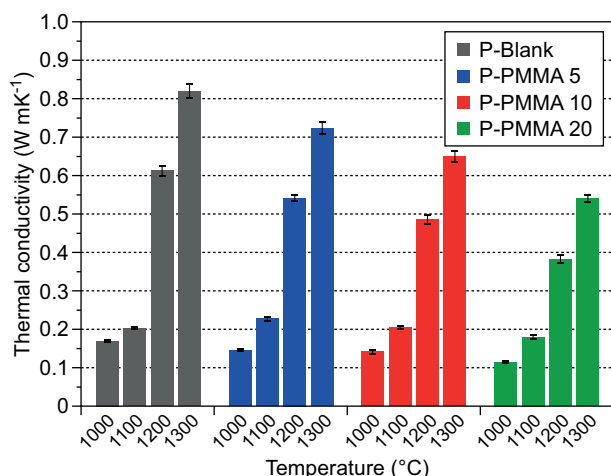


Figure 9. Thermal conductivity of porous ceramic prepared with and without PMMA.

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