

Structure, Microstructure and Properties of Lightweight Aluminosilicate Refractory Aggregates and Castables

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Abstract

Lightweight aggregates and castables are of great significance for energy saving and the reduction of emissions. In this work, lightweight aluminosilicate aggregates were produced using high-quality kaolin and porogenous material by means of high-temperature processing and investigated in terms of XRD mineralogy, bulk density, open porosity, and microstructure. In addition, the influence of the lightweight aggregates on the bulk density, cold crushing strength, microstructure, thermal properties and thermal shock resistance of refractory castables was investigated in detail. For this reason, lightweight castables with different particle size distribution, as per the Dinger and Funk model, were designed to attain very good flow consistency, cast, cured and heat-treated at temperatures between 110–1 200 °C. The lightweight castables with chamotte aggregates presented improved performance, exhibiting bulk density of 1.64 g/cm³ to 1.77 g/cm³, cold crushing strength of 30.3 MPa to 54.8 MPa and thermal conductivity of 0.66 to 0.74 W/m·K measured at room temperature, depending on the firing temperature (850 °C – 1 200 °C) and lightweight aggregates content. The results underscore that the use of microporous aggregates instead of traditional dense aggregates enabled the design of semi-insulating castables for the working lining of kilns and furnaces with improved properties, eliminating the need for porogenic agents.

Keywords: Lightweight aggregate, lightweight castable, monolithic refractories, energy savings, reduced heat loss, university-business cooperation.

1. Introduction

Recently, with the growing demand for the reduction of kiln heat losses, lightweight monolithic working linings, also called semi-insulating monolithic refractories, with both low thermal conductivity and low bulk density have been proposed and developed. These refractories were expected to decrease the kiln heat loss and specific refractory consumption per tonne of products, and to enhance energy efficiency, resulting in reduced energy and fossil fuel consumption. The development of lightweight or semi-insulating castables was followed by aggregates with a high content and optimized pore size, pore diameter distribution and pore structure type ^{1–2}.

Depending on the operating conditions in kilns and furnaces, various types of lightweight aggregates, such as microporous magnesia aggregates ^{3–4}, porous spinel or periclase-spinel aggregates ^{5–7}, microporous or lightweight corundum ^{8–11}, bonite aggregates ^{12–13}, CA₆-MA (C=CaO, A=Al₂O₃, M=MgO) aggregates ¹⁴, lightweight bauxite, ¹⁵ alumina bubbles ^{16–19} and microporous mullite-corundum aggregates ²⁰ can be used in

the production of monolithic linings. The porous components in these refractories have been redesigned to provide improved insulation properties. Applying lightweight aggregates instead of traditional dense aggregate to prepare lightweight refractory castables can significantly reduce thermal conductivity and heat loss into the environment through the furnace lining. Such lightweight aggregates can be effectively obtained mainly by crushing and sieving used insulating refractory firebricks, the *in situ* synthesis decomposition method and calcining of raw materials with a pore-forming additive ^{21 23}. A production process involving mixing the raw clays with porogenous material, extrusion/pelletizing, calcination and sintering, seems to be the most effective in terms of large-scale industrial production.

As is well-known, closed and smaller size pores formed during the *in situ* decomposition synthesis method using kaolin clay and a pore-forming additive are conducive to improving both heat insulation effect and corrosion resistance. Since, lightweight aggregates are mostly used in monolithic refractories, including lightweight castables, a balance between the overall porosity, pore size distribution, physical properties, and corrosion resistance

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should be maintained. Numerous efforts have been made to explain the effect of pore size on the insulating properties of lightweight refractories^{24–25}. Some studies indicate that the thermal conductivity decreases as pore size decreases, with a rapid decline in thermal conductivity occurring when the pore size is less than 10 μm ^{26–27}. To summarize, by incorporating lightweight aggregates with low open porosity and small pore sizes into castables compositions, we can fabricate lightweight wear linings that have low thermal conductivity while maintaining or even enhancing mechanical strength and corrosion resistance. Chamotte is widely used in the monolithic wear lining of kilns due to its excellent thermal stability, good thermal shock resistance, high mechanical resistance and corrosion resistance, especially in contact with alkalis. Compared to the above-mentioned high purity refractory aggregates (e.g. alumina, spinel, magnesite etc.), chamotte is cheaper and, more importantly, exhibits lower thermal conductivity itself. Owing to the differences in the chemical compositions, the sintering mechanism of chamotte is very different from that of high-purity materials, when includes decomposition of aluminosilicate hydrates and transformation to a combination of mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$), cristobalite (SiO_2), and a glassy (amorphous) phase. Here, the primary sintering mechanism could be classified as liquid-phase sintering.

Nevertheless, very limited works are currently available about the effect of temperature on mullite formation and physical properties of lightweight aluminosilicate refractory aggregates. The only study related to this topic in the literature examines the impact of lightweight mullite-silica-rich glass aggregate on the properties of castables, in comparison to a lightweight chamotte aggregate²⁸. Thus, this work fills this gap by fabricating and investigating lightweight aluminosilicate refractory aggregates produced with a firing additive and their influence on the properties of castables designed for refractory working linings. The objective of this work was to design, fabricate and determine the properties of lightweight castables as a novel approach to designing energy-efficient wear refractory linings.

The novel lightweight chamotte refractory aggregates with low bulk density and small pore size were prepared in a high-temperature process. Secondly, the lightweight castables were cast, heat-treated at temperatures between 110 °C and 1 200 °C, and finally characterized in terms of their bulk density, cold crushing strength, microstructure, thermal properties and thermal shock resistance.

II. Experimental

(1) Synthesis and investigation of lightweight aluminosilicate refractory aggregates

The lightweight aluminosilicate refractory aggregates were produced using high-quality kaolin from Jarosław and progenous material in a high-temperature process between 1 150 °C and 1 450 °C. Powdered samples of materials sintered at 1 150, 1 240, 1 350 and 1 450 °C were analysed by means of X-ray diffraction (Panalytical X-ray Diffractometer, $\text{CuK}\alpha$ radiation [$\lambda = 1.5418 \text{ \AA}$], a step size of 0.02°, $2\theta = 10 - 60^\circ$, voltage = 45 kV).

The amount of amorphous phase was determined by admixing an internal standard (100 % crystalline ZnO) in a quantity of 10 wt%. The Rietveld method was used to evaluate the proportion of crystalline and amorphous phases in samples. The Rietveld refinement was done using X'pert High Score Plus software. The bulk density and open porosity of aggregates were measured in accordance with the Archimedes principle using water as the immersion liquid. For further preparation of the castables, aggregates up to 6 mm were produced by crushing and sieving composite material sintered at 1 350 °C.

(2) Preparation of the lightweight castables

Lightweight aluminosilicate aggregates, dense aluminosilicate aggregates, fine particle fractions containing fine alumina, microsilica and calcium aluminate cement and other fine-grained components, additives to induce good flow and setting, and water were used as raw materials to prepare lightweight refractory castables. The Dinger and Funk particle sizing model was employed to design the particle size distribution, using a q value of 0.26 to balance optimal compaction and rheology. **Table 1** presents the details of the formulated lightweight refractory castables with different content of lightweight aluminosilicate aggregates. Two formulations were processed in a Hobart mixer in two sequential steps: (i) homogenization of solid components at a low speed for 4 min and (ii) mixing of solid and liquid components at a high speed for 3 min. Subsequently, prismatic (160 mm × 40 mm × 40 mm) and other cuboid (115 mm × 115 mm × 67 mm) samples were moulded under vibration, and cured at 20–25 °C for 24 h. After this curing step, the samples were demoulded, dried at 110 °C for an additional 24 h, and fired at 850 °C, 1 000 °C and 1 200 °C for 5 h at a heating rate of 3 K/min.

The performance of dried and fired lightweight castables was examined with the following methods: (i) the cold crushing strength (CCS) determined according to the PN EN 993–5 standard, (ii) the bulk density determined based on the Archimedes principle using water as the immersion liquid, (iii) heat transfer properties of lightweight castables were measured at room temperature using a portable system, and (iv) the thermal shock resistance (TSR). Additionally, the reference dense mullite-based castables (Sample R) were prepared for comparison of heat transfer properties.

The TSR of castable beam samples was evaluated with the water-cooling method: the samples were placed in an air furnace at 950 °C for 1 hour; after that, the samples were quickly taken out and cooled with water (below 20 °C) at room temperature for 5 min; this process was repeated five times; after each thermal shock cycle, the residual CCS of samples was tested, and the residual strength rate of CCS of the samples was calculated in accordance with Eq. (1).

$$\text{CCS}_{\text{Re}} = \frac{\text{CCS}_{\text{af}}}{\text{CCS}_{\text{be}}} \times 100 \% \quad (1)$$

where CCS_{Re} is the residual strength rate of CCS in %, CCS_{be} is the CCS value before the thermal shock testing (original material) in MPa, CCS_{af} is the CCS value after each thermal shock cycle in MPa. The original and unmodified procedure is presented elsewhere YB-T-376.1:1995.

The microstructural characteristics of the lightweight refractory castable samples were analysed by means of scanning electron microscopy (SEM, Nova Nano SEM 200 FEI) combined with energy-dispersive X-ray spectroscopy (EDS). Before this analysis, the samples that had been heat treated between 110 and 1200 °C were cut, embedded in resin, ground, finally polished, and coated with a conductive carbon film.

Table 1: Compositions of lightweight refractory castables, concentration of all components in wt%.

Raw material	Index	Concrete code	
		Sample A	Sample B
Lightweight aluminosilicate aggregates	0–5/6 mm	36.0	45.0
Dense aggregates	0–1 mm	16.0	-
Fine particle fractions	<0.01 mm	48.0	55.0
Additives	powders	2.1	2.1
Water	liquid	12.6	16.0

III. Results and Discussion

(1) Mullitization of kaolinite. Phase composition and sintering properties.

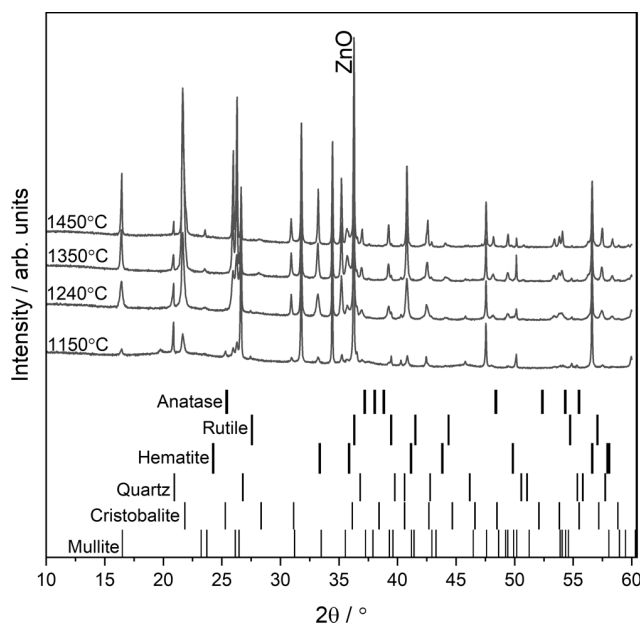


Fig. 1: X-ray diffraction pattern of the mullite formation from natural kaolin from Jarosław. Reference peaks: Cristobalite, SiO_2 (ICDD 01–082–0512); Quartz, SiO_2 (ICDD 01–085–0794); Hematite, Fe_2O_3 (ICDD 01–073–0603); Rutile, TiO_2 (ICDD 01–072–1148); Anatase, TiO_2 (ICSD 98–017–2914); Mullite, $\text{Si}_{1.46}\text{Al}_{4.54}\text{O}_{9.73}$ (ICSD 98–006–6450); ZnO as an internal standard.

All heat-treated kaolin samples were submitted to XRD and the results are shown in Fig. 1, where it can be seen

that the most intense peaks belong to zinc oxide, ZnO (ICSD Card No. 98–005–7450), used as an internal standard. Phase identification was carried out according to the ICSD and ICDD databases. The 100 % peak of ZnO was identified at position 2θ of 36.266°. The X-ray diffraction analysis spectra (Fig. 1) revealed the evolution of the crystalline structure of the samples. Especially, the effect of the sintering temperature on the mullitization behaviour of the kaolinite from Jarosław is obvious. The XRD pattern of the sample fired at 1150 °C shows a strong and amorphous halo due to the presence of a non-crystalline material. Nevertheless, at this temperature, the very low-intensity peaks correspond to the initial formation of mullite (the germination of the mullite). The mullite was identified according to ICSD Card No. 98–006–6450 and the 100 % peak position located at 26.297°. The intensity of the mullite peaks is maximized for sintering temperatures between 1350 °C and 1450 °C. Two secondary phases, quartz and cristobalite, were identified in the XRD pattern of the sample heat-treated at 1150 °C, as well as three admixture phases, anatase, rutile and hematite. Between 1350 °C and 1450 °C, hematite appeared as an admixture phase, only.

Fig. 2 presents the refinement results obtained with the Rietveld method. The R_{wp} values for all fittings were below 5 %, indicating a good fit with the observed XRD spectra. The sample heat-treated at 1150 °C almost consisted of amorphous phases, with an identified amorphous content of 63.78 %. Only 9.22 % mullite, 15.33 % cristobalite, 8.34 % quartz, 2.78 % anatase, 0.33 % rutile and 0.22 % hematite were present. In the case of the sample heat-treated at 1240 °C, five crystal phases – mullite, cristobalite, quartz, rutile and hematite – were identified, with determined contents of 46.67 %, 10.89 %, 8.44 %, 0.33 % and 0.89 %, respectively. The remaining 32.78 % was composed of amorphous phases. The concentration of amorphous phase, cristobalite and quartz varies, whereas the amount of mullite (> 44 %) remains almost constant between 1240 °C and 1450 °C (Fig. 2).

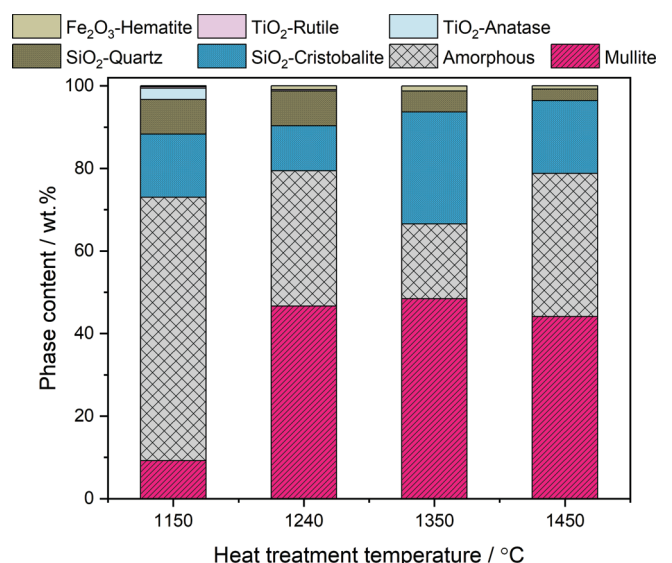


Fig. 2: Quantitative phase-analysis by the Rietveld method using X-ray powder-diffraction data.

Fig. 3 shows the bulk density and open porosity of lightweight aluminosilicate refractory aggregates prepared from natural kaolin from Jarosów versus temperature. This figure shows the general trend of bulk density increase and open porosity decrease versus increasing temperature. At a low sintering temperature of 1 150 °C, the sample exhibits the highest open porosity and lowest bulk density. When the sintering temperatures reaches 1 450 °C, the bulk density increases and the open porosity decreases. Since, no significant differences in bulk density, open porosity and mullite content were found, when the sintering temperature was varied from 1 350 °C to 1 450 °C, the sintering temperature of 1 350 °C appears to be adequate from a technological perspective.

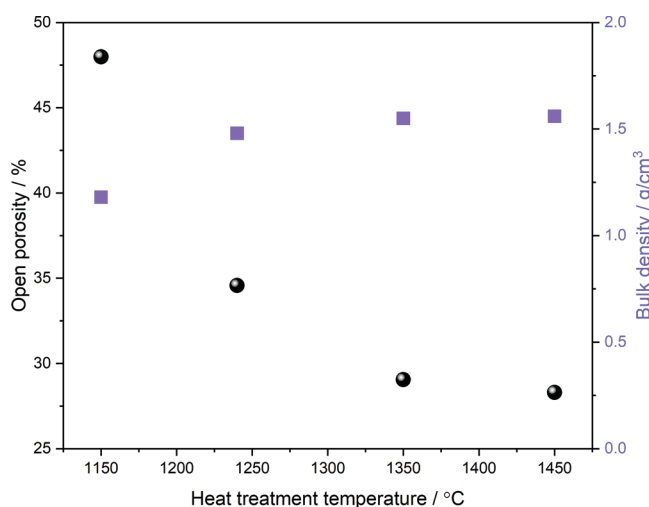


Fig. 3: Physical properties of lightweight aluminosilicate refractory aggregates prepared from natural kaolin from Jarosów versus temperature.

(2) Castables

(a) Physical properties

The physical properties and mechanical properties of the lightweight aluminosilicate castables are presented in Fig. 4 and Fig. 5, respectively. As expected, Sample B specimen showed both lower bulk density and cold crushing strength owing to the higher addition of lightweight porous aggregates, in contrast to Sample A. Interestingly, after heat treatment at temperature of 850 °C, the bulk density of all the specimens decreased sharply and their strength also decreased owing to the thermal decomposition of a castable matrix. After sintering, the castables containing 36 wt% lightweight aggregates had the highest strength, and their strength reached 47.0 MPa and 54.8 MPa after treatment at 1 000 °C and 1 200 °C, respectively, which was due to the *in-situ* chemical reactions. As also expected, the castables containing 45 wt% lightweight aggregates had lower strength, and their strength reached 43.2 MPa and 51.7 MPa after treatment at 1 000 °C and 1 200 °C, respectively. It was found that there is no significant difference in the bulk density of the two castables produced at temperatures 850 °C, 1 000 °C and 1 200 °C, but a slight increase was found after heat treatment at 1 000 °C.

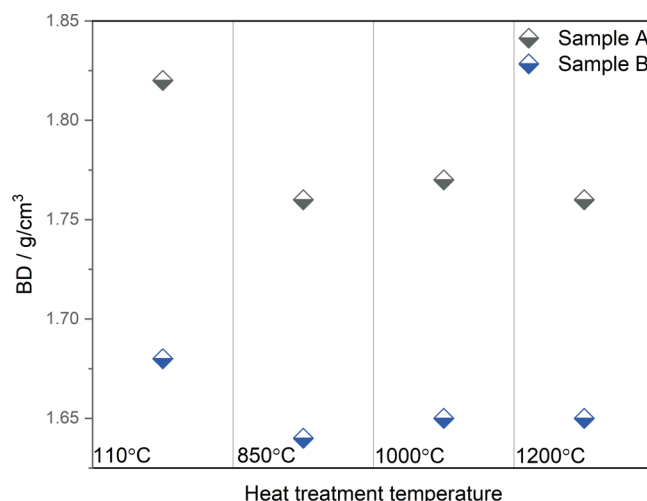


Fig. 4: Bulk density (BD) of lightweight castables heat-treated between 110 °C and 1 200 °C.

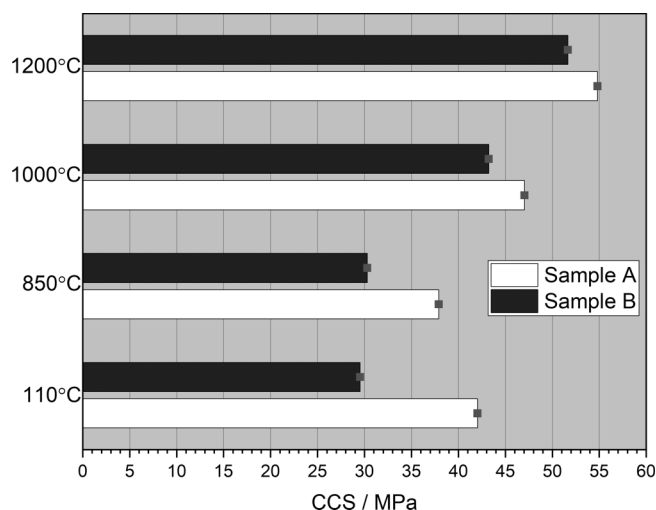


Fig. 5: Cold crushing strength of lightweight castables heat-treated between 110 °C and 1 200 °C.

(b) Microstructure

The effect of the heat-treatment temperature on microstructure evolution was examined using SEM. Fig. 6a – d shows an overview of the microstructure of the castables obtained at 200x magnification for the samples dried at 110 °C, and fired at 850 °C, 1 000 °C and 1 200 °C, respectively. As shown in the micrograph, both dense and lightweight aggregates were bonded by a continuous and relatively dense matrix at 110 °C. As expected, the obvious difference lay in the porosity of dense and lightweight aggregates. As revealed from these micrographs, the lightweight grains contain many intragranular pores, whereas dense aggregates have relatively low porosity. Due to the high temperature of 850–1 200 °C, the matrix of the samples appeared more porous, maintaining good contact and interaction between both the lightweight and dense aggregates. This increase in porosity is directly connected with removal of free water, dehydration of hydrates and reactive sintering of fine-grained components.

Detailed observations of the matrix could be identified at higher magnification. **Fig. 7a – d** presents the microstructure details of these samples recorded at magnification of 1000x. The lightweight aggregates contain mainly closed pores of 1–20 microns in diameter. Chemically, the porous grains primarily consist of Si, Al, and O, as shown in the EDS spectrum (**Fig. 7e**, point 1 in **Fig. 7a**). Significant changes can be observed in the microstructure in the matrix after firing at 1200 °C (**Fig. 6d** and **Fig. 7d**), where the boundaries between aggregates and matrix are

difficult to distinguish, accentuating the distinctive porous structures achieved. Therefore, it can be believed that the larger contact area between the porous aggregates and the matrix of the castables may contribute to the formation of sintering necks at elevated temperatures, which facilitates the sintering process and ultimately enhances the strength of these castables. Moreover, these microstructural characteristics contributed to achieving a lower thermal conductivity value for the lightweight castables compared to the dense ones.

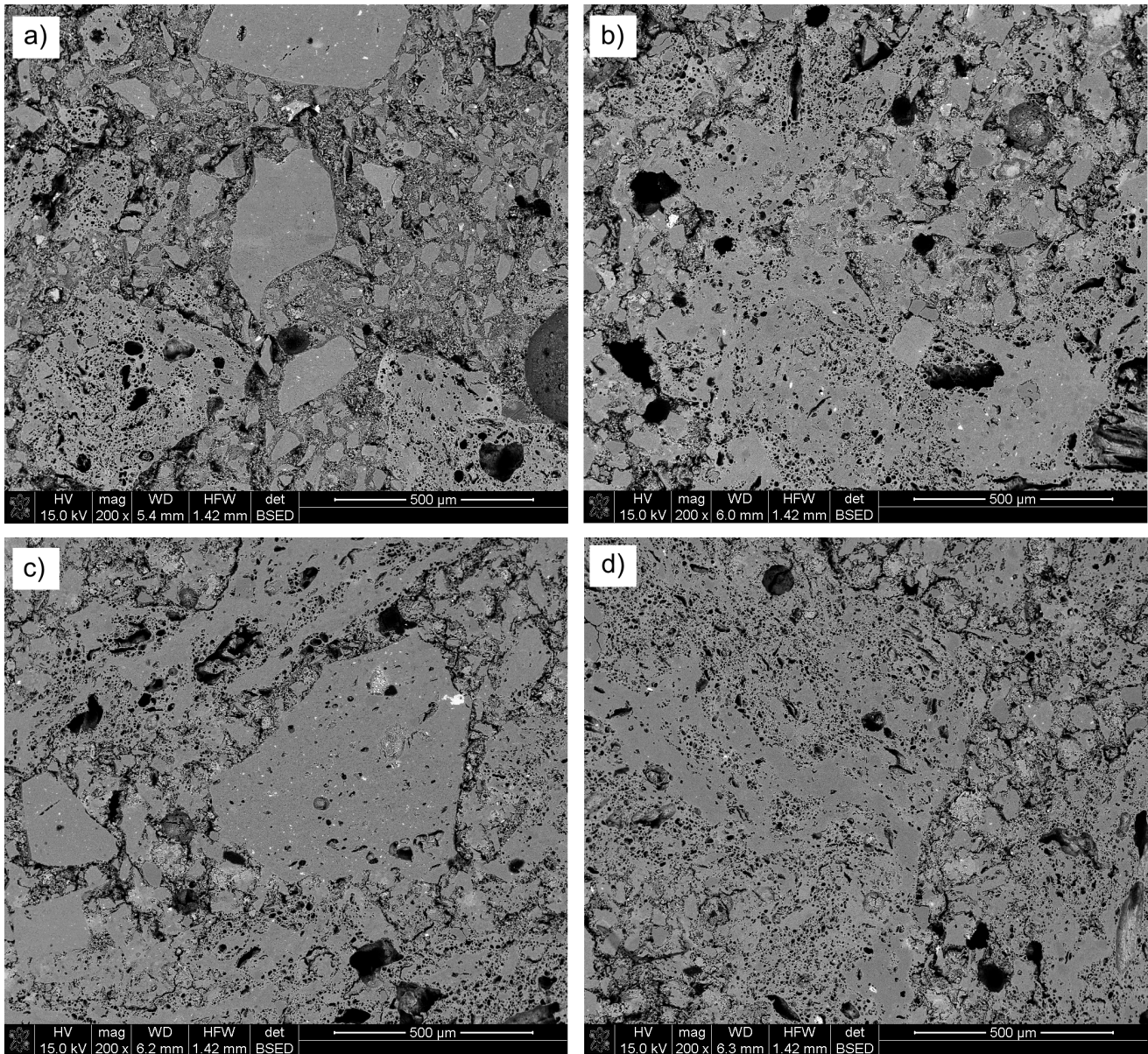


Fig. 6: Backscattered electron detector (BSED) images from SEM of the Sample A after drying at 110 °C and heat-treatment at 850, 1000 and 1200 °C (a-d). Overview of microstructure obtained at 200x magnification showing the characteristic microstructure of sample with three regions: both lightweight porous aggregates and dense aggregates randomly distributed and surrounded by regions of matrix with fine particles.

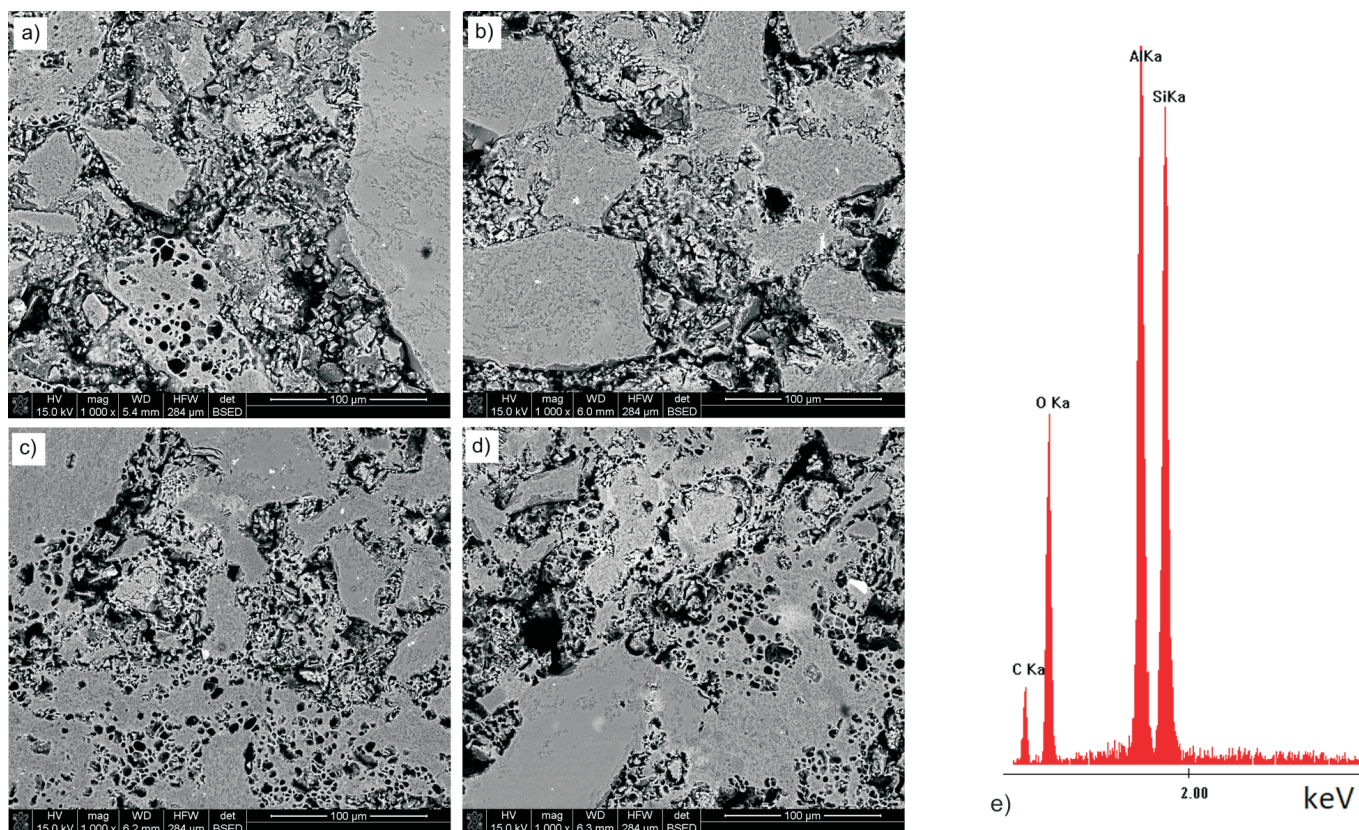


Fig. 7: The microstructure details of castables (Sample A) after drying at 110 °C and heat-treatment at 850, 1000 and 1200 °C (a-d) recorded at magnification of 1000x showing three regions: both lightweight porous aluminosilicate aggregates and dense aluminosilicate aggregates randomly distributed and surrounded by regions of matrix with fine particles of calcium aluminate cement residues, alumina and microsilica powders and other fine-grained components. EDS spectrum of lightweight porous aggregates taken from microstructure of castable dried at 110 °C (e).

(c) Results for the thermal properties

As can be seen in Fig. 8 and Fig. 4, a strong correlation exists between the thermal properties of castables containing as-synthesized aggregates both dense and porous (fine and coarse) and their bulk density (Samples A and B). Moreover, the thermal conductivity of dense castables, designed as the Sample R is inversely correlated with higher density ($BD = 2.52 \text{ g/cm}^3$ for the sample heat-treated at 850 °C).

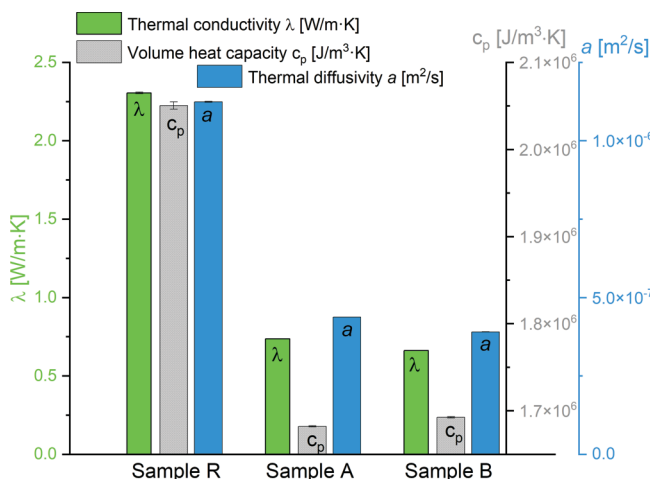


Fig. 8. Thermal properties of castables fired at 850 °C measured at room temperature.

(d) Results for the thermal shock resistance

The thermal shock resistance of lightweight castables heat-treated at 1000 °C is shown in Fig. 9. This figure shows the CCS before and after thermal shock together with the residual strength ratio of samples. It can be clearly seen that the refractories showed lower CCS after the thermal shock test.

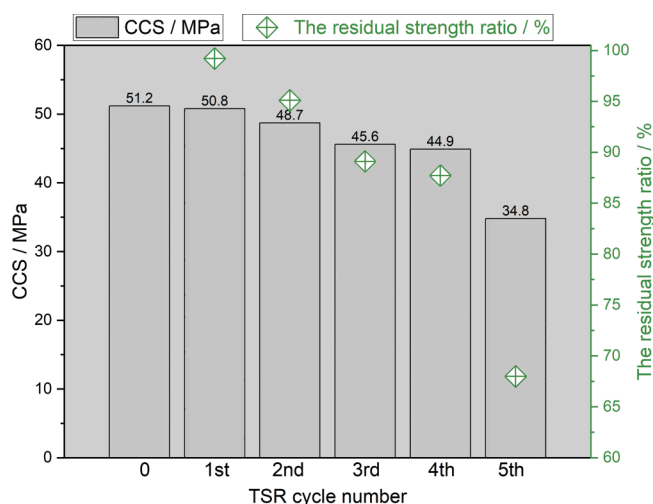


Fig. 9. The CCS of lightweight castables (Sample A) heat-treated at 1000 °C specimens before and after thermal shocks (950 °C-water) and their residual strength ratio. .

Moreover, it can be seen that the residual strength ratios of the samples constantly decreased with the TSR cycle number. After four cycles of thermal shock testing, the sample possessed CCS above 44 MPa. Nevertheless, the CCS was significantly reduced after five thermal shock cycles. Therein, the residual strength ratio of castables sample reached 68.0 %. During the cooling process of the castables in water, the low surface temperature caused the sample to shrink inward. However, the high temperature inside the sample prevented surface shrinkage. As a result, the castables' surface is subjected to tensile stress while the interior experienced compressive stress, leading to a decrease in the castables' performance due to thermal stress. These thermal stresses cause the formation of some cracks on the sample surface (Fig. 10). Nevertheless, the good thermal shock resistance of castables with lightweight aggregates can be primarily attributed to the amorphous phase content, which helps absorb thermal shock stress.



Fig. 10 . Lightweight castables specimen after two thermal shock cycles.

IV. Conclusions

The effects of the addition of lightweight chamotte aggregates on the phase compositions, microstructure, mechanical properties and thermal shock resistance of castables were investigated. The following conclusions were drawn:

- This study evaluated the thermo-physical-mechanical properties of semi-insulating refractory castables composed of lightweight chamotte aggregates with many closed pores of a size below 20 μm .
- The incorporation of lightweight aggregates did not noticeably alter the phase composition of aluminosilicate refractories. However, their rough surface and increased porosity significantly improved the bonding interface between the aggregates and the matrix.
- The results revealed that the content of the chamotte lightweight aggregates and heat-treatment temperature strongly affect the bulk density, cold crushing strength, microstructure, and thermal properties of refractory castables. As expected, with increasing content of lightweight aggregates, the bulk density, cold crushing strength and thermal conductivity decreased slightly.
- The lightweight castables designed here presented a remarkable performance, manifesting an optimal combination of CCS (30.3 MPa to 54.8 MPa), low bulk density (1.64 g/cm³ to 1.77 g/cm³), low thermal conductivity at room temperature (0.66 to 0.74 W/m·K) and good thermal shock resistance.

- These properties depend also on both the firing temperature (850 °C – 1 200 °C) of the castables and the content of lightweight aggregates.
- These attributes accentuate the prospective application and utility of the studied castables in a monolithic refractory working lining.

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