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Microwave and conventional treatment of low-cement high-alumina castables with different water to cement ratios; Part I. Drying

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ABSTRACT

The aim of this study was to determine the effect of the microwave and conventional drying method on the strength, porosity and composition of low-cement alumina castables with various water to cement ratios (wcr). High-alumina low-cement castable samples were prepared with different w/c ratios: 0.64, 0.75, 0.82 and 1.13. Changes in wcr were effected through volumetric replacement of cement with 0–0,045-mm tabular alumina having a comparable particle size. Water content in all the composition was constant (4,5%). After curing, the samples were dried conventionally in a laboratory electric drier or in a laboratory microwave drier. After drying open porosity and modulus of rupture were determined. The pore size distribution, pore median and tortuosity of the samples were measured by the mercury porosimetry method. Phase composition was determined using X-ray diffraction. The Rietveld method was used for quantitative analysis. It was found that at low wcr (0.62) the main hydrate formed in the castable was C_3AH_6, which caused a release of a smaller amount of water during the drying process, mainly pore water, resulting in lower open porosity and lower pore size than in the castables with a high wcr (1.13). At a low wcr, the strenght of castable was higher due to a higher amount of hydrates, low porosity and small pore size. On the other hand, at a high wcr, the strength of castable was lower owing to a higher amount of water released in the drying process, which led to loosening the structure of castable. With an increased water-to-cement ratio the degree of CA_2 hydration decreased. The temperature rise due to cement hydration probably influenced the kinetics of this process.

1. Introduction

In the ceramic industry, microwaves are mainly used for the drying and heating of ceramic blanks. In contrast to conventional drying, during which the heat is supplied from outside through the surface of the material, in the process of microwave the microwaves penetrate into the material and heat its entire volume. Dielectric polarization is responsible for microwave heating. It is caused by the polarization of chemical compound molecules by an electric field [1]. As a result of water dipoles friction in a rapidly changing electromagnetic field, heat is produced in the material's entire volume. The amount of heat generated in the material depends on its dielectric losses $\varepsilon_r \tan \delta$ (where ε_r – dielectric constant, $\tan \delta$ – dielectric loss factor) [2]. Temperature distribution in the samples heated by microwaves is different than in the samples heated conventionally.

The use of microwave drying has many advantages. This kind of drying treatment is characterized by a very high heating rate, which results from the elimination of thermal resistance occurring during heat transport from the source to the material in the process of convection heating. Furthermore, in conventional drying the highest temperature is outside the dried material, which leads to faster drying of the outer layers and hampers transport of moisture from the central areas. In microwave drying, the material is simultaneously heated in its entire volume, which allows accelerating the drying process.

Other advantages of microwave heating include: elimination of external heat sources, reducing of scrap and low specific energy consumption.

Several studies have been performed on the microwave drying of castables [3–8]. The results revealed that microwave drying of monolithic refractory castables is possible in a short period of time and without steam explosion risk.

Refractory castables are subjected to a constant development process, aimed at reducing the demand for water and cement. The reduction of water demand is possible by optimizing the particle size distribution and development of high-performance dispersing agents [9–11]. This way the water demand was reduced from over 14% to approximately 4.4%.

The water-to-cement ratio was practically neglected in

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Table 1
Chemical composition of raw materials (according to suppliers).

Material	Chemical composition [%]				
	Al_2O_3	SiO_2	CaO	Na_2O	Fe_2O_3
Tabular alumina T−60	99.5	≤ 0.09	–	≤ 0.40	Fe magnetic ≤ 0.02
Calcined alumina CTC20	99.7	0.03	0.03	0.12	0.03
Reactive alumina RG4000	99.8	0.03	0.03	0.08	0.02
Alumina cement Secar 712	68.7–70.5	0.2–0.6	28.5–30.5	Na_2O + K_2O < 0.5	0.1–0.3

investigations into the reduction of water and cement in modern refractory castables. Recently, the first systematic study on the setting of refractory castables with different water-to-cement ratios at a fixed water content has been presented [12].

The aim of this study was to determine the effect of methods of drying, microwave and conventional, on strength, porosity and composition of low-cement alumina castables with various water-to-cement ratios (wcr).

2. Materials and methods

To prepare the castable samples, tabular alumina T-60 with various grain size, calcined alumina CTC20, reactive alumina RG4000 and alumina cement Secar 712 with chemical compositions given in Table 1 were used. The mineralogical composition of alumina cement consisted of 56–61% CA and 39–44% CA_2.

Samples were prepared with different water/cement ratios of 0.64, 0.75, 0.82 and 1.13. Changes in wcr were effected through volumetric replacement of cement with 0–0,045-mm tabular alumina having a comparable particle size (Table 2). Water content in all the compositions was constant (4,5%).

Samples were prepared by mixing raw materials with an Eirich PV 02 mixer and applying high speed to obtain good dispersion, homogeneity and flow of the samples. Dry mixing was carried out at tool velocity 450 rpm and plate velocity 32 rpm for 1 min and, next, wet mixing was conducted at tool velocity 450 rpm and plate velocity 64 rpm for 4 min. The samples were cast in the shape of bars – format D (40 × 40 × 160 mm) according to EN ISO 1927-6 standard and cured in a climatic chamber at 20 °C and 95% of relative humidity for 48 h.

After curing, the samples were divided into two groups, the samples in one group were dried conventionally in a laboratory electric drier at 110 °C for 24 h and in the other – in a PG 99 laboratory microwave drier. The microwave drier was equipped with two microwave generators, each having the power of 700 W. Dried bars were placed on a scales pan connected with the scale, so that weight loss in the drying process could be measured. The temperature of the dried samples' surface was measured with a pyrometer installed in the doors of the drier. The drying process was carried out with microwave power gradually increased from 30 W to 120 W and the holding time of 10 h at 120 W.

Table 2
Composition of castables with different water-to-cement ratios.

Component	Composition [%]			
	wcr 0,64	wcr 0,75	wcr 0,82	wcr 1,13
Tabular alumina T60 0,02–3 mm	69	69	69	69
Tabular alumina T60 0–0045 mm	**6.6**	**7.8**	**8.4**	**10.2**
Alumina CTC20	10	10	10	10
Reactive alumina RG4000	7	7	7	7
Cement Secar 712	**7**	**6**	**5,5**	**4**
Dispersant FS60	0.15	0.15	0.15	0.15
Deionised water	4.5	4.5	4.5	4.5

After drying open porosity and modulus of rupture were determined according to EN ISO 1927-6 standard. The pore size distribution, pore median and tortuosity of the samples were measured by mercury porosimetry method using AutoPore IV 9500 device. Phase composition was determined using X-ray diffraction (PANalytical X'PERT PRO MPD) and Cu Kα radiation (45 kV, 35 mA). The Rietveld method was used for quantitative analysis.

3. Results

Progression of castables' microwave drying is presented in Fig. 1.

The course of the samples' temperature rise was very similar, regardless of the water-to-cement ratio, whereas in the case of their weight loss a considerable wcr impact was observed. The correlation between the weight loss and the wcr was linear. The decreasing cement content led to an increased weight loss (Fig. 2).

The weight loss in the drying process increased as the water-to-cement ratio rose and the cement content decreased, i.e. the amount of water remaining in the castable in the form of hydrates was reduced (Fig. 3).

On the basis of weight loss values, the amount of water remaining in the samples and wcr in the dried castables were calculated (Table 3).

The effectiveness of cement hydration after setting, curing and microwave drying, calculated as water-to-cement ratio in the dried castables, increased from 0.51 to 0.67 with a decrease in the cement contents. This trend is consistent with the results obtained in investigations into the setting and hardening the castables with different water-to-cement ratios [12]. The pore water concentrations in the tested green samples after 24 h were slightly (0.1%) higher than in the ones after drying, presented above in Table 3.

The average values of modulus of rupture were similar for most of the samples with the same wcr regardless of the drying method used. The downward trend of bending strength, particularly above 0.75 wcr was observed (Fig. 4).

In Fig. 5 an increase of open porosity with an increased wcr is presented. At wcr equal to 0.64 the measured porosity was almost identical for the samples dried by the microwave and conventional methods. As the cement content decreased, the castables dried by the microwave method demonstrated a lower porosity and the differences become more visible at the highest wcr.

The porosimetric investigations revealed that in all the castables subjected to testing the diameters of most pores were below 0.25 μm (Figs. 6 and 7). At a low w/c ratio, pores with a diameter below 0.05 μm were dominant and, as w/c ratio increased, the dominant pores had larger diameters. As a result, the median pore diameter of castables increased with an increase in wcr (Tables 4 and 5). These dependences were linear with high correlation coefficients (Fig. 8) and almost identical regardless of the drying method. The pores' tortuosity decreased with an increase in the w/c ratio and open porosity

Quantitative X-ray phase analysis allowed comparing the fraction of hydrated and non-hydrated components of cement in the dried castables (Table 6, Fig. 9). The other phases present in the castables were corundum and β-Al_2O_3 ($NaAl_{11}O_{17}$), the components of sintered tabular alumina used for the castables preparation. The fractions of the β-Al_2O_3 were between 3 − 4%, depending on the amount of tabular alumina added to the castables. Quantitative analysis was performed using Rietveld refinement. The standard deviations given in Table 6 are related to the methodology of calculations and refer to a single measurement.

All the castables subjected to testing contained CA and CA_2 phases, which proves that full hydration of the cement components was not

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