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Sintering of Whiteware Body Depending on Different Fluxing Agents and Binders

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Additional information is available at the end of the chapter

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Abstract

The sintering of whiteware (porcelain) body can be affected by using fluxing agents or binders. The chapter describes the sintering process of porcelain body in case of different fluxing agent (different feldspar rocks, bone ash, zeolite) and binder (kaolin vs. calcium aluminate cement) utilization in the porcelain raw material mixture. Sintering process is presented according to thermodilatometrical curves and sintering temperatures especially.

Keywords: whitewares, feldspar rocks, zeolite, bone ash, kaolin, calcium aluminate cement, sintering temperature, water absorption, mineralogical composition

1. Introduction

Whiteware is a traditional ceramic material used to make pottery and porcelain. Traditional raw material mixture for whiteware (porcelain) production covers kaolin or/and kaolin clay, quartz and feldspar rock at a composition about 50:25:25 wt.%. Typical properties of porcelain body are low porosity (below 0.3%), high mechanical strength (bending strength over 40 MPa, Young Modulus over 60 GPa), firing temperature about 1300°C and high whiteness and translucency [1–3].

Feldspar rocks are used in the fine ceramic industry as a fluxing agent to form a glassy phase for accelerating of sintering process. Feldspar rocks are a mixture of pure feldspars, quartz and mica especially from the mineralogical point of view. Pure feldspars are divided into potassium feldspars (orthoclase, microcline), sodium feldspars (albite) and calcium feldspars (anorthite). Solid solutions between K-feldspar and albite are called alkali feldspars, and solid solutions between albite and anorthite are plagioclase feldspars.

The plagioclase series follows according to percentage of anorthite in parentheses [4]. Feldspar rocks are usually used as a source of alkali oxides (Na_2O , K_2O) and alumina (Al_2O_3) for the preparation of glazes [5]. Suitable choice of feldspar rock can significantly affect the properties of the ceramic body [6], firing temperature and soaking time [10]. The densification of green body, cleanability and the stain resistance of polished sintered ceramic tiles is influenced by particle size distribution of used feldspar rocks [7]. Feldspar rocks may be successfully replaced by LCD waste glass [8]. Wollastonite is very suitable material for acceleration of sintering process in porcelain body. Only 1 wt.% addition of wollastonite is able to decrease firing temperature (about 25°C) in the mixture with kaolin, quartz and potassium feldspar rock [9].

Bone ash is fluxing agent for artistic porcelain especially known as bone china. The amount of bone ash in the raw material mixture of bone china is about 50% [11]. Bone ash (cattle bones calcined at around 1000°C) consists predominantly of hydroxyapatite. The reactions of bone ash in porcelain body were studied in detail in Refs. [12, 13]. Bone ash—fluxing agent for bone porcelains (bone china)—is usually produced by the calcination of bovine bones at the temperature of 1100°C . The melting point of bone ash is about 1670°C [14]. The mineralogical composition of bone ash consists of tricalcium phosphate in the form of hydroxyapatite $\text{Ca}_5(\text{OH})(\text{PO}_4)_3$.

Very useful fluxing agent for sintered ceramic body production is zeolite, which is able to accelerate the sintering process very intensively. Zeolite is a natural mineral with exceptional physical properties that follow from its specific crystal structure. The latter consists of a 3D lattice of silicate tetrahedrons $(\text{SiO}_4)^{4-}$ mutually connected by oxygen atoms, with part of silicon atoms replaced by aluminum atoms $(\text{AlO}_4)^{5-}$. Zeolite has a wide range of applications in agriculture, breeding, civil engineering, protection of environment, wastewater purification, and in various industrial sectors. In civil engineering, it began to be used as a partial replacement of cement in the production of concrete [15–19]. Different Italian low-cost natural zeolitic rocks as a substitute of feldspar rocks in porcelain raw materials mixture were investigated. Zeolitic rocks increased the slip viscosity during wet grinding with a coarser grain size distribution. The technological properties (strength, porosity, resistance etc.) of zeolite-based porcelain bodies are similar to current traditional porcelain bodies made in the system kaolin—feldspar rock—quartz [20]. The aim of the study [21] was to investigate the effect of natural zeolite addition on the sintering kinetics. Clinoptilolite, which is a type of natural zeolite, was added partially or fully in replacement of quartz at selected electro-porcelain composition. It was found that the sintering activation energy decreased with increasing zeolite addition. Replacement of quartz with zeolite decreases activation energy for the start of sintering process in electro-porcelain body—firing temperature (about 50 – 100°C) and soaking time were reduced. In the study [22], the effect of natural zeolite addition on the electrical properties of porcelain bodies was investigated. The resistivity of samples increased at 50°C temperature after zeolite addition, while it was decreasing after zeolite addition at higher temperatures. The resistivity of samples depends on sintering temperature. Low-cost naturally occurring mixtures of feldspar and zeolite occurring in epiclastic rocks were promising substitutes for conventional quartz-feldspathic fluxes in ceramic bodies. Different epiclastic outcrops, with a different zeolite-to-feldspar ratio, were tested in porcelain stoneware

bodies. The addition of an epiclastic rock (20 wt.%) brought significant advantages (better grind ability, lower firing temperature with improved mechanical strength and lower porosity) and disadvantages (increasing of slip viscosity, worse powder compressibility, higher firing shrinkage, and a darker color of the body due to high amounts of Fe_2O_3) [23, 24].

Anorthite type of whiteware body on the basis of raw materials mixture of feldspar rock, quartz and calcium aluminate cement (CAC) was developed at the firing temperature of 1300°C . Calcium aluminate cement (substitution of traditional kaolin or quartz) increases the strength of green body (**Figure 1**) and lowers the density due to formation of anorthite in all the fired bodies. An optimal ratio between quartz and feldspar rock for optimal sintering of the body was found (**Figure 2**) [25].

Whiteware body based on anorthite was developed from the mixture of ball clay, alumina, quartz, wollastonite and magnesia mixture. Sintered whiteware body (1220°C) has approximately two times higher modulus of rupture (110 MPa) than traditional porcelain body based on mullite due to lower content of glassy phase (only 30% for anorthitic whiteware body) [26]. Deflocculation of raw materials mixture based on calcium aluminate cement for the production of whiteware body with low porosity is necessary [27]. Carboxylic acids [28], polyethylene glycol, polyacrylate derivatives and aqueous solutions of sodium carboxylate [29] for optimalization of rheological properties of aluminous cement pastes were tested.

Direct sintering is very effective method how to decrease the energy consumption during the firing of porcelain. Direct sintering reduced total processing time by ~50% and also lowered the sintering temperature from 1200 to 1175°C [30].

For the description of sintering process, the thermodilatometrical analysis and sintering temperature are used primarily. Sintering temperature is defined as temperature when the fired body has water absorption exactly 2%.

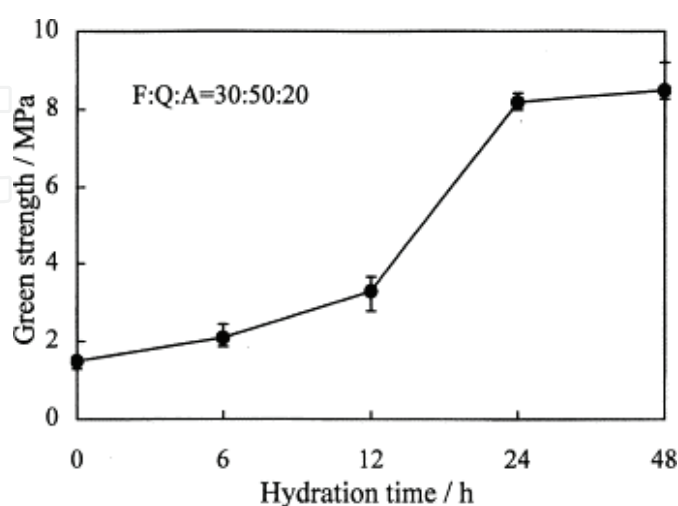


Figure 1. Variation of flexural strength of green body with hydration time. F, feldspar; Q, quartz; A, calcium aluminate cement (CAC) [25].

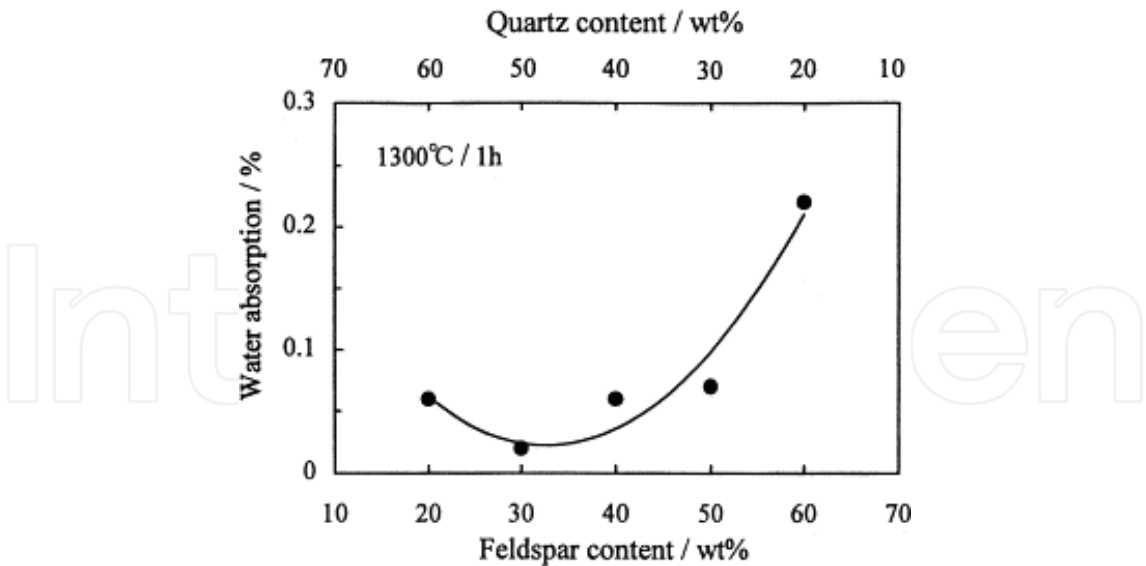


Figure 2. Water adsorption with various compositions in fired bodies with 20% of CAC [25].

2. Sintering of whiteware body depending on fluxing agent (feldspar rocks, bone ash, zeolite)

Sintering and melting of feldspar rocks depend on many aspects, such as the fineness of milling (granulometry), the rate of heating and finally the content of alkali oxides, because it directly creates the melting effect. Very useful is to compare the sintering activity of different typical feldspar rocks with different content of pure K-feldspar, Na-feldspar and Ca-feldspar using for the industrial production of whitewares. The comparison is performed for pure feldspar rocks and for mixtures of feldspar rocks with kaolin. For the comparison, next feldspar rocks were used:

- Sodium-potassium feldspar rock F-KNa with mineralogical composition: K-feldspar (microcline) 20.0%, Na-feldspar (albite) 22.6%, Ca-feldspar (anorthite) 2.4% and quartz 55.0%.
- Potassium feldspar rock F-K with mineralogical composition: K-feldspar (microcline) 57.2%, Na-feldspar (albite) 16.0%, Ca-feldspar (anorthite) 1.5%, quartz 21.3% and mica (muscovite) 4.0%.
- Sodium-calcium feldspar rock F-NaCa with mineralogical composition: Na-feldspar (albite) 60.3%, Ca-feldspar (anorthite) 21.0%, quartz 13.8% and mica (muscovite) 4.9%.

The chemical composition of compared feldspar rocks (**Table 1**) reflects their mineralogical composition and volume of different types of pure feldspars (microcline, albite, anorthite). Granulometry of industrially milled feldspar rocks (the equivalent mean spherical diameter of particles $d(0.5)$ in **Table 1**) is very similar and does not affect the presented results.

Sintering activity of dry pressed test samples based on tested pure feldspar rocks (**Table 1**) was determined according to dependence of water absorption (EN ISO 10545) on the firing

	F-KNa	F-K	F-NaCa	Zeolite
SiO ₂	79.76	70.96	66.67	68.20
Al ₂ O ₃	12.37	16.10	20.11	12.40
Fe ₂ O ₃	0.42	0.10	0.26	1.40
TiO ₂	0.05	0.04	0.04	–
CaO	0.48	0.30	4.23	3.30
MgO	0.10	0.06	0.07	1.00
K ₂ O	3.35	10.36	0.83	2.80
Na ₂ O	2.67	1.90	7.13	1.00
LOI	0.80	0.20	0.74	–
<i>d</i> (0.5) [μm]	20.8	18.4	16.6	20.0

Table 1. Chemical composition of used feldspar rocks and zeolite in weight% (LOI = loss of ignition) and the equivalent mean spherical diameter *d*(0.5).

temperature (**Figure 3**). The most intensive sintering activity of the pure feldspar rock body shows potassium-sodium feldspar rock F-KNa—dry pressed test samples have the lowest water absorption, the highest bulk density and modulus of rupture in all firing temperatures in the range of firing at temperatures 1120–1210°C. Sodium-calcium feldspar rock F-NaCa begins sintering at much higher firing temperatures. Sintering temperature (**Figure 3**) of tested alkali feldspar rocks F-KNa and F-K is significantly lower than oligoclase type of feldspar rock F-NaCa.

The mixtures of feldspar rocks with kaolin (40 wt.%)—samples FK-KNa, FK-K, FK-NaCa—totally change (increase) the sintering temperatures (**Table 2**) of alkali feldspar rocks F-K and F-KNa. The most intensive fluxing agent in case of pure feldspar rock body (F-KNa) exhibits the lowest sintering activity in the mixture with kaolin with the highest sintering temperature. This fact is confirmed according to therm dilatometrical curves (**Figure 4**). Conversely, the mixture with kaolin decreases the sintering temperature of oligoclase F-NaCa with the highest content of pure feldspars. The sintering temperature of F-NaCa mixture with kaolin is lower (about 20°C) than pure feldspar rock F-NaCa.

The difference between the sintering of pure feldspar rocks (F-KNa, F-K, F-NaCa) and mixtures of feldspar rocks with kaolin (FK-KNa, FK-K, FK-NaCa) is evident from the therm dilatometrical curves (**Figure 4**). The highest content of quartz and muscovite in feldspar rock F-KNa caused high expansion of the body during firing in the range of 200–900°C in comparison with other tested samples based on feldspar rocks F-NaCa and F-K. Dry pressed body based on pure feldspar rock F-KNa shows the best sinterability of all compared feldspar rocks with maximal firing shrinkage (about 5%—**Figure 4**). Very significant is quartz transformation at the temperature 573°C on cooling part of therm dilatometrical curves (**Figure 4**) depending on quartz content (**Table 1**) in individual tested feldspar rocks. The quartz transformation is most visible for F-KNa feldspar rock with maximal (55%) content of quartz.

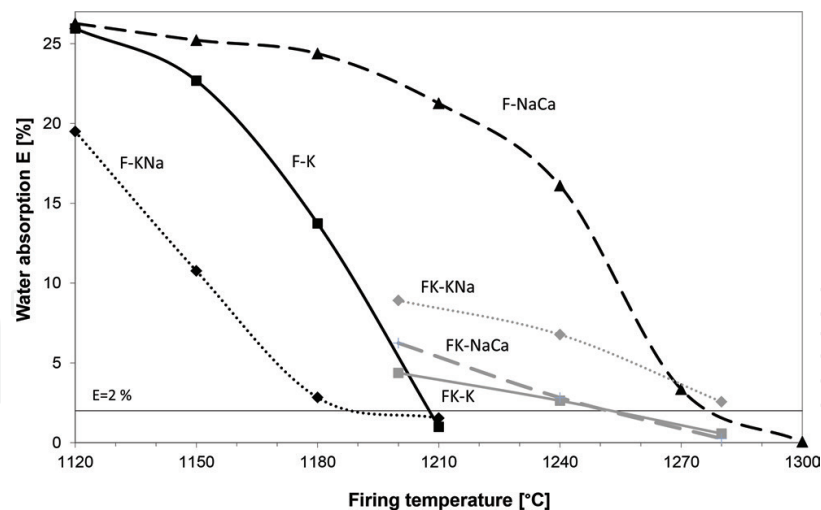


Figure 3. Water absorption E depending on the firing temperature. Determination of sintering temperature ($E = 2\%$).

Mixture	Sintering temperature (°C)	Mixture	Sintering temperature (°C)
F-K	1205	FK-K	1250 (+50)
F-NaCa	1275	FK-NaCa	1255 (−20)
F-KNa	1190	FK-KNa	1285 (+95)
		FK-B	1200

Table 2. Sintering temperatures of tested samples based on different feldspar rocks and mixtures of kaolin (60%) with feldspar rocks (40%) or bone ash (FK-B).

The feldspar rock F-KNa with the lowest sintering temperature based on microcline and albite is typical by the quickest disappearing of feldspars during the sintering. Sintering temperature (1190°C) means the existence of only quartz and amorphous glassy phase without any feldspars (**Figure 5**). Quartz, amorphous glassy phase, and microcline are represented in the body F-K after the firing at sintering temperature (1205°C). It is not possible to find an explanation of this fact in granulometry parameters of used feldspar rocks, which influence sintering and melting of feldspars very much, but in the equilibrium phase diagrams (**Figure 6**). Mixed sodium-potassium feldspar rock generated low melting eutectic melts, which accelerate the sintering and melting process of feldspars. It is surprising that leucite generating during the potassium feldspars melting according to theoretical assumptions [4] is not detected even in sintered body F-KNa or sintered body F-K, both based on the potassium feldspar microcline. After the firing of F-NaCa sample at sintering temperature (1275°C), the body contains anorthite (calcium feldspar) with high theoretical melting temperature of about 1550°C [4] and albite (**Figure 5**).

The more intensive fluxing agent than feldspar rocks for the sintering process in the system kaolin-fluxing agent is bone ash (**Figure 4**)—the mixture containing bone ash FK-B (**Table 2**) shows sintering temperature 1200°C. That is about 50°C lower compared with the most intensive feldspar rock-based mixture (FK-K) with potassium feldspar rock F-K containing 75% of pure microcline (**Table 2**). After the exceeding, the temperature 1200°C is visible intensive

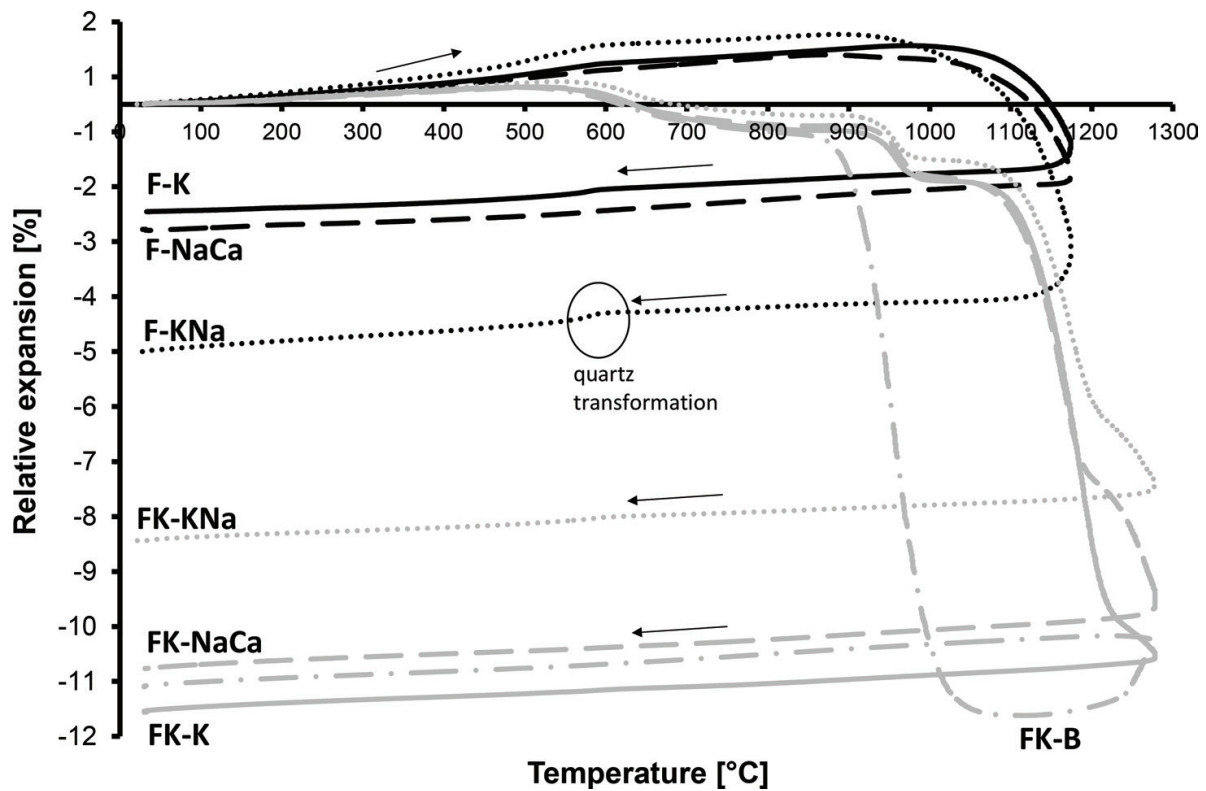


Figure 4. Thermodilatometric curves of pure feldspar rocks (F-K, F-KNa, F-NaCa) and the mixtures of feldspar rocks with kaolin (FK-K, FK-KNa, FK-NaCa) (10°C/min without soaking time on the maximal temperature).

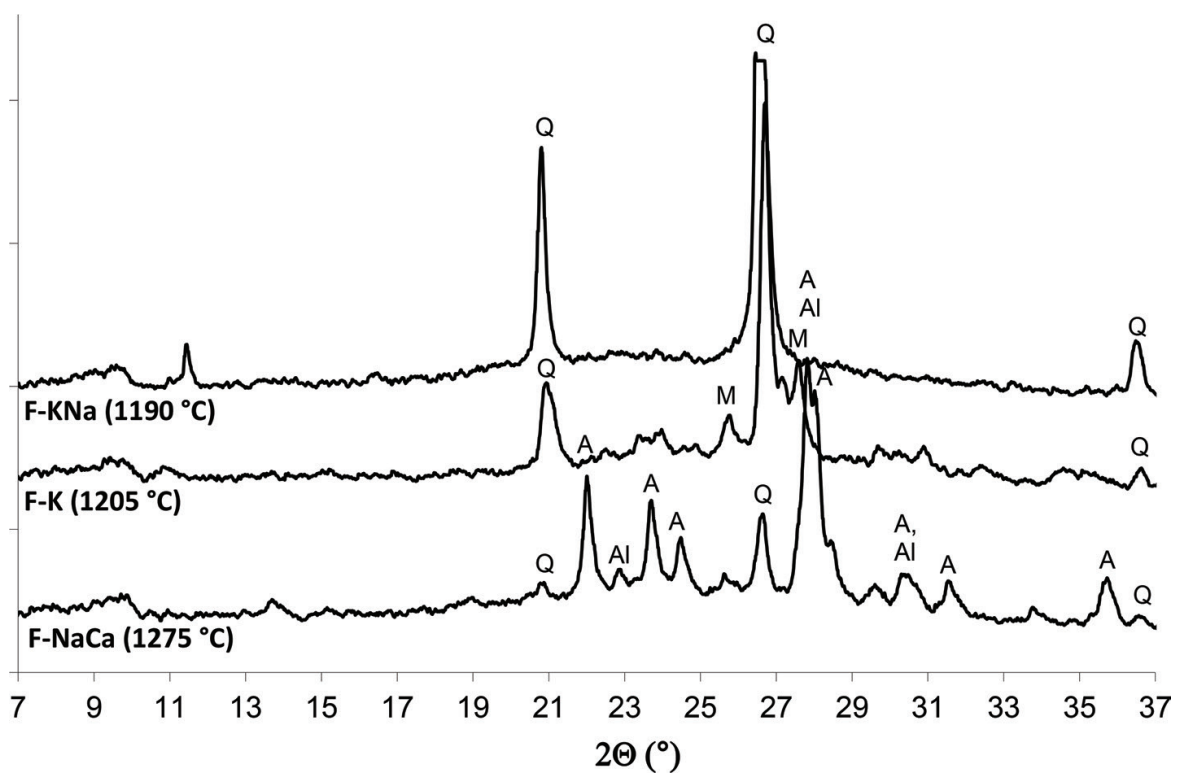


Figure 5. XRD patterns of sintered feldspar rocks at sintering temperature: M, microcline; Al, albite; Q, quartz; A, anorthite.

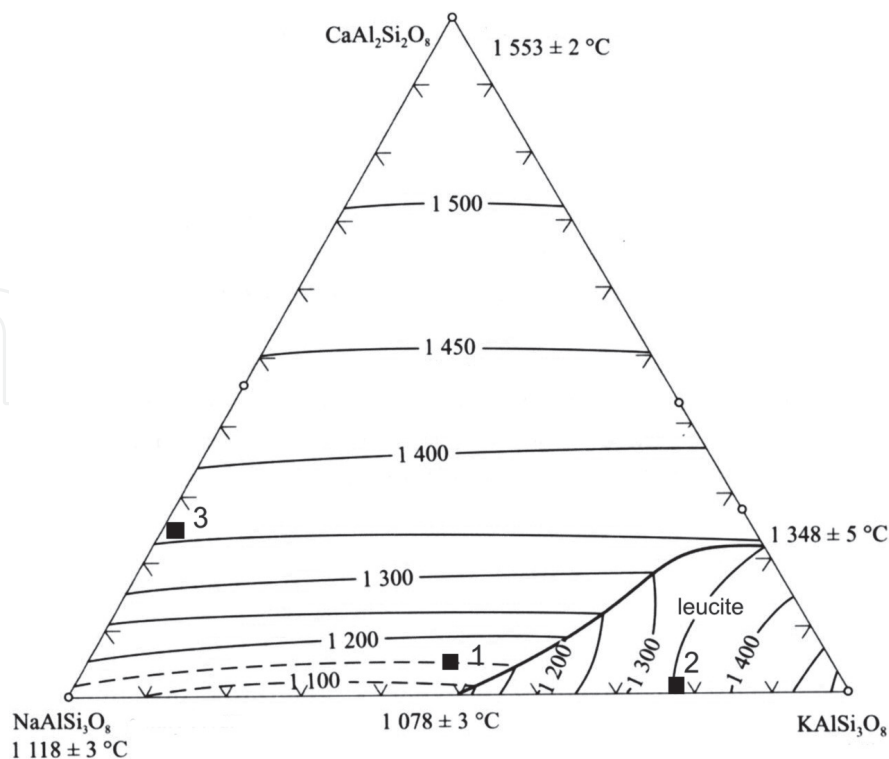
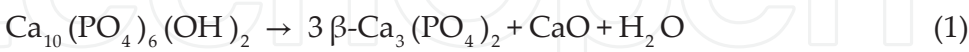


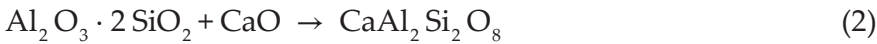
Figure 6. Phase diagram $\text{NaAlSi}_3\text{O}_8 - \text{KAlSi}_3\text{O}_8 - \text{CaAl}_2\text{Si}_2\text{O}_8$ and theoretical melting temperature of used feldspar rocks (1: F-KNa, 2: F-K, 3: F-NaCa).

bloating of the bone ash bodies, which is typical by creating of secondary porosity and increasing in water absorption (**Figure 4**).

Different mineralogical composition between feldspar rocks and bone ash-based porcelain sintered bodies is possible to document according to XRD analyses. Traditional porcelain with high content of feldspar rocks in the raw materials mixture contains mullite and quartz as main mineralogical phases. Mineralogical composition of porcelain body based on bone ash is totally different—typical is high content of β -tricalcium phosphate and anorthite. Bone ash in bone porcelain bodies decomposes into β -tricalcium phosphate $\text{Ca}_3(\text{PO}_4)_2$, lime CaO and water at around 775°C according to Eq. (1) [14]:



Lime reacts with metakaolin from clay relicts to form of anorthite $[\text{CaAl}_2\text{Si}_2\text{O}_8]$ according to Eq. (2) [14]:



Eutectic composition in the ternary system of bone china ($\text{Ca}_3(\text{PO}_4)_2 - \text{CaAl}_2\text{Si}_2\text{O}_8 - \text{SiO}_2$) is about 11% tricalcium phosphate, 51% anorthite and 38% silica with a melting temperature of $1290 \pm 5^\circ\text{C}$ [14].

Zeolite rock was investigated as a fluxing agent for sintered ceramic body and its effect in the sintering process. The thermodilatometric heating and cooling curves dL/L_0 of two different

samples according to fluxing agent utilization (zeolite vs. feldspar rock F-KNa) are shown in **Figure 7**. During the firing, there is evident (**Figure 7**) that zeolite (in mixture Z) is more intensive fluxing agent compared to feldspar rock F-KNa (mixture F) for the creation of sintered body with low porosity. High firing shrinkage is typical for the sintering—the raw materials mixture not C, but Z with zeolite content starts intensive shrinking from temperature of about 900°C. Compared mixture F based on traditional ceramic fluxin agent - potassium feldspar rock F-K - starts the sintering process at a higher temperature (about 1100 °C).

The quartz transformation at 573°C (change of the fired body volume) is visible on cooling part of thermodilatometric curve of the mixture F (**Figure 7**) due to high portion of quartz in the mixture F based on the mixture kaolin-quartz-feldspar. This phenomenon is not presented on the cooling curve of the sintered body Z based on zeolite—the raw material mixture not contains quartz, which is advantageous for lower relative expansion (coefficient of linear thermal expansion) of sintered body Z (**Figure 7**).

Sintering temperature of tested samples based on zeolite is 1180°C, which is about 100°C lower than for the sample based on standard flux feldspar rock F-K (mixture F). From the picture (**Figure 7**), there is evident different coefficient of linear thermal expansion α (in the temperature range of 30–500°C) of both compared sintered bodies (Z vs. F):

- feldspar + quartz body: $\alpha_{30-500^{\circ}\text{C}}(\text{F}) = 70 \times 10^{-7} \text{ K}^{-1}$
- zeolite body: $\alpha_{30-500^{\circ}\text{C}}(\text{Z}) = 48 \times 10^{-7} \text{ K}^{-1}$

The sintered body based on zeolite shows the lower coefficient of thermal expansion α compared with feldspar sample due to the formation of anorthite in the sample Z and absence of quartz.

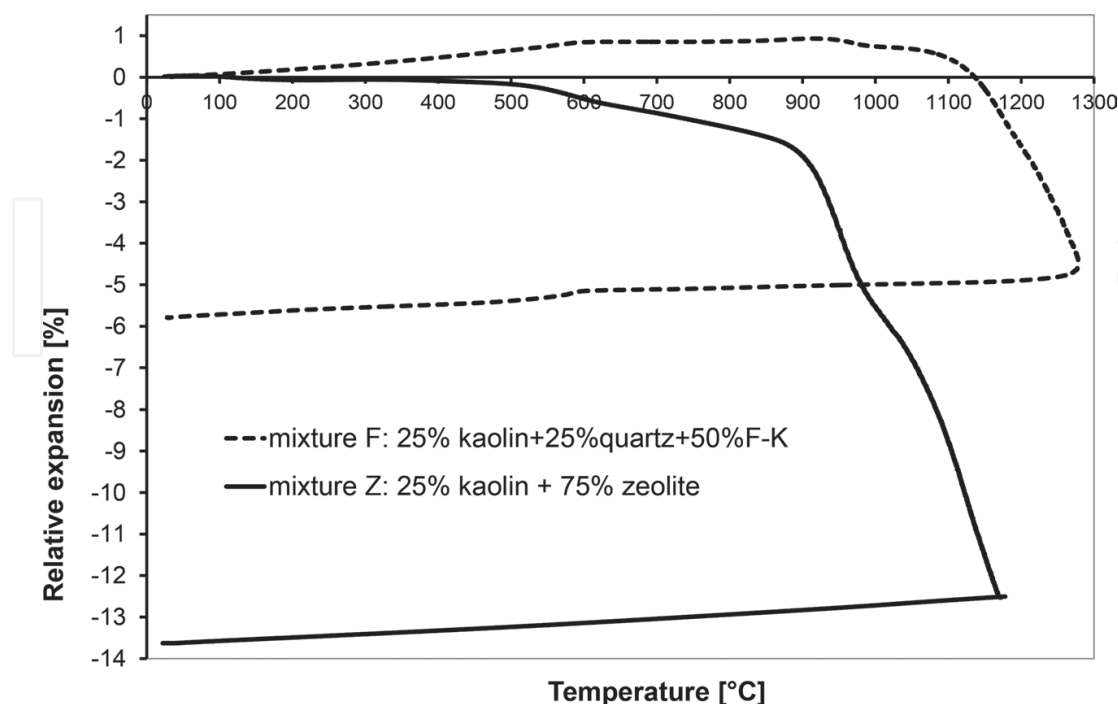


Figure 7. Thermodilatometric analysis of compared samples with different fluxing agent (zeolite vs. feldspar rock F-KNa) during the firing (5°C/min without soaking time).

Important technical property of anorthite is its low coefficient of linear thermal expansion of $48.2 \times 10^{-6} \text{ K}^{-1}$ [31] (mullite $60 \times 10^{-7} \text{ K}^{-1}$ [32]). The mineralogical composition of both bodies after firing in both cases is characterized by the existence of mullite and glass phase. The sintered body (fired at 1200°C —mixture Z or 1300°C —mixture F, respectively) based on feldspar and quartz (mixture F) also contains quartz, and the body made from zeolite contains anorthite and cristobalite.

Sintered body based on zeolite (mixture Z in **Figure 8**) as a fluxing agent not creates white body, which is typical for sintered body of the mixture F based on F-KNa feldspar rock (**Figure 8**). This situation corresponds to chemical composition of natural zeolite with higher content of Fe_2O_3 (**Table 1**).



Figure 8. Color of sintered bodies with water absorption below 2%.

3. The effect of calcium aluminate cement as a binder for the sintering of whiteware bodies

The sintering process of whiteware (porcelain) body is affected by the used binder—we can use traditional plastic material (kaolin) or calcium aluminate cement (CAC) according to latest research [25, 33]. Comparison of the properties of both types (**Table 3**) of porcelains made by pressing from dry granulate is documented.

The difference in sintering process of two whiteware bodies with different binder kaolin vs. CAC (**Table 3**) is documented according to thermogravimetric curves (**Figure 9**). The sintering activity of both compared mixtures is very different when the firing temperature exceeds 1200°C —the system based on CAC (mixture CAC) is more able to sinter—we can observe higher firing shrinkage.

Significant decrease of the coefficient of linear thermal expansion in the temperature range $30\text{--}500^\circ\text{C}$ is evident (**Figure 9**) when calcium aluminate cement CAC is used as binder compared with kaolin based body. The explanation of this fact we can find in the formation of anorthite in the CAC-based sample (**Figure 10**). The fired body based on kaolin also contains mullite and quartz as a main mineralogical phases. Anorthite exhibits lower coefficient of linear thermal expansion of $48.2 \times 10^{-6} \text{ K}^{-1}$ [31] than mullite $60 \times 10^{-6} \text{ K}^{-1}$ [32].

Mixture	Content (%-mass)
K	25% kaolin + 50% F-KNa + 25% quartz sand + 0.35% sodium hexametaphosphate (deflocculant)
CAC	25% CAC + 50% F-KNa + 25% quartz sand + 0.35% sodium hexametaphosphate (deflocculant)

Table 3. Composition of raw material mixtures (test samples).

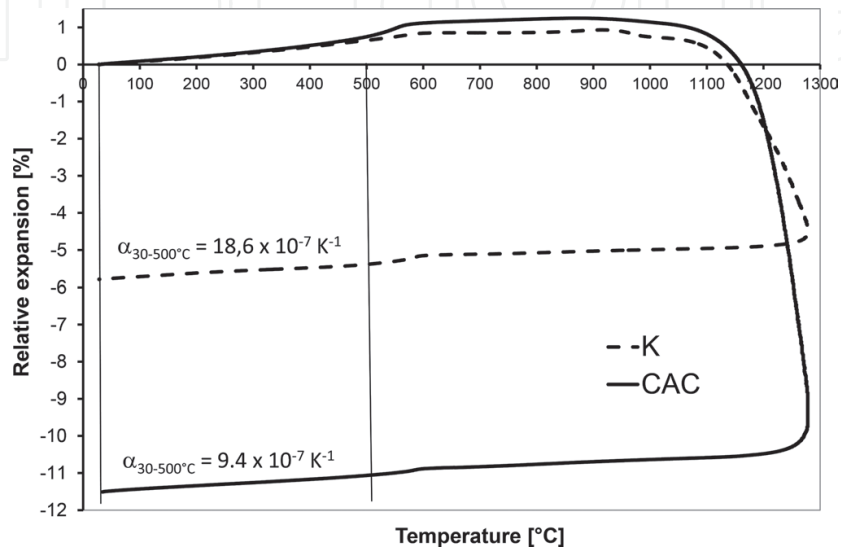


Figure 9. Thermodilatometric analysis of kaolin (K) and calcium aluminate cement (CAC)-based bodies during the firing (1280°C, 3°C/min without soaking time). Determination of the coefficient of linear thermal expansion in the range of temperatures 30–500°C.

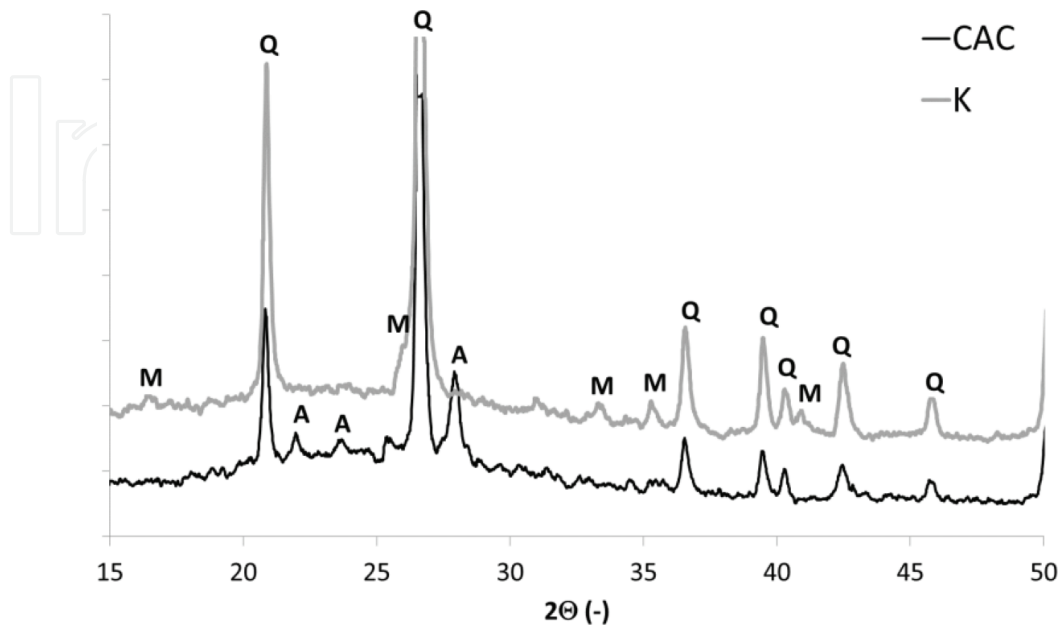


Figure 10. XRD of fired bodies based on different binder kaolin or CAC (M, mullite; Q, quartz; A, anorthite).

Sample	Firing temperature (°C)					
	1250			1280		
	Ev (%)	MOR (MPa)	B (kg m ⁻³)	Ev (%)	MOR (MPa)	B (kg m ⁻³)
K	6.4	29.4	2180	1.9	38.5	2280
CAC	1.3	58.9	2360	Melting of test samples		

Table 4. Physicomechanical properties of fired bodies K and CAC depending on firing temperature: Ev—water absorption, MOR—modulus of rupture, B—bulk density.

The CAC mixture shows significantly higher sintering activity according to measured parameters of porosity—the prepared samples of a mixture CAC have a lower water absorption Ev and higher bulk density B (according to EN ISO 10545) than mixtures based on kaolin (K) after firing at the same temperature (**Table 4**). Higher modulus of rupture MOR of fired bodies is achieved for anorthitic type of body (CAC) compared with mullite whiteware body (K) when MOR values for samples with similar porosity are compared (K-1280°C and CAC-1250°C in **Table 4**). Similar results are published in Ref. [25].

4. Conclusions

For sintering and melting of pure feldspar rocks, not just the total content of feldspar components is important, but also the ratio between potassium, sodium and calcium feldspars. At the appropriate ratio, low melting eutectics can be expected to rise, with a melting temperature substantially lower than the theoretical melting temperature of pure feldspars. The presence of calcium feldspar significantly reduces sintering ability and melting of feldspar rocks. Totally different results we can expect for the mixtures of feldspar rocks with the plastic part of whiteware raw materials mixture—kaolin. The reactions between feldspar rocks and kaolin (Al_2O_3 , SiO_2) during the sintering process are the cause of low melting eutectics, which accelerate sintering.

Natural zeolite is very intensive fluxing agent for ceramic technology. Using zeolite we can reduce the sintering temperature of the body of about 100°C, compared with traditional ceramic fluxing agent—potassium-sodium feldspar rock F-KNa. The sintered body (with water absorption below 2%) based on zeolite has lower coefficient of linear thermal expansion. The presence of zeolite in raw materials mixture significantly changes mineralogical composition of fired whiteware body—mullite, anorthite and cristobalite are the main mineralogical phases instead of mullite and quartz, which are typical for a standard whiteware bodies made from raw material mixtures based on kaolin, quartz and feldspar. The limiting factor for the use of natural zeolite as a flux for whiteware is its coloring effect.

Calcium aluminate cement CAC with high content of Al_2O_3 (70%) in the raw materials mixture for whiteware production is suitable alternative to kaolin—higher strength of green and fired body, more intensive whiteness of body after firing and lower coefficient of linear thermal expansion is possible to expect using CAC. The sintering activity of the whiteware body is

accelerated when calcium aluminate cement is used as a binder instead of kaolin—the bodies can be fired at lower temperatures. Calcium aluminate cement significantly changes mineralogical composition of fired body—anorthite is the main mineralogical phase, mullite is typical phase for standard porcelain bodies made in the system of kaolin-quartz-feldspar rock.

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