Examination of mullite ceramic specimens made by conventional casting method from kaolin and sawdust

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Abstract. Using relatively low-cost raw materials (conventional kaolin and sawdust powders) and simple technology, the authors have developed new ceramic composite materials which can successfully meet different industrial requirements. Casting masses (slurries) were made by mixing and milling different compositions of the powders with distilled water. The ceramic specimens were made by conventional gravitation slipcasting method, after drying of the green specimens, the samples were sintered in an electric kiln under oxidation and reduction atmosphere at 1250°C. The prepared and sintered specimens were tested based on geometrical sizes, microstructures and morphologies using scanning electron microscopy. In this work, the authors present some parts of the results of their research and investigation.

1. Introduction

Nowadays using and researching ceramics and ceramic composite materials is momentous [1-15]. From these the kaolin and mullite are important materials not only in the traditional but also in the modern technical ceramic industries [16-19]. The research and applications of clay-based composite materials have drawn a great deal of attention in the recent years [20-24]. Studying the different properties of clay minerals and the kaolin are very important for successful preparation of desired ceramic products [25-30]. The kaolin and the materials which formed through the thermal decomposition of kaolin (metakaolin, mullite) are well-known materials [31-32] which can be found in the Al₂O₃-SiO₂ system [23-28]. Most of the Mullite or Mullite containing ceramics are formed from kaolin at above 1000°C. Due to the excellent thermochemical properties of mullite crystals, a wide range of lightweight ceramic composites can be formed [33-37]. Ceramic and composite materials have to be developed and produced to meet the increasing needs of these materials [38-44]. Nowadays, high attention has been paid to produce mullite based porous ceramic materials [45-46]. Mullite-containing ceramics can be made using a variety of traditional and modern forming technologies, for instance, casting which can be used to produce high purity mullite ceramic products [47].

In this research work, the authors would like to utilize the favourable properties of mullite based ceramics to make porous ceramic product using conventional slipcasting method. Moreover, reduction sintering process is used to create ceramic composite materials. The author’s future plan is to produce...
mullite matrix composite reinforced with SiC, Si$_2$ON$_2$, Si$_3$N$_4$ or SiAlON using carbothermal reduction process.

2. Materials and experiments
In this research the authors made casting masses (slurries) by milling different compositions of the powders with distilled water. *Zettltitz Sedlecky ml* kaolin and fine grain oak sawdust were used as starting raw materials. According to the composition in Table 1 the dry materials were taken into the jar of the Retsch PM 400 planetary ball mill. 100 g of the dry mixture and 175 g (0.175 l) of distilled water were mixed at 150 rpm for 20 minutes.

The prepared slurries were used to form green ceramic specimens by conventional slipcasting method. The slurries were filled into the gypsum molds in a way that the level of the paste was constantly refilled to get terse ceramic specimens. The density of the used slurries was 1.3-1.4 kg/l. Figure 1 shows the laboratory casting process.

### Table 1. The dry material composition of the slurries

| Sign of mixture | Quantity, m% |
|-----------------|--------------|
|                 | Kaolinite    | Sawdust      |
| A               | 100          | 0            |
| B               | 95           | 5            |
| C               | 90           | 10           |
| D               | 85           | 15           |
| E               | 80           | 20           |

![Figure 1. The process of laboratory casting](image)

After the drying process, the specimens were sintered in an electric kiln under oxidation and reduction atmosphere at maximum sintering temperature of 1250°C with 100°C/h heating rate.

3. Results and discussions
Depending on the different types of the sintering atmospheres, the prepared specimens show different colors. Figure 2 shows the dried and the sintered specimens which made with 20 m% sawdust. In oxidation atmosphere all specimens became white, while the specimens sintered in reduction
atmosphere were discolored. Increasing the sawdust content of the casting mass resulted in an increasing number of grey-black areas, which were mainly located at the bottom of the specimens.

![Figure 2](image)

**Figure 2.** The raw casted specimen (a), the sintered specimens using oxidation (b) and reduction (c) atmosphere

The volumetric shrinkages and weight losses of the casted specimens due to the drying and sintering processes were determined (Figure 3). The degree of volumetric shrinkage due to drying decreases as the sawdust content increases. Based on the diagram, the water loss (mass loss) increases to 10 m% of sawdust during the drying process, which can be explained by the fact that the sawdust added to the kaolin mass can result in a better capillary system that facilitates the drying of the sample.

15 m% and 20 m% sawdust contents are already good for this process, which normally show lower weight loss due to residual moisture because the sawdust absorbs some of the water and cannot dry completely. It can be clearly seen that the weight loss increased proportionally during the sintering with the added sawdust content.

![Figure 3](image)

**Figure 3.** The volume shrinkage and the weight losses of the sintered specimens

The fracture surfaces of the specimens made with 0 and 20 m% sawdust were examined using scanning electron microscopy with 250-fold magnification. For the test, 20 m% sawdust sample was taken from the black part of the specimen after sintered in reduction atmosphere. The effect of the additive addition and the sintering process on the material structure of the products can be clearly seen on the SEM images (Figure 4). On the fracture surface, large pores (20-100 µm) can be observed as a result of the heat treatment, the sawdust retained its characteristic microstructure and converted to ceramic in the sample [48-49].
Based on Table 2, sawdust added to the oxidation sintering did not affect the elemental composition of the samples; in both cases the same amount was measured. Sintering in reduction atmosphere leads to an increase in the carbon content (C) of the specimens. Using 20 m% sawdust leads to formation of more than 18 m% carbon which incorporated into the black area of the sintered specimen. Based on the elemental composition, it is likely that the carbon or carbon-containing phase(s) provide the black color of the sample.

**Table 2** The elemental composition of the fracture surface of the sintered specimens

| Sawdust content | 0 m% | 20 m% |
|-----------------|------|-------|
| **Sintering process** | **oxidation** | **reduction** | **oxidation** | **reduction** |
| **Elemental composition (EDAX), m%** | | | | |
| C | 0 | 0 | 0 | 18.06 |
| O | 20.60 | 28.81 | 20.50 | 31.10 |
| Mg | 0.26 | 0.35 | 0.27 | 0.34 |
| Al | 33.71 | 30.74 | 33.74 | 22.12 |
| Si | 41.63 | 36.56 | 41.68 | 26.40 |
| K | 2.63 | 2.19 | 2.63 | 1.17 |
| Ca | 1.18 | 1.36 | 1.19 | 0.81 |

Figure 4 shows the microstructure of the fracture surface of the sintered specimens made by 0 and 20 m% sawdust.

**Figure 4.** The microstructure of the fracture surface of the sintered specimens made by 0 and 20 m% sawdust.

Figure 5 shows the crushed surface of the specimen containing 10 m% sawdust. It can be clearly seen that there are three areas which can be separated by the color of the sample. There is a black section in the center of the specimen where there was not enough oxygen during the sintering to completely burn the sawdust. Most of the sample became grey, forming a transition between the inner black and outer white parts of the sample. The points shown in the figure indicate the location of the
electron microscope images. Thus, the authors examined three distinct areas (white, grey or transient and black) based on their color in a sample.

Figure 5. The fracture surface of the specimens containing 10 m% sawdust (a) and the measured areas (b)

The evolution of the microstructure and the elemental composition of different colored areas is shown in Figure 6 and 7. The measured elemental compositions prove that the carbon content of sawdust increases as the sample darkens (black). For 10 m% sawdust, 3.71 m% of carbons were measured, while 20 m% sawdust was mixed with 18.06% wt. Based on these, it appears that in the case of 10% sawdust, most of the carbon could still be burned out of the material (formed CO or CO$_2$) [50-51].

Figure 6. The elemental composition of the different areas

Figure 7. The microstructure of the signed areas: white (a), transition (b) and black (c)
4. Conclusions
In the oxidation atmosphere, the sintered specimens became white in all cases, while the samples sintered in reduction atmosphere became discolored (black). The microstructure of the fracture surface of the samples clearly show the effect of the additive and the sintering process. Using 20 m% sawdust leads to formation of more than 18 m% carbon which incorporated into the black area of the sintered specimens.

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