Effect of composition and heat treatment on porosity and microstructures of technical ceramics made from kaolin and IG-017 additive

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Abstract. Based on conventional kaolinite and IG-017 bio-original additives of IGREX Ltd. the authors have developed new ceramic composite materials for different industrial purposes. In this work, different powder mixtures of kaolinite and IG-017 bio-original additive were milled and uniaxially pressed at different compaction pressures into cylindrical discs, after compaction, the discs were sintered in electric kiln under oxidation atmosphere and in oxygen-free atmosphere. Using the oxygen-free sintering process, new high porosity ceramic materials were created. Through the examination of the microstructure of the produced specimens the authors have found that the ceramic structure is reinforced with carbon nanofibers.

1. Introduction
Recently, large amount of ceramic materials and ceramic matrices composites (CMC) are developed [1-11] and used in the industries because of their low density [12-15], hardness [17-20], wear resistance [21-29], toughness [30-34], mechanical strength [35-44], chemical [45-49], biological [50-52] and physical [53-60] properties. Many researches have been conducted to find low-cost solutions for the manufacture of porous ceramic composites, such as the combination of kaolin and alumina raw materials, to increase the toughness [61-63]. For these purposes the authors generally use highly expensive materials and costly technical equipment and processes [64-71]. Nowadays, it is necessary to produce high performance ceramic composites with reasonably priced complex materials for extreme consumptions and applications [72-85].

In this research work, our aims are to develop high porosity ceramic matrix materials on the basis of not expensive conventional kaolinite and IG-017 additive powders and to find optimal material and morphological structures.

2. Materials and experiments
To develop new technical ceramic materials with high mechanical performance commercial kaolinite powders KKA-HB and IG-017 refined bio-original additives of IGREX Ltd were used. Figure 1 shows the grain size structures and distributions of used materials made by Malvern Mastersizer X Type Laser Granulometer.
To optimize the heat treatment, sintering temperature curves and cycles, preliminary thermo-analytical tests were done for both components before their mixing and mechanical activating (Fig. 2). The thermo-analytical properties were measured by MOM Derivatograph-C. The achieved DTA curve of our high purity kaolinite powder was fully confirm the research works of Gabriel Varga and Igor Štubna [86, 87], where the dehydration process starts at about 452 °C and finish at 973 °C. In the present case the dehydration process is mostly intensive at 529.7 °C, meanwhile the IG-017 additive is highly loss the surface-absorbed moisture at 87.4 °C. At the same time the DTG curve shows that, the starting temperature of outgassing process of organic components is 242.8 °C, while the most intensive outgassing temperature is 281.6 °C. The organic component is mostly burned and generated organic gasses at 320.5 °C. Later, the temperature zones which achieved on the derivatograph were taken into the consideration during sintering of the uniaxially compacted cylindrical specimens.
7 different mixtures were prepared from the kaolin and IG-017 additives using the compositions in Table 1. The different prepared mixtures were mechanically activated in a planetary ball mill for 20 minutes at 200 rpm. After mechanical activation of the mixtures 24 cylindrical disc specimens with diameters of 20 mm for each mixture were compacted uniaxially using 100 kN mechanical pull-press.

Table 1. The mixtures compositions in m%

| Number of mixtures | KKA-HB Kaolinite | IG 017 bio original material |
|--------------------|------------------|----------------------------|
| K I                | 85               | 15                         |
| K II               | 75               | 25                         |
| K III              | 65               | 35                         |
| K IV               | 55               | 45                         |
| K V                | 45               | 55                         |
| K VI               | 35               | 65                         |
| K VII              | 25               | 75                         |

Three different compaction forces (50 kN, 60 kN and 70 kN) were used to develop compression pressures of 156.21 MPa, 187.45 MPa and 218.69 MPa to produce cylindrical discs of 8 grams in each case. After compacting half of the specimens were fired (sintered) in normal (oxidation) atmosphere while the other half of the specimens were sintered in oxygen-free (quasi-reduction) atmosphere. In both cases the maximum sintering temperatures were 1250 °C on heating rate of 100 °C/hours. This sintering temperature was high enough for formation of mullite crystal phases from kaolinite, but not enough for creation of cristobalite crystals from the formed free SiO₂ component which is created from the degraded kaolinite at about 1080 °C [88]. In their previous work, the authors have already presented the color change, volume shrinkage, loss of mass and density of ceramic specimens depending on firing temperatures and atmospheres [89]. During the sintering processes the sintered specimens have changed the color (Figure 3), the specimens which sintered in normal (air) atmosphere give white color, while the specimens which sintered in oxygen-free (quasi-reduction) atmosphere give black color.

3. Results and discussions

The porosities of sintered specimens were determined based on water absorption capacity (Table 2) and the following formula was used for the calculations:
\[ P = \frac{m_w - m_s}{V_s} \times 100\% \]  

(1)

Where \( P \) the porosity, \( m_s \) the mass of sintered specimens, \( m_w \) the mass of the tested specimens after boiling in distilled water for 2.5 hours and \( V_s \) the geometry volume of the sintered specimens.

### Table 2. The porosities of the sintered specimens in %

| Compacting pressures | 156.21MPa | 187.45MPa | 218.69MPa |
|----------------------|-----------|-----------|-----------|
| Sintering in         | oxidation atm. | oxygen-free atm. | oxidation atm. | oxygen-free atm. | oxidation atm. | oxygen-free atm. |
| Bio-origin additive, m% | 15       | 10.02     | 5.86      | 3.34     | 11.72     | 3.34     | 17.57     |
|                      | 25       | 6.45      | 16.64     | 6.47     | 13.87     | 9.71     | 16.64     |
|                      | 35       | 16.28     | 15.74     | 16.28    | 18.37     | 19.54    | 20.99     |
|                      | 45       | 26.75     | 20.42     | 23.41    | 20.42     | 30.10    | 22.97     |
|                      | 55       | 26.13     | 18.17     | 33.60    | 23.36     | 37.34    | 38.94     |
|                      | 65       | 31.83     | 21.99     | 40.93    | 27.48     | 45.48    | 41.23     |
|                      | 75       | 16.38     | 20.90     | 38.22    | 32.84     | 60.06    | 47.76     |

The porosities of sintered samples at the low mixing ratio of IG-017 did not depended greatly on the compacting pressures (Table 2 and Figure 4). In the case when the specimens have contained 55-75 m% bio materials, the degrees of porosity were significantly influenced by the applied compaction pressures.

![Figure 4. The porosity of the sintered test pieces in the oxidation and oxygen-free atmosphere](image)

The surfaces of the sintered samples were examined by scanning electron microscopy to determine how the microstructures and the elemental compositions were changing depending on sintering conditions (atmosphere) and applied mixture compositions. In Figure 5 and in Table 3 big differences can be seen in the fracture surfaces due to the difference in the material compositions of specimens which were presintered in normal atmosphere and in oxygen-free atmosphere. With increasing the volume of IG-017 additives in the mixtures, the porosity of the sintered samples has increased significantly.

The used amount of IG-017 additives have strongly influenced the carbon content of the sintered specimens in the oxygen-free atmosphere (23 % carbon in the case of samples with 75 % IG-017 content). Moreover, the different heat treatment conditions have a significant effect on the microstructure and elemental composition of the prepared and sintered ceramic specimens.

The chemical and the phase compositions of the sintered specimens were determined by XRD tests. In each cases the main phase was found to be mullite, together with a large amount of amorphous glass.
and small amount of quartz or cristobalite. During oxygen-free sintering, carbon fibers were formed (Table 4).

**Table 3.** The chemical compositions of the specimens from KI and KIV mixtures determined by EDAX

| Mixture      | Sintering atmosphere | C   | O   | Al  | Si   | K   | Ti  | Fe  |
|--------------|----------------------|-----|-----|-----|------|-----|-----|-----|
| **KI**       | normal (oxidation)   | Wt %| 4.62| 34.96| 24.22| 32.19| 1.89| 0.91| 1.21 |
|              | At %                 |     | 8.17| 46.47| 19.09| 24.37| 1.03| 0.40| 0.46 |
|              | oxygen-free          | Wt %| 6.93| 31.06| 23.72| 32.96| 2.52| 1.13| 1.67 |
|              | At %                 |     | 12.31| 41.40| 18.75| 25.03| 1.37| 0.50| 0.64 |
| **KIV**      | normal (oxidation)   | Wt %| 1.51| 34.62| 25.67| 33.79| 2.13| 1.18| 1.11 |
|              | At %                 |     | 2.77| 47.63| 20.94| 26.48| 1.20| 0.54| 0.44 |
|              | oxygen-free          | Wt %| 23.29| 29.24| 18.94| 25.21| 1.47| 0.63| 1.23 |
|              | At %                 |     | 35.65| 33.6 | 12.91| 16.5 | 0.69| 0.24| 0.41 |

**Table 4.** The oxide- and phase composition of the sintered specimens from KIV mixture

| Sintered atm. | Phase % | SUM | Mullite | Quartz | Cristobalite | Amorph glass | Carbon fiber |
|---------------|---------|-----|---------|--------|--------------|--------------|--------------|
| **Oxidation** |         |     |        |        |              |              |              |
| SiO₂          | 61.95   | 53  | 2       | 5      | 40           | 0            |
| Al₂O₃         | 38.05   | 38.05 |        |        |              |              |
| **Oxygen-free** |       |     |        |        |              |              |              |
| SiO₂          | 50.54   | 48  | 4       | 1      | 32           | 15           |
| Al₂O₃         | 34.46   | 34.46 |        |        |              |              |
| CO₂           | 54.96   |     |        |        |              |              | 54.96        |
4. Conclusions
In this research work, the authors have successfully developed and produced a high porosity ceramic matrix material using conventional KKA-HB kaolin and IG-017 bio-original additives. The volume of porosity is found to be very strongly depending not only on compacting pressures (156.21 MPa, 187.45 MPa and 218.69 MPa) and mixture ratio but also on sintering conditions as well. The samples which sintered in oxygen-free atmosphere using 156.21 MPa compression pressure give a porosity of 20V% while the same samples which compressed at 218.69 MPa give a porosity of 50 V% depending on quantity of the used additives. The reason of this larger porosity phenomena is that the generated gases from firing of IG-017 additive could not evaporate through the high dense surface of specimens during their sintering in oxygen-free atmosphere.

The carbon contents of specimens became insignificant when normal (oxidation) atmosphere was used for sintering, meanwhile using oxygen-free atmosphere and IG-017 additives lead to increase the quantity and volume of carbon contents of specimens up to 23.29 m%.

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