

University of Belgrade
Technical Faculty Bor

PROCEEDINGS

XXIII International Conference Ecological Truth

Editors
Radoje V. Pantovic
Zoran S. Marković

EcoIst '15

Hotel "PUTNIK", Kopaonik, SERBIA
17-20 June 2015

UNIVERSITY OF BELGRADE
TECHNICAL FACULTY BOR



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"ECOLOGICAL TRUTH"**

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**Radoje V. PANTOVIC
and
Zoran S. MARKOVIC**

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DESIGN OF MICROSTRUCTURE OF CERAMICS BASED
ON WASTE FLY ASH AND CLAY

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ABSTRACT

Ceramics microstructure was designed using waste fly ash (REK Bitola, Macedonia) and clay. The raw materials were characterized from chemical, mineralogical and thermal aspect. The ceramics was designed using the granulation less than 0.063 mm. The clay content varied from 10 to 90 wt.%. The consolidation of the compacts was realized at pressure of 45 MPa and sintering at different temperatures (900, 1000, 1500 and 1100 °C/1h, heating rate of 10°C/min).

The designed microstructure of the composite with composition 60wt% clay and 40wt% fly ash sintered at 1100°C/1h was found as optimal regarding to its properties: density- 2.089g/cm³; water absorption- 7.02%, bending strength – 50.47 MPa and E-modulus - 25,35GPa. Technical coefficient of thermal expansion was $\alpha_{20-600}=7.03 \times 10^{-6}/^{\circ}\text{C}$.

Key words: fly ash, clay, microstructure, sintering.

INTRODUCTION

Fly ash presents the by-product obtained during the production of electricity from the coal in thermal power plants. The raw materials in traditional ceramics are taken from nature i.e. from the deposits of the earth. Concerning the chemical and mineralogical composition there are similarity between the natural raw materials (clay) and fly ash. So, this waste can be successfully used as a raw material for production of traditional ceramics [1-3]. In recent years there is growth interest for utilization of waste fly ash in ceramics. For instance, Cicek and Tanriverdi [4] investigated the possibilities for using fly ash for production of light weight bricks based on 20% sand, 68% fly ash and 12% hydrated lime. Lingling et al. [5] used fly ash as replacement of clay in bricks production, so they proved that fired bricks with high volume ratio of fly ash has high compressive strength and low water absorption. Sokolar and Vodova [6] worked on the investigation of the influence of the addition of fly ash in the raw material mixture and the granulometry of fly ash on the properties of fired fly ash – clay body.

In the present paper, the aim was to design the microstructure of ceramics based on waste fly ash and clay and to optimize the content of clay relating to the properties of the fabricated ceramics.

MATERIALS AND METODS

Characterization of the fly ash and clay

Fly ash from thermal power plant REK Biota, Republic of Macedonia and clay near to the this region were used in this investigation. The particle size distribution for both raw materials was lower than 0.063 mm. The content of clay varied from 10-19wt.% Chemical composition of the fly ash was carried out by X-ray fluorescence (ARL 990XP) and classical silicate analysis was applied for clay. The phase composition of the fly ash and the clay was performed by using X-ray diffraction (Philips, model PV 105-1). The thermal properties of the fly ash and the clay were determined using a heating microscope (Leitz Wetzlar) in the temperature interval from room temperature (RT) to 1400⁰C, in air atmosphere with a heating rate of 10⁰C/min.

Consolidation of the fly ash and the clay

Ceramics was consolidated in laboratory conditions by pressing (Weber Pressen KIP 100) at P=45MPa using PVA as a binder. Sintering was performed in chamber furnace in air atmosphere at temperatures of 900, 1000, 1050 and 1100⁰C, with holding time of 1h at maximum temperature and heating rate of 10⁰C/min.

Characterization of the sintered samples

Bulk density of compacts was determined from the ratio between weight and volume of the compacts. Water absorption of the compacts was determined from difference in dry mass and surface dry mass after immersion in cold water. Water absorption values were determined from weight differences between the as-sintered and water-saturated sintered compacts after immersion in boiling water for 2 h.

Mechanical properties i.e. bending strength and E-modulus of fabricated compacts (6 pices, 50X5X5mm³) were carried out using three point bending tester (Netzsch 401/3) with 30 mm span and 0.5mm/min crosshead speed.

Linear thermal expansion of the obtained ceramic compacts was determined using the dilatometer (Netzsch 402 E) in the air atmosphere and temperature interval RT-600⁰C-RT, with heating rate of 2⁰C/min.

The microstructure of the fractured surface of the compacts was analysed by scanning electron microscopy (Leica S440I).

RESULTS AND DISCUSSION

The chemical composition of the fly ash and clay is shown in the Table 1. It is evident from the Table 1 that in both systems there is a dominant presence of SiO₂, Al₂O₃ and Fe₂O₃, whereas in the fly ash there is a minor content of ecologically risky components like MnO and P₂O₅.

Table 1. Chemical composition of the fly ash and clay

Oxide	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	MnO	P ₂ O ₅	ZnO	PbO	SO ₃	LOI	Σ
Fly ash	52.38	0.09	23.61	7.31	7.42	2.11	0.9	1.67	0.03	0.08	0.01	0.03	1.2	3.12	99.94
Clay	58.48	/	19.8	7.44	6.18	1.43	2.1	2.51	/	/	/	/	/	2.05	99.99

The XRD pattern of the fly ash showed the presence of amorphous phase and minerals such as: quartz, anorthite, hematite, albite and anhydritet. The clay was from illite type with the mineralogical composition: quartz, feldspar, aragonite, illite, chlorite and calcite.

The thermal characteristics of the both raw materials are given on the Table 2.

Table 2. Thermal characteristics of the fly ash and clay

Material	Significant shrinkage [°C]	Softening temperature [°C]	Melting temperature [°C]
Fly ash	1050	1380	1440
Clay	1100	1280	1320

From the Table 2 it can be seen that the region of sintering for fly ash was in the temperature interval from 1050 to 1380°C, while for the clay it was in the temperature interval from 1100 to 1280°C.

The dependence of density and water absorption with sintering temperature of the fly ash-clay compacts with different clay content from 10 to 90wt.% are presented in Figures 1 and 2.

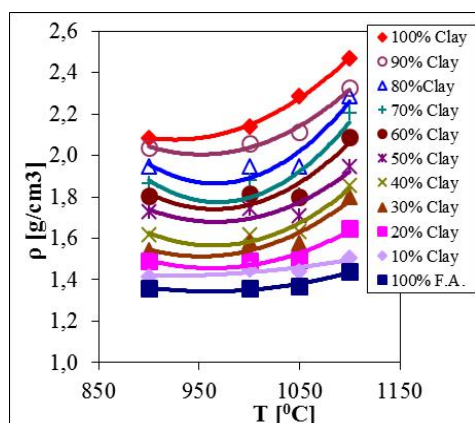


Figure 1. Density of the compacts sintered at different temperature

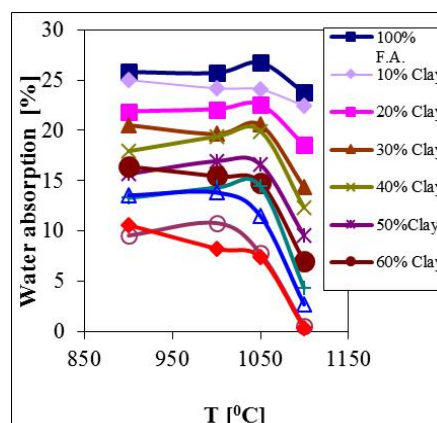


Figure 2. Water absorption of the compacts sintered at different temperatures

It is evident from the Figures 1 and 2 the slight increase of density/decrease of porosity up to 1050°C. At the temperature of 1100°C the density increase rapidly

reaching the value of 2.5 g/cm^3 for the clay compacts and 1.4 g/cm^3 for the fly ash compacts. Generally, for the composites composed with different content of clay (from 10 to 90wt.%) it can be concluded that the clay influenced on the increase of density and decrease the water absorption.

The variation of the mechanical properties i.e. bending strength and E-modulus of the compacts with different clay content (from 10 to 90wt.%) with temperature are presented in Figures 3 and 4. It is evident that the clay influenced on the increase of the mechanical properties. The slight increase of the mechanical properties for the all compacts with different clay content (from 10 to 90wt.%) is evident up to the temperature of 1050°C , but at temperature of 1100°C the rapid increase, almost doubled, of the mechanical properties is obvious. The bending strength and E-modulus of the clay compacts is 90 MPa and 34 GPa, respectively, and for the fly ash compacts the bending strength and E-modulus are 10 MPa and 4 GPa, respectively.

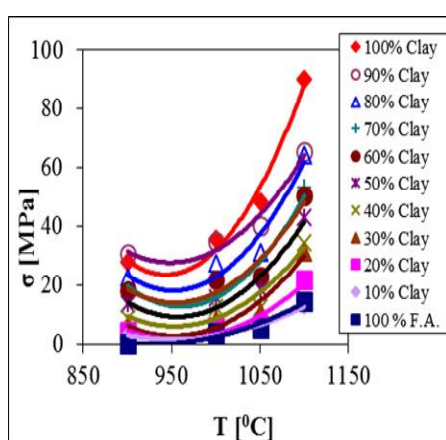


Figure 3. Bending strength of compacts sintered at different temperature

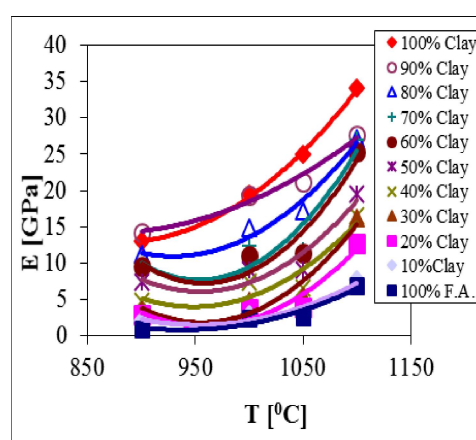


Figure 4. E-modulus of the compacts sintered at different temperature

The composite consisted of 60% clay and 40 % fly ash sintered at temperature of 1100°C with heating rate of $10^\circ/\text{min}$ was chosen as optimal and it was the subject of further investigations.

The microstructure of the composite consisted of 60% clay and 40 % fly ash sintered at the temperature of 1100°C is presented in Figures 5

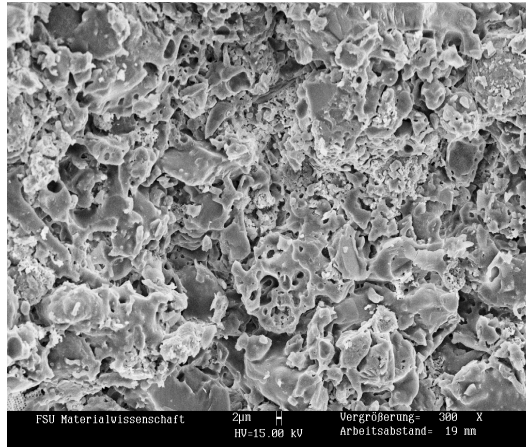


Figure 5. Microstructure of the composite consisted of 60% clay and 40 % fly ash sintered at temperature of 1100⁰C with heating rate of 10⁰/min

The thermal expansion characteristics of the composite consisted of 60% clay and 40 % fly ash sintered at the temperature of 1100⁰C showed absence of hysteresis effect, proving that this material is in thermal equilibrium. The coefficient of thermal expansion for this composite was $7.03 \times 10^{-6}/^{\circ}\text{C}$.

CONCLUSIONS

The designed microstructure of the compacts composed of 60wt.% clay and 40wt.% fly ash sintered at 1100⁰C have the following properties: density: 2.089 g/cm³; water absorption: 7.02%, bending strength: 50.47 MPa, E-modulus: 25.35 GPa and coefficient of thermal expansion of $7.03 \times 10^{-6}/^{\circ}\text{C}$ and it can be potentially used as building material where the waste fly ash (as NORM representative) is used.

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