Influence of Milled Waste Glass to Clay Ceramic Foam Properties Made by Direct Foaming Route

A. Shishkin, V. Mironovs, D. Goljandin, A. Korjakins

Abstract—The goal of this work is to develop sustainable and durable ceramic cellular structures using widely available natural resources- clay and milled waste glass. Present paper describes method of obtaining clay ceramic foam (CCF) with addition of milled waste glass in 5, 7 and 10 wt% by direct foaming with high speed mixer-disperser (HSMD). For more efficient clay and waste glass milling and mixing, the high velocity disintegrator was used. The CCF with 5, 7, and 10 wt% were obtained at 900, 950, 1000 and 1050 °C firing temperature and they have demonstrated mechanical compressive strength for all 12 samples ranging from 3.8 to 14.3 MPa and porosity 76-65%. Obtained CCF has compressive strength 14.3 MPa and porosity 65.3%.

Keywords—Ceramic foam, waste glass, clay foam, glass foam, open cell, direct foaming.

I. Introduction

DUE to a number of exceptional properties such as chemical stability, high permeability, high refractoriness, low bulk density and thermal conductivity as well as specific heat inherent to ceramics [1], [2], these materials attract more attention with each year. The weight reduction, better thermal and acoustic insulation properties [3], application as filters for molten glass and metals [4], [5] and catalyst scaffolds [6], [7] promote research dedicated to porous ceramics. During the past decades, wide variety of manufacturing methods have been investigated and led to development of methods for production of porous ceramics, such as the replica, the sacrificial template, water-oil emulsion and the direct foaming [2], [8], [9]. Nevertheless, most of the mentioned above methods are well investigated and approved for advanced ceramics such as Si_2N_4, SiC and for the geopolymers with the exact oxides molar ratios (SiO_2/Al_2O_3, K_2O/SiO_2), but for clay-based ceramics mostly complicated methods such as freeze-casting [3] or time-consuming biological foaming, through the reaction of yeast with starch in aqueous ceramic suspension, are described [10].

Clay is one of the most available and cheap natural precursors for the production of construction and insulation materials. At the same time, the direct foaming is one of the fast and easiest methods for producing of the foamed clay ceramics, however, number of studies devoted to this topic is limited.

The problem of waste material reuse, particularly the glass (soda-lime glass) is increasing from year to year. In European Union approximately 9 million ton of waste glass was collected in 2010 [11]. A number of researchers have investigated the use of waste glass in ceramic industry: Waste glass incorporated as an alternative ceramic raw material or as a fluxing agent in cement and/or concrete, stoneware tile and brick [12]–[14]. In [15], authors reported that incorporation of 10 wt.% of waste glass into clay bricks and firing at the temperatures of 900-1000 °C enhanced the properties of fired clay bricks – bricks fired at 900 °C had strength comparable to that of conventional clay brick fired at 1000 °C. However, there is still lack of information about clay based foam with waste glass addition. It is necessary to evaluate the effect of waste glass on strength, porosity and firing shrinkage characteristics of fired clay foam. In this work, these effects are assessed using waste milled glass and the firing behavior, physical-mechanical properties and microstructure are discussed.

In previous work [16], the CCF were obtained by direct foaming with High Speed Mixer Disperser with cavitation effect (HSMD). The method is based on the cavitation effects, caused by device rotary elements which lead to multiple hydraulic impacts. Present research is devoted to investigation of influence of waste glass addition on the Latvian clay (from *Liepa* deposition) ceramic foam properties.

II. Materials and Methods

A. High Speed Mixer - Disperser

For experimental studies, we used HSMD as described in previous work [16] at 6000 rpm speed. For clay and waste glass milling and mixing the high velocity disintegrator (HVD) described in previous work [17] was used at 12000 rpm speed.

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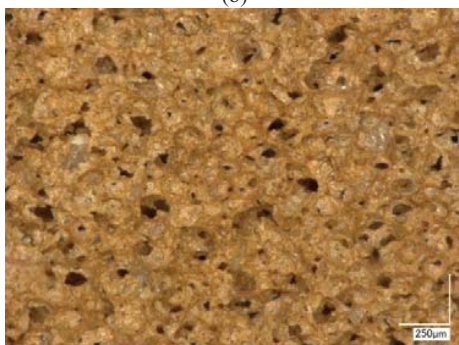
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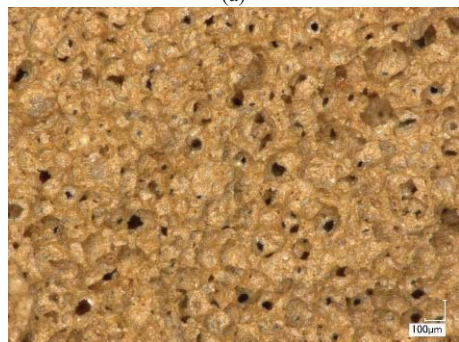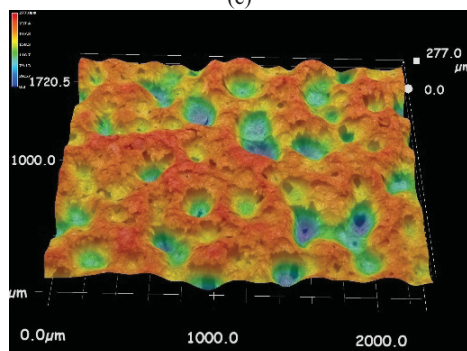
Fig. 1 Optical images of the fracture surface of CCF foams: (a) with 5% WG, fired at 900 °C; (b) with 7% WG, fired at 900 °C; (c) with 10% WG, fired at 900 °C; (d) 3D-height difference image of sample with 5% WG, fired at 900 °C

B. Reagents and Raw Materials

The SCHÄUMUNGSMITTEL W 53 FLÜSSIG (Zschimmer & Schwarz Gmbh, Germany) was used as foaming agent (FA). For CCF preparation, homogenized clay from *Liepa* deposition (Latvia) was used. The clay was obtained from *Lode* Ltd brick factory (Latvia). The clay lumps were dried at 105 °C for 24 h, pretreated in the jaw crusher for particle size 10-30 mm and then milled in HVD in selective mode for particles size < 50 µm. The green bottle glass was used as waste glass (WG) source. Clean, dry bottles were pretreated milled in the jaw crusher for particle size 10-30 mm and then milled in HVD in selective mode for particles size < 50 µm. Tap water – (Riga municipal water supplement system) [18] and DOLAFLUX B 11 (Zschimmer & Schwarz Gmbh, Germany) as dispersant were used for CCF production as well.

C. CCF Obtaining Method

The dry clay-milled waste glass mixtures with 5, 7, 10 wt% were prepared by mixing in HVD. The preparation of CCF comprises of following steps. The HSMD, working at 500 rpm, was filled with 300 ml of tap water and dispersant (1% from clay mass – 6.5 g). Gradually, 700 g of dry clay-glass mixture was slowly added, preventing their agglomeration, and HSMD speed was also gradually changed to 4000 rpm. After that FA (5.5% from clay mass – 38.5 g) was added, mixer speed was set to 6000 rpm and at the same time air was introduced into the system. When the foamed mixture volume increased twofold, it was left in HSMD for 1 min in recirculation mode and ceramic foam was transferred into the mold (corrugated board 150x200x60 mm) and dried for 72 hours at 20 °C. After that, drying sample was removed from the mold and cut into smaller fragments – 55x55x110 and firing) in muffle oven (LH 11 by Nobertherm) at 900, 950, 1000 and 1050 °C (heating rate of 5°C/min) for 30 min.

D. Determination of the Physical and Mechanical Properties

Shrinkage was determined by direct measurement of specimen linear dimensions (X, Y, Z) before and after firing. Archimedes method was used to determine liquid (distilled water) absorption, bulk density, apparent density and apparent porosity.

To determine **the total porosity,** the bulk density of the porous sample was determined by Archimedes method [19] and pycnometry. The apparent porosity is the sum of open and closed porosity ($P_a = P_o + P_c$); for the closed porosity:

$$P_c = P_a - P_o,$$

where: P_a – total porosity, %; P_o – open porosity, %; P_c – close porosity, %.

The apparent porosity P_a is calculated by (1):

$$P_a = \frac{\rho_a - \rho_b}{\rho_a} \cdot 100\% , \qquad (1)$$

where: ρ_a – apparent density, g/cm^3; ρ_b – bulk density, g/cm^3

The bulk density ρ_b in g/cm^3 is calculated according to (2):

$$\rho_b = \frac{m_1}{m_3 - m_2} \cdot \rho_f \qquad (2)$$

The open porosity P_o in volume per cent is calculated by (3):

$$P_o = \frac{m_3 - m_1}{m_3 - m_2} \cdot 100\% , \qquad (3)$$

where: m_1 - mass of the dried sample in air, g; m_2 - mass of the saturated with liquid sample weighed in liquid, g; m_3 - mass of the saturated with liquid sample weighed in air, g; ρ_{fl} - density of the liquid used for buoyancy, g/cm^3; ρ_t – theoretical (pycnometric) density of the solid, determined by pycnometry according to EN 993-2, g/cm^3.

Compressive strengths of sintered cubic shape specimens (50mm X 50 mm X 50 mm) were assessed using Universal Testing Machine (UTM) (Instron: 8801) at room temperature by strain rate 0.01/s. The tests were carried out for a set of five samples in each category.

For **Microstructural characterization,** a Keyence corporation (Japan) VHX-2000 optical microscope with lenses VH-Z20R/W and VH-Z500R/W was used for optical imaging.

III. Results and Discussions

The cubic shape specimens in this research were manufactured from clay and waste glass via direct foaming method using HVD. The as-sintered porous samples were physically characterized in terms of bulk density, total porosity and compressive strength. These properties of porous samples were individually determined in 8 parallel specimens cubic shape (50 X 50 x 50 mm) and the average value was reported. The properties of fired specimens are shown in Figs. 1 (a)-(d).

A. The Pore Structure

The morphology of the CCF is shown in Fig. 1. The microstructures of these foams, obtained by direct foaming, show the presence of interconnected pores, which points at open cell type foam. The pore interconnection is formed due to the thin suspension films between the bubbles ruptured during drying since the displacement of powder from the interface is thermodynamically unfavorable [6]. The WG addition had no significant influence on foam morphology and average cell sizes, which were in the 50-200 μm range for all samples (5, 7, 10% of WG addition) sintered at 900 °C. As shown in Fig. 1 (d), the pores are distributed evenly in the sample.

B. The Apparent Porosity

The porosity is one of the important porous material characterization criteria. The specimens' porosity increases by 2-3% with WG increase from 5 to 7% and by 2-3% with WG increase from 7 to 10% (for all sintering temperatures). At the same time, porosity decreases by 2-2.5% with firing temperature increase by 50 °C for each series. The porosity dependence from firing temperature increases linearly. Insignificant fluctuations of porosity could be explained by specimen inhomogeneity and by the error of measurement range (+/- 0.5%).

C. The Bulk Density

The bulk density alterations follow absolutely different pattern. From one hand the bulk density decreases by 0.05 -0.07 g/cm^3 from series to series for each sintering temperature from sample with 5% WG content to 7%, and by 7-10% for each sintering temperature from sample with 7% WG content to 10% series. (from 0.8 to.73 at 900; from 0.84 to 0.79 g/cm^3 at 950 °C and 1000 °C; from 0.90 to 0.84 g/cm^3 at 1050 °C) between the series, increasing the WG content. But from the other hand all samples series show the same bulk density change pattern: the growth by 0.07 g/cm^3 from 900 °C to the 950 °C, no change from 950 to 1000 °C and continue growth for 0.07-0.9 g/cm^3 from 1000 °C to 1050°.
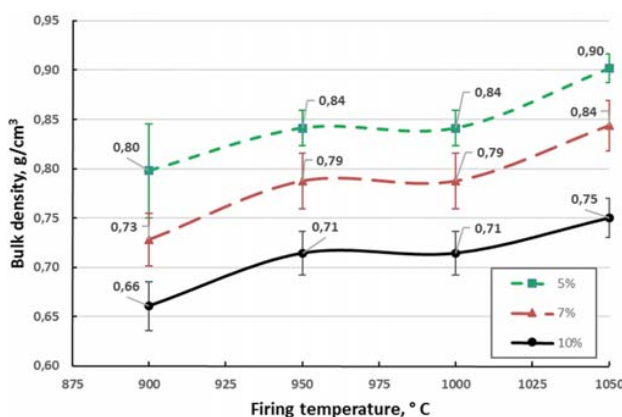

Fig. 2 CF bulk density dependence from firing temperature and the composition
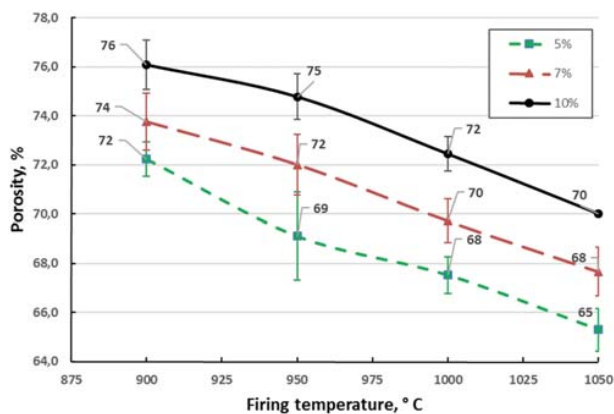

Fig. 3 Total porosity dependence from firing temperature and the composition
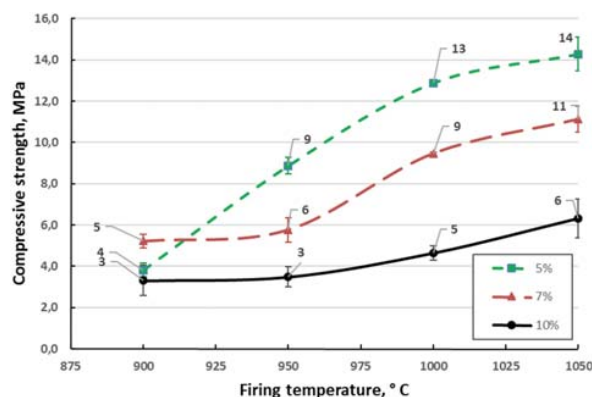
Fig. 4 CF compressive strength dependence of a firing temperature and the composition

D. The Compression Strength

The compression strengths of all three series show three different patterns. Composition with 5% of WG has monotonic growth from 3.3 to 14.3 MPa, with 7% WG almost no change at 900 and 950 C (5.2 MPa and 5.3 MPa respectively), rapid growth from 950 to 1000 °C (5,8 MPa and 10,2 MPa respectively) and almost no change in 1000-1050 °C range (10.2 MPa and 11.1 MPa respectively). The composition with 10% of WG has slight monotonic growth from 3.8 to 6.3 MPa. All three series (5,7 and 10% of WG) show similar trend– the higher content of WD, the smaller compressive strength value is. However, at 900 °C situation changes significantly: the highest value of compressive strength – 5 MPa was found for composition with 7% of WG. This effect could be explained by liquid phase emerging in sufficient amount for the ceramic particle binding. However, in case of 10% WG composition e glassy phase could have a decrease compressive strength – 3 MPa. This information provides valuable information for future technological process optimisation: for cases, where is not necessary achieving compressive strengths higher than 5 MPa, composition with 7% of WG admixture, fired at 900°C, could be used.

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