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MECHANICAL AND FILTERING PROPERTIES OF NON-SINTERED POROUS SOLIDS FOR FILTRATION APPLICATIONS

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Abstract: *This work deals with the mechanical and filtering properties of porous materials based on silicon carbide and aluminate cement for filtration applications. Silicon carbide classified according to particle sizes was used as the main aggregate. The influence of granulometric composition and quantity of aggregate upon porous structure generation was studied and mechanical properties were characterized using a three-point bending test of columns with proportions of 20 × 20 × 100 mm prepared of testing mortars. The input materials were characterized using laser granulometry and X-ray diffraction; the porous structure was studied using mercury porosimetry, and scanning electron microscopy. Disc-shaped barriers with 90-mm diameters were prepared by uniaxial pressing in a mold. They were tested using air flux to determine their effective permeability. Additionally, capillarity testing of the materials was carried out.*

Keywords: Porous material, Porosity, Mechanical properties, Filtration.

1. Introduction

The production of porous supports for membranes as well as macroporous elements for filtration involves firing (Guocheng, et al., 2012). Firing is a basic process in ceramic technology and involves sintering to form strong ceramic bonds between the particles of raw materials (Hanykýř, 2000). The structure of the final product is mainly influenced by the firing temperature, which usually reaches 1100°C for inorganic membrane fabrication. With the increase in temperature, porosity is reduced and flexural strength is increased (Nandi, et al., 2008). The firing process itself can take several hours, and it is very expensive (Hlaváč, 1981, Dong, et al., 2009). The main effort in the field of membrane processes is to reduce the cost of membrane units and thereby reduce the cost of the overall process (Dong, et al., 2009, Sarkar, et al., 2012). One possibility is to skip the heat treatment step, which makes the final products more expensive. However, it is also necessary to preserve porosity and appropriate values of mechanical properties simultaneously. That is the main aim of this work.

2. Materials and Methods

2.1. Materials and preparation of samples

An aluminate cement, Secar 71, was mixed with silicon carbide (Carborundum Electrite a.s., Benátky nad Jizerou). Various particle sizes (Tab. 1) ranging from 33 to 500 μm were used to influence the porous structure of the final material.

Testing samples for evaluating mechanical properties were prepared by mixing water and cement for one minute. The aggregate was added and then homogenized for three minutes and casted in steel molds to form columns with measurements of 100 × 20 × 20 mm. The molds were enclosed in a polyethylene bag to avoid humidity outflow and then demolded. After demolding, the samples were cured at laboratory temperature and pressure in an enclosed plastic bath with increased humidity. Samples for permeability testing were prepared using the same mixing procedure above but the mixture was then pressed into a mold to form disks with 90-mm diameters and a thickness of 5 mm.

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2.2. Characterization methods

The phase composition of starting materials was determined using a PANalytical Empyrean X-ray diffraction spectrometer. Granulometric measurement to determine particle size distribution was performed with a Sympatec HELOS KR laser analyzer with a measuring range between 0.1 to 8750 μm.

Testing of flexural and compressive strengths was performed using the DESTTEST 4310 COMPACT A machine designed by Beton System, which is typically used for testing mechanical properties of building materials. Mechanical strengths were measured after 24 hours, 7 days and 28 days. Characterization of porous structure of the samples was carried out using a Micromeritics Poresizer 9310 mercury intrusion porosimeter. Cross-section of samples and their surfaces were analyzed using a Zeiss EVO LS 10 scanning electron microscope. Porosity was determined using the pycnometric method – the sample was dried at 105°C to a constant weight and left to cool in a desiccator. The sample was then weighed to an accuracy of 1 mg (m_1), put in a pycnometer, filled with distilled water and reweighed (m_2). Finally, the weight of the water-filled pycnometer was measured. Bulk density was calculated according to relation (1). The samples used for determination of bulk density were subsequently put into 50-cm^3 beakers and these were filled with water to completely submerge the samples. The sample beakers were put on a hot plate and the water was boiled for an hour. After the samples were saturated with boiling water, their surfaces were wiped to remove water droplets and weighed (m_4). Absorbability was then calculated according to equation (2). Apparent porosity was calculated using equation (3) (Ptáček et al., 2012).

Finally, the permeability of the prepared materials was determined by air flux with a laboratory filtration apparatus. Mean pore (capillary) radius and the diffusion coefficient of water in the material was determined using a capillarity test. Permeability was tested using the 90-mm disks. The sample was placed into a chamber connected to a compressor air tank with a volume of 0.001 m^3 and connected to the filtration apparatus, and the overpressure (Δp_0) in the range of 120 kPa (compared to atmospheric pressure) was set up. The beginning of the experiment was defined by turning on the valve to the chamber with the disk, and the time of the instantaneous overpressure (Δp) was measured until it was in equilibrium with atmospheric pressure. The results of the measurement were displayed as a linear dependence, described by equation (6). The effective permeability K_{eff} [mol·m^{-1}·s^{-1}·Pa^{-1}] of the material was determined using the angular coefficient β [s^{-1}] of the linear dependence (equation 6), according to relation (7) where A is the disk surface area, h_b is its thickness, V_a is the volume of air tank, R is the molar gas constant (8.314 J·mol^{-1}·K^{-1}) and T is the ambient temperature [K] (Kudová et al., 2004). For the determination of capillarity, a set of samples in the form of columns with dimensions 100 × 20 × 20 mm was used. In a suitable dish, the specimens were put onto glass rods and the dish was filled with water up to the bottom edge of the columns. At the same time, the time measurement was initiated. The height of water raised in the specimen material was measured in three-minute intervals and the measurement was ended after 30 minutes. The values of measured height were used to determine the mean pore (capillary) radius (equation 4) and diffusion coefficient of water in the material of the samples according to relation (5) (Ptáček et al., 2012).

2.3. Equations

$$\rho_{\text{b}} = \frac{m_1}{m_3 - (m_2 - m_1)} \rho_{H_2O} \tag{1}$$

$$A_{\text{C}} = \frac{m_4 - m_1}{m_1} 100 \tag{2}$$

$$\varepsilon_{\text{a}} = \rho_{\text{b}} A_{\text{C}} \tag{3}$$

$$h = \frac{2\gamma \cos\varphi}{r_{\text{p}} g \rho_{\text{l}}} \cong \frac{1.49 \cdot 10^{-5}}{r_{\text{c}}} \tag{4}$$

$$h^2 = D_{\text{g}} t \tag{5}$$

$$\ln\left(\frac{\Delta p}{\Delta p_0}\right) = -\beta t \tag{6}$$

$$K_{\text{eff}} = \frac{\beta h_{\text{b}} V_{\text{a}}}{ART} \tag{7}$$

2.4. Figures and tables

AC-220-53 means sample prepared of aluminate cement (AC) and silicon carbide F220 (see Tab. 1) and AC content of 53 %.

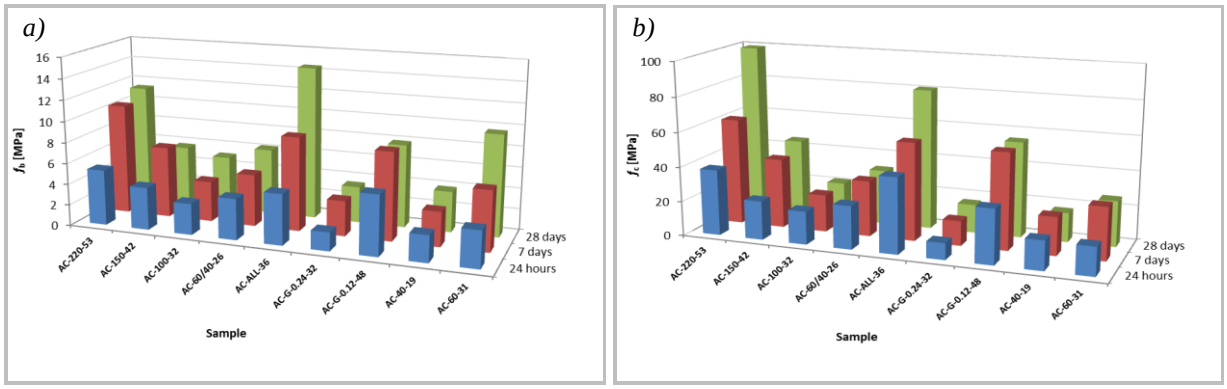

Fig. 1: Development of a) flexural and b) compressive strengths of samples.

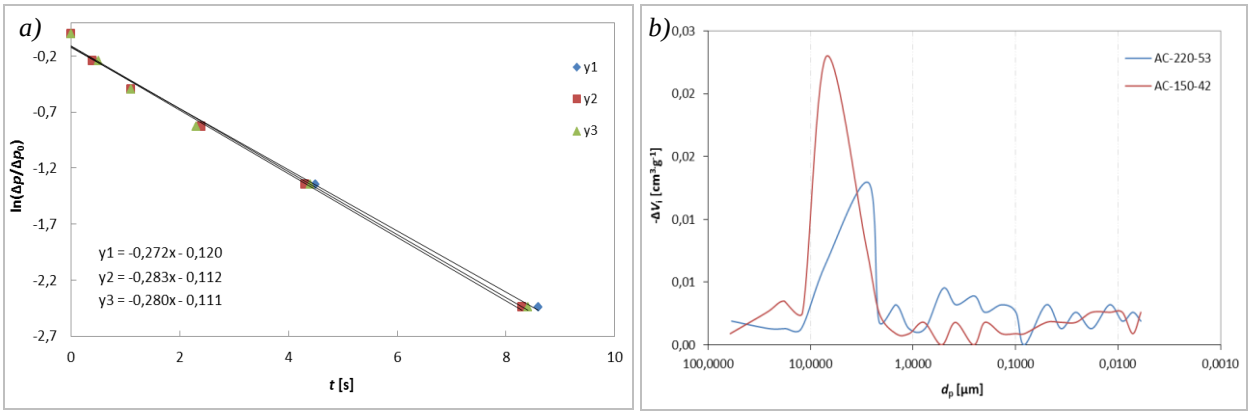

Fig. 2: a) Permeation measurement plot of sample AC-150-42 and b) incremental pore volume distribution of samples AC-220-53 and AC-150-42.

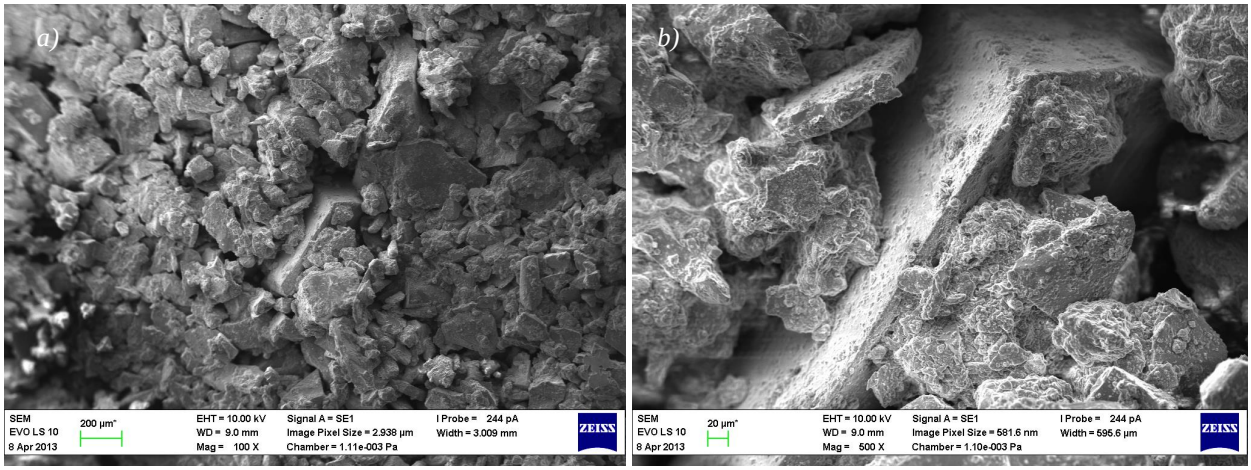

Fig. 3: Surface of sample AC-150-42 – SEM: a) Magnification: X 100; b) Magnification: X 500.

Tab. 1: Particle size of individual silicon carbide classes.

SiC	F40	F60	F100	F150	F220
***d* [μm]**	500 ~ 425	300 ~ 250	150 ~ 125	106 ~ 75	75 ~ 33

3. Conclusions

With the combination of SiC F220, particle-size distribution within 75 – 33 μm and a binder content of 53 % (water-cement ratio = 0.40), an apparent porosity of 18.80 % and average pore diameter of 0.0540 μm were attained. At the same time, the compressive and flexural strengths for this sample were 99.43 MPa and 11.39 MPa, respectively (measured after 28 days). Based on these mechanical properties, the obvious utility for these materials is in filtration. Average pore diameter is decreased due to the high binder content, which had to be increased (compared to the other mixtures) to preserve the rheological properties of the blend. However, the incremental pore volume distribution (Fig. 2a) shows higher increments in the pore diameter range between 10 to 1 μm, so there is a higher amount of pores with diameters in this range, but the smaller pores generally prevail.

The results of permeation measurement show permeability at a relatively high level. The highest value was $3.168 \cdot 10^{-7}$ $mol \cdot m^{-1} \cdot s^{-1} \cdot Pa^{-1}$. The diffusion coefficient for the same sample was 0.034 $cm^2 \cdot s^{-1}$, which means good penetrability of fluid through the material and thus low resistance to fluid flow.

The filtration barriers based on this material have the potential to replace sintered metal or ceramic membranes in certain applications, especially where the use of the sintered materials is too cost-prohibitive.

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