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Potential use of blast furnace slag for filtration membranes preparation: A pilot study

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Abstract. This paper investigates the possibilities of the use of sodium hydroxide activated blast furnace slag for preparation of filtration barriers. The motivation for this research is searching for cheaper and more eco-friendly alternative to sintering, which is commonly used for filtration membranes preparation. Such an alternative could be alkaline activation, which is able to bind slag particles together. Low amount of activator, low water to slag (w/s) ratio together with pressure compaction was used to obtain microstructure with preserved continuous porosity. To confirm this presumption scanning electron microscopy (SEM) and mercury intrusion porosimetry (MIP) analyses were carried out.

1. Introduction

Blast-furnace slag (BFS) is an industrial by-product from production of raw iron. It is used in blended cements and has been a subject of research focused on alkali-activated materials with an application in civil engineering [1, 2]. A lot of attempts to utilize secondary materials from energy (fly ash) and iron production (BFS) industry in advanced applications have been done. Several chemical engineering applications such as adsorption, membrane separations or hot gas cleaning have recently been of growing interest [3–6].

Membrane separations are important chemical engineering operations involving membrane, an active layer/barrier with selective properties. They have widely been used in air/wastewater treatment in tubular [7], flat [8] or hollow fibre geometry [9]. The membrane materials including polymers, ceramics or metals are chosen depending on the process conditions. Ceramic membranes are used mainly for harsh conditions where high temperatures and chemically aggressive environment is expected. Most of them are prepared of expensive materials via several steps one of which includes sintering at temperature around 1,000°C. Some of them may follow a route via hydrothermal reaction [10].

This work is an attempt to prepare a filtration membrane based on alkali-activated BFS without the additional sintering or hydrothermal treatment and thus via easier, cheaper and more eco-friendly process. Two different membranes differing in the initial BFS particle-size distribution were prepared and characterized in terms of pore-size distribution and observed using SEM.



2. Materials and methods

2.1. Materials and their characteristics

Filtration membranes were prepared via alkaline activation of ground granulated blast furnace slag (GGBFS) from Czech production (Kotouč Štramberk, Ltd.) by sodium hydroxide solution. Two series of specimens with different particle size distribution of slag were prepared. The first one contained GGBFS without any modifications, i.e. in the same form as it was obtained from supplier, while for the second series one narrower and coarser GGBFS fraction separated using an Alpine Windsichter ATP50 cyclone classifier was chosen.

Particle size distribution and morphology (Figure 1) of both types of GGBFS were determined using a Sympatec Helos KR laser diffraction analyzer and a ZEISS EVO LS 10 scanning electron microscope, respectively. Blaine fineness of unclassified GGBFS was about 400 m²/kg while its selected fraction had Blaine fineness of about 250 m²/kg. The phase composition was characterized using the X-ray diffraction method (XRD). Both types of GGBFS had the same phase composition containing about 88% of amorphous phase, 9% of akermanite and 3% of calcite. Activating solution consisted of 50% sodium hydroxide solution and demineralized water. Water to slag ratio was 0.095 including water from NaOH and Na₂O to slag ratio was 0.02. Such composition of the mixture resulted in a consistency like that of a wet powder.

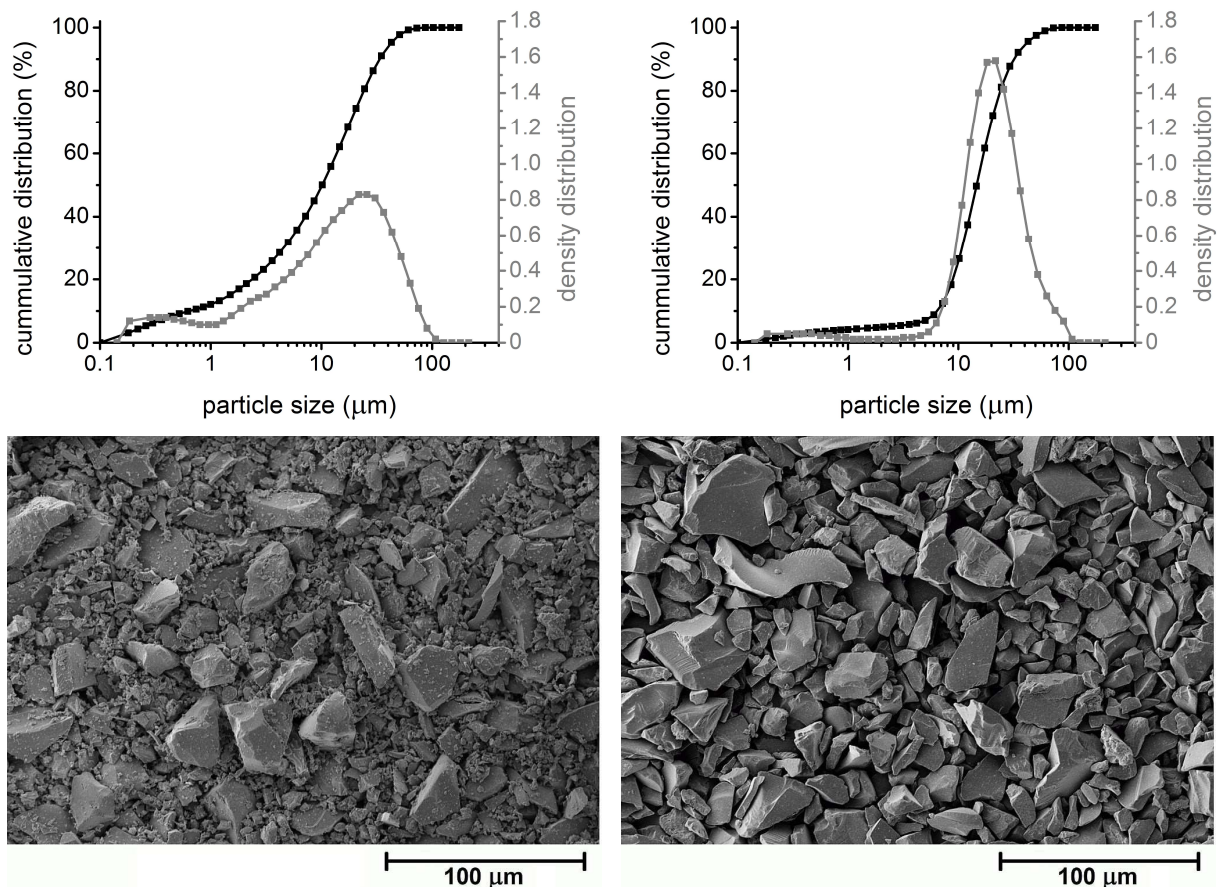


Figure 1. Particle size distribution and particle shape of the used unclassified (left) and classified (right) GGBFS; magnitude 1,000×

2.2. Specimen preparation

Mixture of GGBFS and activating solution was homogenized using a Thinky ARV-310 planetary centrifugal vacuum mixer for 30 seconds at a frequency of 2,000 rpm. Homogenized mixture was cast into round steel molds with a diameter of 25 mm and pressed at a pressure of 40 MPa. Thickness of prepared samples was 2 mm. The fresh samples were then moist cured at 60°C for 24 hours and dried at laboratory conditions. Examples of the final membranes are given in Figure 2.



Figure 2. Examples of the membranes prepared from GGBFS before classification (left) and after that (right). Membrane diameter is 25 mm.

2.3. Scanning electron microscopy

Microstructure of the prepared membranes was investigated using a ZEISS EVO LS 10 scanning electron microscope. Before the observation cross-section of a specimen was smoothed by a sand paper, polished using a JEOL IB09010CP ionic polisher and sputter coated with gold. Observation was carried out in the mode of secondary electrons (SE) at an accelerating voltage of 10 kV.

2.4. Mercury intrusion porosimetry

Pore structure of the prepared membranes was investigated using MIP. For these measurements specimens were abraded by sand paper to obtain small bars with a cross-section of approximately 5×2 mm and a length of 15 mm. Intrusion of mercury into the samples was performed at different pressures up to 225 MPa. Surface tension of mercury was assumed to be 480 mN/m and wetting angle 140° and thus according to Washburn equation the finest pores which were possible to detect had a diameter of 6.5 nm. Four measurements of each mixture composition were done.

3. Results and their discussion

Microstructure overview of the prepared membranes is given in Figure 3. It can be seen that membranes with both GGBFS types were successfully prepared regarding their preserved porosity. The reason for use of classified GGBFS was that membranes based on unclassified one contained a lot of places where potential larger pores were stuck by fine GGBFS particles and thus number of desired pores decreased. Moreover, microstructure of the membrane prepared from unclassified GGBFS is less homogenous compared to classified because many areas with both very low porosity and higher porosity can be found.

On the other hand increase in porosity is generally connected with decrease in strength and despite strength of membranes was not determined those prepared from classified GGBFS were more prone to

cracking during the sample preparation and easier to abrade. Therefore, further study will investigate the effect of increasing activator dose to prepare stronger membranes from various fractions of GGBFS.

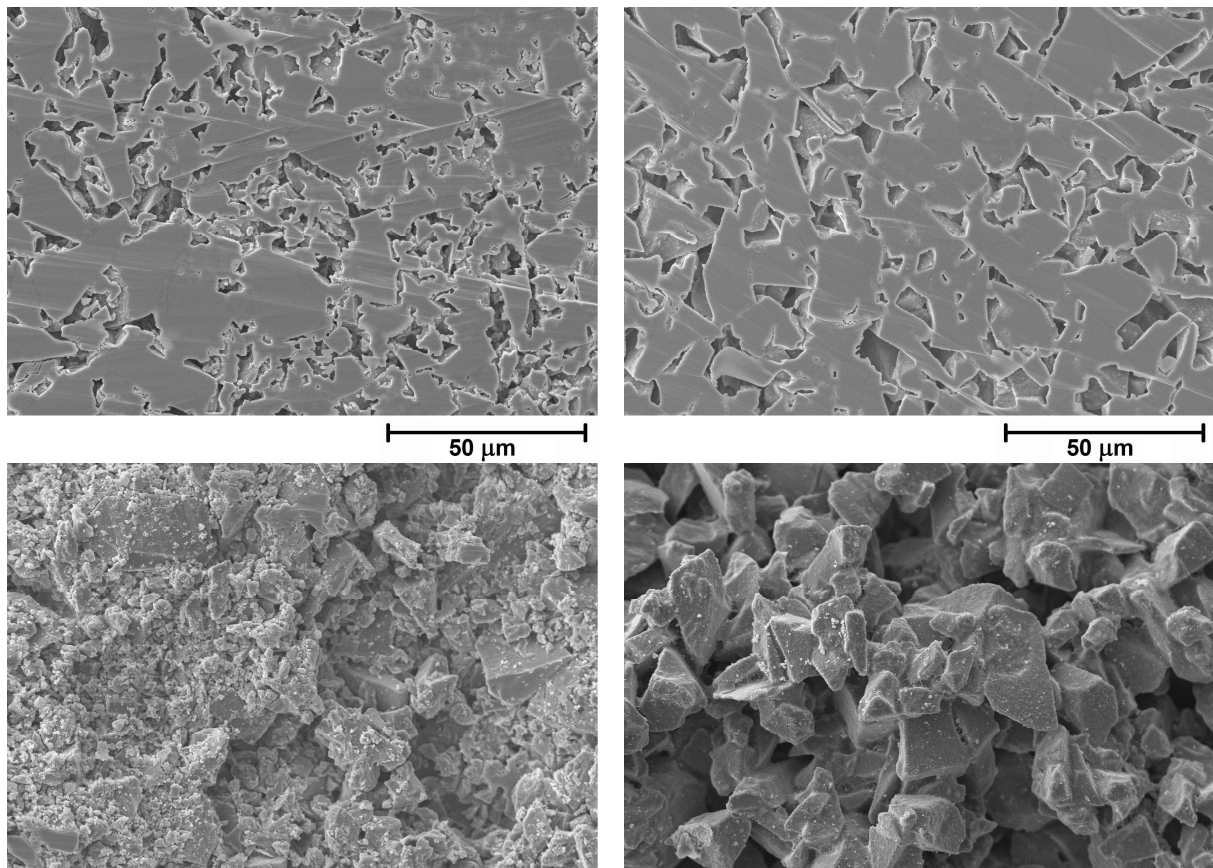


Figure 3. Microstructure of polished (up) and unpolished (down) parts of the membrane from unclassified (left) and classified (right) GGBFS; magnitude 2,000 \times .

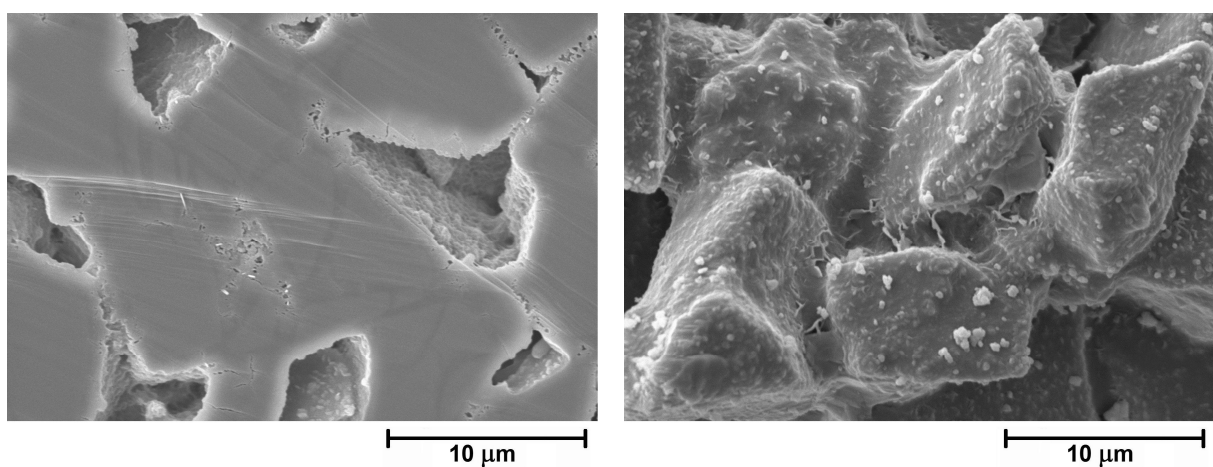


Figure 4. Detail of polished (left) and unpolished (right) part of the membrane from classified GGBFS; magnitude 10,000 \times .

The main binding phase formed during alkaline activation of slag is calcium-aluminium-silicate-hydrate (C-A-S-H) [11, 12]. Thank to this phase, GGBFS grains are connected together and form filtration membrane (Figure 4). Its amount is sufficiently high to facilitate compactness of the membrane but low to determine its composition, e.g. using energy dispersive analysis.

Impact of GGBFS classification on pore structure of the prepared membranes studied using MIP is shown in Figure 5. Both unclassified and classified GGBFS had significant intrusions in the range of pore diameter between 0.2 and 5–10 μm . While the curves of unclassified GGBFS were flat with very diffuse maximum in the range of 0.6–3 μm , for classified GGBFS new very intense sharp peak appeared, whose maximum corresponded to the pore diameter of 6 μm . Classification of GGBFS also resulted in significant total porosity increase from $23.8 \pm 0.4\%$ for unclassified slag to $37.8 \pm 2.0\%$ for the selected coarser fraction.

The main reason for such observations is that unclassified GGBFS contain particles of various sizes and thus finer particles can fill the free space between the coarser ones, which was also confirmed by SEM. General complication of GGBFS for membrane preparation is that its particles are angular. If they are in contact with each other along their edges or walls, continuous porosity can be reduced. More favorable particle shape compared to GGBFS has fly ash which may be an alternative. On the other hand, it contains lower amount of amorphous phase and has variable and inhomogeneous composition.

Due to the lack of results in literature, the determined properties of filtration membranes can hardly be compared to some others. Probably the most similar in terms of the used material and preparation process (but still noticeably different) were membranes prepared by Mohammadi [13]. Their porosity was determined to 45–65% which seems to be higher compared to our membranes from classified GGBFS, but it should be noted that the methods of porosity evaluation were different.

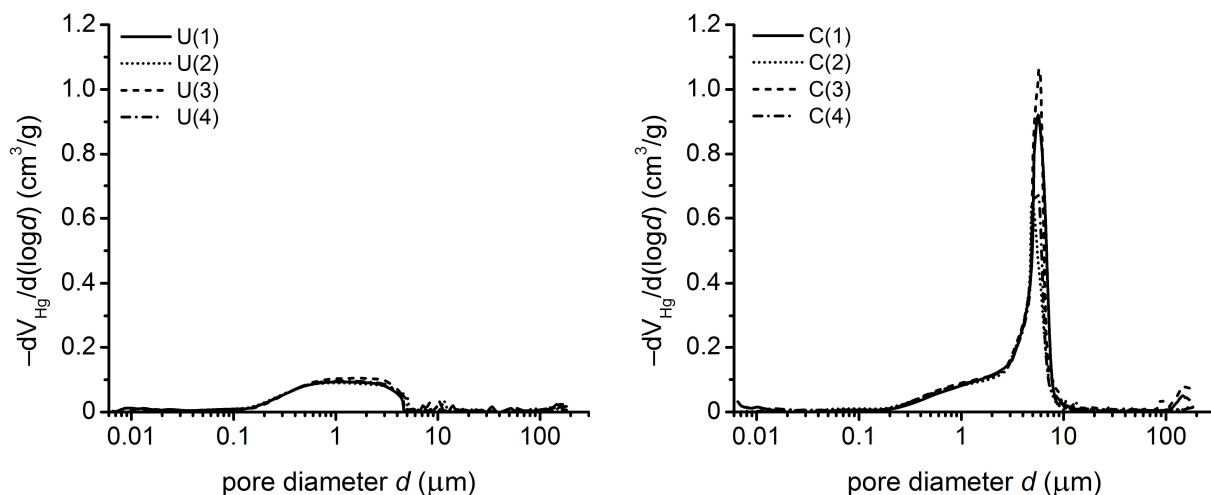


Figure 5. Pore structure of the prepared membranes with unclassified (U, left) and classified (C, right) GGBFS as determined using MIP.

4. Conclusion

This work investigated the possibilities of preparation of alkali-activated slag systems with preserved porosity which can be potentially used for preparation of filtration membranes. The results confirmed that alkaline activation is a possible way to bind the slag particles together at a low temperature compared to sintering. Furthermore, it was found that classification of the used GGBFS is one of important parameters affecting the final properties of the membrane. Membranes based on classified GGBFS had more homogenous microstructure with narrow distribution of the most abundant pore diameters and higher total porosity compared to those with unclassified GGBFS. Further studies will

be focused on the effect of activator dose, different fractions of classified GGBFS and leaching of soluble components on properties of the prepared membranes.

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