The Influence of Different Fibres Quantity on Mechanical and Microstructural Properties of Alkali-activated Foams

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Abstract Alkali activated foams (AAFs) were produced using electric arc furnace steel slag (EAF) and ladle furnace basic slag (LS), obtained from two metallurgical companies in Slovenia. They were activated with a mixture of sodium water glass (Na_2SiO_3) and solid NaOH and foamed with hydrogen peroxide (H2O2). Pores were stabilized with the addition of Triton as a surfactant. Four types of fibres were added to the studied mixture (polypropylene (PP), polyvinyl-alcohol (PVA), basalt (B), and glass wool (GW)) in five different quantities: 0.5, 1.0, 1.25, 1.5 and 2.0 vol % in order to additionally stabilize the structure and thus improve its mechanical properties. The results of mechanical properties showed, that compressive strength was increased in all 20 specimens, partially due to the increased density as well as to the fibre addition. Flexural strength on the other hand was the most improved in the samples where PP and PVA fibres were added. The samples with the addition of B and GW fibres on the other hand showed only small or no improvement in flexural strength in comparison to the referenced sample. Additionally, the microstructure of used fibres and selected foams was also investigated by the means of SEM analysis.

Keywords:
alkali activated materials, lightweight foams, fibres, porosity, mechanical properties

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1 Introduction

Alkali activated foams (AAFs) present a promising material to be used as an insulating and/or acoustic insulation material in the building and construction area. They are produced from different aluminosilicate sources (i.e. slags, fly-ashes, calcined clays) mixed with alkali activator (different water glasses, NaOH, KOH) and foaming agent (such as H_2O_2, metal powders) (Ducman & Korat, 2016). The main advantage of AAFs production is in the lower processing temperatures (up to 100 °C) required to achieve properties similar to traditional foamed glass or ceramics, both of which are produced at highly elevated temperatures (above 900 °C). Furthermore, given their low density, good thermal properties, fire resistance and applicable mechanical properties, AAFs offer an environmentally friendly alternative to ordinary insulation materials such as aerated cement concrete, mineral wool or EPS (Abdollahnejad *et al.,* 2017).

The resulting porous hardened structure contains air voids and could thus be very fragile, therefore the additional stabilization of the structure by the addition of different types of fibres represents one possible way to improve mechanical properties (Mastali *et al.,* 2018). The main tendency found in the literature is the use of locally available fibres from natural bio-resources such as hemp (Li *et al.,* 2004), bamboo (Chen *et al.,* 2018), wood (Lin *et al.,* 1994), cotton (Alomayri *et al.,* 2013), sisal (Almeida *et al.*, 2018) and others due to their renewable and recyclable nature. The second and also very important feature of the fibres used for the AAFs production is their stability with sustainability in a highly alkaline environment when exposed to high temperatures (Abdulkareem *et al.,* 2019). For example, cellulose-based bio fibres usually decompose at temperatures around 360 °C, therefore such foams would not be applicable in the field where high-temperature resistance is required. Hence, some studies in the field of alkali-activated materials' reinforcement are focused also on the use of steel (Rashad, 2020), basalt (Mastali *et al.*, 2018), or glass fibres (Puertas *et al.,* 2006), all of which should survive elevated temperatures. Good performance was achieved also with the use of synthetic fibres such as polypropylene (Wang & Tan, 2011) or polyvinyl alcohol (Nguyen *et al.,* 2020).

In the present study, the influence of different amounts (0.5, 1.0, 1.25, 1.5 and 2.0 vol %) of polypropylene (PP), polyvinyl-alcohol (PVA), basalt (B), and glass wool (GW) fibres on mechanical properties was investigated. Further, also the microstructural incorporation of these fibres into a foamed alkali-activated matrix was assessed by means of SEM analysis.

2 Experimental

2.1 Materials and AAFs preparation

Electric arc furnace steel slag (EAF, designated as Slag A) and ladle furnace basic slag (LS, designated as Slag R), obtained from two metallurgical companies in Slovenia were used as precursors with Slag A/Slag R ratio equals 1, as already discussed in our previous study (Češnovar *et al.*, 2019). Slags were activated with the mixture of 31.5 mass % of Na_2SiO_3 Crystal 0112, 54.2 % aqueous solution with $SiO_2/Na_2O = 1.97$, produced by Tennants distribution, Ltd. and 0.5 mass % of solid NaOH produced by Donau Chemie and foamed with 2.5 mass % of H_2O_2 (30 % aqueous solution, produced by Belinka, Slovenia). Pores were stabilized with the addition of 1.5 mass % of Triton (designated as T; Triton X-100, produced by Merck, Germany) as a surfactant. Four types of fibres were separately added to the studied mixture (polypropylene (PP), polyvinyl-alcohol (PVA), basalt (B), and glass wool (GW)) in five different quantities: 0.5, 1.0, 1.25, 1.5 and 2.0 vol %. For reference also non fibred foam was produced (designated as blank). The list of prepared mixtures with the corresponding designations is shown in Table 1.

The fresh foamed pastes were poured into 20 × 20 × 80 mm^3 moulds and cured at 70 °C for 3 days. Mechanical strengths were determined 3 days after curing at a temperature of 70 °C.

Table 1: Compositions of different AFFs prepared for the investigation; where PP = polypropylene, PVA = polyvinyl alcohol, B = basalt, and GW = glass wool fibres.

Sample designation		Slag A + Slag R [g]	Na_2SiO_3 [g]	NaOH [g]	H_2O_2 [g]	T [g]	Fibres [g]
Blank		192	96	1.5	7.5	4.5	/
PP	0.5 vol %	192	96	1.5	7.5	4.5	0.53
	1.0 vol %						1.06
	1.25 vol %						1.32
	1.5 vol %						1.59
	2.0 vol %						2.12
PVA	0.5 vol %	192	96	1.5	7.5	4.5	0.42
	1.0 vol %						0.84
	1.25 vol %						1.01
	1.5 vol %						1.26
	2.0 vol %						1.68
B	0.5 vol %	192	96	1.5	7.5	4.5	0.32
	1.0 vol %						0.66
	1.25 vol %						0.82
	1.5 vol %						0.98
	2.0 vol %						1.32
GW	0.5 vol %	192	96	1.5	7.5	4.5	0.32
	1.0 vol %						0.66
	1.25 vol %						0.82
	1.5 vol %						0.98
	2.0 vol %						1.32

2.2 Fibres

For this study, four different types of fibres were used as shown in Fig. 1. They are all of elongated shapes and comprised of different sizes (approximately 11, 12, 7 and 1 cm in length for PP, PVA, B and GW, respectively) and with theoretical densities (0.94, 1.19, 2.9 and 2.9 g/cm^3 for PP, PVA, B and GW, respectively) therefore different volumetric percentage (designated as vol %) of each fibre type was added to the mixture. Fibres were added to the activator solution and mixed to obtain as homogenous fibre distribution as possible.

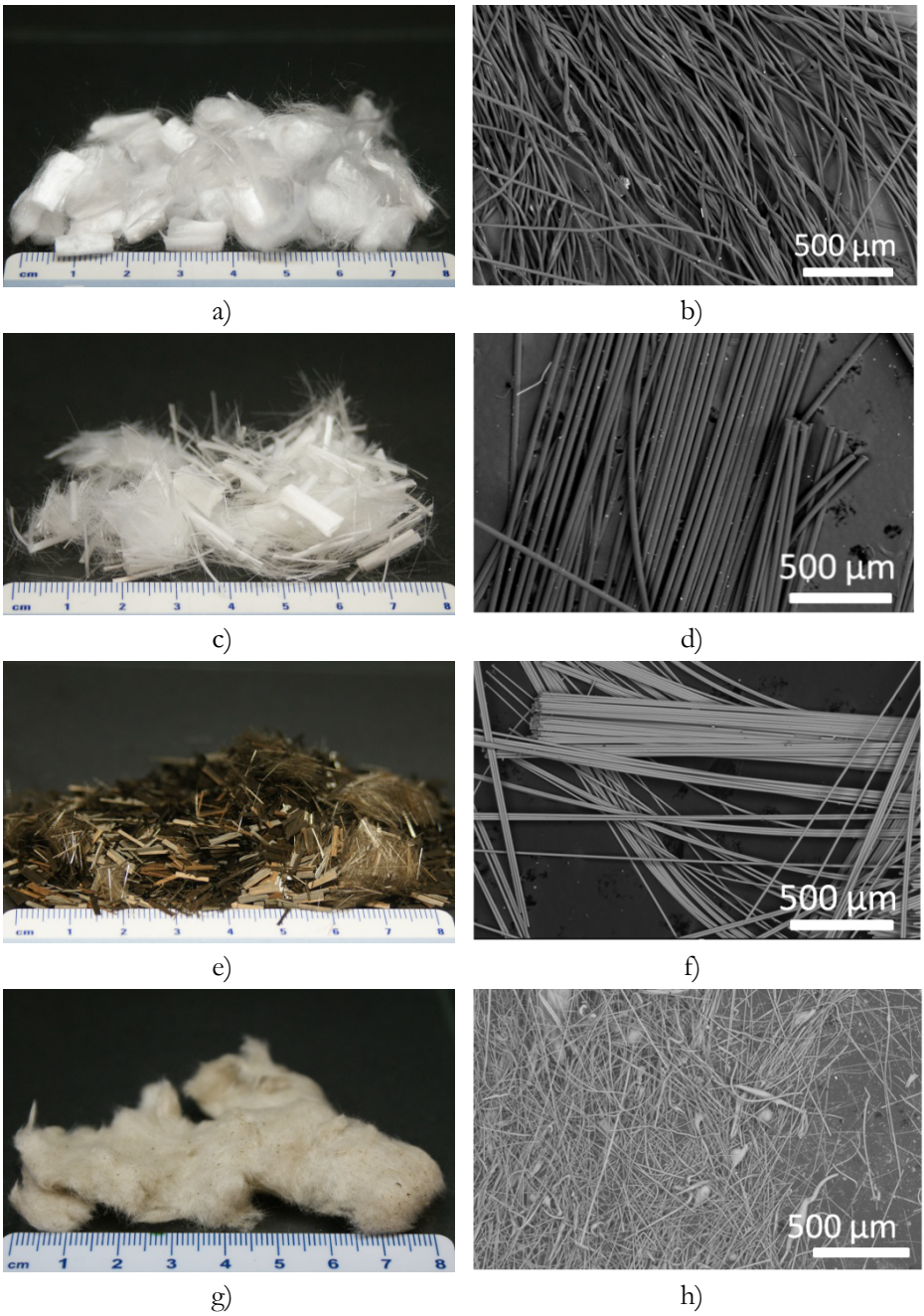

Figure 1: Macro and micro images of polypropylene (a, b), polyvinyl alcohol (c, d) basalt (e, f) and glass wool (g, h) fibres.

Source: own.

2.3 Characterization methods and instruments

The densities of all AAFs were determined by weighing the individual foams and dividing the thus determined weights by the corresponding dimensions of the specimens (i.e. so-called geometrical density).

Flexural strengths (σ_{FS}) and compressive strengths (σ_{CS}) were determined at an age of 3 days by means of a Toninorm test equipment (Toni Technik, Germany), using a force application rate of 0.005 kN/s and calculated according to equations (1) and (2).

$$\sigma_{FS} = \frac{3Fl}{2bd^2} \quad (1)$$

F – force
l – length between support span
b – width of sample
d – thickness of sample

$$\sigma_{CS} = \frac{F}{A} \quad (2)$$

F – force
A – area

The relative densities (rel. ϱ), as well as relative flexural and compressive strengths, were calculated, using the following equation (3).

$$\text{rel. } \varrho = \frac{(\varrho_0 - \varrho_x)}{\varrho_0} \quad (3)$$

ϱ_0 – density of the blank sample

ϱ_x – density of the studied fibres reinforced sample

Microstructural analysis of the fibres, as well as hardened AAFs, was performed by a JSM IT500, JEOL (Japan) with a tungsten filament cathode. Prior to SEM scanning, the samples were vacuum-dried and sputter-coated with gold.

3 Results and discussion

3.1 Mechanical properties

The absolute densities and mechanical properties of the produced AAFs are given in Table 2. Additionally, graphical presentations of the relative (with regard to the measurements of blank foam sample) densities and flexural as well as compressive strengths are shown in Fig. 2. It can be observed, that foam with no added fibres exhibit a relatively low density of 0.39 g/cm^3 and is thus also very fragile with compressive strength of only 0.21 MPa and flexural strength below detection limit. With the addition of fibres the densities are increased in most cases (with the exceptions in specimens 1.25 vol % PVA, 1.25 vol % B, 1.25 vol % GW, and 0.5 vol % B) and are in the range between 0.35 and 0.55 g/cm^3. As expected the foams with the highest density – specimens 1.0 vol % PP, 2.0 vol % PP and 1.0 vol. % PVA, exhibit also the highest compressive strengths (1.24, 1.31 and 1.20 MPa, respectively). These three specimens also showed the best development of flexural strength (0.62, 0.54 and 0.41 MPa, respectively) which could partially be assigned to the density gain and partially to the reinforcement as a consequence of fibres addition. From Fig. 2 could also be observed that all specimens regardless of the fibres type addition showed better compressive strengths. Interestingly, only 26 % density gain for specimen 2.0 vol % PP resulted in 3.3-times higher compressive strength. Further, specimens with PP and PVA addition also exhibit improved flexural strength. The samples with the addition of B and GW fibres on the other hand showed only small or no improvement in flexural strength in comparison to the referenced sample (specimen with no added fibres). Additionally, from this study, some logical correlation between fibres quantity and mechanical strength improvement was not proved, since the maximum flexural strength was reached with the addition of 1.0 vol % of PP and 2.0 vol % of PVA fibres.

Table 2: A list of the absolute densities (ϱ), flexural strengths (σFS), and compressive strengths (σCS) of all samples (n. d. = below detection limit).

		ϱ [g/cm³]	σ_{FS} [MPa]	σ_{CS} [MPa]
Blank		0.39	n. d.	0.21
PP	0.5 vol %	0.44	0.15	0.57
	1.0 vol %	0.55	0.62	1.24
	1.25 vol %	0.43	0.43	0.52
	1.5 vol %	0.42	0.34	0.41
	2.0 vol %	0.49	0.54	1.31
PVA	0.5 vol %	0.39	0.02	0.50
	1.0 vol %	0.50	0.41	1.20
	1.25 vol %	0.38	0.09	0.34
	1.5 vol %	0.41	0.13	0.36
	2.0 vol %	0.45	0.47	0.71
B	0.5 vol %	0.38	n. d.	0.39
	1.0 vol %	0.48	n. d.	0.75
	1.25 vol %	0.35	0.02	0.30
	1.5 vol %	0.40	n. d.	0.29
	2.0 vol %	0.43	0.04	0.26
GW	0.5 vol %	0.40	0.01	0.71
	1.0 vol %	0.47	n. d.	0.85
	1.25 vol %	0.35	0.01	0.41
	1.5 vol %	0.40	n. d.	0.39
	2.0 vol %	0.45	0.03	0.58

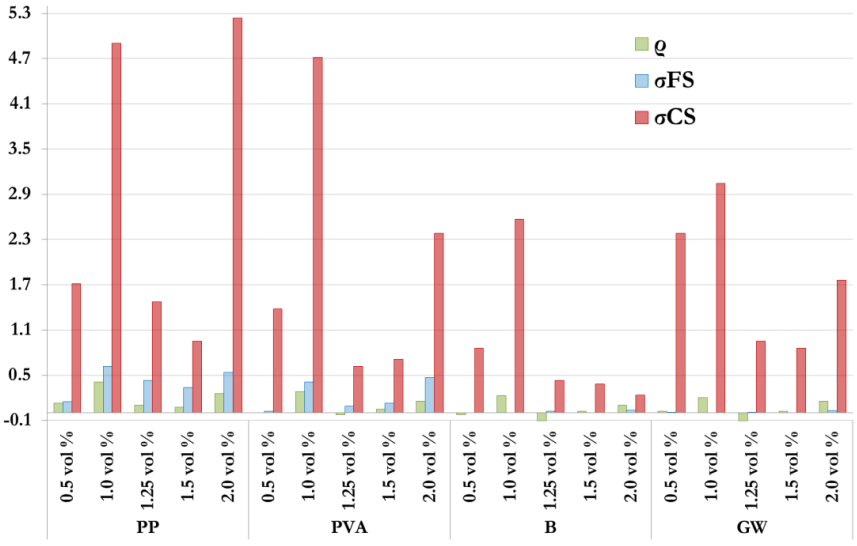

Figure 2: Graphical representation of the relative (with regard to the blank sample) densities (ϱ), flexural strengths (σ_{FS}), and compressive strengths (σ_{CS}).

Source: own.

3.2 Microstructural evaluation

The microstructures of AAFs with different fibres (in all cases the quantity of 2 vol %) and at different magnifications are presented in Figs. 3–6. SEM micrographs of PP fibre-reinforced AAFs are presented in Fig. 3. Fig. 3a shows the evenly distributed PP fibres in the alkali-activated matrix whereas Figs. 3b and 3c show a more detailed view of the boundary between the fibre and the matrix.

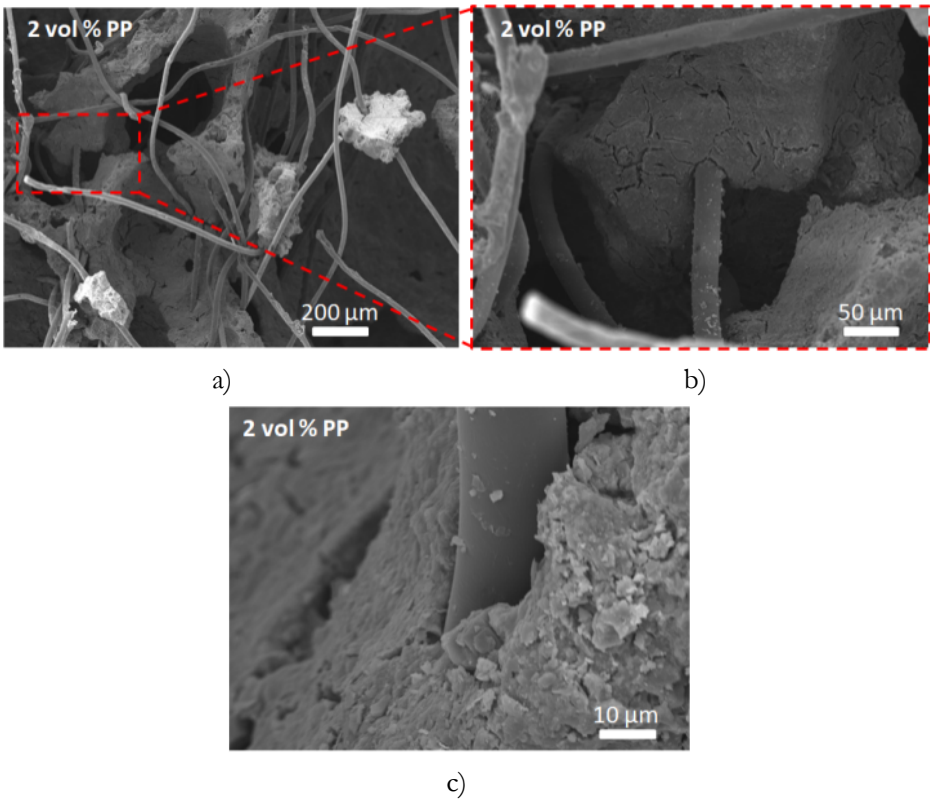

Figure 3: SEM micrographs of polypropylene reinforced AAFs (2 vol %) at different magnifications: a) PP distribution in the matrix; b) and c) boundary between a PP fibre and the matrix.
Source: own.

Similarly, also the distribution of PVA fibres is uniform in the matrix as shown in Fig. 4a and the boundary between the fibre and the matrix is presented in Fig. 4b. Additionally, as observed in Fig. 4c, PVA fibres seem to be less resistant to the highly alkaline environment and/or foaming agent in the fresh alkali-activated slurry,

therefore damages on the fibres may occur as shown in Fig. 4c. However, these damages could also contribute to the better adhesion of the fibres to the matrix resulting in better mechanical properties. After all, specimen 1.0 vol % PVA reaches one of the highest compressive and flexural strengths among studied ones.

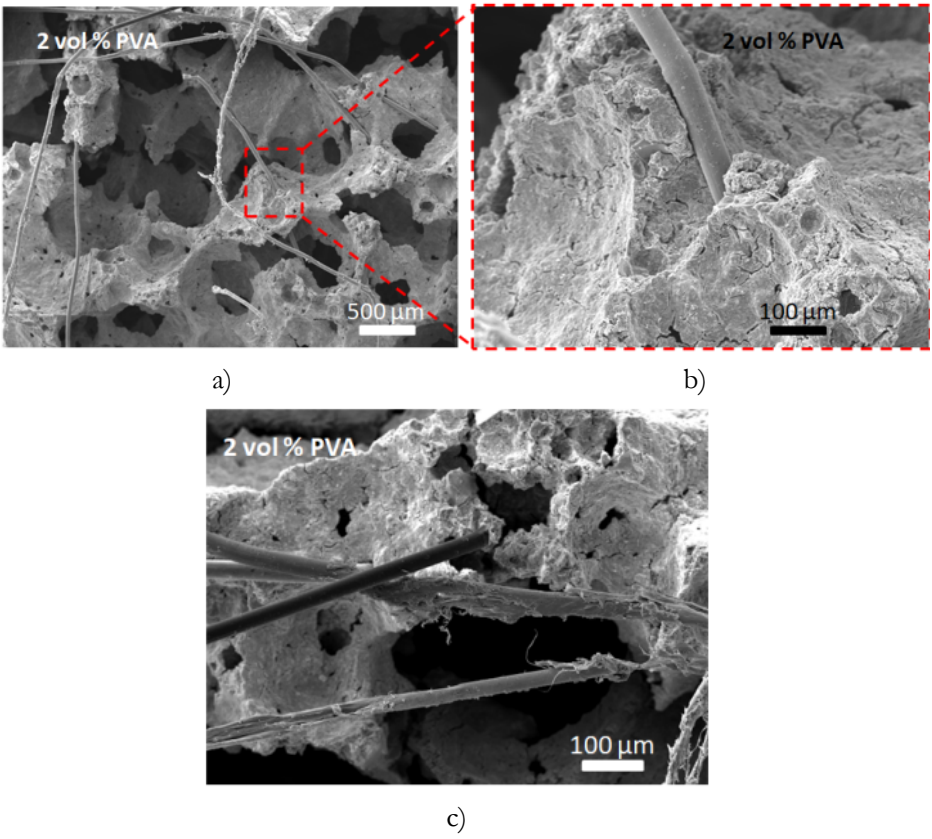

Figure 4: SEM micrographs of polyvinyl alcohol reinforced AAFs (2 vol %) at different magnifications: a) PVA distribution in the matrix; b) boundary between a PVA fibre and the matrix; c) damaged PVA fibres.

Source: own.

Fig. 5a shows the distribution of B fibres in the alkali-activated matrix. Their distribution is also quite uniform. Due to the chemical composition of the basalt fibres (alumino-silicate composition, similar to precursors) these fibres could also react with the alkaline activator and the spots seen on Fig. 5c could be a consequence of these reaction. Similar observations on SEM micrographs of B fibres after

exposure to NaOH solution were detected also by Elshafie & Whittleston (Elshafie & Whittleston, 2016). However, probably due to the more fragile nature of the B fibre, the reinforcement was not as successful as in the case of PP and PVA fibres.

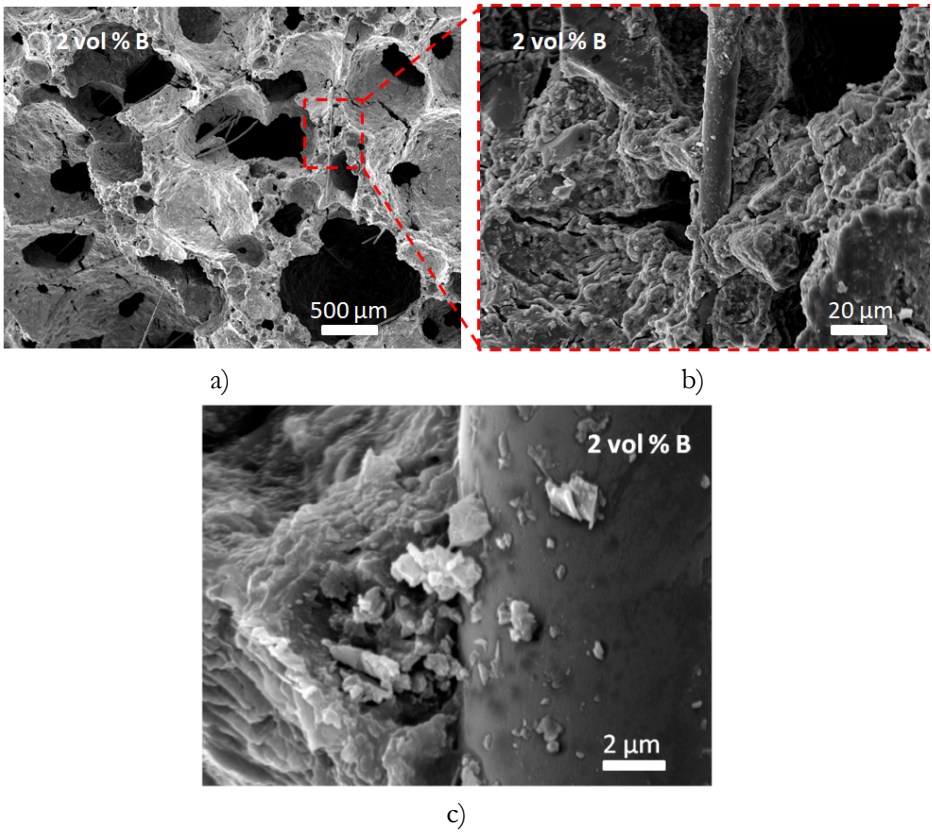

Figure 5: SEM micrographs of basalt reinforced AAFs (2 vol %) at different magnifications: a) B distribution in the matrix; b) and c) boundary between a B fibre and the matrix.
Source: own.

GW fibres are the smallest among the studied ones, therefore they are barely observed in the alkali-activated matrix as shown in Fig. 6a. Regarding to their chemical composition they are comparable to the B fibres. The advantage of such smaller fibres could be firstly in their better incorporation into the alkali-activated matrix (Figs. 6b and 6c) especially in cell and void boundaries and secondly, due to their glassy phase the dissolution in alkalis is also possible. But for the flexural strength improvement probably much higher concentrations should de added.

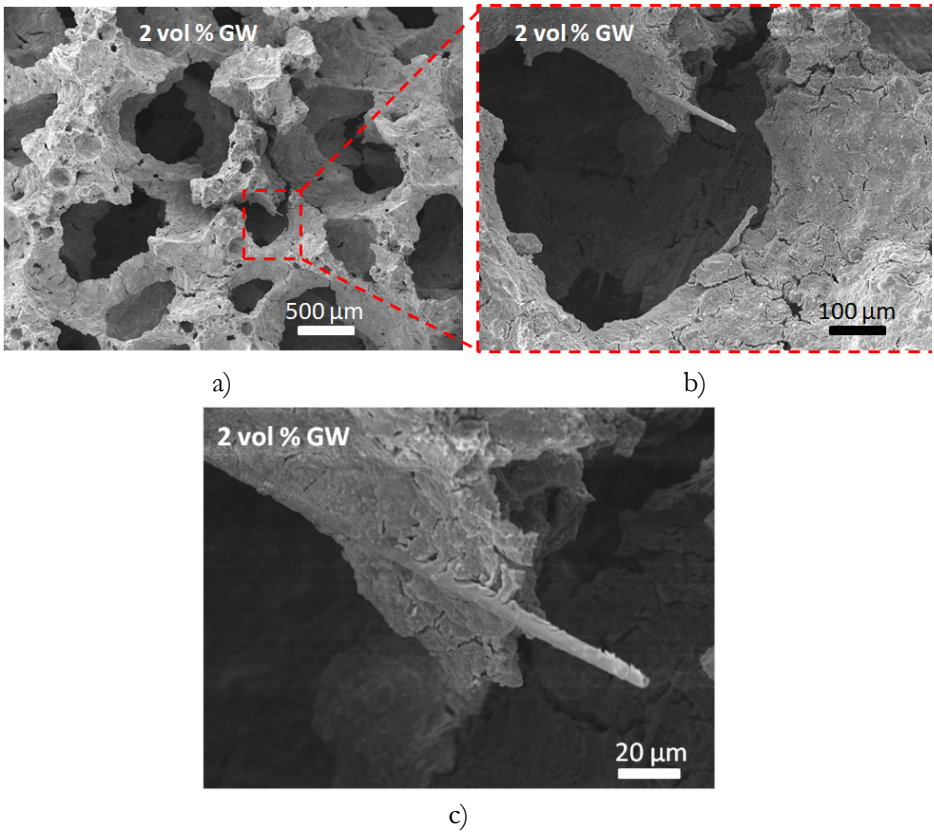

Figure 6: SEM micrographs of glass wool reinforced AAFs (2 vol %) at different magnifications: a) GW distribution in the matrix; b) and c) boundary between a GW fibre and the matrix.
Source: own.

4 Conclusions

With the aim of the mechanical properties improvement, four types of fibres (polypropylene (PP), polyvinyl-alcohol (PVA), basalt (B), and glass wool (GW)) in five different quantities (0.5, 1.0, 1.25, 1.5 and 2.0 vol %) were added to the selected alkali-activated foamed mixture. The results of mechanical properties showed, that compressive strength was increased in all 20 specimens, partially due to the increased density, which is a consequence of fibre addition. Flexural strength on the other hand was the most improved in the samples where PP and PVA fibres were added. The samples with the addition of B and GW fibres showed only small or no improvement in flexural strength in comparison to the referenced sample (specimen

with no added fibres). However, some logical correlation between fibres quantity and mechanical strength improvement was not proved in this study. The best performance in terms of mechanical properties was achieved in the case of the specimen where 2.0 vol % of PP fibres was added. SEM micrographs revealed, that in most cases the fibres are uniformly distributed. In the case of PVA fibres also some damaged fibres were observed which could contribute to better adhesion and therefore improved mechanical properties.

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