Original article

INFLUENCE OF COMBUSTION TEMPERATURE ON THE PERFORMANCE OF SEWAGE SLUDGE ASH AS A SUPPLEMENTARY CEMENTITIOUS MATERIAL

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Abstract

The potential for using sewage sludge ash (SSA) as a supplemenatry cementitious material (SCM) has been investigated. Controlled combustion of sewage sludge collected in Croatia from two wastewater treatment plants (WWTP) produced SSA with different characteristics. These were used to substitute for cement in mortar samples. The chemical composition and physical properties of SSA depends on wastewater composition, the sludge treatment process and the combustion temperature. These factors influence the suitability of SSA to be used as a SCM. For three different combustion temperatures (800°C, 900°C and 1000°C), it was concluded that properties of fresh mortar were not affected while in the hardened state, the most favorable combustion temperature is 900°C regarding mechanical properties. Regardless of combustion temperature, for all types of SSA used in mortars as cement replacement (up to 30%), the average decrease in both, compressive and flexural strength values was less than 8% for every 10% of added SSA. The results presented indicate that using up to 20% replacement of cement by SSA produces mortars that meet the specific technical requirements analyzed in this work.

Keywords: sewage sludge ash, waste utilization, cement mortar, strength, workability

1. Introduction

Wastewater treatment has become an important global issue during the last 20-30 years. This is particularly so for Croatia where the number of wastewater treatment plants (WWTP) is increasing to meet EU requirements [1]. WWTP produce new problems associated with managing the considerable quantities of sludge generated. The current situation in Croatia is that the majority of sewage sludge is disposed of in landfills, with only a very small proportion being used in agriculture. Until recently sludge management was not a major issue, but now the selection of the optimum sludge treatment and the final disposal must be taken into consideration prior to construction of the WWTP.

Sludge incineration is a potential management option for sewage sludge in Croatia. This reduces the mass by approximately 70% mass and the volume by 90%, and produces a residual sewage sludge ash (SSA) [2]. The combustion process destroys hazardous organic components in the sludge and minimises odour problems [3]. Approximately 11.5 Mt dry mass of sewage sludge is produced per annum in the EU, and this is expected to increase to 13 Mt dry mass by 2020 [4]. Currently ~22 % of EU sewage sludge is incinerated [2]. There are currently no sludge incineration plants in Croatia, but the draft version of the National strategy for sewage sludge disposal involves construction of four major sludge incineration plants, located near the four largest cities, Zagreb, Split, Rijeka and Osijek.

The EU Directive 91/271/EEC requires that sludge management meets the requirements of efficient recycling of resources without contaminating the environment with harmful substances. Landfill is not considered a sustainable solution for sewage sludge or SSA, and previous research has investigated use in other applications such as construction materials [5].

Although industrial wastes may be incorporated in cementitious materials by various traditional methods, the proportion is kept relatively low, typically < 20% [6]. SSA may be used as a supplementary cementitious material (SCM) because it can be pozzolanic [6-16]. It also has potential as an inert filler, replacing or partly replacing sand and/or fine aggregate, and as a raw material for the production of lightweight aggregate [17-23].

The main oxides in SSA are SiO_2, Al_2O_3 and CaO, while Fe_2O_3, Na_2O, MgO, P_2O_5, SO_3 and others are present at lower levels [2, 16]. Pretreatment of SSA to extract phosphorus prior to use in cementitious materials is a viable option, given the potential nutrient value [2, 24]. The amorphous content of SSA ranges from 35-75% depending on composition and thermal treatment. Typical particle sizes range from 1 to 100 μm, with a mean of ~26 μm [25, 26].

The addition of SSA to cement mortars causes a decrease in workability [11, 13, 16, 27], an increase in setting times [2, 12, 16, 28], an increase in porosity [16, 29, 30] and a reduction in flexural and compressive strength [1, 6, 10, 12, 13, 15, 16]. At high SSA additions the water requirement of cement mixes increases which also reduces strength [13]. However, mortars containing SSA have also been shown to have equivalent or even improved strengths compared to control samples [1, 29]. This demonstrates that results are influenced by the sludge origin, sludge treatment method and the conditions used to generate the SSA [15]. The research reported in this paper has specifically investigated the influence of sludge type and combustion temperature on the properties of cement mortars containing SSA as a SCM.

2. Materials and methods

2.1 Origin of the sewage sludges

Sewage sludge was collected from two different WWTPs in Croatia.

The Karlovac WWTP operates 3rd stage treatment using conventional activated sludge technology. The maximum capacity is 98,500 population equivalents (PE), and it is currently working at about 70% capacity. Sludge treatment involves dehydration and anaerobic stabilization with lime addition, after which the sludge is stored at the plant before being landfilled. In this research four month old dehydrated and stabilized sludge was used.

The Zagreb WWTP (1,200,000 PE) operates with 2nd stage treatment and uses conventional activated sludge technology. Sludge treatment is based on dehydration and anaerobic stabilization with some lime addition. A schematic diagram showing the processing and where the sludge is generated at the three WWTP is given in Fig. 1. The main difference between the two WWTP is that the WWTP Karlovac uses additional methods to reduce nutrient salts of nitrogen and phosphorus. This is achieved by process modifications inside the aeration tank that include nitrification and denitrification, and also the addition of salts of aluminum and iron to precipitate phosphorus.

Fig. 1 Schematic diagram of WWTP processing showing where the sludge is generated

The collected sludge samples were dried at 105°C for 24-36 hours to constant mass and a dry matter (DM) content of ~90%. Dried sludge samples were then heated to 800°C, 900°C and 1000°C for either 2.5 (900°C and 1000°C) or 3 hours (800°C) using a laboratory electric muffle furnace. The resulting ash samples were lightly ground to form granular materials suitable for testing. This was done to obtain SSA of similar characteristics as the ones that could be expected from modern real scale incinerators, and was necessary as no incinerators were built in Croatia. Also, similar methodlogy was previously used by several authors [3, 11, 29, 31, 32]. This approach is also described in detail by Vouk et al. [33] in the paper that represents the initial part of this study, and where only SSA obtained by incineration of sewage sludge from WWTP Zagreb at 900°C was analyzed. By comparing the results of physical and chemical characteristics of the obtained SSA with tests on the real-scale generated SSA within previously published research, it can be concluded that produced ashes were mostly of similar characteristics so this approach can be considered relevant. Possible minor deviations could be expected in the chemical and crystaline composition and morphology of the SSA particles, but not expected to such an extent that they would significantly affect the obtained results [33].

2.2 Ash characterisation

The density of the SSA samples was determined using the method specified for determining the density of cement (ASTM C-188). This was required to calculate mortar compositions.

The chemical composition of ash samples was determined by inductively coupled plasma optical emission spectrometry (ICP-OES) using the method HRN EN ISO 11885:2010.

The particle size distribution was determined using air jet seiving (A058-05N) in accordance with HRN EN 933-10:2009 for the classification of fillers.

2.3 Preparation of mortar samples

Portland cement containing slag and limestone (CEM II/B-M (S-V) 42.5N) and dolomite sand (0/4mm) were used to prepare mortars with water-binder ratios of 0.45, 0.50 and 0.55. Ordinary tap water at room temperature was used. Samples were prepared with different amounts of SSA partialy substituting for cement (0-30%). The samples were thoroughly mixed for 4 minutes.

2.4 Experimental program

Prior to the preparation of the specimens, the following tests were performed on the fresh mortars:

The temperature of mortars was determined according to HRN U.M1.032 1981 using a pinhole digital thermometer. The thermometer needle was inserted in the middle of the fresh mortar mix just after the mixing was finshed (prior to embeding in the moulds).

The density of fresh mortar was determined from the mass and volume occupied in accordance with HRN EN 1015-6:2000/A1:2008.

The workability of fresh mortar samples was determined using standard cone samples on a flow table using 15 drops. Flow table spread (FTS) was calculated from the average value of the maximum and minimum diameters of the spread cone in accordance with HRN EN 1015-3:2000/A2:2008.

The air content of fresh mortar was determined using the "pressure method" following HRN EN 1015-7:2000. Mortar samples were placed in the special vessel and water was then poured on top of the mortar and the vessel closed. Pressure was then applied frocing the water to fill the pores of the mortar, displacing air. Releasing the valve that separates the chambers allows the pressure decrease to be recorded and the percentage porosity to be calculated.

Specimens for strength testing were prepared as 4 cm x 4 cm x 16 cm prisms. Nine specimens were made from each mix using steel moulds, with three tested at each curing age (1, 7 and 28 days). Specimens for gas permeability testing were 100 mm diameter and 160 mm high cylinders. All the specimens were demoulded after 24 hours and cured in a humidity chamber (relative humidity >95%, temperature 20±2°C). Mechanical tests were performed according to HRN EN 1015-11:2000/A1:2008. Mortar samples were first tested in bending and then the two resulting pieces were tested in compression.

The gas permeability tests were performed on three specimens for each mix. Specimens were oven dried for 24 hours and tested in accordance to RILEM Cembureau method [34] to give gas permeability coefficients.

3. Results and discussion

3.1 Physical and chemical characteristics of SSA

The different SSA samples varied significantly in colour with SSA generated using sludge from WWTP in Karlovac producing a dark grey powder and the one from the WWTP Zagreb was light brown. Chemical composition (oxide % data) of SSA samples produced at different combustion temperatures is given in Table 1.

Table 1 Mean values of shares of significant individual oxides in SSA [mass. %]

The density of SSA samples produced at different temperatures is given in Table 2.

Table 2 Density of different SSA samples [g/cm^3]

Mass loss during incineration was slightly higher (2-3%) for the sludge from WWTP Zagreb. As the combustion temperature increased the mass reduction of sludge increased by about 2% per 100°C. Average mass reduction at combustion temperature of 800°C was about 46%, at 900°C about 48% and it was about 50% at 1000°C.

X-ray diffraction (XRD) data for SSA samples was obtained from the WWTP Karlovac and WWTP Zagreb sludge at three different combustion temperatures (Fig. 2). Results show the significant presence of an amorphous phase and of many crystalline minerals, the main ones being quartz and cristobalite (SiO_2), muscovite, anhydrite ($CaSO_4$) and calcite ($CaCO_3$) for samples from WWTP Karlovac and quartz, lime, anhydrite, calcite and portlandite for samples from WWTP Zagreb. Increasing the combustion temperature thermally decomposed calcite and at 1000°C it had almost completely disappeared. Following the degradation of calcite, a transitional phase of calcium oxide occurs at 900°C, while at the other processing temperatures this was not observed. The intensity of the maximum peak for muscovite falls significantly at 1000°C, indicating advanced degradation at this stage. The intensity of maximum peak for calcium magnesium phosphate increases with increasing combustion temperature, which is consistent with the decomposition of calcite. Based on the stability of phases one might assume that at 900°C sample decomposition is significantly more than at 800°C. At 1000°C, some phases are already formed that are not interesting for the hydration of cementitious materials. It seems that 900°C may be the optimal temperature in terms of quality of ash for use in cementitious materials.

Fig. 2 XRD diffractogram and crystalline composition of the SSA samples obtained by incineration of sewage sludge from different WWTP at different temperatures:

a) Ka_800°C, b) Zg_800°C, c) Ka_900°C, d) Zg_900°C, e) Ka_1000°C and f) Zg_1000°C

All the SSA samples produced at different temperatures had similar morphology. Polydisperse grains are observed and irregularly shaped particles are present with a wide range of particle size distribution and with significant diversities in the form for samples obtained at different temperatures. With increasing combustion temperature reduces the particle size, most likely due to further decomposition. Increasing the combustion temperature also changes the level of agglomeration. It is noticeable (Fig. 3) that the sample obtained at 800°C has the largest agglomerates and at 900°C these agglomerates break. Sample obtained at 1000°C, on the other hand has the smallest particles, but it seems that those also tend to agglomerate due to the formation of the new phases and due to temperatures sufficient for sintering.

When looking at the micrographs of SSA from different WWTP obtained at the same temperature, it can be concluded that SSA sample from the WWTP in Zagreb has somewhat smaller particles which are more agglomerated with less porosity.

Fig. 3 SEM micrographs of the SSA samples obtained by incineration of sewage sludge from the WWTP Karlovac at a) 800°C b) 900°C and c) 1000°C

3.2 Particle size distribution

SSA has a low organic and moisture content. The results, based on 6 samples of analyzed SSA, showed that there are no significant differences in particle size distribution relative to the combustion temperature. Slightly larger differences are present when comparing SSA from different origins (different WWTP). For SSA obtained from the sludge from WWTPs in Croatia, most of the particles are between 20 and 63 µm.

The particle size of SSA ranges between 5 and 500 µm (Fig. 4), with a mean diameter around 50 µm. A relatively small fraction of the particles (less than 2%) are less than 10 µm (Fig. 3). The range of most particles size (up to 80%) of analyzed SSA coincides with a range of ash particles found in previous research (1-100 µm) [25, 26].

Fig. 4 Mean values of particle size distribution for analyzed SSA obtained at different incineration temperatures

According to the results it can be concluded that the obtained SSA, in terms of particle size, can be classified as silt with a significant proportion of particles with the size of sand.

3.3 Effect of SSA on properties of fresh mortars

There was no segregation or bleeding in any of the mixes containing SSA. All mixes showed compliance with reference values with regard to density of fresh mortar and minimum differences between mixes were observed. Density in fresh condition were between 2.25-2.35 g/cm^3 which is in line with the density of reference mixes.

With the addition of the SSA (even in small percentages) a slight increase in the temperature of the fresh mortar was observed, ~1-5°C increase compared to reference mixtures. The trend of increasing temperature with addition of SSA becomes less noticeable for larger additions and the highest temperatures were observed in the mixes with 20% SSA content, and not those with 30% of SSA. Higher reactivity of mixes containing SSA could be attributed to somewhat higher CaO content in the SSA.

Except for low SSA content mixes, there was an increase in water demand of mortars containing SSA. The water demand of SSA is related to the high specific surface area of the SSA particles, which are mainly composed of small agglomerates. Mixes containing higher amounts of SSA showed significantly lower flow table spread (FTS) values. It can be concluded that mortar workability decreases with increasing SSA for all mixes.

Fig. 5 Flow table spread (FTS) of fresh mortar mixes containing different proportions of SSA

Although significant differences in terms of workability for mixes containing SSA obtained at different temperatures were not observed, mixture containing SSA obtained at 900°C showed a slightly larger decline in workability compared to mixes containing SSA formed at the other two temperatures. Generally, the decline in workability is nearly linear, and the avearge rate of decrease in workability is calculated to be 10% for every 10% of SSA (Fig. 5).

The air content of fresh mortar increased roughly linearly with increasing content of SSA. It was found to increase an average of about 0.45% with the addition of 10% of SSA (Fig. 6). There were no significant differences in terms of air content when comparing mixes containing SSA obtained at different combustion temperatures.

Fig. 6 Air content of fresh mortar mixes depending on the proportion of added SSA

3.4 Effect of SSA on properties of hardened mortars

3.4.1 Mechanical properties

Mechanical property test data indicates that, although relatively high strengths are obtained, the increased replacement of cement with SSA led, for all ages of mortar, to a decrease in compressive and flexural strengths.

Furthermore, when using SSA, both 1-day flexural and compressive strengths are lower on average by 10 - 35 % compared to the reference mix (depending also on SSA content). However, the gaps between SSA and reference mortars (in relative comparison, i.e. in percentages) reduced significantly over time (Fig. 7), especially for 7-day results. After 7 days, there were no significant differences between the control mix and mixes with up to 20% replacements of cement by SSA. At the age of 28 days, compressive and flexural strengths are lower on average by only 7 - 12% compared to the reference mix. This could be attributed to a minor pozzolanic effect as reported by other authors [12] and it leads to a conclusion that the short-term negative effect of SSA is significantly reduced by 7 days.

Fig. 7 Effect of curing time on the compressive strength of mortars with 20% of SSA as a substitute for cement

The rate at which SSA mortars gain strength is similar to that for mortars containing coal fly ash, although SSA does contribute to strength development more rapidly. The formation of hydraulic products from the pozzolanic reaction between SSA and cement takes place mainly during medium curing times, suggesting that SSA can be considered as a medium-reactive pozzolan [10].

Considering all the samples that have been tested (Fig. 8 and Fig. 9), the highest 28-days compressive strength was obtained for the sample with 5% SSA obtained at 900°C using the sludge from WWTP Karlovac. This reached 105% of the compressive strength of the reference mix.

The highest flexural strength was obtained for the sample with 5% SSA obtained at 800°C by incineration of the sludge from WWTP Zagreb. This had 113% of the flexural strength of the reference mixture. The lowest 28-days strength values were recorded for samples with 30% of SSA obtained by incineration of sludge from WWTP Zagreb at 800°C for both compressive and flexural strength.

The lowest compressive strength was 66% of the compressive strength of the reference mixture, while the lowest flexural strength was only 60% of the flexural strength of the reference mixture. Overall, the average decrease of compressive strength was less than 10% with the addition of 20% of SSA. The average decrease of flexural strength, with the same SSA proportion, was ~12%.

Fig. 8 Compressive strengths for age of 28 days depending on the proportion and the type of added SSA

Fig. 9 Flexural strengths for age of 28 days depending on the proportion and the type of added SSA

Although the results show considerable diversity (Fig. 8 and Fig. 9), if grouped by different combustion temperatures to determine the mean strengths (compressive and flexural) for individual temperature the best results were obtained for mixes containing SSA formed at 900° C. This agrees with the XRD data that SSA formed at 900° C had the most potential as a SCM. Furthermore, it can be seen that the lowest strength results are obtained with mixtures incorporating SSA obtained at 1000° C and lower combustion temperatures produce SSA with better characteristics for use in cementitious materials.

3.4.2 Gas permeability

Increasing the amount of SSA in mortars tended to result in a decrease in the permeability coefficient (Fig. 10). This is also the case when coal fly ash is used. By using very fine and well-dispersed mineral admixtures, the initial w/c ratio around the aggregates is reduced and the structure of the interfacial transition zone (ITZ) is improved. Although the air content of fresh mortars increased with SSA addition, pozzolanic reaction could be the reason for the decrease in gas permeability when using higher amount of SSA in mortars. This was not found for mixes containing highest proportions (20-30%) of SSA formed at 800°C and 1000°C from WWTP Zagreb. These mixtures show the opposite trend, i.e. increasing SSA addition increased the permeability coefficient of mortars indicating that SSA replacement shares over 20% are probably not feasible. The reason for this could be found in the increased air content in fresh mortars with these SSA (see Figure 6), which is especially expressed for a mix with 30% SSA obtained at 1000°C.

It is also important to note that the class of mortars in terms of gas permeabilty remained the same when SSA was added. Most SSA containing mortars were low resistance mortar while only few samples with the SSA from WWTP Karlovac were classified as medium resistance mortars.

The results shown in Fig. 10 suggest that, although certain differences are present, no conclusions can be drawn considering the influence of different combustion temperatures on the coefficient of gas permeability and therefore durability properties of mortars containing SSA.

Fig. 10 Gas permeability coefficient of mortars containing different proportions of SSA

4. Conclusions

Based on the research conducted using SSA obtained by laboratory incineration of sewage sludge collected from two WWTP in Croatia, the following can be concluded:

- The partial replacement of Portland cement by SSA produced an increase in air content and a reduction in workability, but did not significantly affected the density of fresh mortar.
- In mortar mixes, using SSA as a cement replacement results in lower strength, but the average decrease in both, compressive and flexural strength values is limited to ~12% or less for a 20% addition of SSA.
- Gas permeability data generally shows an increase of mortar resistance with an increase in SSA addition. An average of about a 20% decrease in the coefficient of permeability was observed with the addition of 10% of SSA. However some mixtures containing SSA showed the opposite trend.
- Properties of a fresh SSA-cement mortars were not affected by the temperature at which the SSA is generated, while in the hardened state some differences were noticable. Based on the the mechanical property data obtained the most favorable combustion temperature is 900°C, while the worst mechanical property data was obtained using SSA generated at 1000°C.
- Gas permeability tests generally have shown that the class of mortars remained the same when SSA was added, and for some samples even improved. Also, no conclusions can be drawn considering the influence of different combustion temperatures on the coefficient of gas permeability and therefore durability properties of mortars containing SSA.
- Finally, it can be concluded that excellent mortar properties can be achieved using selected SSA and if the level of cement replacement is kept below 20%.

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Figure 1 Schematic diagram of WWTP processing showing where the sludge is generated

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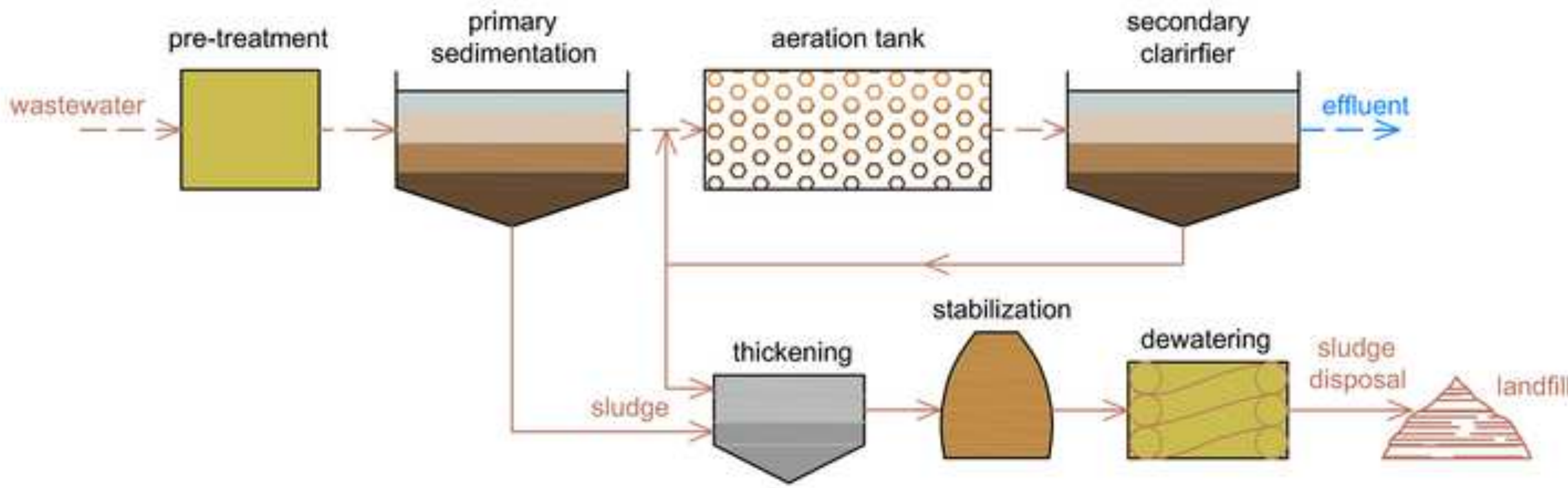

Figure 2

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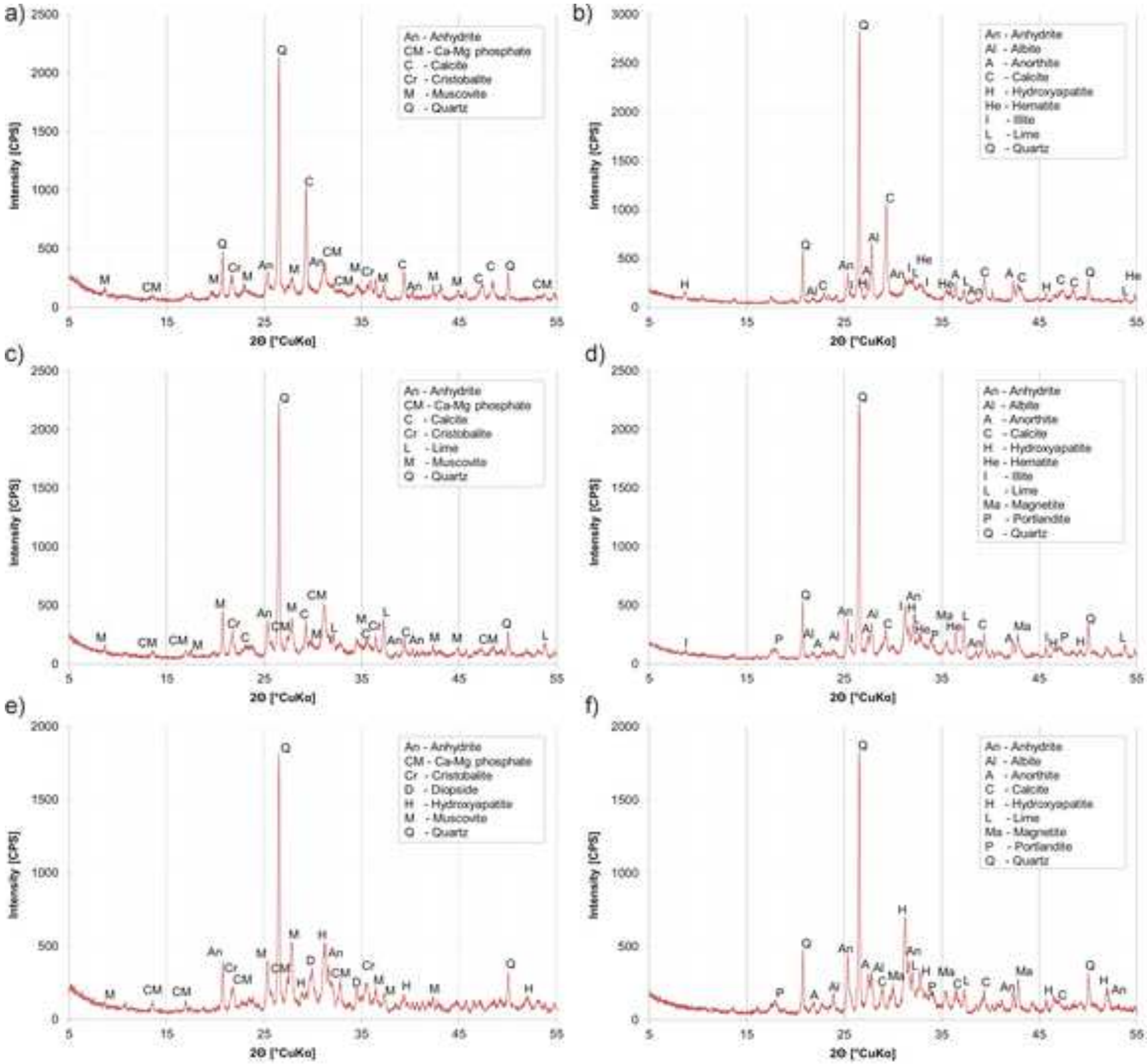

Figure 3 SEM micrographs of the SSA samples obtained by incineration of sewage sludge from the WWTP Karlovac at: a) 800°C, b) 900°C, c) 1000°C

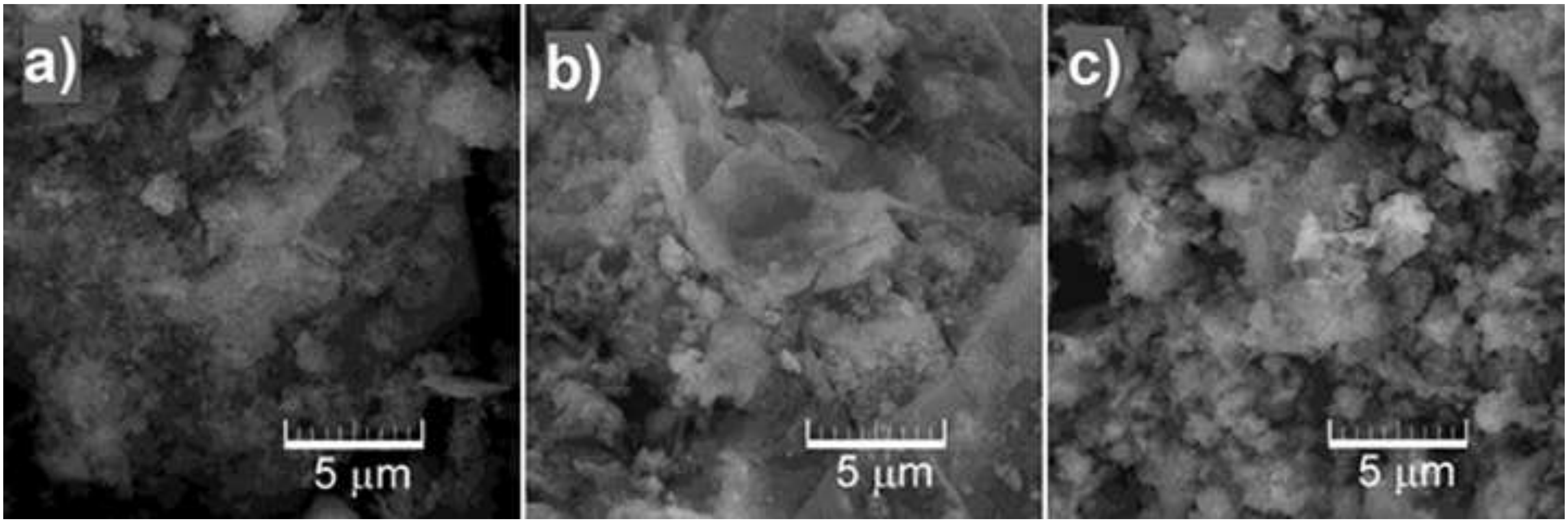

Figure 4 Mean values of particle size distribution for analyzed SSA obtained at different incineration temperatures

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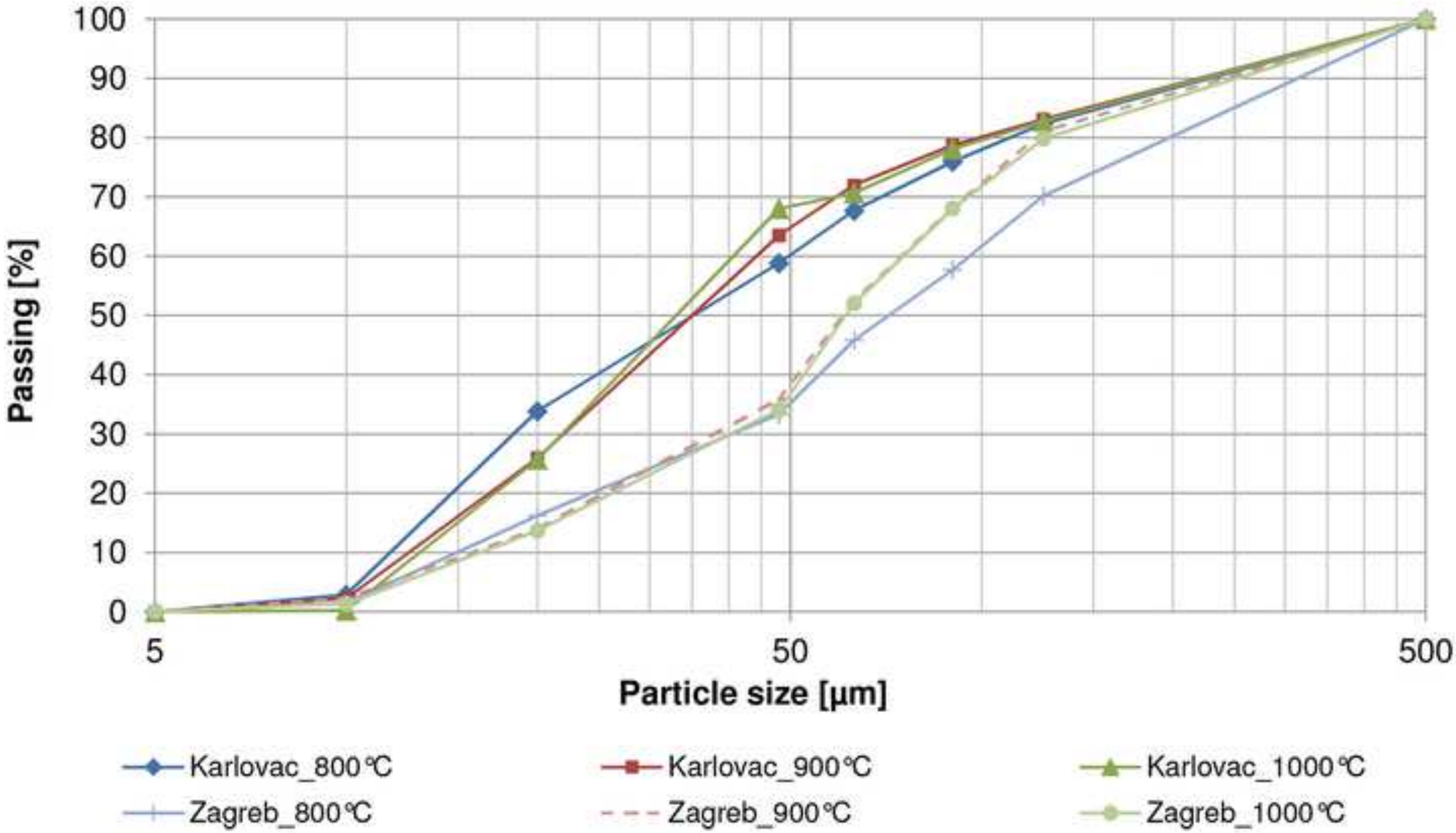

Figure 5 Flow table spread (FTS) of fresh mortar mixes containing different proportions of SSA

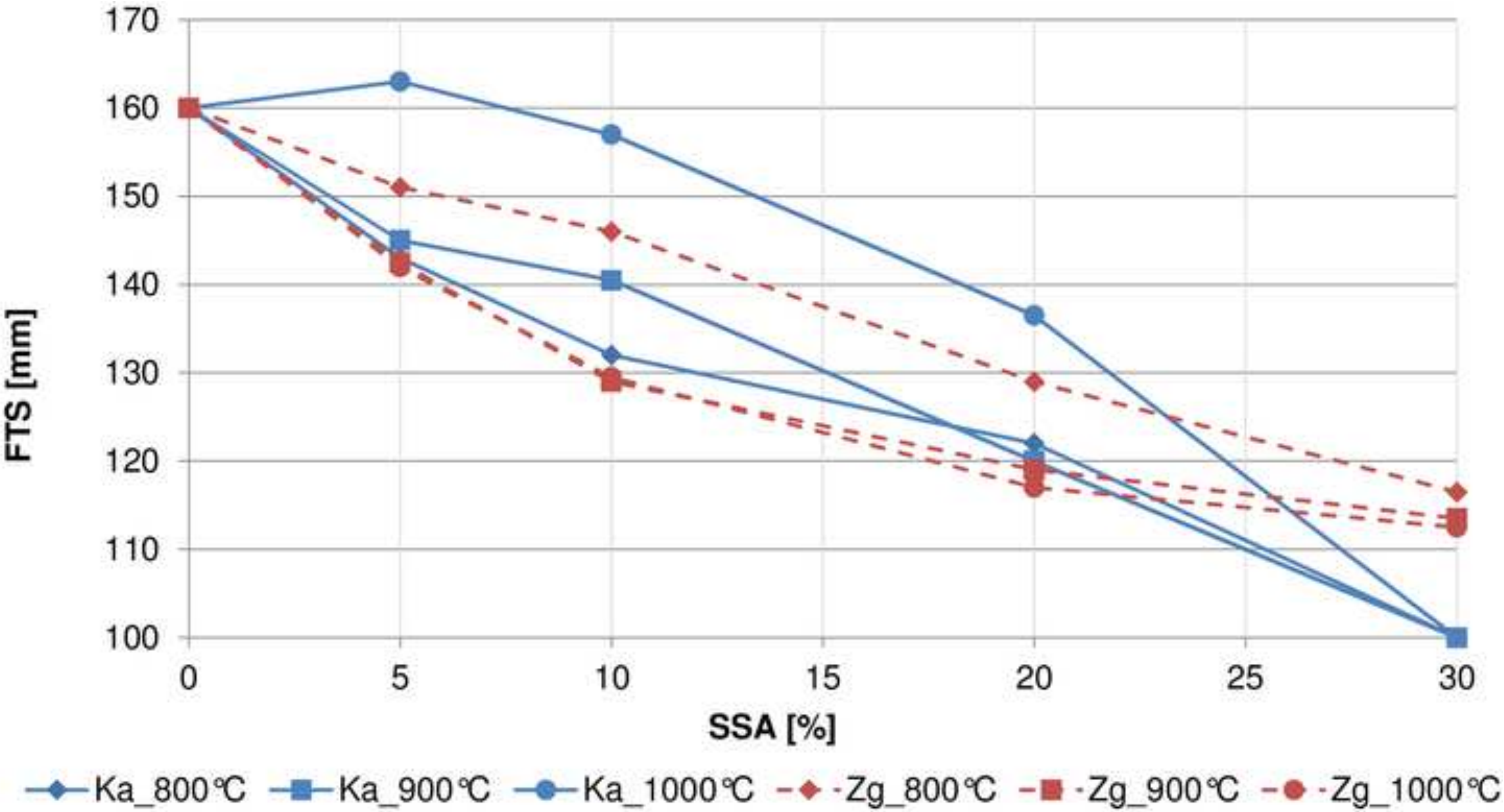

Figure 6 Air content of fresh mortar mixes depending on the proportion of added SSA

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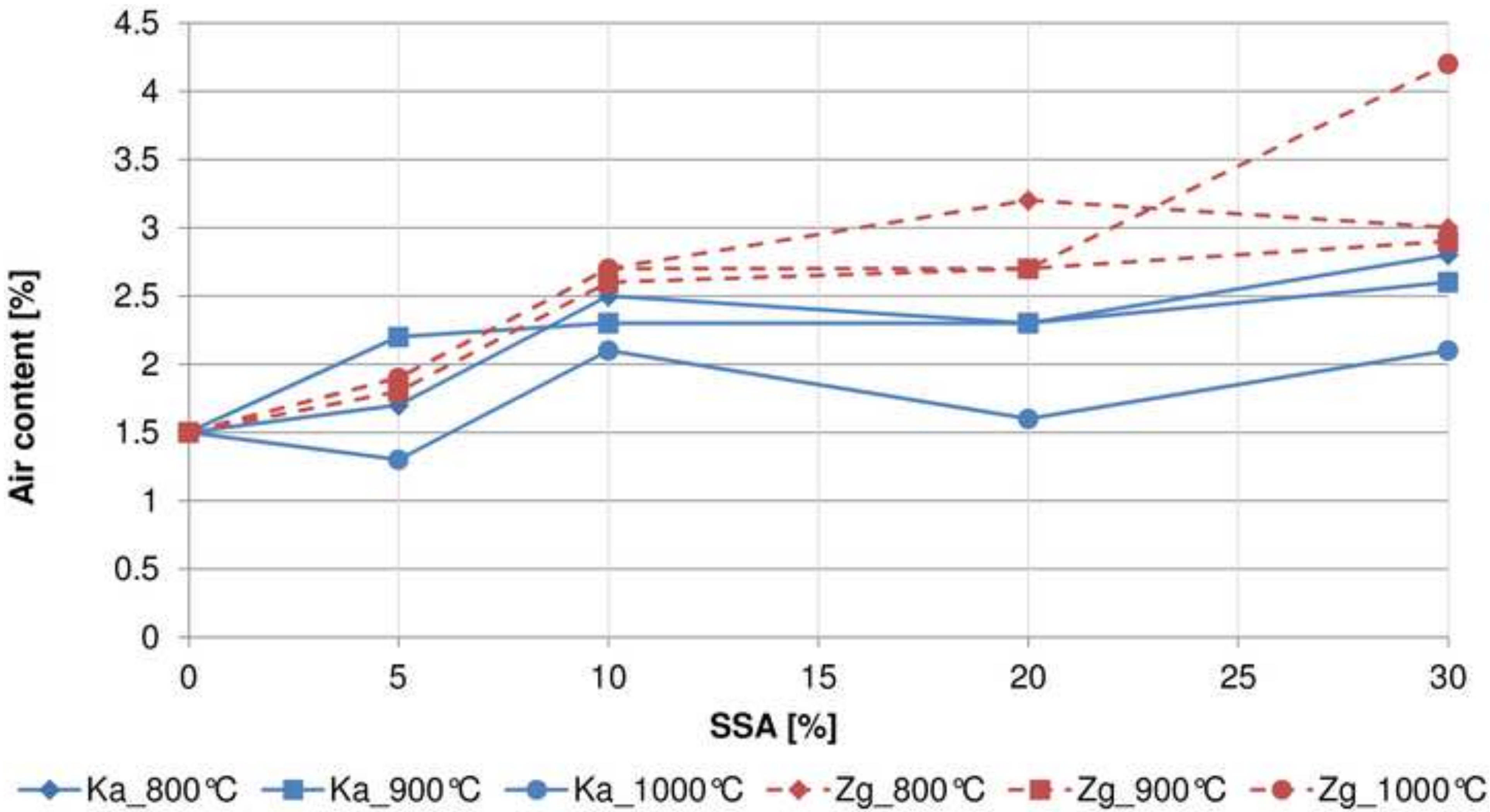

Figure 7 Effect of curing time on the compressive strength of mortars with 20% of SSA as a substitute for cement

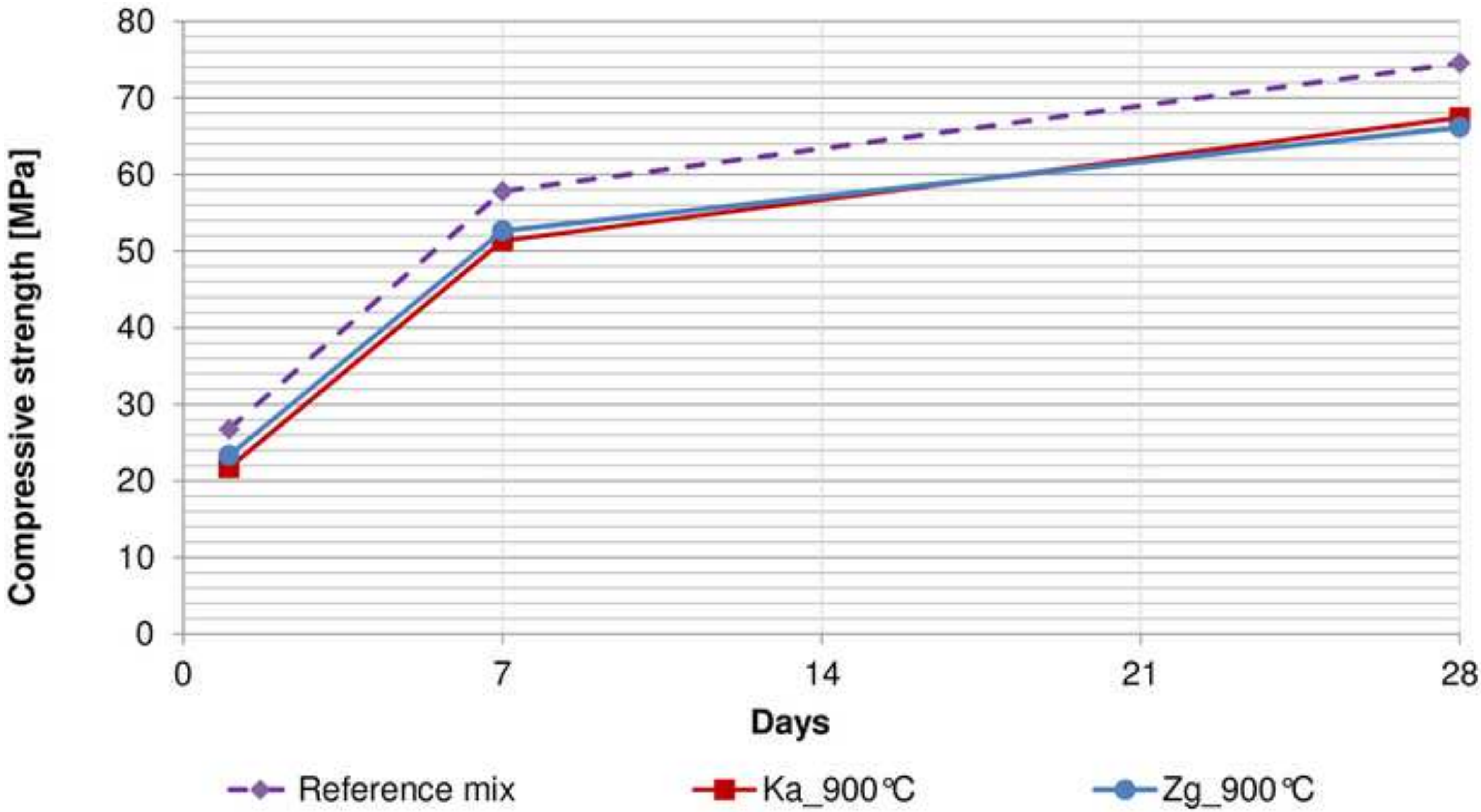

Figure 8 Compressive strengths for age of 28 days depending on the proportion and the type of added SSA

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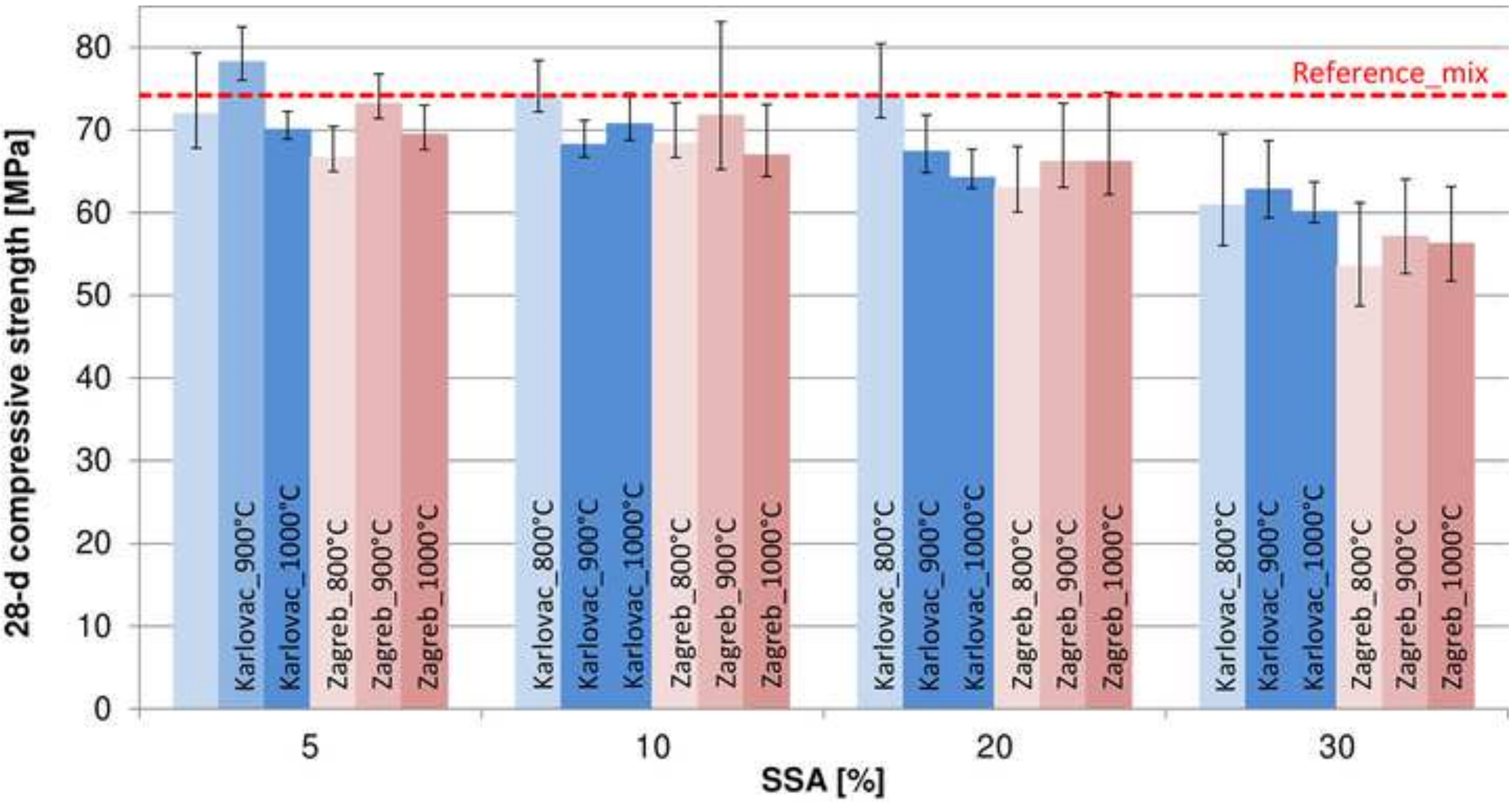

Figure 9 Flexural strengths for age of 28 days depending on the proportion and the type of added SSA

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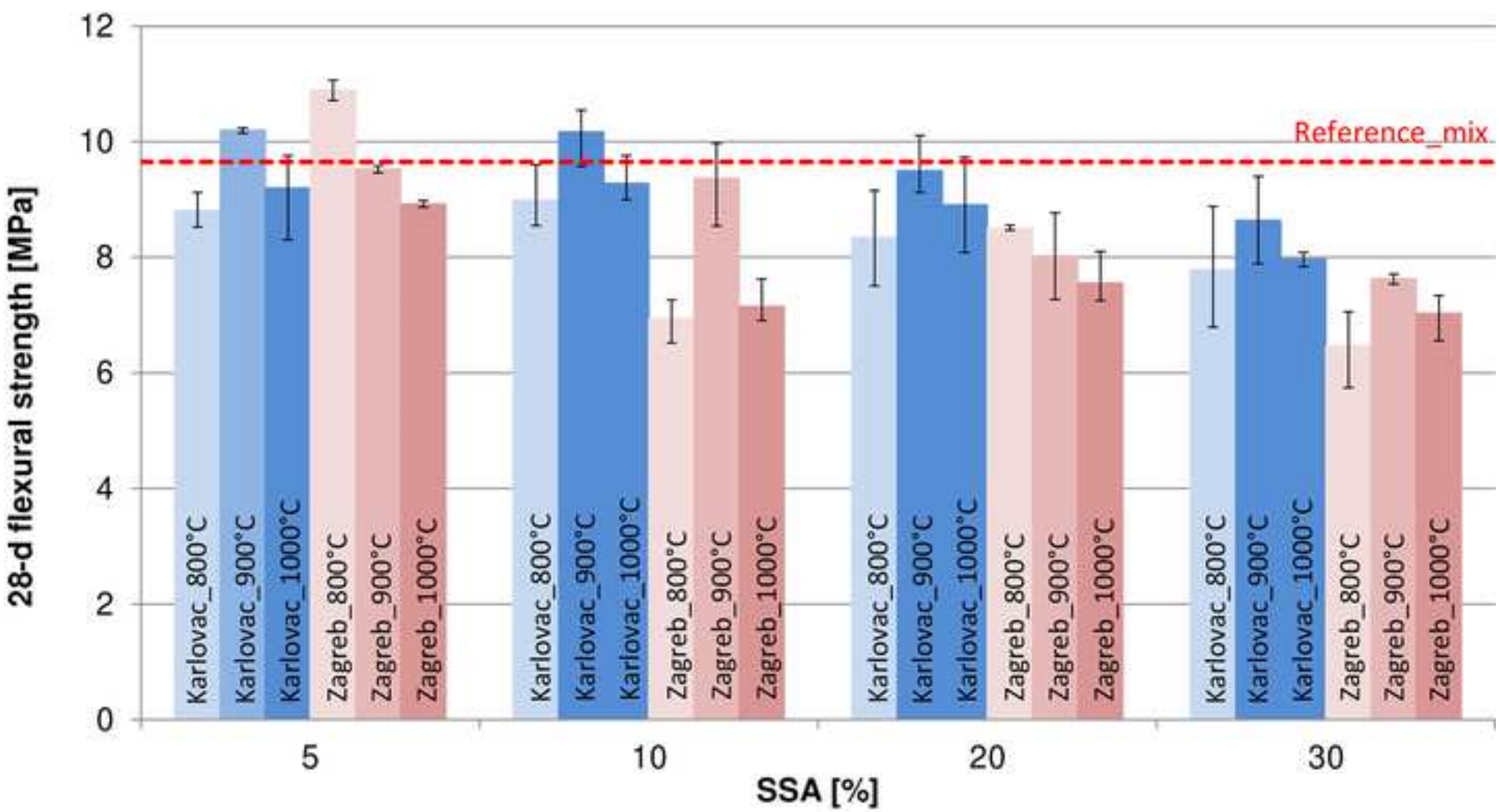

Figure 10 Gas permeability coefficient of mortars containing different proportions of SSA

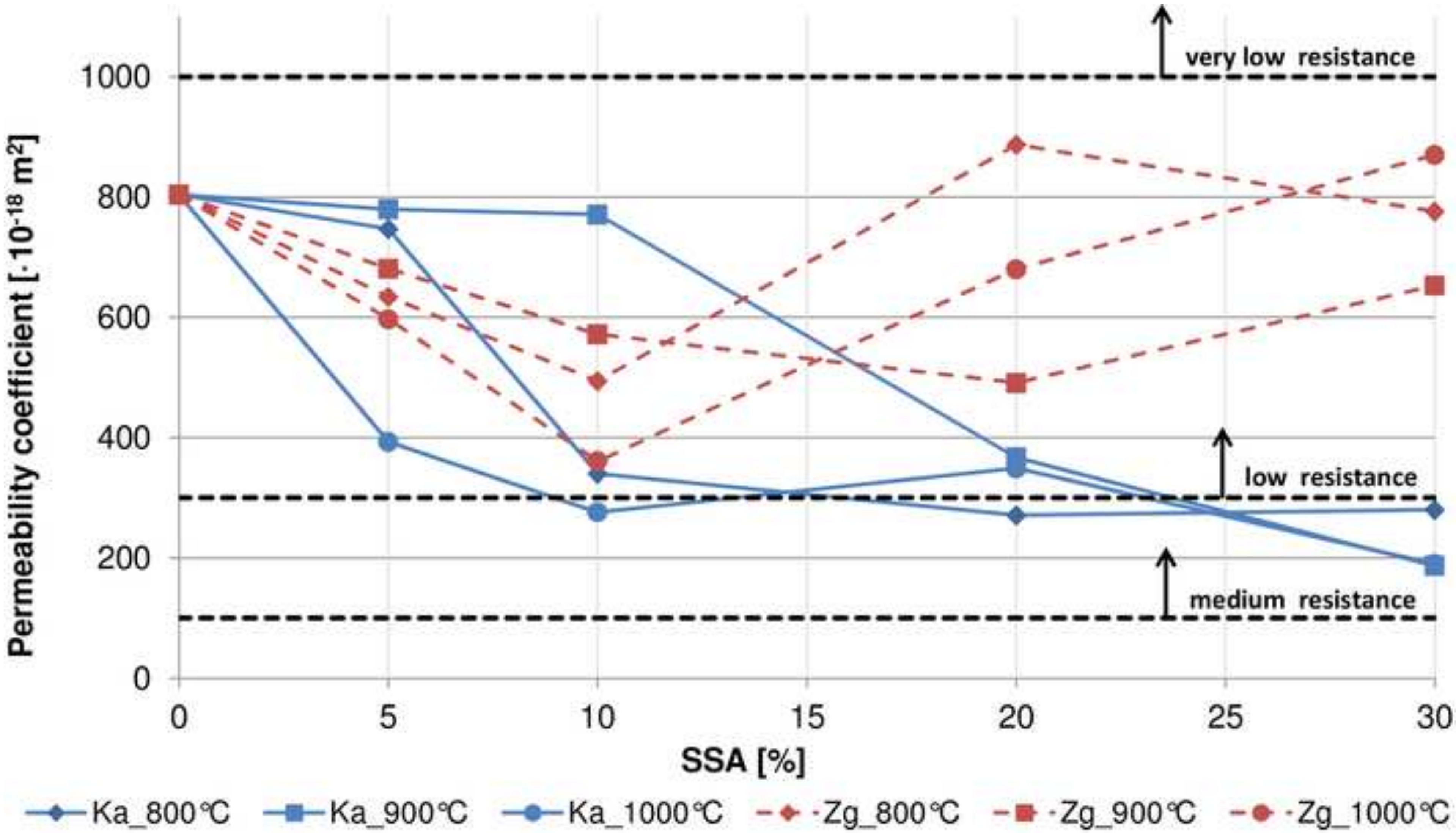

Table 1 Mean values of shares of significant individual oxides in SSA [mass. %]

Oxide	WWTP Karlovac			WWTP Zagreb		
	800°C	**900°C**	**1000°C**	**800°C**	**900°C**	**1000°C**
CaO	37.6	39.4	42.1	23.5	24.6	27.0
SiO_2	7.9	3.9	2.9	20.8	23.0	25.7
Al_2O_3	16.5	14.3	11.7	7.5	7.6	8.5
Fe_2O_3	8.2	8.2	9.5	5.7	5.9	7.0
MgO	4.2	4.3	4.5	2.5	2.9	3.0
P_2O_5	16.0	16.1	17.2	10.4	11.0	12.0
TiO_2	0.8	0.8	1.0	0.4	0.4	0.5
Na_2O	0.3	0.3	0.3	0.2	0.2	0.2
K_2O	1.3	1.3	1.3	0.5	0.6	0.6
SO_3	5.8	6.3	7.7	4.8	5.2	5.9
remaining	1.3	4.8	1.9	23.8	18.6	9.7

Table 2 Density of different SSA samples [g/cm3]

Sludge origin	**Combustion temperature**		
	800°C	**900°C**	**1000°C**
WWTP Karlovac	2.67	2.73	2.83
WWTP Zagreb	2.69	2.75	2.77