

Primljen / Received: 10.7.2017.

Ispravljen / Corrected: 7.11.2017.

Prihvaćen / Accepted: 22.11.2017.

Dostupno online / Available online: 10.5.2018.

Management of sewage sludge – new possibilities involving partial cement replacement

Authors:



Domagoj Nakić, PhD. CE
WYG Savjetovanje d.o.o., Zagreb
domagoj.nakic@wyg-c.eu



Assist.Prof. Dražen Vouk, PhD. CE
University of Zagreb
Faculty of Civil Engineering, Department of Water Research
dvouk@grad.hr



Prof. Nina Štirmer, PhD. CE
University of Zagreb
Faculty of Civil Engineering, Department of Materials
ninab@grad.hr



Assist.Prof. Marijana Serdar, PhD. CE
University of Zagreb
Faculty of Civil Engineering, Department of Materials
mserdar@grad.hr

Original scientific paper

Domagoj Nakić, Dražen Vouk, Nina Štirmer, Marijana Serdar

Management of sewage sludge – new possibilities involving partial cement replacement

An increasing trend of sludge generation at wastewater treatment plants (WWTP) has been observed in developing countries like Croatia. Thermal processing of sewage sludge facilitates its further management, although ash is generated as new waste in the process. The proposed approach, while eliminating the need to dispose ash at non-hazardous waste disposal sites, directly reduces not only the sludge and ash disposal costs, but also the raw cement production costs. All analysed technical and environmental requirements are met when 20 % of cement is replaced with ash.

Key words:

sludge from wastewater treatment plants, ash, incineration, cement, strength, leaching

Izvorni znanstveni rad

Domagoj Nakić, Dražen Vouk, Nina Štirmer, Marijana Serdar

Gospodarenje muljem s UPOV-a - novi uvid u mogućnosti njegove uporabe kao zamjenskog cementnog materijala

U zemljama u razvoju, kakva je i Hrvatska, prisutan je trend povećanja količina mulja koji se generira na uređajima za pročišćavanje otpadnih voda (UPOV). Termičkom obradom mulja olakšava se njegovo daljnje gospodarenje, a pritom se generira pepeo kao novi otpad. Ovakvim pristupom izbjegava se potreba za odlaganjem pepela na odlagalištima neopasnog otpada, a time se direktno smanjuju troškovi ne samo odlaganja mulja, odnosno pepela, nego i proizvodnje sirovog cementa. Uz zamjenu 20 % cementa pepelom, svi analizirani tehnički i okolišni zahtjevi su zadovoljeni.

Ključne riječi:

mulj s uređaja za pročišćavanje otpadnih voda, pepeo, spaljivanje, cement, čvrstoća, izluživanje

Wissenschaftlicher Originalbeitrag

Domagoj Nakić, Dražen Vouk, Nina Štirmer, Marijana Serdar

Entsorgung des Schlamms aus der Kläranlage – neue Möglichkeiten für deren Verwendung als alternatives Zementmaterial

In Entwicklungsländern wie Kroatien besteht ein Trend der Erhöhung der Menge an Schlamm, der in den Kläranlagen generiert wird. Durch thermische Bearbeitung des Schlamms erleichtert man dessen weitere Entsorgung und dabei wird Asche als neuer Abfall generiert. Mit einem solchen Ansatz vermeidet man die Notwendigkeit der Entsorgung von Asche auf Deponien für nicht gefährliche Abfälle, wodurch nicht nur die Kosten für die Entsorgung des Schlamms beziehungsweise der Asche direkt gesenkt werden, sondern auch der Produktion von Rohzement. Mit dem Ersatz von 20 % Zement durch Asche werden alle analysierten technischen und umwelttechnischen Anforderungen erfüllt.

Schlüsselwörter:

Klärschlamm, Asche, Verbrennung, Zement, Festigkeit, Auslaugung

1. Introduction

Wastewater treatment and management of related by-products has become a very important global problem, especially in developing countries like Croatia, where the number of wastewater treatment plants (WWTP) is increasing to meet stringent EU requirements. Sewage sludge is generated as a by-product of accumulation of solids during physical, biological and chemical processes, primarily through separation from primary and secondary settling tanks. The construction of new WWTPs and stringent water treatment requirements in locations where most often only mechanical pre-treatment was previously used (lower BOD and SS limits of final effluent) have resulted in the production of increasing quantities of sewage sludge. Today at the EU level about 11.5 Mt of dry mass of sewage sludge is produced annually, which is expected to increase to over 13 Mt of dry mass by 2020 [1]. In most EU countries, the problem of adequate disposal of sewage sludge is a continual challenge and cost burden to water utilities since it has not been comprehensively solved, nor has it been regulated by the rules, instructions or guidelines. However, it is necessary to emphasize that there are also some positive experiences in dealing with this issue. Due to various technological possibilities of wastewater and sludge treatment and sludge disposal, and possible negative social and environmental impacts, a consensus should be reached between scientists and professionals in each country about acceptable solutions and acceptable levels. The EU Directive 91/271/EEC states that sustainable sewage sludge handling should be a socially acceptable and cost-effective method that meets requirements of efficient recycling of resources, while at the same time ensuring that harmful substances are not transferred to humans or the environment. Thermal processing of sewage sludge can facilitate its further management by reducing the total mass and volume. The calorific (thermal) value of sewage sludge is also extremely important from the aspect of its thermal processing (gasification, pyrolysis, incineration, wet oxidation) [2]. Calorific value of sludge is close to that of brown coal, but it should be kept in mind that this refers to the calorific value of the organic component of sludge, while the non-organic part is devoid of calorific value. Therefore, it is usually necessary to bring sludge to the level of no less than 28 – 33 % of dry matter (DM) to enable auto-thermal combustion without adding external fuel to maintain the process [3]. Lime stabilization reduces bad odours and kills pathogens, but also reduces calorific value of the sludge, and so the calorific value of the stabilized and dehydrated sludge ranges from 10 to 20 MJ/kg. About 22 % of sewage sludge is incinerated at the EU level [4], and this number is constantly increasing. This is especially the case with the growing concern on the use of stabilized sludge in soil as well as on the restricted sewage sludge landfilling that goes towards complete ban of such practices. Sewage sludge ash (SSA) is the main by-product of the combustion processes, and it also requires disposal. Incineration reduces the mass by approximately 70 % and the volume by 90 % [4] while also destroying hazardous organic components in

sludge and minimising odour problems [5]. This residual product (SSA) is characterised by a potential for reuse. There are five main oxides in the SSA: SiO_2 , Al_2O_3 , CaO , Fe_2O_3 and P_2O_5 , while Na_2O , MgO , SO_3 and others are present at lower levels [4, 6, 7].

The fact that most of the SSA generated worldwide is still landfilled represents a significant additional cost for utilities since it is usually classified as a non-hazardous waste, depending on the results of leaching tests. Therefore, the SSA utilisation is desirable and it may be possible to use it in concrete industry as a partial replacement for cement. Although industrial waste may be incorporated in cementitious materials by various traditional methods, the proportion is kept relatively low, typically at less than 20 % [8]. The SSA may be used as a supplementary cementitious material because it can be pozzolanic [6-10]. It also has a potential as an inert filler, replacing or partly replacing sand and/or fine aggregate, and as a raw material in the production of lightweight aggregate [11-15].

Main problems encountered with regard to the SSA use in concrete industry may be sociological, i.e. due to negative perception of incinerators, as well as insufficient public awareness about the safety and ecological acceptability of the final construction products containing the SSA. Also, there is some uncertainty about readiness of the market to accept the SSA as a supplementary material. The lack of uniform regulations and guidelines is one of the main aggravating circumstances too. For example, EN 197-1 standard, which is based on decades of research, only recognizes a defined range of supplementary cementitious materials (e.g. coal, fly ash, slag). At the same time, this standard only recognizes other potential supplementary cementitious materials when added in quantities of up to 5 %. Therefore, the conduct of detailed technical and environmental studies on the SSA use in concrete industry is the basic precondition for overcoming the described problems.

This research evaluates potential reuse of laboratory produced SSA from WWTP Karlovac, Croatia, as a partial replacement of cement in mortars and concrete, and includes determination of the effect of ash on mix properties. A series of tests is conducted as a part of technical and environmental study on possible SSA use in concrete industry. The results aim to provide an indication of the potential viability of the use of ash generated at any industrial scale mono-incineration plant that might be built in Croatia in the future. Positive results of the research on the possibilities of SSA use in concrete industry, from both technical and environmental points of view, could serve as a good basis in the decision-making process for SSA management should Karlovac WWTP opt for sewage sludge incineration as an alternative sludge disposal and management option. The influence of incineration temperature on the SSA characteristics, and on the characteristics of mortar and concrete that contain the SSA, is also investigated. This could serve as a basis for deciding on sludge treatment parameters aimed at generating ashes that would be as convenient as possible for their subsequent use, while taking into account environmental impacts due to an increase in combustion temperature.

2. Materials and methods

2.1. Origin of sewage sludge

Sewage sludge was collected at the WWTP in Karlovac, Croatia. The Karlovac WWTP operates with tertiary treatment using conventional activated sludge technology, with biological nitrogen removal (nitrification-denitrification) and combined biological-chemical removal of phosphorus, for which aluminium and iron salts are also used. The maximum capacity is 98.500 population equivalent (PE). Sludge treatment involves anaerobic stabilization with some lime addition and dewatering, after which the sludge is temporarily stored at an open covered area within the plant perimeter before being permanently landfilled. When operating at full capacity, and taking into account daily production of about 55 g of DM per capita, this WWTP would produce each year about 20 kg DM of sludge per capita or in total about 2,000 t DM of stabilized sludge. Analyses show that about 1,000 t of SSA per annum would be generated if sludge is thermally treated in a mono incineration plant. At a gate fee cost of 50 €/t, the disposal of SSA to non-hazardous landfill in Croatia would generate an annual cost for the utility of 50,000 €, just from such gate fee (i.e. ignoring transport costs and landfill taxes). This cost would directly be avoided if SSA is used as a supplementary cementitious material.

Four month old dewatered and stabilized sludge was used in this research. The collected sludge samples were dried at 105 °C for 24–36 hours to constant mass and to a DM content of ~90 %. Dried sludge samples were then heated to 800 °C and 900 °C for 2.5–3 hours using a laboratory electric muffle furnace. These temperatures and combustion times were previously established as sufficient for complete decomposition of organic matter in sludge. Also, these temperatures are usually selected as maximum combustion temperatures in real-scale industrial incinerators. The resulting ash samples were lightly ground to form granular materials suitable for testing (Figure 1). The SSA obtained in this way exhibits characteristics similar to the ones that could be expected in case of sludge incineration in modern real-scale incinerators. The described methodology was used as no incinerators have so far been built in Croatia, and as it has already been presented by several authors [5, 7, 9, 16–19]. By comparing results obtained by testing physical and chemical characteristics of SSA with the results of similar tests on the SSA generated at real scale, it can be concluded that characteristics of the ashes produced were mostly similar. Possible minor deviations could be expected in the chemical and crystalline composition and morphology of the SSA particles but such small deviations can not significantly affect the results [19].

Also, it must be noted that the laboratory incineration process provides a substantially longer residence time compared to industrial scale incinerators, which may result in somewhat

lower concentrations of heavy metals such as As, Cd, Hg, Pb and Se that can be volatilized at incineration temperatures [20].



Figure 1. Granular ash obtained by incineration (up) and lightly ground ash used for testing and partial cement replacement (down)

2.2. Ash characterisation and preparation of mortar and concrete samples

The SSA sample density was determined using the method specified for determining the density of cement (ASTM C-188) since SSA is used as a partial cement replacement. This was required to calculate mortar and concrete compositions. The chemical composition of ash samples was determined by inductively coupled plasma optical emission spectrometry (ICP-OES) according to HRN EN ISO 11885:2010. The particle size distribution was determined using air jet sieving machine in accordance with HRN EN 933-10:2009 for the classification of fillers. Leaching tests were based on the compliance leaching test for granular waste materials and sludge (EN 12457-2). The samples tested were SSA obtained at 900 °C and samples of crushed cement mortars cured for 28 days containing 20 % of SSA obtained at 900 °C.

Table 1. Composition of mortar mix with 20 % SSA obtained at 900 °C

Component		Mass [kg]	Density [kg/dm ³]	Volume [dm ³]
Cement		405.44	2.95	137.58
SSA (20 %)		101.36	2.69	37.68
Water		253.40	1.00	253.40
w/b = 0.50				3.45
Air (2.5 %)				
Superplasticizer (0.75 % mass of the binder)		3.80	1.10	25.00
Aggregate	0-4 mm	1492.94	2.75	542.89
Total		2256.94	2.26	1000.00

Table 2. Composition of concrete mix with 20 % SSA obtained at 900 °C

Component		Mass [kg]	Density [kg/dm ³]	Volume [dm ³]
Cement		240.00	2.95	81.44
SSA (20 %)		60.00	2.69	22.30
Water		150.00	1.00	150.00
w/b = 0.50				
Air (2.5 %)				25.00
Superplasticizer (1.6 % mass of the binder)		4.80	1.10	4.36
Aggregate	0-4 mm (49 %)	966.01	2.75	351.28
	4-8 mm (20 %)	394.29	2.75	143.38
	8-16 mm (31 %)	611.15	2.75	222.24
Total		2426.26	2.43	1000.00

Portland cement containing slag and limestone (CEM II/B-M (S-V) 42.5N), dolomite sand (0/4mm) and ordinary tap water, with a water to binder ratio (w/b) of 0.50, were used for the preparation of mortar and concrete samples. Mortar batches were prepared in a 5-litre mixing bowl, with 2534 g of binder (cement and substitution with SSA). Concrete samples were prepared in a 75-litre mixing bowl, with 22.5 kg of binder (cement and substitution with SSA). The samples were thoroughly mixed for 4 minutes. The mortar and concrete samples were prepared as reference (0 % SSA) and with 20 % of SSA as a partial substitution for cement. In order to overcome the deterioration of workability (when SSA was used), a superplasticizer (Master Glenium SKY 629, BASF Chemicals) was also added in an amount of up to 1.6 % by mass of the binder. The composition of mixtures with 20 % of SSA obtained at 900 °C is given in Table 1 (mortar) and Table 2 (concrete).

2.3. Experimental program

The temperature of mortar and concrete samples was monitored following HRN U.M1.032 1981 using a pinhole digital thermometer. The workability of fresh mortar samples was measured using standard cone samples on a flow table. The 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

workability of fresh concrete samples was determined using a standard truncated cone for slump testing in accordance with HRN EN 12350-2. The air content was determined using the pressure method following HRN EN 1015-7:2000 for fresh mortars and HRN EN 12350-7 for concrete.

Mortar specimens (Figure 2 left) for strength testing were prepared as 4 x 4 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 100 mm in diameter and 160 mm in height were used for gas permeability testing. 15 cm concrete cubes were used for compressive strength and water penetration depth testing (Figure 2 right), while 10 x 10 x 40 cm prisms were used for flexural strength and shrinkage testing. All specimens were demoulded after 24 hours and cured in a humidity chamber (relative humidity >95 %, temperature 20±2 °C). Mechanical tests were performed following HRN EN 1015-11:2000/A1:2008 on mortars and according to HRN EN 12390-3 and HRN EN 12390-5 on concrete. The bending strength of mortar samples was tested and then the two resulting parts were tested in compression.

Gas permeability tests were performed on three specimens of each mortar mix, using the RILEM Cembureau method [21]. Test specimens were oven dried at 105 °C for 24 hours. The pre-conditioning of samples did not follow the procedure described in the RILEM recommendations. All specimens



Figure 2. Mortar specimens (left) and concrete cubes for compressive strength and water penetration depth testing (right)

were pre-conditioned using the same procedure so that the gas permeability coefficient obtained can be used to compare different mixes. Water penetration depth was determined on three concrete specimens of each mix in accordance with HRN EN 12390-8.

Total shrinkage of concrete was tested following HRN U.M1.029. Each test result was the average of tests on three 10 x 10 x 40 cm prisms. Specimens were de-moulded 24 hours after casting and two sets of measuring pins were glued onto two opposite sides of the specimens and these were then stored in a chamber at a controlled temperature of 20 °C and relative humidity of 60 %. The mean value of two shrinkage measurements on opposite sides of the prisms was registered to determine shrinkage of each specimen. Measurements were performed every 7 days for 90 days.

3. Results and discussion

3.1. Physical and chemical characteristics of SSA

As the combustion temperature increased from 800 °C to 900 °C, the mass reduction of sludge increased by about 2 %, and so the average mass reduction at 800 °C and 900 °C was ~42 % and ~44 %, respectively.

The ash in form of dark grey powder, with low organic and moisture content, was obtained by incineration. The density of SSA samples produced at different incineration temperatures increased from 2.62 g/cm³ to 2.69 g/cm³ at 800 °C at 900 °C, respectively.

The particle size of SSA ranges between 5 and 500 μm (Figure 3), with approximately 50 μm in mean diameter. A relatively small fraction of the particles (less than 2 %) are less than 10 μm in size, while the size of most particles ranges between 20 and 63 μm. The range of most particle sizes (about 90 %) of analysed

SSA coincides with the size range of ash particles found in previous research (1-100 μm) [22, 23]. Chemical composition of SSA samples produced at two combustion temperatures is given in Table 3.

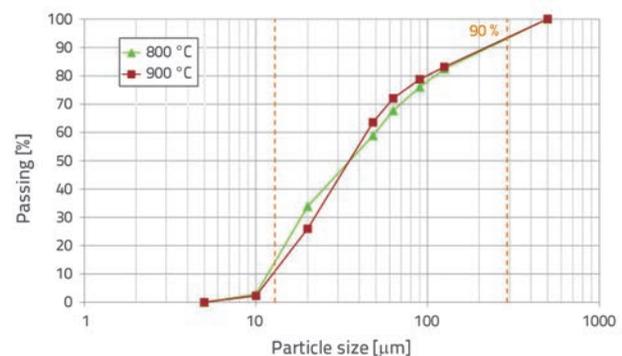


Figure 3. Particle size distribution of SSA obtained at different incineration temperatures

Table 3. Mean values of weight percentages (wt. %) of major oxides in SSA

Oxide	Average value of oxide content [wt. %]	
	800 °C	900 °C
CaO	37.6	39.4
SiO ₂	7.9	3.9
Al ₂ O ₃	16.5	14.3
Fe ₂ O ₃	8.2	8.2
MgO	4.2	4.3
P ₂ O ₅	16.0	16.1
TiO ₂	0.8	0.8
Na ₂ O	0.3	0.3
K ₂ O	1.3	1.3
SO ₃	5.8	6.3
Remaining	1.3	4.8

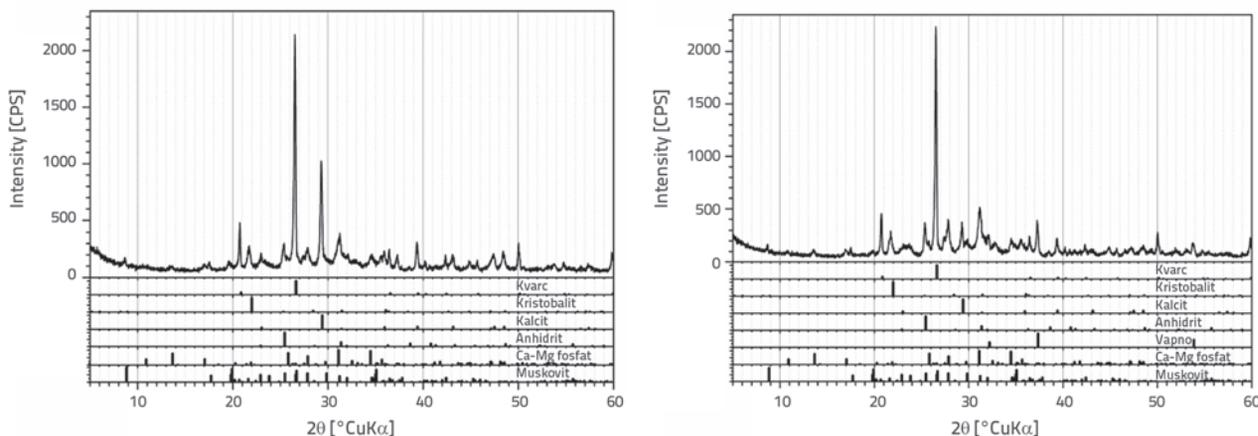


Figure 4. XRD diffractogramme and crystalline composition of SSA samples obtained by incineration of sewage sludge from WWTP Karlovac at 800 °C (left) and 900 °C (right)

X-ray diffraction (XRD) data (Figure 4) for SSA samples reveal a significant presence of an amorphous phase and many crystalline minerals, the main ones being quartz and cristobalite (SiO₂), muscovite, anhydrite (CaSO₄), and calcite (CaCO₃). Increasing the combustion temperature thermally decomposed calcite, which is evident from a decrease of the intensity of 30° peak. One might assume that free lime is forming, leading to enhanced reactivity of SSA. It can therefore be concluded that 900 °C may be a better incineration temperature in terms of quality of ash for use in cementitious materials.

Based on SEM micrographs, all SSA samples produced at different temperatures reveal similar morphology. Polydisperse grains are observed and irregularly shaped particles are present with a wide range of particle size distribution (Figure 5) and with no significant diversity in the

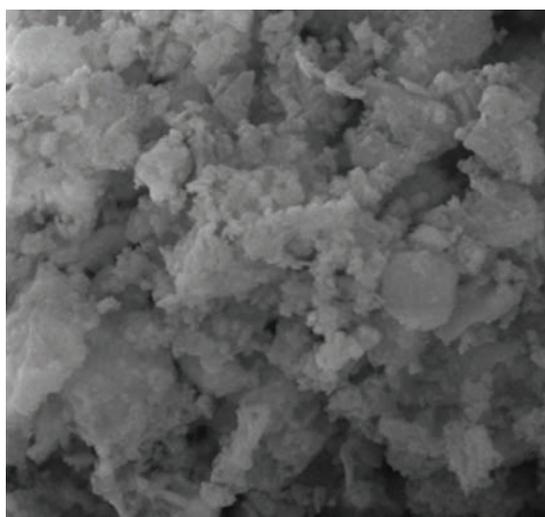


Figure 5. SEM micrograph of SSA sample obtained by incineration of sewage sludge (900 °C) from WWTP Karlovac

form of samples obtained at different temperatures. The particle size slightly reduces with an increase in combustion temperature.

The EN 12457 leaching test was used to determine the type of landfill (inert, non-hazardous or hazardous) for a particular type of waste material. Test results demonstrate that the laboratory obtained SSA could be classified as non-hazardous but not as inert waste due to leached Cl⁻ and Mo levels (Table 4). Crushed mortars based on cement containing SSA could however be classified as inert waste at their end-of-life, and sent to inert waste landfill or used as aggregate in new construction. The fact that the SSA is therefore categorized as a non-hazardous material may lead to conclusion that there are some environmental impacts of the material, i.e. that there is a risk that some elements and compounds present in the SSA might reduce the quality of the environment in the landfill area. Also, the disposal of non-hazardous waste is more expensive compared to inert waste. All this points to the logic of using the SSA in cement-based materials, since in them the soluble elements, representing a potential environmental hazard, could be effectively immobilized within an alkaline Portland cement matrix and therefore disposed of in a more inert form. The same trend was also reported in a similar previously published research [6, 23, 24].

The above presented results clearly show that the leaching of elements that were flagged with SSA alone (Mo and Cl⁻) is well in compliance with the stricter limits for inert waste landfill when the SSA is used in cement mortar. In addition, it seems that cement contributes more than SSA to soluble Mo and Cl⁻ concentrations since their leaching is higher from reference compared to SSA-mortar. On the other hand, SSA increases the leachable Zn, Pb and Cr content considerably, but these concentrations are still well below limits that could be of concern.

Table 4. Leaching concentrations [mg/kg] of selected elements from SSA obtained at 900 °C and crushed SSA-mortars with liquid to solid ratio L/S = 10 l/kg (leaching of all other analysed elements was below detection limits)

Parameter	SSA	Crushed SSA-mortars	Crushed reference mortar	Inert limit	Non-hazardous limit
Cl ⁻	2.150	28	59	800	15.000
F ⁻	9	2.1	1.6	10	150
Zn	< DL	0.38	0.02	4	500
Pb	< DL	0.0038	0.001	0.5	10
As	0.0287	< DL	< DL	0.5	2
Cr	0.3933	0.0466	0.0047	0.5	10
Se	0.0621	0.0094	0.0086	0.1	0.5
Mo	1.698	0.1105	0.2216	0.5	10

< DL – below detection limit

3.2. Effect on properties of fresh mortars and concrete

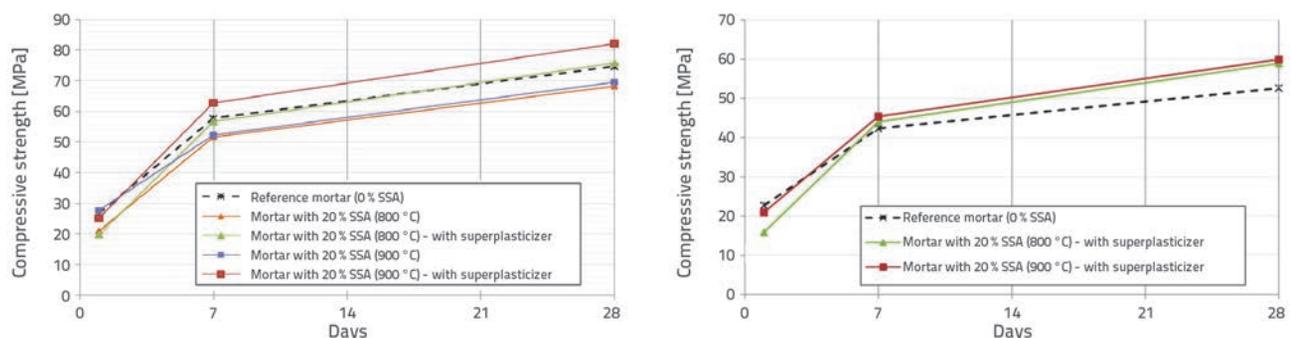
No segregation or bleeding was observed in mixes containing the SSA. All mixes containing the SSA showed compliance with reference values for density of fresh mortar and concrete, and minimum differences between mixes were observed. Densities of mortar and concrete amounted to $\sim 2.3 \text{ g/cm}^3$ and $\sim 2.5 \text{ g/cm}^3$, respectively. With the addition of the SSA a slight increase in temperature of fresh mortar and concrete was observed, i.e. the increase amounted up to 4°C compared to control mixes. This is probably due to the fact that the sludge used was lime stabilized and that a significant fraction of this lime/calcite was converted to free lime during incineration in laboratory. There was also an increase in water demand for mortars and concrete containing the SSA. Mixes containing higher amounts of the SSA exhibited significantly lower flow table spread (FTS) and slump values. This was successfully overcome by adding superplasticizer in the quantity of 0.75 % by mass of binder in mortars, and up to 1.6 % by mass of binder in concrete. It can be concluded that the mortar and concrete workability decreases with an increase in the SSA quantity for all mixes, which is in accordance with previous research when different SSA were used as partial cement replacement [6, 7, 25].

The air content of fresh mortar/concrete increased with an increase in SSA content. The increase is not linear and it is

less pronounced at higher replacement rates. There were no significant differences in air content for SSA mixes obtained at different combustion temperatures. For mortar and concrete mixes containing 20 % of SSA the air content was up to 2.6 %, i.e. it was $\sim 75\%$ higher when compared to the control mixes.

3.3. Effect on properties of hardened mortar and concrete

Mechanical property data for mortars reveal that the SSA use as partial cement replacement slightly decreases the compressive and flexural strength values. Nevertheless, the flexural and compressive strength values of all specimens increase with curing time (Figure 6). Thus, the decline in the strength of mortars with SSA, as related to the reference mix, is the most pronounced in the early stages of hydration (1-day strengths), while this decline gradually reduces at subsequent hydration times. In contrast, when superplasticizer was used in the SSA-mortar mixes, the strengths obtained were on a par with or even exceeded the reference mixture. The use of superplasticizer facilitated and improved mortar placing into the moulds, which ultimately contributed to strength development. Considering all mortar samples tested (Figure 6 left), the highest 28-day compressive and flexural strengths were obtained for the mix with the 20 % SSA produced at 900°C and with the addition of superplasticizer. These strengths were $\sim 10\%$ greater compared

**Figure 6. Compressive strength development for experimental SSA-mortars (left) and SSA-concrete (right) and their comparison to reference mixes**

to those of the control mix. The lowest strength values were recorded for samples with SSA produced at 800 °C and without superplasticizer (decrease in strength amounts to 10-15 % compared to the reference mix).

On the other hand, both experimental concrete mixtures (with 20 % SSA) were found to be superior to the reference mixture except for the early strength (1-day). At the age of 28-days, both experimental mixes exhibit the strength that is approximately 12 % greater compared to the reference mix. The strengths recorded using the SSA produced at 900 °C were only slightly higher.

Finally, it can be concluded that mixtures (both mortar and concrete) in which up to 20 % of the cement is replaced by the SSA, and in which superplasticizer is used, exhibit mechanical characteristics equal to or even better than the reference mixes. Also, these results differ from most of the previously published research where mostly the adverse impact of SSA addition on mortar and concrete strengths was reported.

The gas permeability coefficient decreases with an increase in the quantity of the SSA in mortars. Similar results were recorded with the SSA produced at 800 °C and 900 °C. This is a positive effect of the SSA on the durability properties of mortars, as the permeability coefficient was lower compared to the control mortar.

In terms of gas permeability, the class of mortars containing the SSA [26] remained the same as the reference mortar ("low resistance" mortars), but these values were close to the limits for "medium resistance" mortars (Figure 7). The reduced permeability of the SSA containing mortars is explained with the "filler effect", where SSA particles fill the gaps between larger aggregate grains.

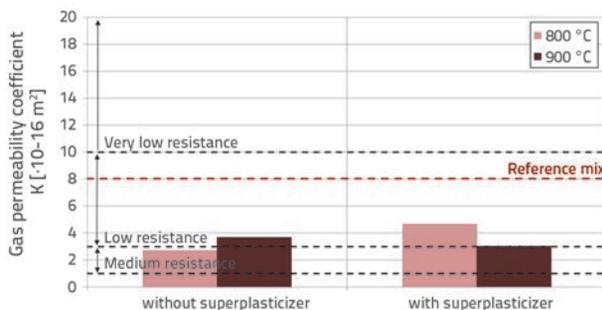


Figure 7. Gas permeability coefficient of mortars containing SSA

There were no significant changes in maximum water penetration depth for concrete samples when the SSA was used (Figure 8). When 20 % of the SSA incinerated at 800 °C was used in concrete the water penetration depth was slightly higher (24.10 mm) compared to the control mix (22.23 mm), but when 20 % of the SSA incinerated at 900 °C was used, these values even slightly decreased (21.00 mm).

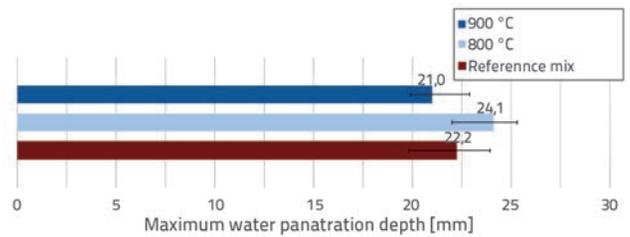


Figure 8. Maximum water penetration depth of concrete depending on type of SSA

Shrinkage data obtained by drying concrete samples is shown in Figure 9. The SSA use has no major adverse effect on the volume deformation of hardened concrete. Total shrinkage of concrete samples with 20 % of the SSA incinerated at 800 °C increased after 90 days by more than 20 % compared to the reference concrete. However, the effect of the SSA incinerated at 900 °C on the volume deformation of concrete was negligible since the obtained values are almost equal to those of the reference concrete.

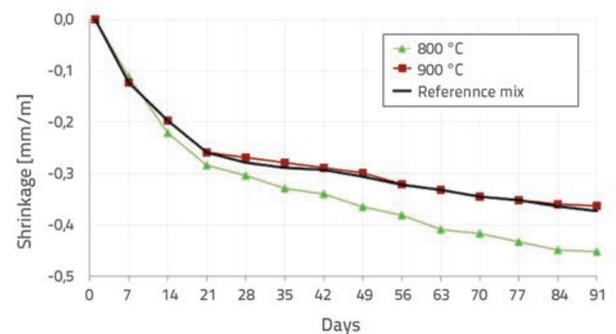


Figure 9. Shrinkage of concrete depending on SSA type

Higher combustion temperature (900 °C) and use of superplasticizer seem to eliminate all detrimental effects on technical properties of SSA mortars. Also, as shown in Table 4, leaching of analysed elements from SSA-mortars does not differ significantly from the reference mortar, pointing to the conclusion that their use would be safe from the environmental and health perspectives.

4. Conclusion

It may be expected that the recycling and use of sewage sludge will gain more importance worldwide in the near future, especially in developing countries. In case the incinerator construction concept is adopted, the SSA use in concrete industry will provide environmental and economic benefits, primarily through conservation of raw materials, but also through reduced quantity of waste sent to landfills. According to the principles of sustainable development, the sludge or SSA use closes almost completely the wastewater

treatment cycle, generating negligible quantities of waste to be landfilled.

Main detrimental effects are associated with an increased water demand and reduced workability of SSA-mortars, but this can successfully be overcome by the addition of superplasticizer. Based on the mechanical and durability data, 900 °C proved to be a more favourable combustion temperature compared to 800 °C. Compressive and flexural strengths of mortar and concrete mixes with the 20 % SSA content are mainly within the range of strengths (± 15 %) obtained on reference mixes. An increase in the quantity of the SSA in mortars results in a decreased gas permeability coefficient and hence in better durability properties, while water permeability and shrinkage data obtained on concrete samples were not significantly affected by the addition of the SSA. As to technical requirements, management of selected SSA and its use in the production of mortars and concrete seems feasible, practical and justified if the level of cement replacement is kept below 20 %. Soluble elements like Mo and Cl⁻, which represent potential environmental hazards when leaching from SSA alone, are

effectively immobilised within cement matrix and are therefore disposed of in a more inert form. Also, leaching of elements of concern from crushed SSA-mortars is of the same order of magnitude as leaching from the reference mortar. It can be concluded that the SSA has the potential for satisfactory use in cement mortars and concrete for selected applications.

This research may serve as a good basis for decision making on the SSA disposal method, should Karlovac WWTP opt for sewage sludge incineration as an alternative sludge disposal and management option. Moreover, the presented methodology can be directly applied to other WWTPs, but at the same time a particular emphasis should be placed on the need for separate analysis of each of the SSA used.

Acknowledgements

This work has been fully supported by the Croatian Science Foundation under the project "7927 - Reuse of sewage sludge in concrete industry: from microstructure to innovative construction products".

REFERENCES

- [1] Milieu Ltd: Environmental, economic and social impacts of the use of sewage sludge on land, Final Report, Part III: Project Interim Reports, Brussels, 2010.
- [2] Fyttili, D., Zabaniotou, A.: Utilization of sewage sludge in EU application of old and new methods – A review, *Renewable and Sustainable Energy Reviews*, 12 (2008), pp. 116-140, <https://doi.org/10.1016/j.rser.2006.05.014>
- [3] Donatello, S., Cheeseman, C.R.: Recycling and recovery routes for incinerated sewage sludge ash (ISSA): a review, *Waste Management*, 33 (2013), pp. 2328-2340, <https://doi.org/10.1016/j.wasman.2013.05.024>
- [4] Lynn, C.J., Dhir, R.K., Ghataora, G.S., West, R.P.: Sewage sludge ash characteristics and potential for use in concrete, *Constr. Build. Mater.*, 95 (2015), pp. 767-779, <https://doi.org/10.1016/j.conbuildmat.2015.08.122>
- [5] Tantawy, M.A., El-Roudi, A.M., Abdalla, E.M., Abdelzاهر, M.A.: Evaluation of the pozzolanic activity of sewage sludge ash, *Chem. Eng.*, Article ID 487037, 8 pages, 2012.
- [6] Cyr, M., Coutand, M., Clastres, P.: Technological and environmental behavior of sewage sludge ash (SSA) in cement-based materials, *Cem. Concr. Res.*, 37 (2007), pp. 1278-1289, <https://doi.org/10.1016/j.cemconres.2007.04.003>
- [7] Vouk, D., Serdar, M., Nakić, D., Anić-Vučinić, A.: Use of sludge generated at WWTP in the production of cement mortar and concrete, *GRADEVINAR*, 68 (2016) 3, pp. 199-210, <https://doi.org/10.14256/JCE.1374.2015>
- [8] Baeza-Brotons, F., Garcés, P., Paya, J., Saval, J.M.: Portland cement systems with addition of sewage sludge ash. Application in concretes for the manufacture of blocks, *J. Clean. Prod.*, 82 (2014), pp. 112-124.
- [9] Pan, S.C., Tseng, D.H., Lee, C.C., Lee, C.: Influence of the fineness of sewage sludge ash on the mortar properties, *Waste Management*, 33 (2003), pp. 1749-1754, [https://doi.org/10.1016/S0008-8846\(03\)00165-0](https://doi.org/10.1016/S0008-8846(03)00165-0)
- [10] Donatello, S., Tyrer, M., Cheeseman, C.R.: Comparison of test methods to assess pozzolanic activity, *Cem. Concr. Compos.*, 32 (2010), pp. 63-71, <https://doi.org/10.1016/j.cemconcomp.2009.10.008>
- [11] Khanbilvardi, R., Afshari, S.: Sludge ash as fine aggregate for concrete mix, *J. Environ. Eng.*, 121 (1995), pp. 633-637, [https://doi.org/10.1061/\(ASCE\)0733-9372\(1995\)121:9\(633\)](https://doi.org/10.1061/(ASCE)0733-9372(1995)121:9(633))
- [12] Cheeseman, C.R., Virdi, G.S.: Properties and microstructure of lightweight aggregate produced from sintered sewage sludge ash, *Resour. Conserv. Recy.*, 45 (2005), pp. 18-30, <https://doi.org/10.1016/j.resconrec.2004.12.006>
- [13] Chiou, I.J., Wang, K.S., Chen, C.H., Lin, Y.T.: Lightweight aggregate made from sewage sludge and incinerated ash, *Waste Management*, 26 (2006), pp. 1453-1461, <https://doi.org/10.1016/j.wasman.2005.11.024>
- [14] Jamshidi, A., Mehrdadi, N., Jamshidi, M.: Application of sewage dry sludge as fine aggregate in concrete, *J. Environ. Stud.*, 37 (2011), pp. 4-6.
- [15] Kosior-Kazberuk, M.: Application of SSA as Partial Replacement of Aggregate in Concrete, *Polish J. of Environ.*, 20 (2011), pp. 365-370.
- [16] Fontes, C.M.A., Barbosa, M.C., Toledo Filho, R.D., Goncalves, J.P.: Potentiality of sewage sludge ash as mineral additive in cement mortar and high performance concrete, *Intern. RILEM Confe. on the Use of Recycled Materials in Buildings and Structures*. pp. 797-806, 2004.

- [17] Naamane, S., Rais, Z., Taleb, M.: The effectiveness of the incineration of sewage sludge on the evolution of physiochemical and mechanical properties of Portland cement, *Constr. Build. Mater.* 112 (2016), pp. 783-789, <https://doi.org/10.1016/j.conbuildmat.2016.02.121>
- [18] Wang, K.S., Chiou, I.J., Chen, C.H., Wang, D.: Lightweight properties and pore structure of foamed material made from sewage sludge ash, *Constr. Build. Mater.*, 19 (2005), pp. 627-633, <https://doi.org/10.1016/j.conbuildmat.2005.01.002>
- [19] Vouk, D., Nakić, D., Serdar, M., Donatello, S.: Evaluation of using sewage sludge ash in the cement industry: Case study of Zagreb, Croatia. The IWA Specialist Conference on Sludge Management: Sludgetech 2017, London, UK, 2017.
- [20] Nakić, D., Vouk, D., Donatello, S., Anić-Vučinić, A.: Environmental impact of sewage sludge ash assessed through leaching, *Engineering Review*, 37 (2017), pp. 222-234.
- [21] Geiker, M., Grube, H., Luping, T., Nilsson, L.O., Andrade, C.: Laboratory test methods. Rilem Report, No 12 (1995), pp. 144-147.
- [22] Yusuf, R.O., Moh'd Fadhil, M.D., Ahmad, H.A.: Use of sewage sludge ash (SSA) in the production of cement and concrete – a review, *Int. J. Global Environ. Issues*, 12 (2012), pp. 214-228, <https://doi.org/10.1504/IJGENVI.2012.049382>
- [23] Coutand, M., Cyr, M., Clastres, P.: Use of sewage sludge ash as mineral admixture in mortars, *Constr. Mater.*, 159 (2006), pp. 153-162, <https://doi.org/10.1680/coma.2006.159.4.153>
- [24] Chen, M., Blanc, D., Gautier, M., Mehu, J., Gourdon, R.: Environmental and technical assessments of the potential utilization of sewage sludge ashes (SSAs) as secondary raw materials in construction, *Waste Management*, 33 (2013), pp. 1268-1275, <https://doi.org/10.1016/j.wasman.2013.01.004>
- [25] Monzo, J., Paya, J., Borrachero, M.V., Girbes, I.: Reuse of sewage sludge ashes (SSA) in cement mixtures: the effect of SSA on the workability of cement mortars, *Waste Management*, 23 (2003), pp. 373-381, mixtures: the effect of SSA on the workability of cement mortars, *Waste Management*, 23 (2003), pp. 373-381, [https://doi.org/10.1016/S0956-053X\(03\)00034-5](https://doi.org/10.1016/S0956-053X(03)00034-5)
- [26] AFGC (Association Française de Génie Civil): Concrete design for a given structure service life, State of the art and Guide for the implementation of a predictive performance approach based upon durability indicators, Scientific and technical documents, April 2007.