REUSE OF SEWAGE SLUDGE – PROBLEMS AND POSSIBILITIES

Authors:
Drazen Vouk [a], *Domagoj Nakic* [b], *Nina Stirmer* [c]

[a] Assist.Prof.Ph.D., University of Zagreb, Faculty of Civil Engineering, Water Research Department, Kaciceva 26, 10000 Zagreb, dvouk@grad.hr

[b] mag.ing.aedif., Professional assistant, University of Zagreb, Faculty of Civil Engineering, Water Research Department, Kaciceva 26, 10000 Zagreb, dnakic@grad.hr

[c] Associate.Prof.Ph.D., University of Zagreb, Faculty of Civil Engineering, Department of Materials, Kaciceva 26, 10000 Zagreb, ninab@grad.hr

Abstract

Each stage of wastewater treatment generates certain quantities of sewage sludge that need to be adequately treated at wastewater treatment plants and disposed of in the environment.
Final disposal of sewage sludge is important not only in terms of satisfying the regulations, but the aspect of choosing the optimal wastewater treatment technology, including the sludge treatment. Special emphasis is given on the possibility for reuse of sewage sludge. The paper presents the possibilities of reuse of sewage sludge or byproducts of its treatment (e.g. ash). In the process of sewage sludge incineration special form of ash is generated – incinerated sewage sludge ash (ISSA), which is three to five times less in volume compared to stabilized and dehydrated sludge. ISSA also need to be disposed of in accordance with certain legislation. In this paper special emphasis is given on the possibility and feasibility of reuse of ISSA. Results of the research carried out on cement mortars point to the great possibilities of ISSA incorporation in the concrete industry.

Key words: sewage sludge, wastewater, WWTP, sludge reuse, ISSA, cement mortar

1. Introduction

Wastewater treatment and management of products generated in the process is an actual problem on the worldwide level, in particular during recent 20-30 years. Construction of wastewater treatment plants (WWTP) on one hand represents a favorable effect on the environment, and initiates, on the other hand, new problems manifested by generation of considerable quantities of sewage sludge which must be adequately managed. The process of wastewater treatment, regardless of the technology applied, generates some sludge primarily through sludge separation in primary (primary sludge) and secondary (biological sludge) sedimentation tanks. Generated sludge must be adequately treated at WWTP and disposed into the environment in accordance with the legislation. On the average unit quantities of sewage sludge are generated of 35 to 85 g DM/PE·day [1]. In Croatia, according to available data from existing WWTP, this value is about 50 to 55 g DM/PE·day

The sludge generated at WWTP is the byproduct of solid substance accumulation during physical (sedimentation), biological (microbiological activities) and chemical processes (coagulation, flocculation). The sewage sludge is of composite texture and represents a mixture of organic and non-organic matter dispersed in water and contains pathogenic microorganisms, parasites, viruses and numerous potentially toxic elements and compounds (heavy metals etc.)

In recent practice of WWTP construction the efforts have been concentrated primarily on the water line, striving to achieve the prescribed quality of treated water and meet the criteria of treatment efficiency. In some of EU countries, for instance, the sewage sludge is still being disposed on solid waste disposal landfills and in other, often inadequate and illegal ways. This approach is still present, although the professional public has beer for comparatively long time informed about European guidelines for final disposal of sewage sludge which render conventional disposal on landfills practically impossible.

It must be pointed out that designs for construction of WWTP that do not contain final sludge disposal cannot be considered complete, because they do not include technological solutions and costs related thereto. Accordingly, efficiency of public sewerage and price of wastewater treatment (expressed as unit price per population equivalent or as volume of wastewater) cannot be based exclusively on costs generated within WWTP, but should include all costs until final sludge disposal. Costs of sewage sludge treatment and disposal are not negligible, and in WWTP sized from 5,000 to 200,000 PE they amount to approximately 50% of total operating costs [2], and in certain circumstances (export from Croatia) they can be considerably higher, with increased negative sociological impact [3].

The fact is that in many developing countries, as well as in some developed countries the problem of sewage sludge management has not been solved comprehensively, nor regulated by rules, instructions or guidelines. As construction of WWTP is becoming more intensive on a worldwide level, sludge disposal will become a burden to many water utilities managing sewerage and wastewater treatment systems.

The choice of optimum sewage sludge treatment process depends, among other things, on final disposal, and this should be taken into consideration already at the construction stage. Some study analyses [3,4], following complete analysis of various solutions, taking into account also the costs of environmental protection, conclude that incineration would be an acceptable concept of final sludge treatment in larger capacity WWTP. Sewage sludge incineration considerably facilitates further management of the newly generated product (incinerated sewage sludge ash - ISSA), mainly as the effect of considerable reduction of mass and volume of the final by-product. Thermal processing reduces the total mass of sludge by up to 85% [5]. The volume of the waste matter is also reduced, toxic organic components are destroyed, odours are reduced to minimum, further management is facilitated, and power generation is also possible [6].

Thermal processing of sewage sludge generates the byproduct (ash) which must be properly managed in an adequate way in the final stage, and its quantities are not negligible. For example, the wastewater treatment plant of the capacity of 1,500,000 PE will generate about 18,000 tons of stabilized dehydrated sludge per annum (with 30-35% of DM). Donatello and Cheeseman [7] estimate that at present, at the global level, thermal processing of sludge from WWTP generates 1,700,000 tons of ISSA per annum (mainly in USA, EU and Japan). Construction of new and reconstruction of existing WWTP will result in constant increase of this number. For example, in Croatia in 2018 there will be treatment plants in operation with the total capacity of 4,500,000 PE, generating the total quantity of stabilized and dehydrated sludge of about 250,000 per annum (160,000 m^3 of ISSA per annum). If thermal processing of sludge is chosen, the annual production of ISSA would be 57,000 tons (21,000 m^3).

The possibility of reuse of sewage sludge (ISSA) greatly depends on its composition, mainly chemical. Therefore the results of research carried out on sewage sludge of different composition should be taken with caution. For example, during the past two decades industrial production in some EU countries has been considerably reduced, which resulted in considerable changes of quantities and composition of wastewater arriving to WWTP, and consequently, changes in sludge composition. Likewise, the composition of sewage sludge and ISSA depends also on the technological process of water treatment and sludge treatment (for instance, whether lime is added to sludge in the final phase of treatment, etc.).

Final sewage sludge management is not only important from the standpoint of meeting of legal requirements, but also from the aspect of selection of optimum concept of treatment, including sludge treatment. In addition to problems described above, the paper also presents the possibilities of reuse of sewage sludge, or by-product of its treatment (ISSA), and results of research carried out so far, which should intensify thinking about the profitability and feasibility of reuse (recycling) of sewage sludge. Based on results of researches with cement mortar, the possibility and feasibility of sludge/ISSA reuse in concrete industry will be demonstrated.

2. Possibilities of reuse of sewage sludge

Adequate management of sewage sludge represents a challenge to all stakeholders involved in sewerage and wastewater treatment, as well in waste management. Final disposal of sewage sludge from WWTP is a costly and environmentally sensitive procedure, a problem faced by many developed and developing countries even at present time. It is important to keep in mind various possibilities of reuse of sewage sludge, limited by certain treatment processes, and by resulting properties of treated sludge. The present worldwide practice offers several possible solutions of reuse of sewage sludge and by-products of its treatment (e.g. ISSA), including the following:

1. Use in agriculture
2. Phosphorus extraction
3. Use in construction
   3.1 Soil improvement
   3.2 Road construction
   3.3 In production of bricks
   3.4 In concrete industry (production of concrete, cement mortars, etc.)

2.1. Use of sewage sludge in agriculture

The use of sewage sludge in agriculture and for similar purposes such as flower gardening, forestry, pastures, belongs to the most economically profitable ways of final sludge disposal, with manifold favorable and adverse effects. The use of sewage sludge in agriculture is based on the nutrient contents in sludge. Organic matter contained in sludge is important for plant growth. By decomposition of organic matter into non-organic it is incorporated in clay and humus particles, where it is retained and becomes available for use and for plant growth. Organic matter in sludge improves the soil texture and facilitates soil aeration. Simultaneously, it improves retaining of moisture in soil. The use of sludge in agriculture also recovers phosphorus in nature. In addition to nitrogen (2-7% of dry matter content), phosphorus (2-7% of dry matter) and potassium (<1.5% of dry matter) sludge contains other substances required for plant growth such as microconstituents (iron, manganese, zinc, copper, boron, molybdenum) [8]. The use of sludge in agriculture renews phosphorus in nature.

Table 1 Comparison of nutrients in mineral fertilizers and sludge, percent [9]

Product	Nitrogen	Phosphorus	Potassium
Mineral fertilizer	5 %	10 %	10 %
Stabilized sewage sludge (dry matter)	3.3 %	2.3 %	0.3 %

Legislation related to the use of sewage sludge in agriculture, including the use of sludge in compost form mainly sets the limitations regarding sludge composition, soil where sludge is to be used, crops and areas of use. For instance, in the Republic of Croatia the possibility of sludge use in agriculture is defined by the maximum quantity on the annual level of 1.66 tons of dry matter per hectare of agricultural land. Sludge is used in agriculture only a part of the year, and as it is generated all year round, it must be stored. The possibility of storing, costs of transport to agricultural land, as well as the method of spreading is dependent on the type of sludge. Transport costs are lower with lower water content in sludge. The use of sludge in agriculture is not the solution that may be applied on a larger scale, in particular after passing of more stringent requirements in numerous countries worldwide, and in present practice in EU decreased use of sewage sludge in agriculture may be noticed.

It should be pointed out that, in addition to legal constraints, there is also a certain sociological problem regarding the use of sewage sludge in agriculture.

2.2. Extraction of phosphorus from sewage sludge

Phosphorus in wastewater represents a significant renewable resource and there is no environmental or technical reason why phosphorus can't be recycled. There are many potential environmental benefits from reducing reliance on phosphate rock and associated impurities, a practice that is ultimately unsustainable. There are several stages of both, wastewater and sludge treatment process where phosphorus could be recovered. Recently, actual interest has been related to the possibility of extraction of phosphorus from ISSA, as natural resources of phosphorus are assessed as limited and insufficient for rapid development that includes the use of phosphorus as a resource [7]. With the present rate of phosphorus use, natural resources are estimated as sufficient for only 50 to 100 years of economically sustainable use [10, 11, 12].

The use of recovered phosphates is a priority for the European phosphate industry [13]. Incineration does not lower phosphate extraction potential regarding to thermal stability of phosphates up to high temperatures which means it does not volatilize during incineration at 800-900°C. Proportion of phosphate in ISSA (as P_2O_5) is typically 10 - 25% by weight [12].

There are few options for recovering phosphorus from ISSA and they can be divided into two groups: wet extraction methods [11, 14, 15, 16, 17, 18, 19, 20] and thermochemical methods. Wet extraction method includes leaching of ISSA with acids or bases. The efficiency of the process depends on the type and concentration of the extractant, pH value, the extraction time and the incineration temperature of sewage sludge. The wet chemical methods of extraction are based on the solubility of salts as a function of pH value [21]. Main problem within this option is that a number of other metals simultaneously dissolve [7]. An optimum incineration temperature for recovery of phosphorus by acid leaching is around 950°C [20]. The acid washing process is the simplest and cheapest option, but the major concerns are connected with the remaining acid insoluble ISSA residue generated [7]. According to these authors the main alternative for phosphorus recovery from ISSA are thermochemical methods. In this process ISSA is mixed with a chloride donor and subsequently heated within a temperature range of 900-1050°C, leading to the transformation of phosphate compounds into a bioavaliable form while heavy metals present in the ISSA react with the chloride donor resulting in the formation of volatile chlorides, which evaporate and hence the heavy metal free ash is obtained [22].

The technologies for phosphorus recovery from sewage sludge/ISSA must take into account the wastewater and sludge treatment technologies applied at the WWTP.

2.3. Use of sewage sludge in construction

Construction industry is an important consumer of natural resources and materials, which makes it a sector with enormous potential for use of waste material generated within construction activity, but also in other sectors. The use of such materials helps reduction of energy consumption, contributes to conservation of natural (non)renewable resources, and reduces enormous quantities of waste material disposed into the environment (in landfills). Basic aspects related to the possibility of sewage sludge use in civil engineering are given below.

2.3.1. Use of sludge for soil improvement

Soil stabilization is a usual engineering practice used to improve physical characteristics of poor foundation ground. There are numerous mechanical and chemical methods of soil stabilization: adding of lime, flying ash, cement, natural gypsum, combined lime and gypsum, lime and ash, ash from rice husks, flying ash from coal and steel [23].

Clayey soils are characterized by high swelling and shrinking potential, which may cause numerous problems in their use for foundation of engineering structures such as buildings, roads and other projects. With adding of hydrated lime it is possible to add ISSA for stabilization of soft cohesive foundation soil. Increasing of ISSA content in poor foundation soils decreases the value of the plasticity index of the soil [23]. Further, increasing of ISSA content up to 7.5% increases maximum dry density, while ISSA content above 7.5% causes the decrease of dry density. This trend is in accordance with the behaviour of rise/fall of soil compressive strength with adding of ISSA (up to 7.5% results in rise, and above 7.5% causes the fall of dry density of soil). Soil swelling potential is considerably reduced by adding ISSA. Basic properties of soft foundation soil have been successfully improved by using of the cement/ISSA mixture. In soil stabilization with adding of ISSA, considerable drop of the soil plasticity index has been noticed, leading to the shift from the category of poor bearing capacity to medium bearing capacity [24,25]. Influence on soil swelling has also been noticed, i.e. the higher the percentage of added ISSA/cement, the lower soil swelling, while the compressive strength of the soil is increased, as well as the overall strength and bearing capacity of the foundation soil. According to the results of triaxial compressive tests [24], using of the mixture of ash and hydrated lime as stabilizer of poor foundation soils, it is possible to increase the sliding strength of such soil by 30 to 50-70 kPa. With such measures, with adding of ISSA it is possible to classify poor foundation soils into the category of good foundation soils and achieve good effects related to soil stabilization.

2.3.2. Use of sewage sludge in road construction

A number of scientific researches has been oriented to testing of the possibility of substituting of powder limestone in asphalt mixtures by ISSA, which in this case plays the role of mineral filler [26]. Sato [27] has concluded that ISSA may be used as filler in asphalt mixtures, but that the quality of mixtures with ISSA is somewhat lower than conventional mixtures (using powder limestone only). Mixtures using ISSA without grinding required considerably higher portion of asphalt, and as asphalt is expensive, the effect is unfavorable. Mixtures using ISSA without grinding showed a considerable drop of dynamic stability and strength (the higher ISSA content in the mixture, the larger the drop of the properties). Some of these problems may be eliminated by using ISSA that has been ground in order to get finer particles. In this way the required quantity of asphalt in mixture is reduced, while dynamic stability and tearing strength are increased. The use of ISSA as substitute for a part of the aggregate in asphalt mixtures is also possible [5, 26].

2.3.3. Use of sewage sludge in brick industry

Bricks and other ceramic materials occupy a very important place in the field of masonry materials, thanks to their physical, mechanical and chemical properties, in particular as regard to strength, durability and compactness. This is the reason for efforts to recycle waste materials in brick industry.

Substituting of a part of raw clay in brick industry (production of bricks, roof tiles, facade tiles, etc.) production of ceramic materials by waste materials (sewage sludge or ISSA) contributes to reduced abstraction of raw clay from nature, and consequently to conservation of natural resources. This reduces costs of clay abstraction from nature, and, equally important, solves a great deal of the problem of management and disposal of sewage sludge. Further, such practice may even contribute to improvement of the properties of conventional bricks.

Adding of incineration material (dried sewage sludge) in the brick baking process results in formation of pores, reduction of the density and thermal conductivity of the product, but also in deterioration of mechanical resistance by modification of composition and microstructure [28]. Therefore, for production of competitive bricks it is necessary to reach a compromise between thermal and mechanical properties. Reduced density of the resulting material, along with replacement of a part of original raw material by sewage sludge and higher porosity, is reflected on the mechanical resistivity of the final product. Still, traditional bricks, in order to meet the strength requirements, should not have compressive strength below 100 kg/cm^2, which means that bricks made with 10% portion of sewage sludge meet this condition [29]. The portion of sewage sludge that may be incorporated into clay for various brick materials depends a great deal on the properties of sewage sludge (particle size, chemical and mineralogical composition), but even more on characteristics of the raw material used [30].

ISSA has some properties similar to materials used in production of bricks and other ceramic materials. This refers primarily to the content of iron, calcium and phosphorus whose compounds have been recognized as compounds of important ceramic characteristics, considerably contributing to material properties in the baking process. In Japan some bricks have been made of ISSA only, but with special processes of ISSA compaction at very high pressures, which makes this technology irrationally expensive for European conditions [31].

ISSA may be incorporated into ceramic materials as substitute for a part of clay or sand [32]. Reduced density of material obtained by substitution of a part of original materials by ISSA due to higher porosity is greatly reflected on mechanical resistance of the final product. According to research by Anderson and Skerratt [31], adding of 5% by weight of ISSA to the brick mixture does not show any important influence on properties during formation and baking of final products. The general conclusion is that shrinking during drying is considerably reduced by adding of ISSA, which might be a considerable advantage, because it reduces the time required for drying. Adding of ISSA also has the tendency of increased shrinking during baking, as well as increased porosity, which at the final stage means a mild reduction of strength and increased absorption of water.

2.3.4. Use of sewage sludge in concrete industry

At present, concrete is the most used artificial construction material in the world. Mineral additives to concrete are defined as non-organic materials, pozzolanic materials or latent hydraulic materials which, finely ground, may be added to concrete and/or mortars based on Portland cement, in order to improve certain properties or to achieve certain characteristics [33]. Apart from direct incorporation into concrete and cement mortars, sewage sludge may be used also in cement industry, as the unavoidable segment of concrete industry.

There are three basic principles of using of waste materials in cement industry: as raw material for formation of clinker, as alternative fuel in the production process, or as a substitution material in cement/concrete mixtures, replacing a portion of Portland cement.

The basic chemical elements present in Portland cement are calcium, silica, aluminum and iron. These elements, as already mentioned, are present to a considerable extent in the ISSA [7].

Thanks to considerable calorific value of dried sewage sludge, there is an interesting possibility of its use as alternative fuel in cement industry in production of clinker. Non-organic ash, which is the byproduct in the process, possesses significant pozzolanic properties, may be further used as substitute for a part of raw material from nature, required for production of cement. Namely, when the organic part of sewage sludge burns out, non-organic fractions remaining in the form of ash are incorporated in the cement clinker. It has been estimated that such approach in cement production may reduce the requirements for fossil fuels in modern cement kilns by 70% [34]. This approach to sewage sludge management is assessed as highly acceptable, because sewage sludge is used as alternative fuel, and the product of incineration (ash) is directly incorporated into clinker, without separate depositing. This simultaneously reduces the requirements for fuel in the process of incineration, and the requirements for raw material from nature for production of cement.

The use of sewage sludge in the process of cement production requires changing of a part of physical-chemical characteristics of sewage sludge, such as reducing of humidity and organic matter content, and increasing of the content of calcium compounds. These aims are achieved by previous stabilization of sewage sludge, among others by adding alkaline compounds, e.g. lime [35]. In this process, chemical and thermal energy caused by reaction of sewage sludge and lime leads to evaporation of humidity from sewage sludge and mineralization of organic matter.

Still, the largest potential of sludge/ISSA reuse in concrete industry refers to the possibility of using as a substitute of a part of original raw materials in concrete and cement mixtures. ISSA may be used in concrete as pozzolanic active material, partly replacing cement, or as inert filler, replacing sand and/or fine aggregate. Several authors [1, 7, 36, 37, 38, 39, 40] have published that partial replacement of Portland cement by ISSA influences the workability and strength of cement pastes, mortars and/or concrete.

Considerable contents of SiO_2 and Al_2O_3 in ISSA guarantee the possibility of use as pozzolanic material. Mainly adverse effects of substitution of a part of cement by ISSA are attributed to increased water requirements due to irregular morphology of ash particles, partial reduction of flexural and compressive strength and concrete workability, and longer binding times have also been recorded [7].

The following effects have been recorded in connection with substitution of a part of cement by ISSA in cement pastes, mortars and concretes: longer binding times [7,39,41], reduced settling time and increased total porosity [42], reduction of workability [37,39,40], and increased water requirements [7,37]. Monzo et al. [37] have concluded that the decrease of workability of mortars with ash is non-lineary, and that with larger portions of ISSA the decrease of workability is less important. These drawbacks may be compensated by alternative chemical additives such as plasticizers and superplasticizers [37], higher fineness of ash particles [38] and even by adding of flying ash [43]. For example, by adding of 20% of flying ash it is possible to compensate the above adverse effects caused by replacing of 10% portion of cement by ash. It is important to stress that increased fineness of ash particles extends the binding time and settling time, i.e. water adsorption is increased (due to larger total free area of particles), and this in turn causes greater pozzolanic activity and developing of higher pressre strengths of the resulting concrete.

As regards concrete strength, two significant trends may be pointed out: increasing of ISSA content replacing cement reduces the concrete strength, and by increasing of fineness of ISSA particles concrete strengths become higher. Comparison of results by separate authors shows considerable differences of absolute values. For instance, replacement of 20% of Portland cement by ISSA leads to different reductions of compressive strengths: 20% [44], 52% [38], 32% [45]. The paper by Jamshidi et al. [1] states that replacement of 10% of cement by ISSA causes reduction of compressive strength by 8%, and therefore the concretes containing more than 10% of ISSA may be used only as non-constructive. The obvious differences are primarily the consequence of different sample sizes and different w/c ratios. The important factor is also the process of ISSA generation, kiln type, temperature, influence of various additives during incineration, etc.

According to Chen et al. [46], the strength of analyzed mortars decreases linearly with increase of ISSA portion substituting cement. This influence on hydraulic properties is explained by surplus water required in ISSA-containing mixtures to maintain workability and CaO content in ISSA (less than 10%).

The type of commercially available cement also influences the behavior of mortars in which a part of cement is replaced by ISSA [40]. According to this work, cement CEM II/B-M (V-LL) has been pointed out as the most suitable (among analyzed cement types) for mortars with certain percentage of ISSA. Highest compressive strength values were achieved in mortar samples prepared with cement CEM I 52.5 R, because nominal strength of this cement is also highest. Lowest values were achieved with cement CEM II/B-L 32.5 N. In all mortars, compressive strength was reduced by increasing the portion of ISSA.

Additional possibility of using of ISSA in concrete refers to substitution of a part of fine aggregate [47,48] and sand [7]. Also, it is possible to apply ISSA in the process of production of light aggregates [48].

According to research by Baeza-Broton [49], samples with a portion of ISSA as substitute for cement in concrete up to 5% have shown even somewhat higher compressive strengths compared to reference samples. Portions of ISSA above 5% give somewhat lower values of compressive strength, but still above 90% of compressive strength of reference samples.

There are also possibilities of using ISSA in mixtures with returned concrete (remaining concrete on site and returned to the batching plant), which are fragmented after drying to make ground material for bearing layers [27]. In this case better properties of the final material were obtained using ground ISSA.

Environmental effects of using ISSA in cement materials have been processed by analyzing leaching from concrete samples, and the general conclusion is that it is, according to all parameters, within allowed limits [7,46].

3. Characteristics of ISSA

ISSA is primarily a powdery material with some particles sized like sand, and with negligible content of organic matter and humidity [23]. The particle size of ISSA ranges from 1 to 100 μm, with mean diameter value of about 26 μm [42,50]. There is a comparatively high percentage of particles (even up to 90% for some ISSA) under 75 μm [42]. ISSA consists of irregular particles with large specific area, which results in higher water requirements when used in cement mortars and concretes.

Precise range of ash particle sizes depends on the sludge processing procedure, percentage of industrial water in wastewater, and the type of the sewerage system [7].

Actual specific gravity of ISSA varies, according to different authors (but not significantly) 2.3 – 3.2 g/cm^3 [46], 2.62 g/cm^3 [40], 2.86 g/cm^3 [51].

ISSA density, determined according to ASTM C-188 [52] from incineration of sludge from WWTP Karlovac (Croatia), used in the experimental research within this paper, is given in Table 2. There is a noticeable trend of increasing of ISSA density with increased temperature of incineration.

Table 2 Density of ISSA obtained by incineration of sludge from WWTP Karlovac (Croatia) at different temperatures

Temperature of incineration [°C]	Density of ash [g/cm^3]
800	2.67
900	2.73
1000	2.83

Temperature of incineration of dried sludge has a considerable influence on microstructure and pozzolanic properties of ash. At temperatures above 900 - 950°C clinker formation occurs, i.e. increased crystallization and weakening of pozzolanic properties of ISSA [6, 51].

According to some research, optimum temperature of sludge incineration, from the standpoint of preserving of pozzolanic properties of ash is about 800°C [6].

Major chemical elements contained in ISSA are silica (Si), calcium (Ca), iron (Fe), aluminum (Al), and phosphorus (P). Crystaline forms of Si, Ca and Fe are unchangeable quartz (SiO_2), calcium phosphate ($Ca_3(PO_4)_2$), and hematite (Fe_2O_3).

If tertiary sludge is used as well, it is necessary to pay attention to salts for chemical precipitation of phosphorus on iron and aluminum basis, which can in this case considerably increase the content of these metals in ISSA. Even in WWTP operating in stationary conditions the share of main chemical elements contained in ISSA may vary a great deal [31, 52]. Elements present in ISSA with smaller shares may vary even more and be considerably influenced by the nature of industrial activity in the area gravitating toward the sewerage system.

Heavy metals like mercury (Hg), cadmium (Cd), antimony (Sb), arsenic (As) and lead (Pb) should burn out during incineration [53]. Still, according to some researches, traces of evaporated metals are found in ISSA due to condensation on ISSA particles after the temperature in the thermal processing plant is lowered. If ash after incineration is to be deposited, the problems regarding leaching of heavy metals are antimony (Sb), molybdenum (Mo) and selenium (Se). According to this criterion such ISSA in most countries would not be suitable for disposal on deposits of harmless waste [44,46].

Basic minerals that make ISSA are SiO_2 and Al_2O_3 which creates good preconditions for use in the form of mineral additive to Portland cement-based composite materials. The share of CaO, SO_3, P_2O_5 i Fe_2O_3 is also significant [49]. Donatello et al. [7] point out that ISSA contains high shares of phosphates, usually 10% to 20% of mass share in the form of P_2O_5. Chemical analysis of ISSA from WWTP Karlovac (Table 3) according to HRN EN ISO11885:2001 has shown that the resulting ISSA considerably differs from ISSA analyzed in previous researches. It is necessary to point out considerably higher shares of CaO (which may be attributed to considerable quantities of lime added in sludge processing procedure), and considerably lower shares of SiO_2.

Table 3 Minimum, maximum and mean values of shares of individual oxides in ISSA on the basis of results of previous analyses [6, 37, 38, 39, 40, 43, 48, 49, 50, 51, 54, 55, 56] and comparison with results of chemical analysis of ISSA obtained by incineration of sludge from WWTP Karlovac

Oxide	**Oxide percentage in ISSA [%]**				
	Min	**Max**	**Average**	**Karlovac * 800 °C**	**Karlovac * 1000 °C**
SO_3	0.60	12.40	5.74	5.83	7.66
Fe_2O_3	4.70	20.00	11.47	8.21	9.46
SiO_2	17.27	50.60	33.13	7.94	2.87
CaO	1.93	31.30	16.74	37.64	42.12
MgO	1.48	3.22	2.17	4.23	4.53
Al_2O_3	6.32	19.09	12.43	16.46	11.72
P_2O_5	1.67	18.17	10.70	16.02	17.21
TiO_2	0.29	1.00	0.65	0.76	1.03
Na_2O	0.32	1.26	0.73	0.28	0.28
K_2O	0.62	1.76	1.29	1.31	1.26

Note: * …. Results of chemical analysis of ISSA obtained by incineration of sludge from WWTP Karlovac (incineration temperatures 800 °C and 1000 °C), used in experimental research the results of which are shown in this paper

4. Incorporation of ISSA in cement mortar

Experimental research was carried out for the purpose of this paper, including testing of the effect of incorporation of ISSA on properties of cement mortar in fresh and hardened condition. Description of research results includes comparison with results from recent worldwide practice.

4.1. Experimental research

4.1.1. Materials

The ISSA used in this research was obtained by incineration of stabilized and dried sludge from WWTP Karlovac in Croatia. Karlovac has a combined sewerage system, which means that WWTP along with wastewater from households and industrial wastewater also receives rain water. Maximum capacity of the wastewater treatment plant in Karlovac is 98,500 PE, and at present it operates with about 70% capacity. The plant includes the third stage of treatment. After the wastewater treatment process, primary and secondary sludge is dehydrated and anaerobically stabilized, with production of biogas which covers a part of power requirements of the plant. Dehydrated and stabilized sludge is temporarily stored at the plant location on a sheltered open spoil bank before final transport to the municipal mixed waste dump. For the purpose of this paper, dehydrated and stabilized sludge four months old from the temporary spoil bank was used. The sludge was additionally dried in laboratory drier at the temperature of 105°C, to reach the level of dry matter content above 90%. Dried sludge was then incinerated in laboratory conditions at different temperatures: 800°C, 900°C and 1000°C. Further, for preparing of cement mortar, cement CEM II/B-M (S-V) 42.5N was used, and dolomite sand 0/4 mm.

4.1.2. Dosage and experimental program

For the purpose of tests with cement mortar 36 different mixtures were prepared. The starting, i.e. reference mixture (without added ISSA) was marked as M0. Further, a separate mixture was prepared for each of the combinations of incineration temperatures (800°C, 900°C and 1000°C), w/c ratios (0.45, 0.50 and 0.55) and percentages of cement substituted by ISSA (5%, 10% and 20% of cement mass). Additionally, mixtures with 30% of ISSA were analyzed for incineration temperatures of 800°C and 900°C (and all three w/c ratios). Table 4 shows the marks and characteristics of all obtained mixtures. The reference mixture is marked M_0, and all others as $M_{n,,}$ where n represents the mass percentage of cement replaced by ISSA (5%, 10%, 20% and 30% respectively). Marks A, B and C define different w/c ratios (0.45, 0.50 and 0.55), while incineration temperatures of ISSA used are represented by asterisk (*) with mixture mark.

Table 4 Cement mortar mixtures

Mixture	ISSA share [%]	w/c ratio	Incineration temperature	Mixture	ISSA share [%]	w/c ratio	Incineration temperature
M_0 - A	0	0.45	-	M_{10} – C	10	0.55	800 °C
M_0 - B	0	0.50	-	M_{20} – A	20	0.45	800 °C
M_0 - C	0	0.55	-	M_{20} – B	20	0.50	800 °C
M_5 – A	5	0.45	800 °C	M_{20} – C	20	0.55	800 °C
M_5 – B	5	0.50	800 °C	M_{30} – A	30	0.45	800 °C
M_5 – C	5	0.55	800 °C	M_{30} – B	30	0.50	800 °C
M_{10} – A	10	0.45	800 °C	M_{30} – C	30	0.55	800 °C
M_{10} – B	10	0.50	800 °C	$M_5 – A^{*}$; $M_{20} – C^{**}$			

Note: * mixtures with ISSA obtained by incineration of dried sludge at 900 °C

** mixtures with ISSA obtained by incineration of dried sludge at 1000 °C

The following tests were carried out with each mixture

- setting time (initial and final)
- porosity
- workability (consistence)
- mechanical characteristics (compressive and flexural strength after 1, 7 and 28 days).

4.1.3. Setting time

In cases when a part of cement was substituted by ISSA in cement mortars, longer setting times were noticed [7,39,41]. According to some researches, with replacement of 20% of cement by ISSA initial and final setting times of cement mortars longer up to 2.5 times were noticed [57]. Also, greater fineness of ISSA particles elongates setting time due to higher water adsorption [38].

Initial and final setting times in this test were determined by the Vicat device by penetration of the needle of 1 mm cross-section into the body of molded cement mortar according to EN 480-2:1996, and the results are shown in Fig. 1.

Analysis of obtained results reveals the general trend of delaying of the initial and final setting time with increasing of the w/c ratio, but also with increased temperature of sludge incineration. The longest setting times were shown by mixtures with 10% and 20% shares of ISSA.

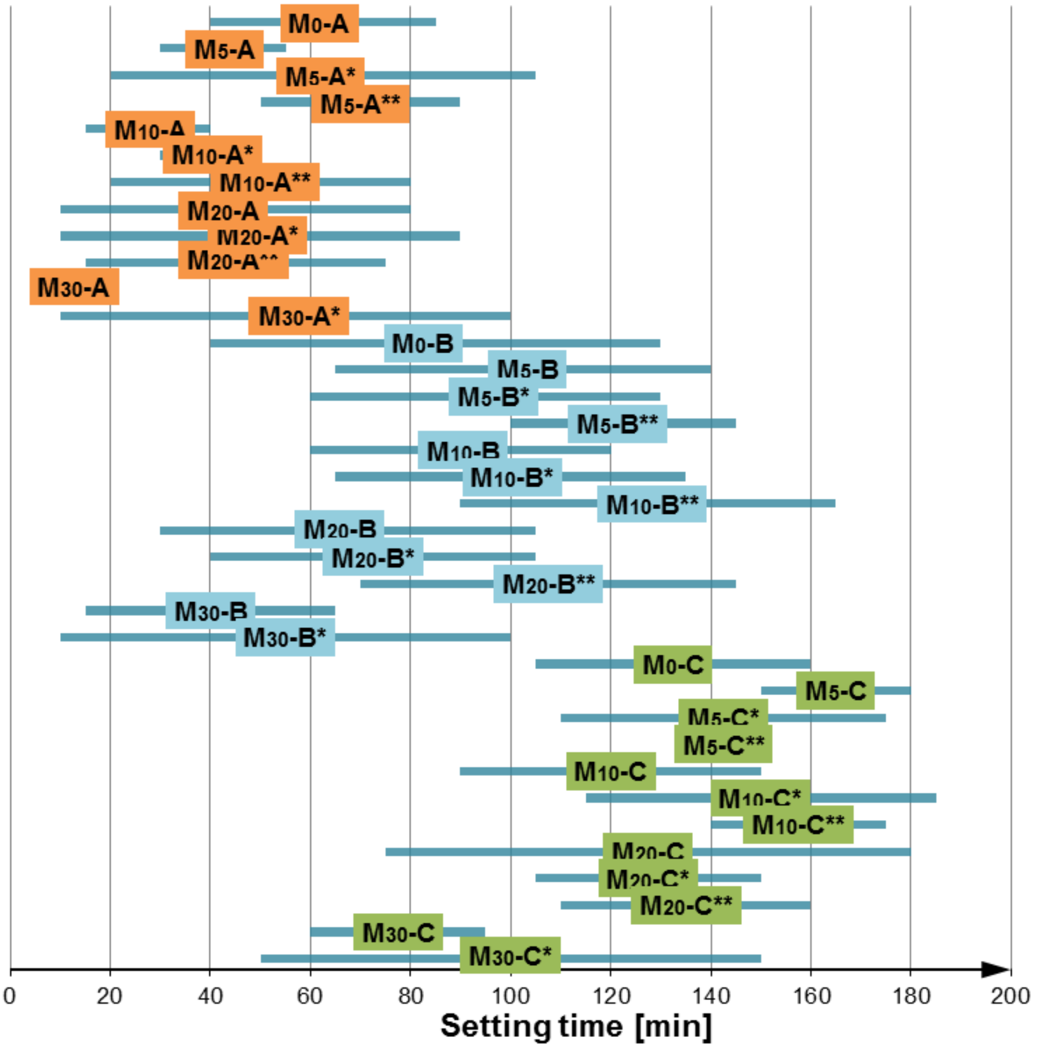

Fig. 1 Initial and final setting time for all analyzed mixtures

4.1.4. Porosity

Results of research carried out so far have shown that increasing of the share of ISSA leads to increasing of total porosity in relation to the reference mixture. Also, at higher shares of ISSA water requirements increase rapidly, becoming the critical factor leading to expressedly weak compactness of the sample [40]. According to research by Fontes et al. [56] it has been concluded that significant differences in porosity in relation to the reference mixture occur at ISSA shares of 10% and more.
Further, with increased temperature of incineration, the mass of ISSA decreases due to additional oxidation of organic matter. Porosity of ISSA is decreased with increasing of incineration temperature. The lowest porosity was reached for temperature of 1000°C [58].

This research has proved the general trend of porosity increase (determined according to HRN EN 1015-7:1998) with increased share of ISSA in cement mortar mixtures. Also, there is a porosity decreasing trend with increased temperature of sludge incineration. It is necessary to point out some inconsistencies in mixtures with lowest w/c ratio and higher shares of ISSA (in particular mixture M_{30}), because their difficult workability.

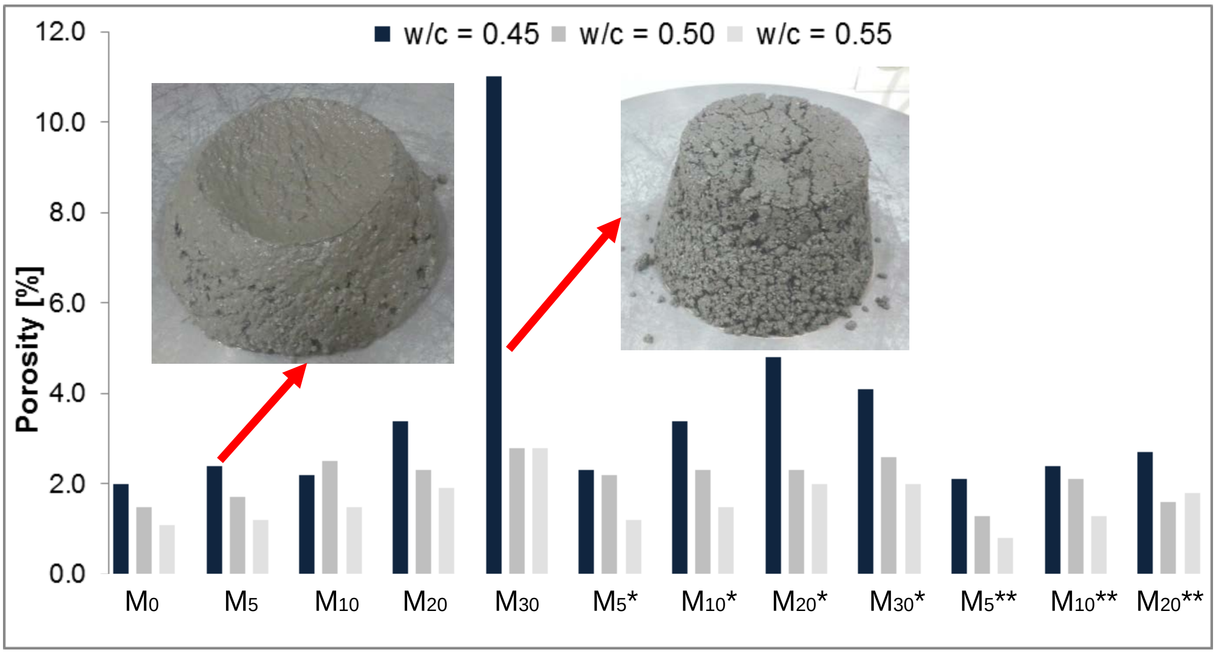

Fig. 2 Porosity for all analyzed mixtures

4.1.5. Workability (consistence)

Usual problems encountered when replacing a part of cement in cement mortars and concrete by ISSA are related mainly to reduced workability and increased water requirements, which may be compensated by alternative chemical additives. According to research by Monzo et al. [37] non-linear decline of workability is noticed in cement mortars with ISSA; at higher percentages of ISSA in cement mortar the decline of workability is less significant. Irregular morphology of ISSA particles causes the decline of workability, even at lower ratios of cement substitution by ISSA. According to some authors, poor workability may be compensated by increasing of ISSA particles fineness [38], adding of superplasticizer [37], or by adding flying ash of coal into the cement mortar mixture [43]. In the last case even some additional benefits are possible, such as a greater portion of replaced cement, extended hydration time and synergic effects of several materials with pozzolanic properties on the final product. It has been demonstrated [43] that adding of 20% of flying ash can compensate adverse influences in cement mortar caused by adding of 10% of ISSA as substitute for cement.

The workability of the mortars in this research was determined through the flow table test according to HRN EN 1015-3:1999.

Results obtained in this research prove increased water requirements and reduced workability with increased percentage of ISSA in cement mortar (Fig. 3 and Fig 4). Also, a workability improvement trend is noticed with increased temperature of sludge incineration; thus, the best properties regarding workability are shown by mixtures with ISSA produced at the temperature of 1000°C. It is important to note that there were considerable problems due to reduced workability with the mixture with lowest w/c ratio (0.45) and highest percentage of ISSA (30%).

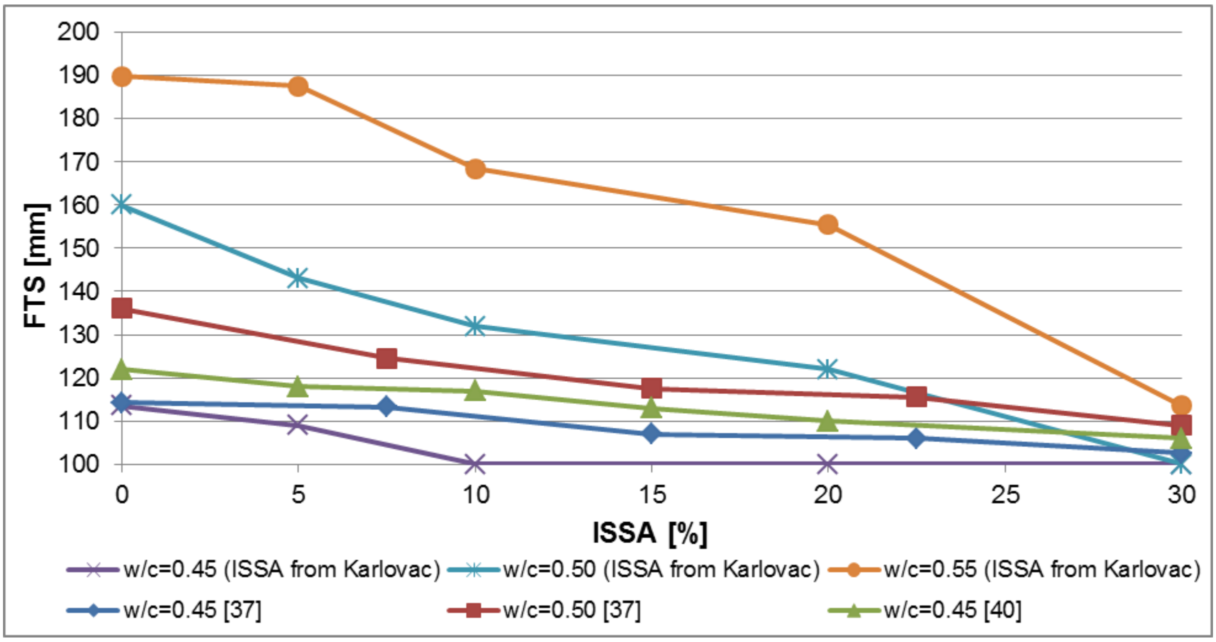

Fig. 3 Comparison of flow table spread (FTS) for ISSA (800°C) - cement blends with different w/c ratios with previous research results [37, 40]

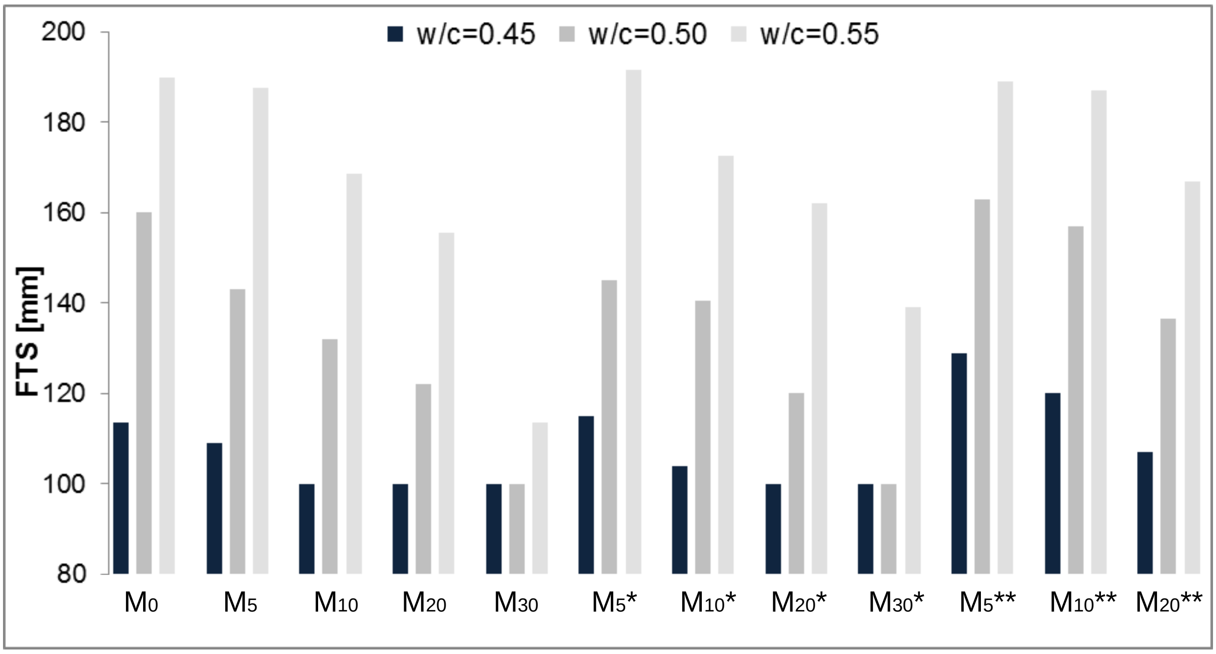

Fig. 4 FTS for all analyzed mixtures

4.1.6. Density of mortars

In this research, in accordance with different resulting densities of ISSA at different sludge incineration temperatures, minimum differences were noticed between densities of cement mortars in fresh conditions. The lowest density values in fresh conditions were obtained for M_{30} mixtures, i.e. mixtures with highest proportion of ISSA obtained at lowest incineration temperatures. For all other mixtures, mainly uniform values of density in fresh condition were obtained, in line with the density of reference mixture.

The same trend of mortar density reduction was noticed in hardened condition as well. In this case the lowest values were also obtained for mixture M_{30} -A (1.99 g/cm^3) which is about 85% of density of the reference mixture.

4.1.7. Testing of mechanical properties

In testing of mechanical samples of cement mortars, standardized 4x4x16 cm samples were used, like by the majority of other authors dealing with this problem [39, 40, 46, 50, 48, 59]. The samples were mechanically mixed and then compacted. After the curing time (1, 7 and 28 days), both flexural and compression strength tests were performed. All the procedure was carried out in accordance to the European (and Croatian) Standard HRN EN 1015-11:1999 and EN 1015-11:199/A1:2006. Three specimens of each mixture were prepared. Specimens were stored in a moisture room from their fabrication day until the test date (Fig. 5).

Fig. 5 Mortar specimens prepared for mechanical strength tests in the laboratory at the Faculty of Civil Engineering University of Zagreb

All researches carried out so far show the general trend of reduction of compression and flexural strength with increased proportion of ISSA. Still, comparing of results by different authors one may notice considerable differences in absolute values of the results. For instance, substitution of 20% of Portland cement by ISSA results in different reductions of compression strength: 24% [44], 52% [38], 32% [45].

It is important to note that adverse effect of adding ISSA loses importance in later stages of curing (the influence on reduction of 28-day strength is considerably lower in relation to strengths after 7 days) [39].

In some researches, mortars with ISSA have shown equal or even higher strengths in particular phases of curing. According to research by Fontes et al. [56] cement mortars in which 10-30% of cement was substituted by ISSA have shown flexural strengths equal to those of referent mortars only at later curing time (28 days), while compression strengths of reference mortars were achieved in considerably shorter times (up to 7 days)

Another important factor is the influence of the type of commercially available cement on behavior of mortars in which a part of cement is replaced by ISSA [40]. ISSA is particularly compatible with cements with high content of C_3A in cement mortars, and in such cases the decline of mechanical properties after 28-day curing has not been recorded [42].

According to Chen et al. [46] the strength (compression and flexural) of analyzed mortars decreases linearly with increased proportion of ISSA as replacement for cement in the mixture. This is explained by two hypotheses: too much water required to maintain workability, and CaO content in ISSA (less than 10%) which influence the hydraulic properties. According to this research for mixtures with ISSA percentage of 10%, the decrease of flexural and compression strength was less than 25% in relation to control samples (without ISSA).

The results published by Monzo et al. [36,60,61] are particularly outstanding, because they record moderate increase of compression strength of mortars with ISSA in relation to reference samples. They show average strength growth of mortars from 8.3% to 15.3% when 15% of cement is replaced by ISSA in mortars with mixing ratio 3:1. These samples were cured by immersion in water at 40°C, and the increased temperature of curing is considered the reason for different recorded results.

According to research by Baeza-Brotons et al. [49], samples with up to 5% of ISSA have shown even somewhat higher compressive strengths in relation to reference samples. ISSA percentages above 5% give somewhat lower values of compressive strengths, but still above 90% of compressive strength of reference samples. This research has observed the growth of flexural strength with increase of curing time, but flexural strengths in all cases remained lower than those of the reference samples (between 71% and 85% of reference values).

Compressive strength of mortars with ISSA increases with increased fineness of ISSA particles (smaller granulation). Improvements are present due to pozzolanic properties of ISSA particles [44]. All obtained values of flexural strength are shown in Fig. 6, and compressive strength in Fig 7.

Regarding to both, flexural and compressive strength, it can be seen in all cases that the values are positively increased with the curing time. This means that the pozzolanic reaction in mortars with ISSA positively influences the increase of flexural and compressive strength. Early strengths (1 day) were particularly sensitive to adding of ISSA. The highest flexural strength after 1 day was that of the reference mixture, and the highest compressive strength was that of the mixture M_5-A, i.e. with minimum ISSA content. The poorest results of 1 day strengths were those for mixture M_{30}-A (flexural strength and compressive strength were about 65% of reference mixture strengths)

Best values of late strengths (28 days) were shown by mixtures M_{20}-A* for flexural strength, and mixture M_5-A* for compressive strength (both 121% of strengths of the reference mixture).
Analysis of all results shows that the best results regarding compression and flexural strengths were those of mixtures with smallest w/c ratio (0.45), and the worst results were those of mixtures with highest w/c ratio (0.55).

From this set of data it is not possible to draw a general conclusion regarding the effect of incineration temperature on mechanical properties of cement mortar samples with incorporated ISSA, and in this field the need for additional research is envisaged, but it can be concluded that already at 800°C, satisfactory results are obtained.

In the entire scope of research it is necessary to point out particularly the mixture M_{20}-A* which has shown in all phases of curing higher values of both compressive and flexural strength in relation to the reference mixture. The only negative deviation of this mixture in relation to the reference mixture was for 1 day flexural strength. After 28-days curing, flexural strength of mixture M_{20}-A* was 121% of reference flexural strength, while for the same period its compressive strength was 111% of referent compressive strength. Fig. 8 shows the flexural strength as a function of curing time for the reference mixture and mixtures with 20% of ISSA for different incineration temperatures and w/c ratios, while Fig. 9 shows the same comparison for compressive strength. Therefore the conclusion arises that exactly this mixture is optimum regarding mechanical properties, i.e. w/c ratio of 0.45, sludge incineration temperature of 900°C and ISSA portion of 20% of cement mass.

It is also a very positive fact that all the specimens with an age of 28 d exceed the strength class of the cement used (42.5 MPa).

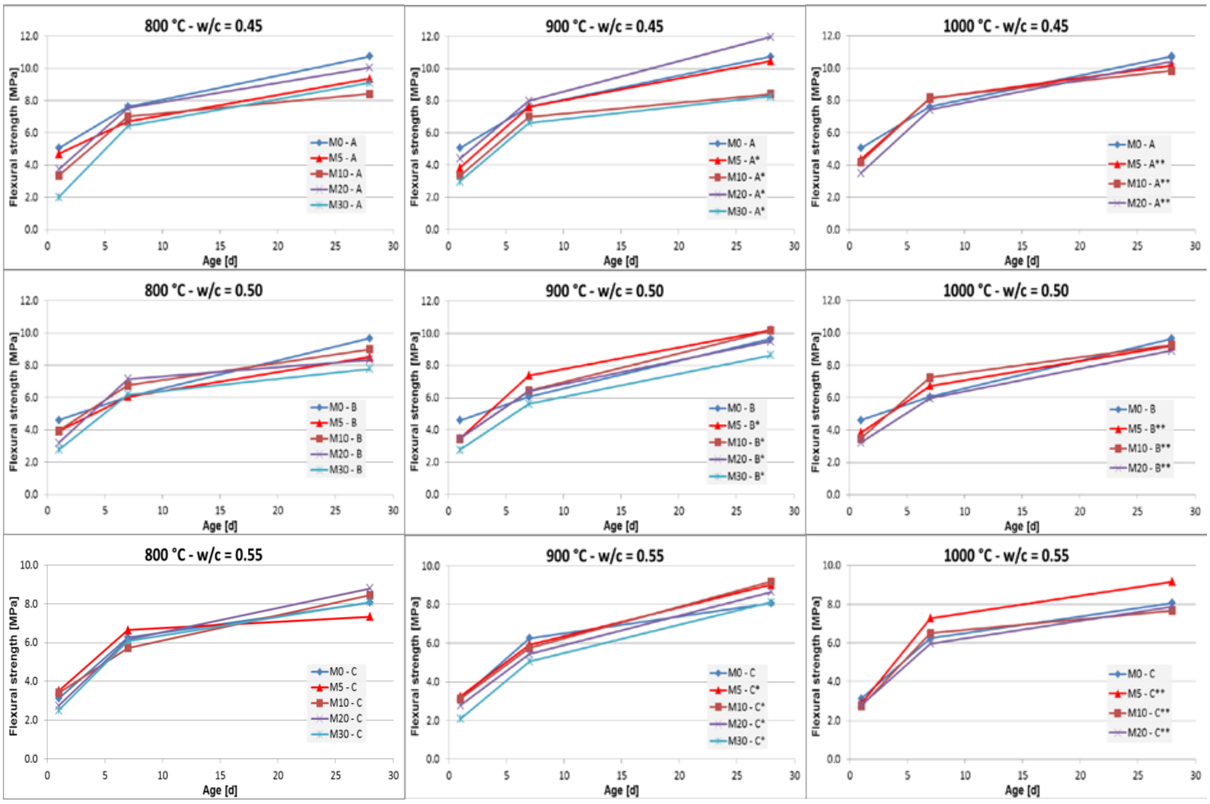

Fig. 6 Flexural strength as a function of curing age

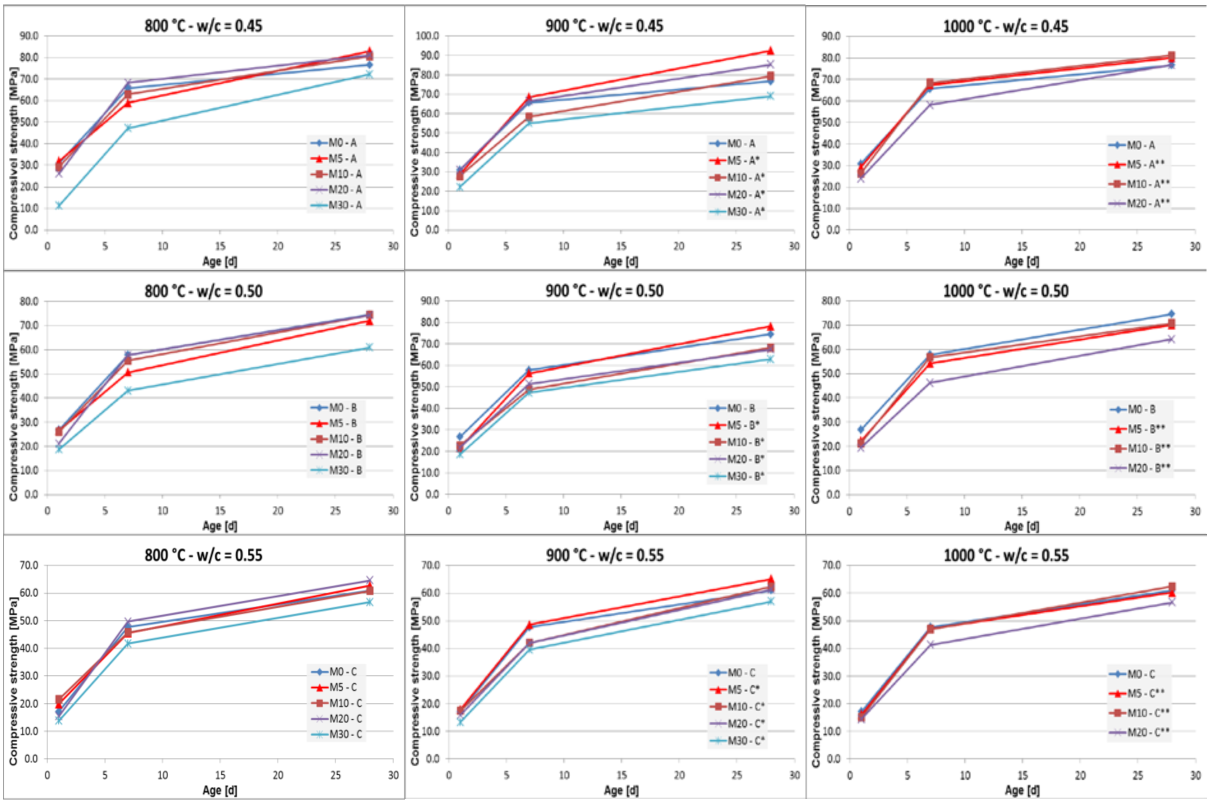

Fig. 7 Compressive strength as a function of curing age

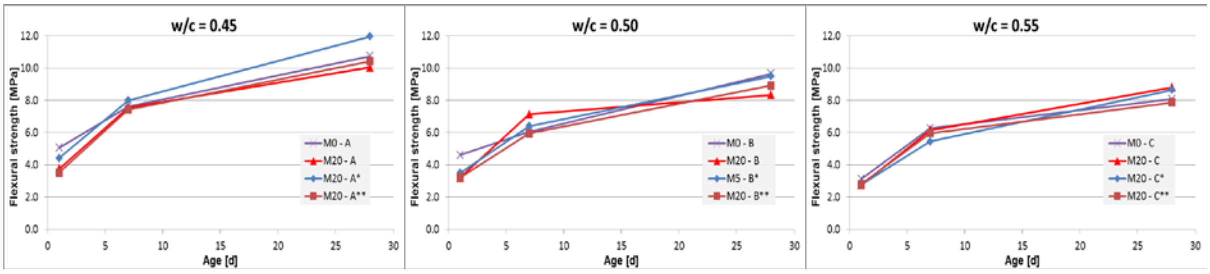

Fig. 8 Flexural strength of the mixtures with 20% ISSA for different w/c ratios and incineration temperatures

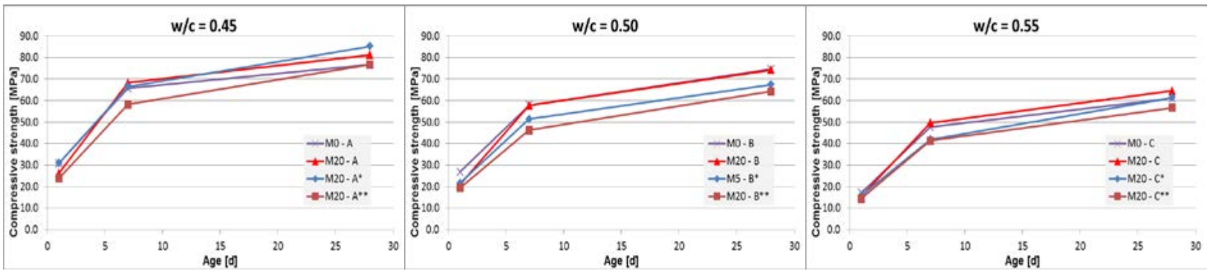

Fig. 9 Compressive strength of the mixtures with 20% ISSA for different w/c ratios and incineration temperatures

5. Possible problems in ISSA recycling

Hereafter, the basic aspects of potential problems in obtaining and recycling of ISSA are given.

Sludge incineration – production of ash
It is a fact that the content of flying ash in exhaust fumes of the thermal processing plant is high, and in flying ash contains low level of heavy metals, and therefore the gases generated by combustion must be treated. Still, according to available data about modern technological solutions by using best available technology [62] such emissions are always below legal limits. According to the data of the European Commission [63], when sludge is incinerated without mixing with other waste, concentration of dioxins and NO compounds in the exhaust fume is low enough, not requiring additional treatment above regular.

Leaching of heavy metals
The environmental impact of ISSA when used in cement materials has been elaborated by several authors, first of all by analyzing of leaching from concrete samples, and the conclusion has been reached that according to all parameters, it is within permissible limits, unlike leaching from ash in the spoil bank, where excessive leaching of some heavy metals occurs, in particular Mo, Sb and Se [46, 64].

Loss of source of phosphorus
The major drawback of ISSA use in civil engineering is the loss of potentially valuable phosphorus contained in ISSA. This possibility of additional use of ISSA for obtaining of phosphorus, as a non-renewable and limited resource on Earth, and important in many branches (in particular in agriculture) has recently become a topic [7].

Legislation
Another limiting factor in the use of ISSA in concrete industry is the lack of comprehensive legislation and guidelines at EU level that would define the possibilities and fields of use.

Sociological impacts
It is necessary to mention also the adverse sociological impacts accompanying the use of such materials, due first of all to inadequate information level of the population about health, safety and ecological acceptability of final products with incorporated ISSA. With respect to this, the question arises about readiness of the market (cement factories, concrete batching plants) to accept certain quantities of ISSA for use in concrete industry.

6. Conclusion

Due to the increasing attention given by water utilities to incineration of sewage sludge and as traditional disposal routes become increasingly restricted there has been many research on recycling and recovery of ISSA lately. Most of them focused on the use of ISSA as a partial cement replacement material in concrete industry. Results showed that mortar materials prepared by substituting part of cement with ISSA exhibited similar mechanical properties as reference mixtures without ISSA addition.

The partial replacement of Portland cement by ISSA promoted an increase in the total porosity of the reference mixtures. Initial and final setting time was increased with increasing the addition of ISSA to cement mortar mixtures. Workability of cement mortars was significantly affected with larger ISSA additions, especially for the least w/c ratio (0.45). However, these negative impacts are expected to be conducted by addition of superplasticizer.

Still, these recycling applications fail to consider the potentially valuable phosphorus content of ISSA and also may encounter market and wider public acceptance due to the lack of information provided. A high level of environmental protection during the incineration process and meeting all environmental requirements for use of ISSA in concrete industry is required. Unique legislation on this issue at EU level would also contribute to its wider acceptance.

Considering technical specification, the utilization of ISSA in concrete industry therefore appears possible and feasible.

For the conclusion may be mentioned that in the definition of best recovery procedure for the sewage sludge, in accordance with the principles of energy efficiency and minimum environmental impact, thermal recovery with the use of ISSA in the construction industry is acceptable solution.

Acknowledgments

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