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CRUSHED SILICOMANGANESE SLAG AS AGGREGATE FOR ASPHALT AND CONCRETE

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Abstract: *The study of aggregate made of crushed silicomanganese slag is a pioneering work because, according to available informations, such material has so far never been produced and used in the world. The aggregate was tested according to European standards for asphalt and concrete, and with other technological and environmental regulations for industrial materials. The test results confirmed that this aggregate from Šibenik TEF Factory can be used in asphalt and concrete production so it was used as an ingredient in asphalt placed at the Vinjani Donji - Vinjani Gornji road near the Croatia and Bosnia-Herzegovina border.*

Key words: *silicomanganese (SiMn) slag, crushing facilities, screening plant, industrial aggregate.*

1. INTRODUCTION

The Šibenik-based Factory of Electrodes and Ferroalloys (TEF) was founded in 1904 during the Austro-Hungarian monarchy, and was closed in 1994 in the Republic of Croatia. During these 90 years of production, manganese ore was delivered from various countries, and used in the process of manganese alloys factory manufacture. The slag was dumped next to the factory, at the sea coast. The quantity of slag dumped in this way is estimated at 800000 tons.
The colour of the freshly fractured slag is green or blue (Figure 1) but later the manganese particles on the surface oxidise, and the colour changes to gray-black.

Figure 1. Coarse aggregate of SiMn slag

2. EXTRACTION OF MANGANESE REMAINS FROM THE SLAG

It was established in 2007 that the slag still contains 5-15 mas.% of manganese alloys, so the English company Wesbrooke Resources, in cooperation with the Zagreb-based company MLM d.o.o., decided to separate and use the remaining manganese. Thus the companies Visoki Napon d.o.o. and Gratex d.o.o. were engaged to crush the slag in the primary jaw crusher down to the particle size of max 150 mm. The crushing continued in cone crushers which produced the crushed SiMn slag in 0/25 mm particle size. This material was then screened by wet process into two fractions, 0/5 and 5/25 mm, and the fine muddy particles were separated by sedimentation. A special plant was installed in Šibenik in order to extract manganese alloys from the coarse 5/25 mm fraction (Figure 2).

Figure 2. Screening plant for separation of 0/5 and 5/25 mm fractions, with extraction of manganese alloys from the coarse fraction

Since the density of manganese and manganese-alloy grains is greater than the rest of the slag, they were separated from the remaining aggregate by sedimentation in water through a special screen. The remaining 5/25 mm fraction grains were used as raw material in the production of aggregate fractions for asphalt, concrete and pavement subbase.
The manganese and manganese-alloy grains has not as yet been separated from the finer 0/5 mm fraction since a new plant still has to be installed for that purpose.

3. CRUSHING AND SCREENING OF THE SLAG

The SiMn slag, with very hard and strong grains, was crushed in heavy-duty cone crushers manufactured by the Swedish company Svedala, and a favourable cubic shape was obtained by gradual adjustment of operating regime of the crusher.
The crushing and screening plant produces aggregate fractions 0/4, 4/8, 8/11, 11/16 and 16/22 mm for asphalt and concrete (Figure 3).

Figure 3. Crushing and screening plant for industrial aggregate. The manganese extraction plant can be seen in the background.

4. AGGREGATE TESTING

In the period from year 2008. to 2010., the physicomechanical, mineralogical, chemical, radiological properties and influence on environment of the crushed industrial aggregate [1] were tested in the following Croatian institutions: CSS d.o.o. (physicomechanical properties); Faculty of Science - Geological Department (mineralogical analysis and determination of harmful ingredients); Ruđer Bošković Institute (radiological analysis, analysis of heavy metals in raw sample and in eluates); Institute of Public Health - City of Zagreb (analysis of harmfull compounds in raw sample and in eluates), and Cemtra d.o.o. (chemical analysis).
Old regulations from Croatia and Bosnia & Herzegovina excluded industrial aggregate from use, so we didn't have any experience with the crushed slag aggregate. Although new European standards allow use of industrial aggregate, they do not mention the SiMn slag. The technical committee CEN/TC 154 hadn't any experience with such a material, which is also confirmed by the lack of literature about this topic. The information however exists that the SiMn slag has been tested as pozzolan additive to cement [2], but not as aggregate.

5. PHYSICOMECHANICAL PROPERTIES OF AGGREGATE

Physicomechanical properties of aggregate were tested according to EN 13043 and EN 12620 standards in CSS central laboratory in Zagreb. Significant physicomechanical properties of aggregate are described and the corresponding results are presented in Table 1.

5.1 Grain size distribution

Fine aggregate 0/4 mm belongs to the grading category G_F 85 with the fineness modulus of FM = 2.88 ± 0.12 (category: CF), while coarse fractions above 4 mm belong to the grading category G_C 90/15.
The quantity of fine particles ≤#63 μm in the 0/4 mm fraction ranged between 4 and 8%, and amounted to less than 1 % in the coarser fractions. The quality of fine particles, tested by the methylene blue method (category MB_F 10 was achieved) and sand equivalent method (SE≥80 was obtained, while ≥60 % hight is required for asphalts), is considered favourable since the fine particles consist of dust appeared after the crushing of SiMn slag.

5.2 Particle shape

The particle shape and angularity of fine aggregate 0/4 mm in size was determined by measuring the time needed for the aggregate to pass through the standard funnel. The category E_{CS} 20 was achieved.
The particle shape of coarse aggregate was checked by determining the flakiness index and the shape index. Categories FI_{20} and SI_{20} were achieved, which meet requirements for asphalts and concrete.

5.3 Density and water absorption

The SiMn aggregate is characterised by a very low porosity. The density was expressed as particle density on an oven-dried basis (ρ_{rd}) and particle density on a saturated surface-dry basis (ρ_{ssd}). The densities (ρ_{rd} and ρ_{ssd}) vary from 2.94 to 3.00 Mg/m3, so the fractions were classified as normal weight aggregates (2.00-3.00 Mg/m3).
The water absorption for fine aggregate ranges from 0.5 to 0.9 mas.%, while this value for coarse aggregate varies from 0.2 to 0.6 mas.%. The results obtained are compliant with the category $WA_{24}1$.

5.4 Shrinkage

The drying shrinkage of SiMn aggregate amounts to 0.020%, which is much less than the allowed ≤0.075% for concrete.

5.5 Resistance to fragmentation, wearing and abrasion

The resistance of aggregate to fragmentation (Los Angeles), wear (Micro Deval) and abrasion (AAV) were tested and it was established that SiMn slag is highly resistant in wearing courses of asphalt and concrete in all types of traffic load, but also that this aggregate is also highly resistant to other types of abrasion.

Table 1. Physicomechanical properties of aggregate and categories of aggregate fractions for asphalt and concrete

FRACTION # mm	AGGREGATE PROPERTIES	STANDARD	UNITS	RESULT RANGES	CATEGORIES EN 13043, EN 12620
0/4	Grain size distribution (G_F)	EN 933-1	# mm	0 - 5.6	G_F85; f_{10}
	Flow coefficient (E_{CS})	EN 933-6	s	23 - 24	$E_{CS}20$
	Sand equivalent test (SE)	EN 933-8	%hight	80 - 85	SE ≥ 80
	Methylene blue test (MB_F)	EN 933-9	g/kg	0.2 - 0.4	MB_F10
	Particle density (ρ_{rd})	EN 1097-6	Mg/m^3	2.94 - 2.98	$(2.96±0.05)^{+}$
	Particle density (ρ_{ssd})	EN 1097-6	Mg/m^3	2.96 - 3.00	$(2.97±0.05)^{+}$
	Water absorption (WA_{24})	EN 1097-6	%mas.	0.6 - 0.8	$WA_{24}1$
	Fineness modulus (FM)	EN 12620	-	2.76 - 3.00	CF
4/8	Grain size distribution (G_C)	EN 933-1	# mm	2 - 8	$G_C90/15$; $f_{1.5}$
	Flakiness index (FI)	EN 933-3	%mas.	15 - 18	FI_{20}
	Shape index ≥3:1 (SI)	EN 933-4	%mas.	14 - 17	SI_{20}
	Particle density (ρ_{rd})	EN 1097-6	Mg/m^3	2.95 - 2.97	$(2.96±0.05)^{+}$
	Particle density (ρ_{ssd})	EN 1097-6	Mg/m^3	2.96 - 2.98	$(2.97±0.05)^{+}$
	Water absorption (WA_{24})	EN 1097-6	%mas.	0.4 - 0.6	$WA_{24}1$
8/11	Grain size distribution (G_C)	EN 933-1	# mm	4 - 11	$G_C90/15$; $f_{1.5}$
	Flakiness index (FI)	EN 933-3	%mas.	8 - 11	FI_{20}
	Shape index ≥3:1 (SI)	EN 933-4	%mas.	7 - 8	SI_{20}
	Particle density (ρ_{rd})	EN 1097-6	Mg/m^3	2.94 - 2.96	$(2.95±0.05)^{+}$
	Particle density (ρ_{ssd})	EN 1097-6	Mg/m^3	2.95 - 2.97	$(2.96±0.05)^{+}$
	Water absorption (WA_{24})	EN 1097-6	%mas.	0.2 - 0.5	$WA_{24}1$
	Polished stone value (PSV)	EN 1097-8	-	35 - 36	PSV_{34}
	Aggregate abras. value (AAV)	EN 1097-8	-	1.6 - 2.0	AAV_{10}
	Aggregate-bitumen affinity *	EN 12697-11	%	80 - 85	>80
	Agg.-polymer bitum. affinity *	EN 12697-11	%	>90	>90
11/16	Grain size distribution (G_C)	EN 933-1	# mm	8 - 16	$G_C90/15$; $f_{1.5}$
	Flakiness index (FI)	EN 933-3	%mas.	11 - 14	FI_{20}
	Shape index ≥3:1 (SI)	EN 933-4	%mas.	6 - 13	SI_{20}
	Resistance to wear (M_{DE})	EN 1097-1	-	5.4 - 6.3	$M_{DE}10$
	Resist. to fragmentation (LA)	EN 1097-2	%mas.	15 - 16	LA_{20}
	Particle density (ρ_{rd})	EN 1097-6	Mg/m^3	2.94 - 2.96	$(2.95±0.05)^{+}$
	Particle density (ρ_{ssd})	EN 1097-6	Mg/m^3	2.95 - 2.98	$(2.96±0.05)^{+}$
	Water absorption (WA_{24})	EN 1097-6	%mas.	0.2 - 0.5	$WA_{24}1$
	Resist. to freezing-thawing (F)	EN 1367-1	%mas.	1.2	F_1
	Magnesium sulphate test (MS)	EN 1367-2	%mas.	9 - 11	MS_{18}
16/25	Grain size distribution (G_C)	EN 933-1	# mm	8 - 25	$G_C90/15$; $f_{1.5}$
	Flakiness index (FI)	EN 933-3	%mas.	11 - 16	FI_{20}
	Shape index ≥3:1 (SI)	EN 933-4	%mas.	9 - 13	SI_{20}
	Particle density (ρ_{rd})	EN 1097-6	Mg/m^3	2.93 - 2.97	$(2.95±0.05)^{+}$
	Particle density (ρ_{ssd})	EN 1097-6	Mg/m^3	2.94 - 2.98	$(2.96±0.05)^{+}$
	Water absorption (WA_{24})	EN 1097-6	%mas.	0.4 - 0.7	$WA_{24}1$

** Refers to asphalt only; $^{+}$ Declaration of the aggregate producer*

The resistance of aggregate to fragmentation is highly dependent on grain shape, whereas the resistance to wear and abrasion is mostly dependent on mechanical properties of slag.
The tested aggregate belongs to the categories LA_{20}, $M_{DE}10$ and AAV_{10}, respectively. Results obtained by testing resistance of aggregate to fragmentation (LA) varied from 15 to 16 mas. %, which is compliant with Croatian regulations relating to wearing courses of asphalt in road construction.

5.6 Resistance to frost

The resistance to frost was tested by the $MgSO_4$ crystallisation method (magnesium sulphate test) and by the freezing/thawing method. The results obtained are compliant with the stringent category MS_{18} and F_1.

5.7 Resistance to polishing

In the scope of preparations for the testing, aggregate grains glued onto rounded rectangular bakelite plates were subjected to two-staged wet polishing, first by rough and then by fine corundum sand, using rotary rubber wheels. The resistance to polishing of polished SiMn slag aggregate was estimated by making comparison with the reference quartz dolerite aggregate of controlled origin, from Great Britain. The polishing stone category PSV_{34} was obtained.

5.8 Aggregate-bitumen affinity

The adhesion of bitumen to aggregate grains was tested with an ordinary bitumen and with polymer bitumen. Enveloped aggregate particles were subjected to mechanical treatment aimed at separating the bitumen film from the particle surfaces.
Test results were compliant with requirements for bituminized load-bearing layers (>80% of grain surfaces were enveloped), while polymer bitumen results were compliant with requirements for wearing courses (>90% of grain surfaces were enveloped) according to Croatian regulations.

5.9 Aggregate-mortar adhesion

As to concrete, it was established that the cement matrix adheres poorly to glassy silicate aggregate grains, which negatively affects its tensile strength.

6. INFLUENCE OF AGGREGATE ON THE VOLUME OF CONCRETE AND ASPHALT

The influence of the crushed slag aggregate on the increase in volume of concrete and asphalt is due to subsequent hydration of individual reactive oxides, or to alkali-silica reaction of the non-crystallized glassy base with alkalis from cement.

6.1 Stability of volume

Unlike natural stone aggregate, the crushed slag aggregate must obligatorily be subjected to the volume stability testing. In fact, the asphalt and concrete containing industrial aggregate may be subject to subsequent hydration of free MgO (periclase) or CaO (lime), which is why the change in volume is checked by accelerated hydration based on overheated water vapour according to EN 1744-1. The testing of volume stability of the SiMn slag aggregate has revealed that the increase in volume ranges from 0.5 to 1-8 vol. % (category $V_{3.5}$), which is a favourable result for asphalts, while the corresponding requirements have not as yet been set in the European standard for concrete.

6.2 Potential alkali-silica reaction

The SiMn slag aggregate contains considerable quantities of the amorphous silicate matter in which manganese and synthetic mineral particles are finely dispersed. It was necessary to demonstrate that this phase of slag does not react with alkalis (Na_2O and K_2O) from cement, which would cause an alkali-silica reaction, or that it does not cause a subsequent increase in the volume of concrete due to humid environment.
Test results according to ASTM C 1260-94 did not confirm the alkali-silica reaction.
About 35 years ago, concrete cubes were made with the SiMn slag aggregate and they were used to construct the pavement structure within the TEF factory area. After the long-term use in variable hygroscopic conditions, there is now no indication of alkali-silica reaction on the pavement structure, except for the surface peeling of cement mortar that was exposed to heavy traffic load.

7. MINERALOGICAL AND CHEMICAL ANALYSES

The mineralogical and chemical analyses of the SiMn slag phase system were conducted by X-ray diffraction, electronic microscopy and standard chemical analyses.

7.1 Mineralogical analysis

The SiMn slag consists of the glassy silicate substance mixed with dispersed manganese particles and various nano- to micro-crystalline phases (Figure 4), which were formed by reaction in melt or during subsequent re-crystallisation.
Here we find synthetic minerals (Figure 5): clinopyroxene, akermanite, high-temperature quartz, ramsdellite and, in smaller quantities, calcite, strichtite, gupeite, barite, merwinite and brucite. Some of these phases originate from ores used in the silicomanganese extraction technology, such as for instance alabandite (MnS) and rutile (TiO_2).

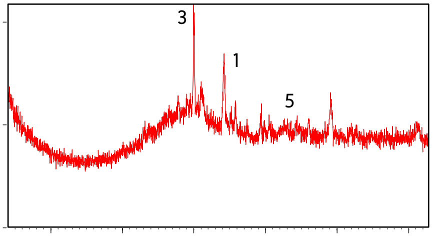

Figure 4. Sample rich in silicomanganese glass which causes increase in the base line. The presence of alabandite (1), clinopyroxene (3) and ferosilicomanganese (5) can be seen on the diffractogramme.

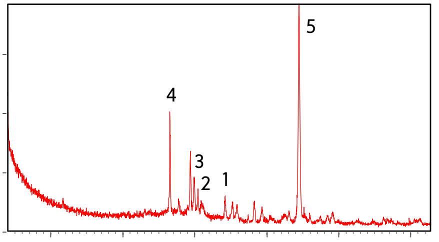

Figure 5. Synthetic minerals: alabandite (1), akermanite (2), clinopyroxene (3), high-temperature quartz (4) and ferosilicomanganese (5) identified by the X-ray diffraction

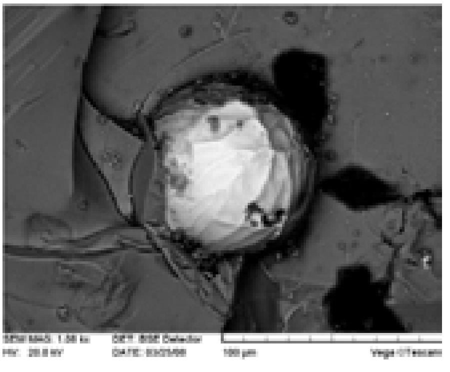

Figure 6. Ferosilicomanganese nodule ø 60 µm in the glassy slag base, recorded using scaning electrone microscope.

The slag contains residual quantities of manganese, iron and silicon alloys. These alloys occur in form of balls (Figure 6) several µm to several mm in diameter, and less often in form of lumps.

Alloys are not of uniform composition, and the prevailing one is ferosilicomanganese ($Fe_{1.6}Mn_{8.4}Si_6$). Manganese alloys were mostly extracted by mechanical procedure, and smaller quantities still remain in aggregate.
These phases in SiMn slag are equivalent to natural materials that occur in environments characterized by high-temperature contact metamorphosis, or in basic rocks of volcanic origin.

7.2 Chemical analysis

Average quantities of the following oxides were determined by chemical analysis of the SiMn slag: SiO_2 (34.3%), Al_2O_3 (15.3%), Mn_2O_3 (15.6%), CaO (8.3%), which together amount to 73.5% of the mass, while the oxides MgO (5.1%), K_2O (1.9%), Na_2O (0.6%), Fe_2O_3 (1.1%), TiO_2 (0.7%) were found in smaller quantities. The quantity of insoluble residue was 1.1%.
The pH factor of the eluate solution was 8.8.
The analysis of crushed grains revealed that the total quantity of sulphur is 0.7...1.6% of the mass (the allowable value is $S \leq 2\%$), and that the quantity of sulphate (SO_3) soluble in acid was 0.20...0.24% (the allowable value is 1% for category AS1). The sulphide content was 0.6...1.5% of the mass, which is mostly related to mineral alabandite. Sulphates and sulphides are less harmful in SiMn slag then they are in natural aggregate, as here they occur as finely dispersed particles trapped within a dense glassy base.

8. ENVIRONMENTAL REQUIREMENTS

European standards refer to the Mandate M/125 [3] which contains specifications for testing and evaluation of environmental requirements prior to the use of industrial aggregates.

8.1 Radioactivity of the slag

The radioactivity of aggregate samples involved activity concentration measurement for radioactive elements: potassium (^{40}K), radium (^{226}Ra i ^{228}Ra), thorium (^{232}Th), uranium (^{228}U) and artificial radionuclides, using the multi-channel gamma-spectrometer.
The following limits are applied for construction materials in the Croatian regulations on radioactivity and ionizing radiation: ≤3000 Bρ/kg for ^{40}K, ≤200 Bρ/kg for ^{232}Th i ≤300 Bρ/kg for ^{226}Ra, and the following requirement must also be met:
$$C_{Ra\text{-}226}/3000 + C_{Th\text{-}232}/200 + C_{K\text{-}40}/300 \leq 1$$
The manganese ore was supplied from various countries and so the radioactivity of material is not uniform. Although some samples did exceed the allowable limit, the mixed aggregate, regarded as a whole, is compliant with the above mentioned criteria.

8.2 Releasing of heavy metals

The releasing of heavy metals was tested according

to DIN 38414-4 and EN 1744-3. The analyses were conducted on eluates obtained after aggregate leaching. The eluate analysis according to DIN was conducted using the gas and liquid chromatography and mass spectrometry, and then by absorption and infrared spectrometry. The analysis according to EN 1744-3 consisted of X-ray fluorescence measured on MiniPal 4 spectrometer.
According to test results, it was established that the quantities of heavy metals that could be released into environment by Šibenik SiMn slag aggregate are below the limit values.

8.3 Releasing of PAH-s and other harmful substances

Eluates were tested to determine the content of phenols, fluorides, chlorides, sulphates and dissolved organic carbon. In addition, the presence of following substances was also determined: polycyclic aromatic hydrocarbon (PAH), polychlorinated biphenyl (PCB), aromatic hydrocarbon (BTEX), mineral oil and total organic carbon (TOC).
Test results show that the quantities of PAH-s and other harmful substances that could pollute the environment are below the limit values.

Figure 7. Vinjani Donji - Vinjani Gornji road

9. CONCLUSION

The study and use of crushed SiMn slag aggregate originating from the TEF factory in Šibenik is a pioneering work as, according to available informations, this material has never been used as civil ingeneering aggregate in the world.
Cone crushers were used to obtain a favourable shape of aggregate particles, and then a good quality grading was obtained by sieving into fractions: 0/4, 4/8, 8/11, 11/16 and 16/22 mm. The SiMn slag aggregate is characterized by an exceptional resistance to fragmentation (LA<16%), wear, abrasion and frost, and its water absorption amounts to less than 1 % of the mass. The aggregate density corresponds to normal weight, i.e. it amounts to 2.97 ± 0.03 Mg/m^3. It was established that the aggregate's volume stability remains unaltered when subjected to various physical and chemical tests. The content of harmful substances was within limits allowed for concrete and asphalt.
The Šibenik industrial aggregate consists of glassy silicate mater mixed with fine particles of manganese and various nano- to micro- crystalline phases. These include synthetic minerals: clinopyroxene, akermanite, alabandite, high-temperature quartz, ramsdelite, etc., and ferosilicomanganese alloys.
The surface of aggregate particles is smooth. The adhesion to standard bitumen is compliant with requirements for asphalt base layers and, with polymer bitumen, it is also satisfactory for wearing courses.
According to eluate testing, the SiMn slag aggregate is an environment friendly material. As the manganese ore was supplied from various countries, some slag samples do exceed allowed levels of radioactivity, but the mixed aggregate, taken as a whole, is satisfactory.
It was established that the tested SiMn slag is suitable for use as aggregate in the fabrication of asphalt base layers or concrete in civil engineering applications.
The aggregate was used in the fabrication of the asphalt at the Vinjani Donji - Vinjani Gornji road in Croatia (Figure 7).

10. LITERATURE

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