Concrete containing steel slag aggregate: Performance after high temperature exposure

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ABSTRACT: Due to the fact that steel slags are made at the temperatures up to 1600°C, utilisation in concretes with improved properties after the exposure to high temperatures is considered in this paper. An experimental program was designed and carried out to study properties of four steel slag based concrete mixtures with different types of cement pastes prior and after heating up to 800°C. Dolomite based concrete mixture was studied as a reference mixture. The results obtained have shown that residual mechanical properties of concrete made with steel slag aggregate are comparable to the reference dolomite concrete up to the temperature of ca. 550°C, while at higher temperatures, steel slag exhibits mineral transformation followed by unstable volumetric expansion which reflected negatively on the residual properties of slag based concrete mixtures. This expansion become stable if the slag was previously heated up to 1000°C.

1 INTRODUCTION

Due to demand for reducing overexploitation of the natural quarries, the use of the by-products from different industries has become an increasing practice in the sustainable construction industry.

Steel slag is generated as a melt at about 1600°C during steelmaking from hot metal in the amount of 15%-20% per equivalent unit of steel. The function of this slag is to refine the steel of sulphur and to absorb the oxides formed as a result of deoxidation during steel production. Steelmaking slags are composed principally of calcium silicates, calcium aluminoferrites, and fused calcium oxides, iron, magnesium, and manganese. Organic, semi-volatile and volatile compounds are not present in the steel slag due to the fact that they are made at high temperatures during production process. Practically all steel slags are air-cooled, but the current technology of slag production cannot always provide its immediate cooling which can influence its quality. As a consequence, it is not always suitable for further usage and that is the reason why quality control of steel slag production must be provided (Cerjan et al 1995, National Slag Association 1982). Long since steel slags had been extensively used as protective armour stones for rivers, sea and coastal erosion schemes and in various land reclamation projects due to their high density,

but a certain amount of produced steel slag is still dumped (National Slag Association 1982).

There are two landfills of air cooled steel slag in the Republic of Croatia—near the towns of Sisak and Split. The slag originating from Sisak steel plant is spread out over the area of around 25 ha in the total amount of around 1.5 million tons. At the moment this type of slag is utilised in road construction (as a stabilisation layer) and in agriculture (fine fractions are used for soil improvement). The slag from Split landfill in total quantity of 30.000 tons has not found its application till now. High price of slag disposal imposes the need for finding new fields of its utilisation. Due to high hardness of steel slag its grinding turned out to be unprofitable to be used as cement addition and that is why the intensive research work if focused on the use of steel slag as an aggregate in concrete. Steel slags have already been researched as an aggregate in concrete and results were affirmative (Dunster 2002, Maslahuddin et al 2003, Zelic 2005, Qasrawi et al 2009, Japan Society of Civil Engineers 2011).

Theure primary aim of this study, as a part of E! 4166 EUREKABUILD FIRECON project was to ascertain the properties of concrete prepared with steel slag aggregate after the exposure to high temperatures. Four concrete mixtures, made from steel slag aggregate from a Croatian steel plant

landfill were studied with respect to different types of cement pastes including portland cement, fly ash as 20% replacement of portland cement and polypropylene fibres in the amount of 1% by volume respectively. The fire performance of the steel slag aggregate concretes obtaining compressive strength, modulus of elasticity, weight loss and ultrasonic pulse velocity, was compared with the fire performance of the crushed dolomite aggregate concrete. In order to clarify the obtained results, the SEM analysis of the studied concretes and the dilatometrical analysis of aggregates used in the studied concrete mixtures were conducted.

2 EXPERIMENTAL STUDY

2.1 Concrete mixture details

The mix proportions of the studied concretes are listed in Table 1. All mixtures were prepared with the same binder content (400 kg/m³) and the same water to binder ratio (w/b = 0.43). The reference mixture (M1) was completely prepared with dolomite aggregate, while the other four mixtures, M2 to M5, were prepared from the slag from Sisak steel plant as a coarse aggregate (4–8 mm and 8–16 mm) and dolomite fines (0–4 mm). Slag fines did not meet the requirements needed for concrete aggregate utilization (Netinger 2010).

Cement matrices for slag based mixtures were made of:

1. portland cement (M2),

- 2. portland cement and polypropylene fibres (M3),
- 3. portland cement and fly ash (M4)
- 4. portland cement, fly ash and polypropylene fibres (M5).

Fly ash was added as 20% replacement for portland cement, while polypropylene fibres were added in the amount of 1% by volume in all relevant mixtures. CEM I 52.5N conforming to EN 197-1 (2000+A1:2004) was used. Polypropylene fibers were of 12 mm cut length, nominal diameter 18 microns, tensile strength 557 MPa and melting point of 160°C. Superplasticizer based on the modified polycarboxylic ether (PCE) polymers was added during concrete production in order to improve the workability of tested mixtures. All mixtures were planned to have the same consistency-S4 class according to the slump test (160-210 mm). Fresh concrete properties shown in Table 1 were obtained according to the relevant European Standards EN 12350-2, -6 and -7 (2009).

Each concrete mixture was cast in steel moulds of dimensions of 100/100/400 mm, demoulded 24 h after the casting, placed in a water tank for further 7 days, and thereafter placed in the chamber (temperature of about $20 \pm 2^{\circ}$ C and humidity level of $95 \pm 5\%$) for up to 28 days. Later on, the specimens were kept until 56th day in the air in the laboratory, in which relative humidity and temperature were about 60% and 20°C respectively.

Constituent/mixtures	M1 (D-pc)	Mixtures with steel slag aggregate			
		M2 (SL-pc)	M3 (SL-pc-ppf)	M4 (SL-pc-fa)	M5 (SL-pc-fa-ppf)
Cement (kg/m ³)	400	400	400	320	320
Fly ash (kg/m ³)	_	_	_	80	80
Water (1/m ³)	172	172	172	172	172
Water/binder	0.43	0.43	0.43	0.43	0.43
Superplasticizer (1/m ³)	3.20	3.20	3.20	3.00	3.00
Dolomite, 0-4 (kg/m ³)	807	864	864	864	864
Dolomite, 4–8 (kg/m ³)	367	_	_	_	_
Dolomite, 8–16 (kg/m ³)	661	_	_	_	_
Slag, 4–8 (kg/m ³)	_	422	422	422	422
Slag, 8–16 (kg/m ³)	_	723	723	723	723
Polypropylene fibres (% by vol)	_	_	1	_	1
	Fresh concrete properties				
Mass per unit volume (kg/m ³)	2530	2643	2637	2666	2563
Air content (%)	0.9	2.0	3.5	2.4	2.8
Slump test (mm)	200	200	190	200	190

Upper case letters denote the type of used coarse aggregate (D-dolomite, S-slag), while lower case letters denote constituents of cement pastes (pc—portland cement, ppf—polypropylene fibres, fa—fly ash).

2.2 *Heating and cooling regime* of concrete specimens

After the period of 56 days concrete specimens, with moisture content ranging between 3-4% were exposed to the high target temperatures of 100° , 200° , 400° , 600° and 800° C in the electric test furnace. The specimens were heated to the target temperature which was kept constant for another 1h. As per RILEM TC 200-HTC recommendations, the heating rate was 1° C/min. After the heating process was completed, the furnace was turned off and the specimens were allowed to cool down naturally to the room temperature inside the furnace in order to prevent thermal shock of concrete material. The temperature histories were monitored by means of K type thermocouple mounted in the centre of the specimen before casting.

2.3 Testing details

Upon cooling of the specimens, their properties were tested and compared to the initial values obtained at room temperature before heating.

The tests carried out on the concrete specimens were as follows:

Unstressed residual compressive strength testing was performed according to the procedure described in EN 12390-3 (2009) on 100-mm concrete cubes sawn from the 100/100/400-mm prisms using Toni Technik compression testing machine with 3000 KN capacity.

Static modulus of elasticity testing was performed according to the procedure described in the Croatian standard HRN U.M1.025 (1987) on 100/100/400 mm prisms. Three loading-unloading cycles between 0.5 MPa and the third of the compressive strength obtained previously were performed using Toni Technik compression testing machine with 3000 KN capacity.

Weight losses were obtained by weighing prism specimens using a precision balance before and after each temperature cycle.

Ultrasonic Pulse Velocity (UPV) is measured on the longitudinal direction of prisms according to the procedure described by EN 12504-4 (2004). The specimens are tested before and after the fire exposure using TICO Proceq ultrasonic pulse velocity testing device with transducers of 54 Hz.

SEM analysis was performed on the specimens obtained from concrete slices sawn in the transverse direction of specimens using a JEOL 5600 scanning electron microscope in LV mode and with backscattered electrons.

Dilatometrical analyses of the aggregates used in the studied mixtures were performed on a Netzch 402 device at the heating rate of 10°C/min and up to the temperature of 1000°C.

3 RESULTS AND DISCUSSION

3.1 Residual compressive strength

Figure 1 presents the variation of the residual relative compressive strength (in percent of the initial value) versus the temperature.

All steel slag based mixtures (M3, M4 and M5), apart from mixture M2, have slightly higher relative compressive strength at the temperature of 100°C compared to the mixture with dolomite. At temperatures above 100°C, compressive strength for all mixtures decreases with temperature increase. Up to 600°C, relative residual compressive strength of dolomite based mixture is lower than that of the other slag based mixtures. On the contrary, in the temperature range from 600°C to 800°C dolomite based mixture has higher relative residual strength with the difference of about 15% at the temperature of 800°C.

It is worth pointing out that there is a sharp drop of around 35% for all steel slag based mixtures in that temperature range. Also, the mixtures containing steel slag have very similar values of the residual compressive strength (66–68% at 600°C, to about 28%–30% at 800°C, respectively), no matter what type of binder was used for the mixture. It is obvious that type of the used aggregate has predominant influence on the residual compressive strength of slag based mixtures in the temperature range of 600°–800°C.

3.2 Residual modulus of elasticity

Figure 2 presents the variation of the modulus of elasticity (in percent of the initial value) versus the temperature. Reduction of modulus of elasticity of concrete exposed to high temperatures is caused by breakage of bonds in the microstructure of cement matrix (Bazant & Kaplan 1996).



Figure 1. Relative compressive strengths of the studied types of concrete vs. temperature.



Figure 2. Relative modulus of elasticity of the studied types of concrete vs. temperature.

The evolutions of modulus of elasticity with temperature are comparable within the slag and dolomite based concretes up to 600°C. As per compressive strength, at the temperature of 800°C residual modulus of elasticity of dolomite based mixture is higher (19%) than in the case of all slag based mixtures (0–8.5%). Slag based mixture with portland cement (M2) did not show any residual modulus of elasticity after cooling from 800°to room temperature.

3.3 Weight loss after thermal treatment

The relative weight (in percent of the initial value) of the studied concretes as a dehydration measure and/or some chemical and physical decomposition of concrete with temperature increase is given in Figure 3.

It was observed that up to 600°C, the evolution of the relative weight versus the temperature is very similar for all studied concrete mixture. All concrete mixtures (including reference mixture) experienced almost the same weight loss up to 100°C.

In the temperature range from 200°C to 600°C, slag based mixtures lost slightly more weight than the reference mixture, while in the temperature range from 600°C to 800°C, the reference concrete experienced a greater reduction in weight than slag based mixtures.

At 800°C, there remained 78% of the initial weight of dolomite based mixture (M1) and 84–86% of slag based mixtures respectively. Such a difference in weight loss could be attributed to the type of aggregate.

3.4 Change in UPV after thermal treatment

The ratio of the residual pulse velocity of all concretes after different peak temperatures compared to the pulse velocity at the room temperature (in percent) is presented in Figure 4. The transmission



Figure 3. Relative weight of the studied concrete specimens vs. temperature.



Figure 4. Relative pulse velocity of the studied concretes vs. temperature.

of pulse waves through the concrete mass is influenced by the occurrence of microcracking in the concrete material. Thus, the decrease in pulse velocity with temperature increase could be used as a measure of the microcracking progress in concrete.

It is visible in Figure 4 that in the whole temperature range (20°C–800°C) slag based mixtures (M2-M5) experienced greater reduction in pulse velocity than dolomite based mixture (M1). This is especially pronounced in the temperature range between 600°C and 800°C, which is consisted with the results for compressive strength and modulus of elasticity.

At 800°C reference mixture retained around 55% of initial pulse velocity compared to slag based mixture, in which negligible residual pulse velocity was measured (3-5%). This leads to the conclusion that there was a higher degree of microcracks within slag mixtures compared to dolomitemade mixture, especially in the temperature range between 600°C and 800°C.

3.5 Microstructural investigation

Figure 5 a,b show SEM images of concrete mixtures after the exposure to the temperature of 800°C. The microstructural analysis of the studied concretes is given in detail by (Netinger et al 2010).

According to the results of microstructural analysis, a grain of dolomite suffered more microcracks (Figure 5a) than a grain of slag—the grain of slag remained intact (Figure 5b). Dolomite grain suffers decarbonation at the temperatures higher than 700°C which led to microcracking throughout dolomite grain (Bazant & Kaplan 1996), which spread further through the cement matrix.

Wider microcracks occurred at the interface of the slag grain and cement matrix which also spread out through cement matrix in slag based mixture (Figure 5b). Therefore no reason for lower performance of the concrete containing slag compared to the concrete containing dolomite can be found in the lower performance of slag aggregates.

3.6 Dilatrometrical investigation of aggregate

Most commonly used aggregates experience expansion during heating, while cement paste experiences shrinkage at the same time (Khoury et al 2007), which can reflect negatively on the mechanical properties.

The linear thermal expansion of dolomite and slag grains during the exposure to the temperature of 1000°C is presented in Figure 6 (full lines).



Figure 5. SEM analysis of a) dolomite based concrete and b) slag based concrete after heating to 800°C.



Figure 6. Results of the dilatometrical analysis.

In Figure 6 it can be observed that thermal expansion of dolomite grain is more pronounced compared to steel slag grain, especially to decarbonation in the temperature range above 600°C. Considering the fact that due to decarbonation aggregates expand, crack and spall it is to be expected that more pronounced expansion of dolomite grain should affect the residual mechanical properties of dolomite based mixture more negatively, which is not the case in our study.

Unstable expansion of steel slag grain can be clearly observed in the temperature range between 550° and 800°C in Figure 6. The research presented at Pattek-Janczyk et al. 1999 and Ducman et al. 2011 reported the phase transition of steel slag aggregate from wüstite into magnetite followed by an unstable expansion, which can be responsible for low mechanical properties of slag based mixtures in that temperature range. Also at Ducman et al. 2011 was shown that this expansion is negligible if the slag was previously heated up to 1000°C before utilization in concrete. This observation is shown in Figure 6 where the dotted curve presents the stable expansion of the previously heated slag to the temperature of 1000°C at heating rate of 5°C/min.

It seems that the observed phase transformation of the steel slag aggregate could be connected to pronounced microcracks in the interface aggregatecement matrix which reflect more negatively on the mechanical properties of slag based mixtures compared to the transformations of dolomite based mixture caused by high temperature.

Further research will be based on the utilization of the previously heated slag in concrete mixtures, in which better performance should be expected in the temperature range between 600° and 800°C.

4 CONCLUSIONS

The experimental results and observations in this study can be summarized as follows:

Residual mechanical properties (namely compressive strength and modulus of elasticity) of steel based mixtures are comparable with dolomite based mixture up to the temperature of almost 600°C.

A sharp drop in residual mechanical properties in slag based mixtures is observed in the temperature range between 600°C and 800°C. Due to the fact that similar residual properties were obtained within all slag based mixtures (with different types of cement pastes) it is obvious that after cooling from 600°C and 800°C, the type of aggregate used has a predominant influence on the residual mechanical properties of slag based mixtures.

Mineralogical and dilatometrical analysis show that a steel slag aggregate at the temperature higher than 550°C experience unstable expansive mineralogical transformation found in the available literature as transformation from wüstite into magnetite which results in the pronounced microcracking at the interface aggregate—cement matrix. These microcracks, in turn, affect negatively the mechanical performance of slag based concretes. The literature review shows that this transformation is irreversible and if heated prior to use in concrete up to the temperature of 1000°C the slag become stable which is proved in this study. Further research will be based on testing of the concrete made from the previously heated slag.

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