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EAF Slag Treatment for Inert Materials' Production

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Abstract Danieli & C. in collaboration with Politecnico di Milano, designed an innovative, patent pending, treatment plant to solve some issues associated to metals leaching (i.e., Ba, V, and Cr), and high free lime concentration for carbon steel EAF slag, for the production of artificial aggregates featured by mechanical properties suitable for the building purposes. The plant is featured by an innovative pneumatic transport system to convey the stabilizing agent to intercept the molten slag flux during the deslagging operation. The new system promotes stable microstructural phases' formation, able to retain metallic ions leaching, maintaining the same mechanical properties of the as-cast slag. The main advantages of the new implemented stabilization plant are a simple installation and an easy process operation.

Keywords Carbon steel EAF slag · Stabilization treatment · Pneumatic injection system · Leaching behavior · Pilot-plant

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Introduction

Recently, the countries which are more concerned with environmental protection (European Union countries, USA, Japan, etc.), are following two main routes for handling steel mill by-products: the development and application of innovative and more efficient technologies to decrease liquid, solid, and gaseous emissions; and the valorization of the *waste materials* (in accordance with the Zero Waste Philosophy) both for the recovery of the valuable fractions (e.g., ZnO from the EAF dust) [1] and to enhance, after treatments, the material properties for their reuse as building materials [2].

These virtuous routes lead to the increase of knowledge at the microstructural level of the materials and to the development of more efficient plants to turn waste materials into inert ones, being equivalent for their chemical–physical properties to natural substances used in constructions. The exploitation of the recycled by-products and their use imply several benefits, especially from an environmental point of view: reduction of waste fraction sent to landfill, reduction of the exploitation of natural resources, reduction of activities impacting on the territory (i.e., extraction and drilling), energy saving, and the related decrease of CO_2 emissions.

The recycling of the steel slag as an alternative to the common stone materials is becoming a usual practice all around the world. Since the steel slag produced by the electric arc furnace (EAF) can be considered as natural hard rocks, this by-product could be successfully used to replace inert material in several areas (road construction, concrete production, etc.), and this allows decreasing their disposal costs by giving them a new value [3–6]. Although the real employing of steel slag should depend essentially on its compliance with the technical regulations, some

restrictions introduced by laws for the protection of human health and environment cannot be neglected. In particular, since the steel slag can be assimilated to natural igneous rocks, they could cause perishability problems when coming into contact with water, namely chemical weathering, that might induce heavy metals leaching [7]. Thus, the environmental compatibility of a slag must be assessed according to even more strict limits on pollutants, headed by national regulations for the recycling of the materials considered non-hazardous [8, 9].

Danieli & C., in collaboration with Politecnico di Milano, designed an innovative treatment plant that aims at making the slag produced by the EAF inert, in order to solve some issues associated to metals leaching (i.e., Ba, V, and Cr), free lime concentration decrease, [10, 11] and production of artificial aggregate characterized by mechanical properties suitable for building purposes [12–14]. Such a project is aimed at transforming EAF slag into a safe and inert by-product, and it has been divided in the following steps:

- Steel-plant slag microstructural analysis and leaching behavior characterization;
- Laboratory test: investigations on inertizing additives and on their effects on phase transformation;
- Definition of optimal process conditions for the industrial scaling up;
- Pilot-plant design and construction, to test the inertization technologies into the steel shop;
- Tests in steelmaking plant and industrial technology validation.

The need to develop a new approach to treat carbon steel EAF slag derives from the lack of information about this type of slag, because until now a great effort has been applied mainly to solve environmental problems concerning the Cr^{6+} release. Hexavalent chromium form is the most reactive and dangerous one (it is classified as carcinogenic by the World Health Organization). Several studies aimed at finding a way to reduce or inhibit the leaching of Cr oxyanion. One of the most important studies stated the correlation between chemical composition and chromium leaching, formulating a simply parameter (sp-factor (1)) to forecast the Cr leaching [15].

$$sp\text{-factor} = 0.2 \cdot MgO + 1.0 \cdot Al_2O_3 + n \cdot FeO_n - 0.5 \cdot Cr_2O_3 (wt\%), \quad (1)$$

where n depends on the oxidation state of the slag.

The sp-factor weighs the spinel forming oxides, and the Cr leaching behavior is described with consideration of its weight of influence. Three domains have been distinguished: values of sp-factor <5 wt% imply no or only few Cr-containing spinels; values of sp-factor from 5 to 25 wt%

allow the formation of Cr-spinels; values of sp-factor >25 wt% imply nearly no Cr leaching.

However, the sp-factor, widely used to guarantee the Cr stability in the stainless steel slag, is not effective for low Cr-content slag. As a consequence, the development of a deep empirically studied inertization is mandatory, most of all for the slag originated by carbon and low-alloyed steels.

Different technologies for EAF slag treatment based on the mixing of the liquid slag with stabilizing oxides already exist. The most applied treatments tried to solve the dusting problem due to larnite disintegration and to reduce the total amount of free lime and periclase that cause volume instability due to hydration phenomena [10, 16–19]. The other ones focused on the possibility to increase the hydraulic properties of the slag in order to improve their application in cement production. Only a few examples focused on chromium leaching reduction have reached till now also the goal of conditioning the properties of the slag [20–22]. Regardless of the treatment type and its intended goals, the only opportunity to intervene on the slag quality is to operate while it is still liquid. Then, the treatments can be carried out during or immediately after the spill skimming in the slag pot (or in the slag pit), but certainly never after the slag is already solidified.

The most important improvements described in this paper can be summarized through the following points: the use of the inertizing agent through the pneumatic transport (instead of the discharging by gravity applied till now [11, 23]), to realize an optimum mixture between the liquid slag and the solid oxides thanks to the high kinetic energy of the oxide flow; the decrease of the powdered material losses, caused by the convective air flow and EAF fume plant suction, thanks to the high coherence of the material flow and the right grain size of the powder; and the solution of the issues associated to free lime content, pulverization, and heavy metal leaching.

The designed treatment plant transforms the slag microstructure promoting the formation of low-hydraulic properties phases (i.e., gehlenite) [23–25] and forming complexes that avoid the free lime exceeding fraction.

Experimental Procedure

Experimental tests have been split into two steps: laboratory and pilot-plant testing in the steelmaking plant.

Laboratory Testing

Different slag samples have been selected from various steel plants, and they are featured by Cr, Ba, and V

Table 1 Chemical composition by ED-XRF (**wt%**) of the as-cast slag stabilized in laboratory scale trials

Sample	MgO	Al_2O_3	SiO_2	CaO	FeOx	Cr_2O_3	BaO	V_2O_5
Slag 1	2.7–3.2	3.5–6.7	10.4–17.5	41.1–54.3	9.0–19.6	0.4–1.4	0.30–0.50	0.20–0.25
Slag 2	2.6–3.5	4.7–6.2	14–16.7	42.8–52.6	14.2–19.4	0.8–2.2	0.25–0.35	0.20–0.25

concentrations that can be associated to a potential elution risk (Table 1).

After the chemical and physical characterization and the measurement of the metallic ion concentrations during the leaching test, the slag samples have been subjected to stabilization trials by melting them into a resistive furnace ($T_{max} = 1400$ °C), using a ceramic crucible and adding oxides mixtures such as SiO_2, FeO, and MgO. In Table 2, the conditions experimentally investigated are reported. MgO and iron oxide were principally added to improve the Cr immobilization, whereas SiO_2 was chosen so that the EAF slag chemical composition approaches that of crystalline blast furnace slag, in order to promote the formation of non-hydraulic phases (melilite and merwinite) [26–28]. Additives' efficiency was evaluated as a function of their effect on the slag melting temperature. The melting temperature of slag-additive mix was determined by means of hot stage microscope (HSM). The solid slag was pulverized by a ring mill and mixed with different amounts of the additive powders. For each condition, 50 mg of mixture was analyzed with a heating rate of 10 °C/min, using a HSM MISURA equipped with MISURA 3.32HSM software.

Pilot-Plant Testing

The pilot plant consists of a silo stocking the oxide mix and of a metering valve supplying and conveying it through the pneumatic transport to a nozzle that lies close to the EAF deslagging door. In Fig. 1, the picture of the silo used for the pilot-plant tests is reported. In Fig. 2, the zone of the EAF involved in the injection of the powdered oxide mixture through a dedicated lance is described.

Great efforts have been devoted in studying and developing the injection plant: process parameters, such as transport air pressures, rates, injection nozzle position, and

Fig. 1 Picture of the industrial dispenser
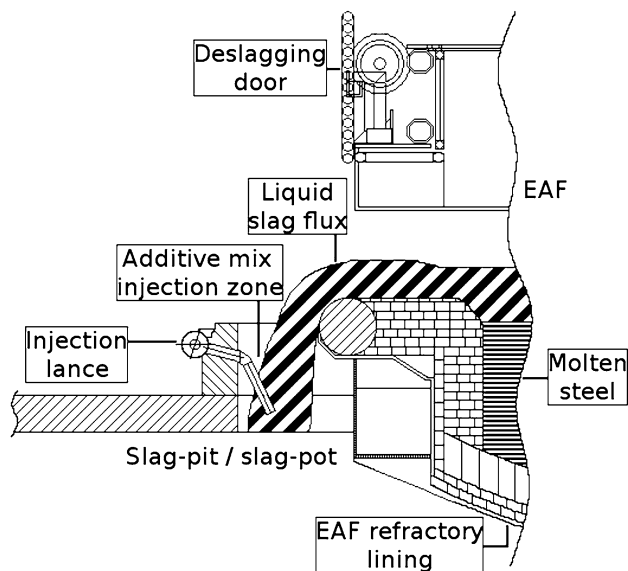
Fig. 2 Sketch of the injection zone of the EAF, reporting a simplified lance

Table 2 Composition of the additives for the liquid slag inertization in the laboratory scale

Oxides mixture	Cooling medium	Results
Mainly SiO_2	Natural convection	Optimum
Mainly MgO		Partial
Mainly FeO		Partial

shape, have been designed in order to improve the mixing of the slag with the inertizing flux. Such efforts aim at enhancing the reaction kinetics of transformation of water-soluble phases (such as larnite, brownmillerite etc.) into stable ones, such as gehlenite, wüstite, and stoichiometric Cr–Mg spinel. A suitable control system guarantees the accurate injection timing, and it optimizes the additive mixture consumption.

A summary of the applied conditions is reported in Table 3.

Chemical and Physical Characterization

Both the sample groups, the one prepared in laboratory and the other coming from the steel plant, have been characterized by these techniques:

- ED-XRF, to evaluate the average chemical composition;
- SEM and XRD, to analyze the nature, the morphology and the composition of the slag phases;
- Leaching test according to UNI EN 12457-2/04;
- Free CaO concentration analysis according to UNI EN 1744-1:2013;
- Los Angeles coefficient determination according to UNI EN 1097-2.

Results and Discussion

Laboratory Testing Results

Laboratory tests allowed studying different stabilizing additives formulations. Additives efficiency was evaluated as a function of their effect on the slag melting temperature. Specifically, substances able to decrease the slag melting temperature should allow to maintain the slag liquid for more time, enhancing diffusion reactions between the different mineralogical phases (formed inside the furnace) also outside the EAF and after the deslagging operation. Moreover, by this way, the promotion in the formation of the eutectic phases is made possible, because

such slag seems able to retain the polluting substances better, avoiding their leaching.

Different oxides were considered during the laboratory analysis, focusing on the same species adopted and described in the literature [10, 16–22]. The main goal of the laboratory campaign is to identify the best material able to solve simultaneously the different issues affecting the slag: volume instability, dusting effect, and heavy metal release. Thus, magnesia (MgO), mill scale (FeO), and pure quartz (SiO$_2$) were tested. These three compounds are easily available in a steel mill, and they have low implementation costs. Actually, SiO$_2$ is the cheaper glass-promoter compound, and it does not imply any particular management problem (supply, storage, and handling). Mill scale could be easily supplied by rolling plants, whereas MgO is chosen because, in some researches, [15, 20] its benefit effect on chromium immobilization has been stated. The melting temperatures of the different slag-additive mixes, determined through the heating microscope, are reported in Fig. 3.

Quartz and quartz-mill scale mix have the same effects on slag melting temperature for all the investigated samples. In particular, an addition between 5 and 15 % wt% of SiO$_2$ is enough to decrease the slag melting temperature by approximately 50 °C. Similar effect, even if less significant, is induced by FeO. On the contrary, MgO addition, as expected, has a very limited consequence because it is an alkaline chemical species and so its excess sorts the opposite effect associated to a temperature increase.

On the basis of the obtained results, stabilizing experiments through slag re-melting have been performed. The as-cast and treated slags have been compared each other, taking into account existing phases and metallic ions leaching. Two different slags featured by high fraction of CaO, low concentration of FeO and MgO, and different Cr$_2$O$_3$ concentrations have been re-melted after different inertizing oxide additions (Table 1). In Fig. 4, a comparison between low Cr$_2$O$_3$ as-cast slag (Fig. 4a) and a laboratory re-melted slag interested by pure quartz addition (Fig. 4b) is reported.

The as-cast slag mainly consists in four crystalline phases: magnesium-wüstite (W), brownmillerite (B), hatrurite—3CaO·SiO$_2$ (H) and larnite—2CaO·SiO$_2$ (L). In high Cr-content slag samples (namely slag 2) calcium chromite was also detected.

Calcium silicates (3CaO·SiO$_2$, 2CaO·SiO$_2$) own the well-known hydraulic properties and the high concentration of heavy metals dissolved in such phases make them potential responsible for Ba and V leaching (Table 4) [24]. Brownmillerite also belongs to the main constituents of the cement and possesses modest hydraulic properties that could heighten the leaching of Ba and Cr dissolved in this

Table 3 Experimental parameters for the pilot-plant testing

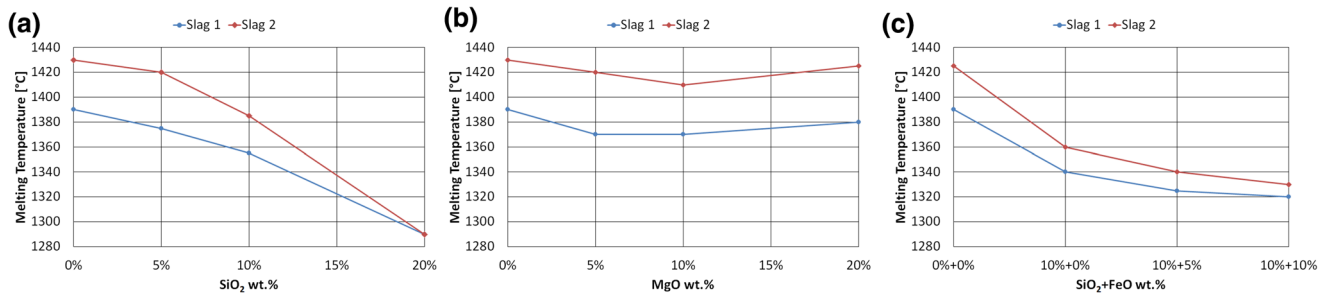| Parameters	Value
Air flow rate (Nm^3/h)	150/300
Dispenser internal pressure (bar)	2/4
Air pressure (bar)	0/3.5
Additive flow (kg/min)	0/140
Optimum grain size range of the oxide mixture (mm)	1/2

Fig. 3 Results of wettability furnaces tests: **a** effect of pure quartz; **b** effect of magnesia; **c** effect of SiO_2-mill scale mix
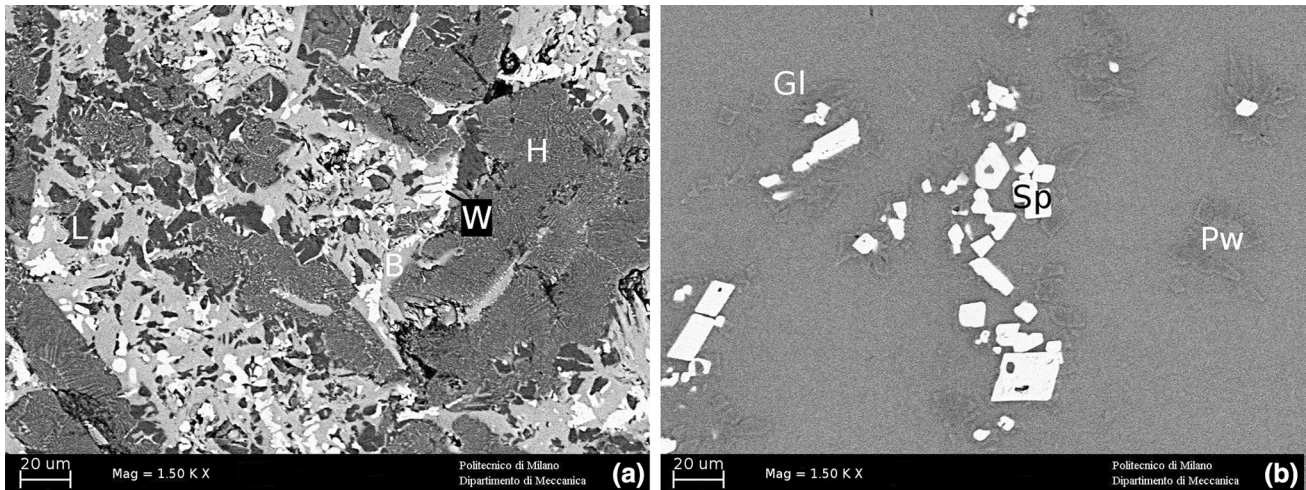

Fig. 4 SEM images of the as-cast slag 1 (**a**) and SiO_2-inertized slag in laboratory scale (**b**). *B* brownmillerite, *L* larnite, *H* Hartrurite, *Gl* glass, *Pw* pseudowollastonite, *Sp* Cr–Mg spinel, *W* Mg-wüstite

Table 4 Oxides partitions in the different phases of the as-cast slag

Phase	Symbol	Cr_2O_3 (%)	$Cr_2O_3^a$ (%)	BaO (%)	V_2O_5 (%)	TiO_2 (%)
Mg-Wüstite	W	0.5	0.5	4.0	6.5	0.0
Brownmillerite	B	99.0	15.0	48.0	32.0	100.0
Hatrurite	H	0.5	0.5	20.0	16.5	0.0
Larnite	L	0.0	0.0	28.0	45.0	0.0
Ca-chromite	Ch	–	84.0	–	–	–

[a] For slag 2 samples characterized by Ca-chromite

phase (Table 4) [29, 30]. The role of Ca-chromite in Cr leaching is well known, too [31, 32].

Through the addition of inertizing oxides, phase transformations occur, and the slag obtains a structure similar to the one in Fig. 4b. Slag microstructure is formed by a Si-rich glassy matrix (Gl), where silicate crystals characterized by high concentration of MgO, Al_2O_3, and FeO (Pw) and Cr-rich spinels (Sp) nucleate. The addition, preferably of quartzite, seems to promote the demixing of chromium that tends to bind with Mg-wüstite forming stable Cr–Mg-spinels that also act as the nuclei where the silicate crystals

start to nucleate and to grow. These structures are stable [25], and, as a consequence, Cr can be considered irreversibly captured. Similar microstructures have been obtained by adding SiO_2–MgO or SiO_2–FeO mix. Thus, MgO and FeO addition do not significantly affect the formed microstructure, while the most significant modifications are induced by SiO_2. Also in those cases, spinels are the only phases rich in Cr, and they seem the nuclei where the silicate crystals start to nucleate and grow. The addition of such stabilizers, mainly pure quartz, also reduces the amount of free lime, from the 2 wt% in the as-

cast slag up to 0.1 wt% in laboratory-treated samples. Macroscopically, the slag featured by MgO addition appears less compact and more porous than the others.

Even if the addition takes place at solid state, the additives' effect on the slag melting temperature is enough to promote the liquefaction of the solid mass. Nevertheless, the added inertizing mix completely dissolves in the slag, and SEM analysis does not point out undissolved SiO_2 or other additive particles. SiO_2 enrichment converts $3CaO \cdot SiO_2$ and $2CaO \cdot SiO_2$ into pseudowollastonite ($CaO \cdot SiO_2$), and the formation of liquid eutectic compound contributes to envelop the new crystalline phases into a glassy matrix, unreactive with water, and Ba, V, and Cr leaching has been effectively avoided (Tables 5 and 6). The higher Cr leaching shown by slag 2 seems correlated to the presence of Ca-chromite. This structural constituent in alkaline circumstances can easily be oxidized to $CaCrO_4$, a compound that easily leaches chromium and builds hexavalent chromium from divalent or trivalent chromium [31, 33, 34]. The high value of $CaCrO_4$ solubility constant indicates the high solubility of this compound [32].

These experimental trials give a clear indication about the feasibility of slag treatment performed through the external addition. In order to discern which additive is better than another, elution tests are an important tool to assess the benefits produced by the different added oxides. Since the as-received slags have environmental problems, mainly related to Ba and Cr releases, all the treated slags do not point out any Ba release higher than the law constraint (Table 6). More criticalities are instead related to chromium and vanadium immobilization. Although for each experimental condition, the elements' concentration in leachate solution always complied with the regulation limits, the samples modified by MgO and FeO additions point out a higher vanadium concentration in leachate than the SiO_2-modified slag. In addition, the stabilization through $MgO–SiO_2$ mix seems less effective on chromium immobilization than the $FeO–SiO_2$ mix, mainly due to the less weight of MgO to contribute in spinel formation, as indicated by sp-factor equation. This observation gives a clear indication about the procedure to follow and to extend the treatment from laboratory scale to the steelwork applications.

Pilot-Plant Results

Pilot scale validation of laboratory investigations has been realized through pneumatic transport of the oxides mix from a storage silo to a nozzle. The nozzle is placed in a position that allows an optimal mixing of liquid slag with the additives to maximize the effects of stabilization. The pilot plant is installed in a steel mill that operates with slag-pit deslagging, so the most important parameter to be controlled is the temperature in the mixing point between the liquid slag and the injected powder. Actually, the best dissolution of the stabilizing agents within the molten slag can be achieved if the conveying nozzle is positioned to intercept the slag flux at its maximum temperature. In this way, enough time is assured for additive-slag reaction before the fast cooling and the solidification induced by the floor contact begins. For these reasons, a dedicated experimental campaign of temperature measurement by thermal camera has been performed. In Fig. 5, the trend of the liquid slag temperature is reported. A temperature in the mixing point, close to 1600 °C has been measured: this is a very good condition to improve the diffusion and the reactivity of the involved phases.

Thanks to the properly designed additive receipt, mainly constituted by pure quartz, the slag melting temperature after stabilizer injection decreased according to the results obtained during the laboratory tests. The average chemical composition has been modified, too. As indicated in the ternary diagrams reported in Fig. 6, the slag composition moved from high melting point area, associated with unstable and leachable microstructure, to low melting temperature area, leading to the formation of more stable microstructure [23–25].

Results obtained in pilot-plant scale have been very positive, as stabilized slag SEM image and XRD pattern were demonstrated (Fig. 7). The experimental evidence demonstrates that silica is able to react with slag. The decrease of $3CaO \cdot SiO_2$ and free CaO fraction represents a proof that silica dissolves and reacts with the molten slag, even if larnite (phase C in Fig. 7b) residual is still visible. The increase in wüstite (W in Fig. 7b) fraction and the formation of Cr–Mg spinels (Sp in Fig. 7b) and gehlenite (G in Fig. 7b) are also unequivocal indexes that quartzite positively modify the slag microstructure. As reported in Table 7, oxides subjected to elution have been incorporated in stable structures (Cr–Mg spinels and gehlenite [9]) decreasing also those dissolved in larnite. Leaching tests' results (Table 8) confirm the occurrence of stabilization. Moreover, the quartz correction contributes to hinder the formation of $CaO \cdot Cr_2O_3$ that has been detected for some

Table 5 Oxides partitions in the different phases of SiO_2-stabilized slag in laboratory test

Phase	Symbol	Cr_2O_3 (%)	BaO (%)	V_2O_5 (%)	TiO_2 (%)
Cr–Mg spinels	Sp	99.0	50.0	35.0	0.0
Glass	Gl	0.4	32.0	50.0	36.0
Pseudowollastonite	Pw	0.6	18.0	15.0	64.0

Table 6 Leaching tests: limit concentrations [8, 9] and ICP-OES results of the as-cast and treated slag (mg/l)

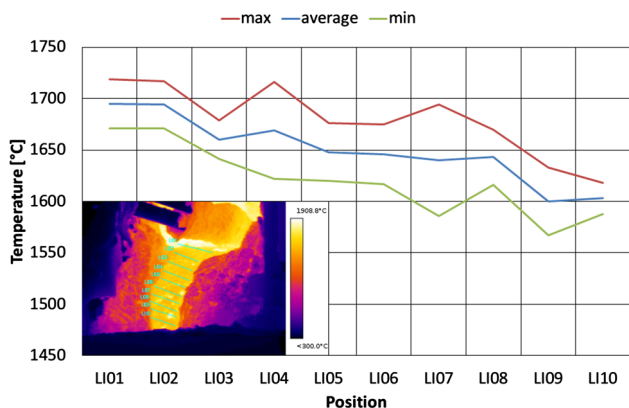| Species	Concentration limit	As-cast 1	Treated 1 SiO$_2$	Treated 1 SiO$_2$ + FeO	As-cast 2	Treated 2 SiO$_2$	Treated 2 SiO$_2$ + MgO
Ba	1.00	**2.31**	0.01	0.01	**1.41**	<0.01	0.01
Cr	0.05	**0.07**	0.04	0.02	**0.17**	0.02	0.03
V	0.25	<0.001	<0.001	0.14	<0.001	<0.001	0.05
pH	>5.5 to <12	**12.20**	10.00	10.20	**12.20**	9.17	10.81

Bold values over the limits

Fig. 5 Thermal camera image of the liquid slag, close to the injection point of the stabilizing additives

as-cast samples (Fig. 7a). Ca-chromite is unstable, and its role in Cr leaching was previously discussed in paragraph 3.1.

The application of stabilization treatment through the pilot-plant leads to the 75 wt% of reduction of final free lime content (Table 9). It is worthy to mention that the limit on admissible free lime content generally adopted is the 1 wt% [35–37]. Generally, the residual content of free lime is reduced through an aging period (3–6 months to atmospheric exposure) by the natural hydration and carbonation processes interesting the CaO. Thanks to the efficacy of the implemented stabilization process, mainly due to the optimized injection device, the aging step can be avoided, having available a stable product both from the chemical and volumetric point of view. This aspect
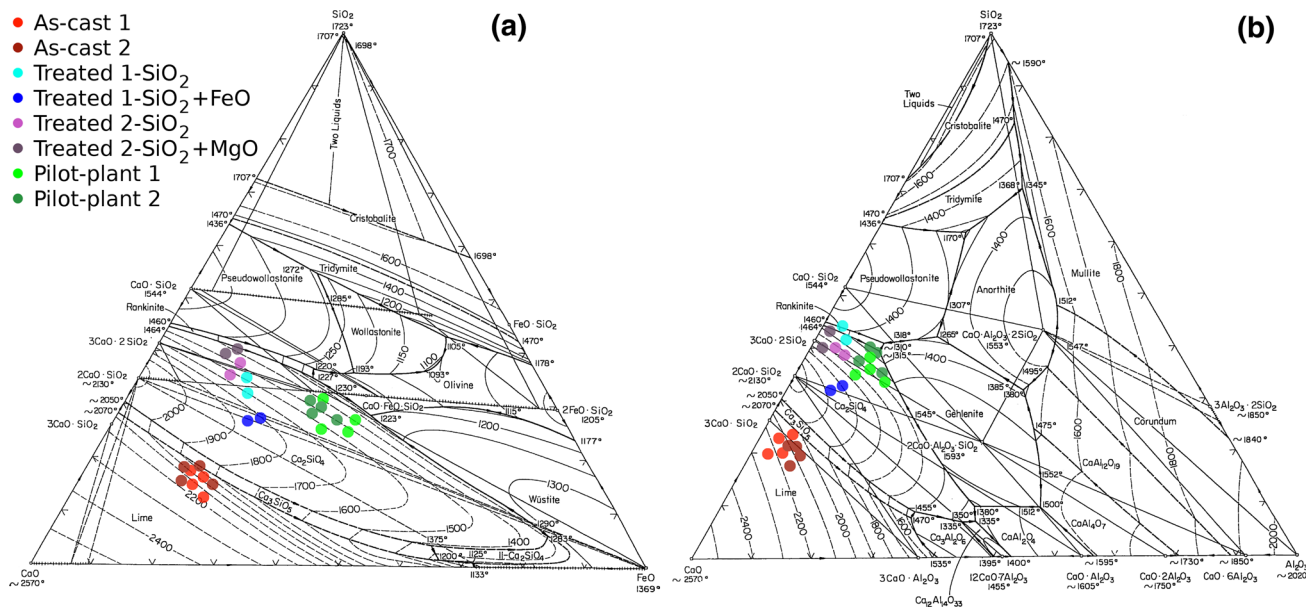

Fig. 6 Ternary diagram CaO-SiO$_2$-FeO (**a**) and CaO-SiO$_2$-Al$_2$O$_3$ (**b**). Four quarters for each slag (two for laboratory-treated slag), sampled according to UNI 10802 standard, were measured by ED-XRF
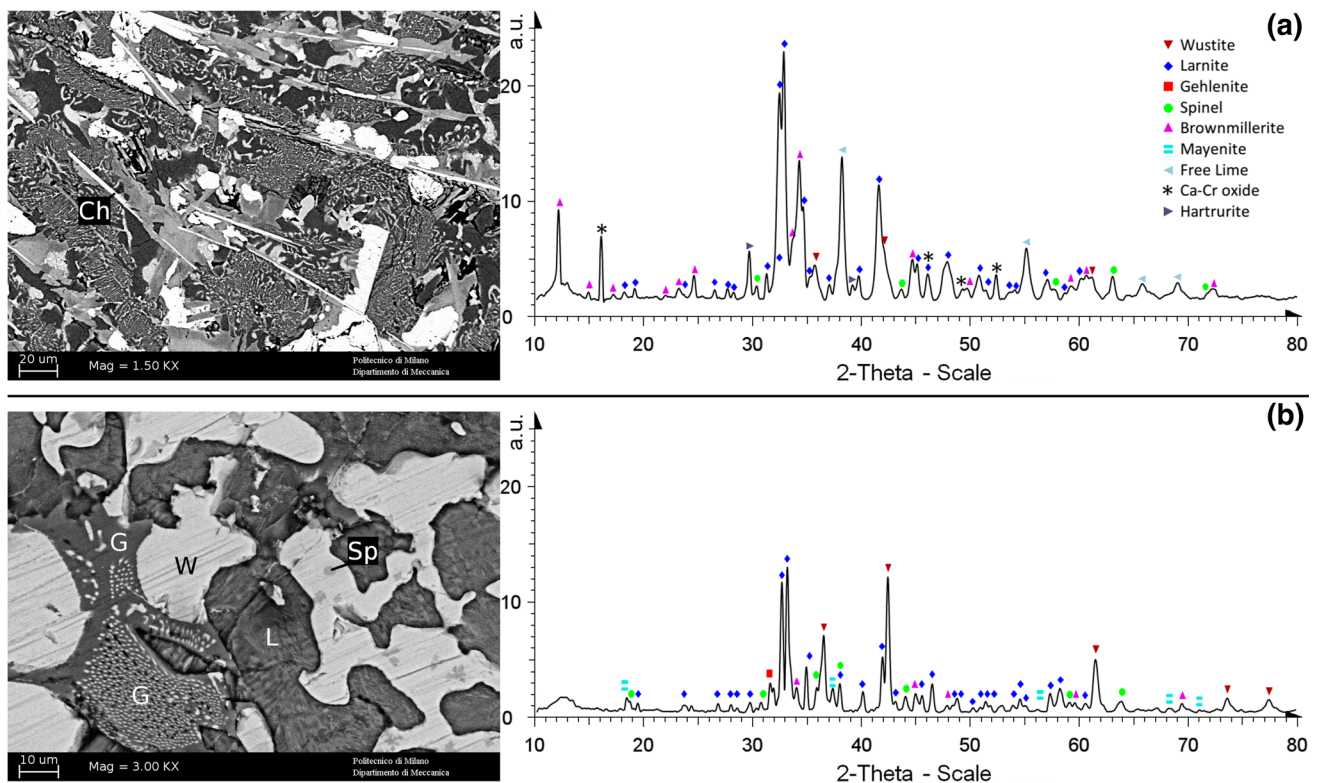
Fig. 7 SEM image and XRD pattern of the as-cast slag 2 (**a**) and the corresponding treated slag produced during pilot-plant trials (**b**). *Ch* Ca-chromite, *L* larnite, *G* gehlenite, *Sp* Cr–Mg-spinel, *W* Mg-wüstite. *Y axis* is in arbitrary unit (a.u.)

Table 7 Oxides partitions in the different phases of pilot-plant stabilized slag

Phase	Symbol	Cr_2O_3 (%)	BaO (%)	V_2O_5 (%)	TiO_2 (%)
Mg-Wüstite	W	8.0	8.0	3.0	0.0
Cr–Mg spinel	Sp	89.0	15.0	92.0	100.0
Larnite	L	0.0	26.0	0.0	0.0
Gehlenite	G	3.0	51.0	5.0	0.0

Table 8 Leaching tests: limit concentrations [8, 9] and ICP-OES results of the as-cast and pilot-plant treated slag (mg/l)

Species	Concentration limit	As-cast 1	Treated 1	As-cast 2	Treated 2
Ba	1.0	**2.31**	0.20	**1.41**	0.40
Cr	0.05	**0.07**	0.03	**0.17**	0.03
V	0.25	<0.001	0.07	<0.001	0.03
pH	>5.5 to <12	**12.20**	11.70	**12.20**	11.90

Bold values over the limits

introduces new benefits for the steel shop, i.e., a size reduction of the slag deposit and a ready availability of a product with specific physical properties.

Finally, mechanical tests performed on both the as-cast and pilot-plant treated samples demonstrated that the inertization process preserves slag properties: treated slag Los Angeles coefficient (LA) is similar to the one of the as-cast slags (LA < 30) (Table 9). Slag with this characteristic could be exploited for concrete pavers or bituminous binder layers, in addition to all the base and foundation layers applied in the road constructions. Although the significant additive amount added during the pilot-plant tests, LA coefficient seems not to be affected by the chemical composition correction. The values reached by the modified slag are in the same range of those associated to the aged slag.

Table 9 Free CaO content and Los Angeles values comparison between the as-cast and stabilized slag

Sample	Free CaO (%)	L.A.
As-cast fresh	1.5–2	28
As-cast aged	<1	25
Treated 1	0.4	25
Treated 2	0.3	27

Conclusions

EAF slag stabilization process and a plant equipped by a pneumatically conveying system that aim the injection of stabilizer oxide mix into the molten slag have been developed by Danieli & C. in collaboration with Politecnico di Milano. Such process and the tested plant promote the formation of stable phases, able to retain metallic ions, so that the leaching is maintained under the prescribed limits, and the slags maintain the same mechanical properties of the as-cast slag. The satisfactory results obtained during the testing of the pilot-plant led to filing an application for a patent, which is currently pending (Patent n° WO2014041418).

This technology expands EAF slag uses as an aggregate in civil engineering, which use has been forbidden so far because the slags featured leaching of heavy metals above the limits.

Simple plant implementation and easy process operation are two key points of this technology.

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