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SIDERURGICAL MORTARS IN SPAIN: REHABILITATION OPPORTUNITIES AND AN OVERVIEW OF PROGRESS

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ABSTRACT

In the present paper, waste products from the steel-making industry are reused as fine raw material in rehabilitation mortars for construction works. Two cement mixes are evaluated: masonry mortars and structural mortars. The study is focused on two steel-making by-products: electric arc furnace slag and ladle furnace slag. The mortar design proposed here incorporates the former in partial substitution of fine aggregates (sand) and the ladle furnace slag in partial substitution of Portland cement and, in some cases, as aggregate (filler). Several partial substitutions of mortar binders/aggregates were prepared which yielded different mixes: 8 masonry mortars whose compressive strengths were below 20 MPa and 12 structural mortars whose compressive strengths were over 50 MPa. At the lab scale, various physical and chemical tests were performed on batches in both the fresh and the hardened state (densities, spreading, mechanical strength, porosity and weathering studies). Our results fully support the use of these siderurgical mortars for architectural (non-structural) rehabilitation purposes and in structural refurbishments for strengthening reinforced concrete elements.

KEYWORDS: Electric Arc Furnace Slag (EAFS); Ladle Furnace Slag (LFS); Cement and aggregate substitutions; Mortar design; Durability.

1. INTRODUCTION

Opportunities for reusing the high waste volumes from the steel-making industry (one of the heaviest in the EU27) in rehabilitation mortars for construction works are studied in this paper. Several mix designs are presented for: Masonry Mortar (MM) and Structural Mortar (SM), the latter, for instance, as a strengthening solution in ancient concrete structures.

As its overall objective, the present research is focused on incorporating wastes from the steel industry (a highly developed sector in Spain) into the production cycle, in the form of noble raw materials (high added-value) and, to do so, in a traditional sector (construction), also a highly intense consumer of both natural resources and energy with some of the highest CO_2 emissions.

The steel-making industry manufactures steel products through the electric steelworks and generates two fundamental types of slag (solid supernatant in liquid steel). The first, black slag (EAFS - Electric Arc Furnace Slag) is a 1st-degree fusion slag due to acidic oxidation in electric arc furnaces melting scrap, with a stony appearance (black) and a gravel-like geometry. It is heavy (20% denser than natural aggregate), porous, hard and of diverse granulometries, ranging from millimetres to several decimetres (Figure 1). The second one, called white slag (LFS - Ladle Furnace Slag), basic in nature, is generated in a ladle furnace (secondary metallurgy, steel refining) with (in general) a powdery appearance, high chemical/crystalline complexity and reactive in water and cement matrices [1].

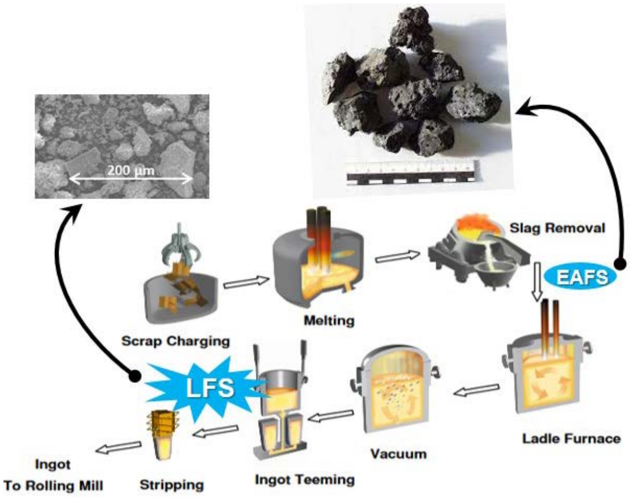

Figure 1: Electric arc furnace steel-making process.

Europe is the 2nd global steel producer with 10% of global production, its most important heavy industry. Spain is the 4th European steel producer and 3rd in terms of electric (scrap iron) production, after Germany and Italy, with 70% of their production: i.e., 0.21 t-steel/inhabitant, compared to the European average of 0.13 t. The Basque Country produces 45% in Spain, that is, 1% of the total electric furnace steel in the world [2].

As an example of construction industry impacts, the industry processed some 121 Mt of natural aggregates (70% by mass of concrete), in 2018 (unconsolidated with 2019/20), according to the Spanish ANEFA association. Moreover, concrete is among the materials that humankind has progressively employed in ever-greater quantities, despite its negative environmental effects having been known for many years: high CO_2 emissions, high (non-renewable) processing of resource and raw-material extraction. Furthermore, at slightly over 5% of Spanish GDP, the construction sector has seen a clear revival of rehabilitation works during the COVID-19 pandemic. Therefore, re-use of EAFS+LFS by-products in construction materials has an obvious environmental advantage, as demonstrated in previous research [3]. Within the geographical scope of this paper (Northern Spain), the annual production of steel-making slags implies some 600,000 t-EAFS and 120,000 t-LFS. Clearly, a material that has significant embedded energy (within itself), rather than wasted, can potentially be reused with a further consequence that its visible impact on the territory/society in the form of slagheaps and landfills will be lessened.

In a territory as small as the Basque Country, it would mean finding an annual productive use for 270 kg-EAFS/person and 110 kg-LFS/person (in Spain: 32kg-EAFS and 13kg-LFS). However, as stated in this research, the environmental/social advantage must neither be proposed at the expense of low/poor performance material (*does it work?*), nor industrial non-viability (*its cost?* ordinary concrete is around 0.04€/kg); in short, we need to promote "integral" sustainability.

As a result of this scenario, the present work is focused on designing siderurgical mortars that incorporate EAFS in partial substitution of fine aggregate (sand) and LFS in partial substitution of Portland cement and aggregates (filler) [4]. In all, several substitutions of mortar binders/aggregates for EAFS/LFS yielded different volumetric mixes: 8 MM mixes (compressive strengths below 20 MPa) and 12 SM mixes (compressive strengths over 50 MPa). The experimental results were based on physical and chemical characterizations of samples from the siderurgical batches of LFS/EAFS and both the fresh and the hardened properties of the MM and the SM siderurgical mortars: densities, spreading, mechanical strength, porosity and weathering studies.

As a concluding remark to this Introduction, the results fully support the use of these MMs for architectural (non-structural) rehabilitation works and the SMs for structural rehabilitation (strengthening techniques), for instance, in low-grade reinforced concrete structures.

2. MATERIALS

2.1. Steel-making slags: EAFS and LFS

EAF slag (EAFS) and LFS slag (saturated -Al/-Si, alumina/silica, respectively) were used in this work after various analyses of their properties: chemical composition, physical performance and main crystalline constituents (XRD), as detailed in table 1.

Table 1: Chemical composition and physical characteristics of slags.

Constituents	EAFS (average values)	LFS-Al	LFS-Si
Fe_2O_3 (%)	22.3	3.8	1.0
CaO (%)	32.9	54.0	59.2
SiO_2 (%)	20.3	3.8	21.3
Al_2O_3 (%)	12.2	28.8	8.3
MgO (%)	3.0	6.4	7.9
MnO (%)	5.1	0.3	0.3
Cr_2O_3 (%)	2.0	--	
Others (P_2O_5+TiO_2+SO_3) (%)	< 1.8	< 3.0	
Water absorption (%)	1.1	--	
Density (kg/dm^3)	3.4	2.8	
Fineness modulus (units)	4.0	0.8	
Fraction sizes (mm)	0/5	0.04/0.06 (prevalence)	
XRD	Wüstite-Ghelenite-Kirsteinite	Olivine-Mayenite-Periclase	

The black slag (EAFS) particles were crushed to a fraction size of 0/5 mm and left outdoors for three months of spontaneous weathering. Volumetric stabilization, which creates surfaces with an angular morphology and small cavities, then took place. Two extreme types of white slag were used in the steel-making factory: high alumina-low silica (LFS-Al) and high silica-low alumina (LFS-Si). The LFS alumina-saturated particles have to be gently crushed to sizes of less than 1 mm, following weathering and agglomeration over several weeks outside the factory. Therefore, LFS has a slight variability in relation to its chemical composition (different types of steel, refining process, etc.) and around 30% of its fraction could be considered hydraulically active as a supplementary binder material [5].

2.2. Natural aggregates, cement and water

Two types of Portland cements, based on UNE-EN 197-1, were used in the present research for MM mixes. The composition by weight of CEM I 42.5 R is: 90% Portland clinker, 5% calcium carbonate powder fines and 5% gypsum, but with a unimodal particle-size distribution of 20µm. Additionally, a CEM II/A-M (V-L) 42.5 R: 80% Portland clinker, 15% fly ash, 2% calcium carbonate, and 3% gypsum, but a bimodal particle-size distribution (34µm and 19µm, more frequently the former). This CEM II (addition of fly-ash) is a promising solution [1] when EAFS siderurgical aggregates release free CaO, improving the quality of the interfacial transition zone, as suggested elsewhere in other research.

In the SM, two Portland cements were applied: CEM IV/B-V 32.5-N and CEM I 42.5 R (as in MM). The former has a composition by weight of 5% CO_3Ca fines, 40% fly ash, 50% clinker and 4% gypsum, which is a promising solution if EAFS aggregates could release free lime, as stated in [6]. A commercial crushed natural limestone was used to promote a flowable cementitious matrix in the fresh state. These fine fractions were sized 0/4 mm (20% less than 0.063 μm) and their main mineral constituents were calcite (95%), with a density of 2.67 kg/m^3 and a fineness modulus of 2.4 units.

The mix water source, from the urban mains supply of the city (Bilbao and Burgos in MM and SM, respectively), contained no compounds with adverse effects on hydraulic mixes. Additionally, no admixtures were added to the MM mortars, in order to avoid undesired effects over the siderurgical aggregates (chemical incompatibilities, etc.) because admixtures increase the complexity of mix behaviour. Nevertheless, a commercial plasticizer was added to the MM admixtures, in order to improve their flowability, and, additionally an air-entrainment admixture to counter decreasing densities.

3. SIDERURGIC MASONRY MORTAR BEHAVIOUR

As described above, LFS could act as an active admixture in Portland cement mixes: mortars and concretes. Besides, adding a powdery mineral product to hydraulic mixes is a novelty approach in construction. However, because of the LFS mineral complexity, the focus must be on its free-lime and periclase components, because these constituents can cause volumetric instability (expansions). In other words, the results of the durability study are key.

3.1. Mix designs

The MM mixes are presented in table 2 with their different binders (CEM I and CEM II) and both LFS types in 10% and 20% cement substitutions (by weight): *i.e.*, I-MM10Al% means that 10% of the MM CEM I was substituted by LFS-Al. Several tests on 40×40×160 mm specimens were performed. A standardized reference mortar (M7.5) was produced for comparison with each MM. The reference specimens followed the specifications of EN 998-2 code, based on EN 196-1: pattern mortars with a workability of 170±3 mm (EN 1015-3/A1:2007) and a minimum compression strength (28 days) of 7.5MPa (as specified in EN 1015-11). As a natural aggregate, limestone sand (0/4) was mixed. Other properties tested were: fresh density (EN 1015-6), spreading (EN 1015-3/A1:2007) and mechanical strengths (EN 196-1:2005).

Table 2: MM mixes design and main properties.

Mixes	Dosage (kg/m^3)				Fresh stage		Mechanical Strength (MPa)	
	Water	CEM	Sand	LFS	Density (kg/dm^3)	Spreading (mm)	Compression	Flexion
I-MM0%	275	I-205	1840	0	2.18	175	17.6	4.9
I-MM10Al%		I-185		20	2.17	178	15.0	4.4
I-MM20Al%		I-165		40	2.18	191	11.8	3.3
I-MM10Si%		I-185		20	2.14	178	12.2	3.7
I-MM20Si%		I-165		40	2.14	177	10.2	2.9
II-MM0%	265	II-205	1840	0	2.21	175	17.7	4.3
II-MM10%		II-185		20	2.21	180	13.7	3.7
II-MM20%		II-165		40	2.18	178	11.4	2.9

As table 2 indicates, all the mixes developed compressive strengths over the required threshold (>7.5MPa, as 6 tests average). The tests showed a low scatter of the MM under bending loads (An average value from three tests in shown in table 2). Moreover, the textures of the failed surfaces may be inspected following the flexural tests. In view of its mechanical behaviour, LFS high alumina-low silica (LFS-Al) appears better suited as an addition than LFS high silica-low alumina.

Although outside the scope of this study, other LFS contents were analysed and, because of the low expectations obtained in 30% and 40% cement substitutions, we have only presented the 20% LFS and 10% LFS maximum content. As a concluding remark, 20% LFS (alumina or silica, respectively, LFS saturated) appears the maximum advisable substitution of Portland cement for commercial masonry mortars, focused on rehabilitation works (architectural purposes). However, as previously stated, durability issues should still be analyzed.

3.2. Durability studies: dimensional variations and wetting-drying cycles

DIMENSIONAL VARIATIONS

As previously outlined, the complexity of the LFS compounds (periclase, free-lime, etc.) means that it is advisable to analyze both shrinkage and expansion of the siderurgical masonry mortars. These dimensional tests are specified in the following codes: ASTM C596-07 and ASTM C490-93. The high expansiveness of the LFS constituents when wet meant that only three of the above mixes were selected: I-MM0%, I-MM10Al% and I-MM10Si%. It may be underlined that a maximum drying shrinkage of around 0.5mm/m is acceptable for ordinary MM (1 mm/m is an acceptable maximum figure for SMs).

Table 3 presents these results in specimens measuring 25×25×285 mm. The measured values were almost stabilized as from 75 days (each measurement is an average value from 4 samples). Higher expansiveness may be observed in low silica LFS (alumina-saturated slag). In contrast, when less than 10% of LFS was substituted for cement, regardless of the LFS type, an average expansive effect of 0.12 mm/m was observed, in partial compensation of the ordinary drying shrinkage of an MM.

Table 3: MM dimensional variations.

MM	Length variation (mm/m) along days												
	0 d	5 d	10 d	15 d	20 d	25 d	30 d	45 d	60 d	75 d	90 d	105 d	120 d
I-MM0%	0.00	+0.01	-0.02	-0.02	0.00	-0.01	-0.02	+0.01	+0.01	+0.01	+0.01	0.00	0.00
I-MM10Al%	+0.02	+0.05	+0.06	+0.08	+0.09	+0.10	+0.11	+0.12	+0.12	+0.13	+0.13	+0.13	+0.13
I-MM10Si%	+0.02	+0.03	+0.04	+0.05	+0,06	+0,07	+0.08	+0.09	+0.10	+0.11	+0.11	+0.11	+0.11

ACCELERATED WETTING-DRYING CYCLES

This study (see table 4) in the laboratory simulates rainy days and spontaneous drying according to ASTM D–559-03 (30 cycles/24h: 16 hours in-water, 2h stove-drying and, finally, 6 hours al 20ºC in-air). These effects were tested by measuring the changes to three main siderurgical MM characteristics: weight variations, porosity (Mercury Intrusion Porosimetry, MIP) and mechanical strengths (compression and bending). As previously declared, the LFS-Al showed a higher dimensional sensibility so I-MM10Al% and I-MM20Al% were analyzed and contrasted with the reference specimen I-MM0%.

Table 4: Physical and mechanical properties of each MM after wetting-drying cycles.

MM	Physical properties (from 0 days to 1 year)			Mechanical strength (MPa)	
	Weight (%)	Porosity		Compression	Flexion
		Pore diameter (μm)	Content (%)	(from 0 days to 1 year)	
I-MM0%	+0.08	0.35 to 0.38	21.8 to 22.6	17 to 19	5 (constant)
I-MM10Al%	+0.09	0.48 to 0.44	23.1 to 22.3	14 (constant)	4 (constant)
I-MM20Al%	+0.13	0.67 to 0.44	24.1 to 22.0	11 (constant)	3 (constant)

Table 4 presents the test results measured at the start of the test (day 0) and after one year. The table shows how higher amounts of LFS-Al meant higher specimen weights, because of the long-term hydraulicity of these white slags. Additionally, these siderurgical mortars presented higher porosities than the reference MM, in view of the accessible MIP porosity measurements of LFS-Al, in addition to the capillary porosity of the mortar. As regards mechanical strength, substitution of 20% of the cement for LFS-Al, after one year of weathering, reduced the strength by up to 40% (the link between the mechanical expectations and the pore structure is well-known: the higher the porosity, the lower the mechanical strength).

4. SIDERURGIC STRUCTURAL MORTARS BEHAVIOUR

4.1. Mixes design

Table 5 presents twelve SM mixes, designed for structural refurbishment, with improved workability. Seven mixes were based on a combination of EAFS plus LFS-Si (silica-saturated) and the remaining five, a reference specimen without EAFS aggregates but with 10%, 20%, 30% and 40% LFS-Si as binder (partial cement substitution).

Table 5: SM mix designs and main in-fresh properties.

Mixes	Dosage (kg/m^3)							Fresh state	
	Water	CEM	Sand	LFS-Si	EAFS	Plasticizer	Air entraining	Density (kg/dm^3)	Spreading (mm)
I-SMref	220	I-550	1585	--	--	1.20%	--	2.45	194
I-SM1		I-500	1470	167	--	1.20%	--	2.42	191
I-SM2		I-550	630	--	1260	1.60%	0.1%	2.54	194
IV-SM2		IV-550	630	--	1260	1.50%	--	2.62	204
I-SM3		I-500	515	167	1260	1.50%	--	2.71	184
I-SM4		I-515	550	117	1260	1.50%	--	2.71	189
IV-SM4		IV-515	550	117	1260	1.50%	--	2.62	199
I-SMref1	255	I-513	1540	--	--	1.20%	--	2.32	170
I-SM10%Si		I-462		51	--	1.25%	--	2.32	171
I-SM20%Si		I-411		102	--	1.35%	--	2.31	171
I-SM30%Si		I-360		153	--	1.40%	--	2.30	172
I-SM40%Si		I-309		204	--	1.40%	--	2.29	172

In terms of densities, the presence of EAFS aggregates implied almost 10% heavier mortars in relation to the reference specimen (non-slags). However, the decrease in these specific gravities when dosed with air-entrainment admixtures was around 12%. In terms of workability aspects (spreading), if the EAFS contents are higher than 50% by weight, approaching a self-compacting performance will almost be impossible.

Authors conducted an in-depth study of the EAFS+LFS-Si mixes and their comparison with others (I-SMref1, I-SM10%Si, I-SM20%Si, I-SM30%Si and I-SM40%Si, where neither stiffness - modulus of Elasticity E - nor porosity were tested), because the former mortars were better suited for structural applications (improved mechanical strengths), as table 6 shows. On the other hand, following the same logic, the durability studies were focused on the structural mortars of higher strength (I-SMref, I-SM1, I-SM2, IV-SM2, I-SM3, I-SM4 and IV-SM4). These mixes were analysed under hardened conditions, evaluating their mechanical behaviour and their durability expectations. In addition, the physical and mechanical properties (aged from 7 days to 90 days) are presented in table 6, following ASTM C 348 and ASTM C 349 codes, but the specimens were kept immersed in water at room temperature. The mechanical strengths were tested on 40×40×160 mm specimens and SM stiffness or modulus of elasticity (E) was measured using ultrasonic pulse propagation velocity techniques. The physical properties were obtained from the MIP test (a well-suited technique for these sorts of materials).

Having observed the physical properties in the hardened state, its density values led to similar conclusions, as previously stated (table 5). Besides, the porosities were well-performed with values between 5% to 10%, except in mixes that included additives (fly ash) and admixtures (plasticizer and air entraining).

Table 6: Physical and mechanical properties of hardened SM from 7 days to 90 days.

SM	Physical properties		Mechanical properties (MPa)		
	Bulk density (kg/m^3)	Porosity (%)	Compression	Flexion	E (180d)
I-SMref	2.38	5.3	72.3 to 93.7	10.8 to 13.38	55,800
I-SM1	2.38	5.4	55.5 to 90.5	9.9 to 11.4	53,700
I-SM2	2.49	12.6	58.7 to 77.6	9.9 to 12.0	49,500
IV-SM2	2.48	12.2	53.5 to 81.6	8.8 to 11.5	56,100
I-SM3	2.69	7.5	76.6 to 92.2	10.6 to 13.4	59,300
I-SM4	2.67	9.4	77.9 to 97.7	12.8 to 14.9	61,500
IV-SM4	2.53	13.2	40.9 to 68.1	6.8 to 12.7	52,400
I-SMref1	2.30	--	58.0 to 72	9.5 to 11.0	--
I-SM10%Si	2.30	--	53.0 to 67.0	10.0 to 11.0	--
I-SM20%Si	2.29	--	48.0 to 60.0	9.0 to 11.3	--
I-SM30%Si	2.29	--	39.0 to 53.0	9.0 to 11.0	--
I-SM40%Si	2.30	--	35.0 to 40.0	7.3 to 10.0	--

At 90 days, the compression strengths when the LFS-Si substitution by weight of cement was less than 30%, remained well above 50MPa, a well-suited property for structural applications, although air-entraining admixtures must be minimized. The strength development of SM with LFS-Si might be indicating that the hydraulic activity from this slag has a positive (and visible) effect at a medium term (90 days), but negative at early ages (7d). Additionally, the positive effect of the presence of fly ash (I-SM2 vs IV-SM2) was noted in the medium term.

Similar comments to those on the results of compressive strength may be echoed in relation to flexural strength, although the enhanced tensile strength of SM that contains EAFS may be underlined. The behaviour of stiffness (E) under compression was likewise coherent with the compression strength.

4.2. Durability studies: shrinkage and dimensional stability

SHRINKAGE TESTS

Four of the above mixes (I-SMref, I-SM1, I-SM2 and I-SM3) were used in the shrinkage test with 3 specimens measuring 25×25×287.5 mm per test (exposure environment). With regard to measurement of sample elongation, the ageing conditions were: in-air (270 days at room temperature, T_{amb}) and in water (120 days, T_{amb}). After 120 days in water, the expansions were almost negligible, but in-air, the specimens developed a none-too negligible shrinkage effect, reaching values in the region of 0.75 mm/m, but lower than 1.2 mm/m obtained in similar works [4].

DIMENSIONAL STABILITY

In these weathering studies two experiments were completed: the autoclave test (48 h at 130ºC/0.2MPa) and the water immersion test at 70ºC over 150 days (based on ASTM D-4792). These tests were used to evaluate any eventual bulk disaggregation of SMs under accelerated weathering.

The autoclave test yielded good results for the integrity of the sample specimens and only negligible detached materials confirmed the long-term integrity of these siderurgical mortars. In the hot-water study, after immersion in water (for the shrinkage test) over 120 days, all samples were subsequently immersed in hot water for 150 days. Satisfactory results were obtained following these immersions, because of the superposition of both effects (wet-expansion vs dry-contraction), however this superposition might entail a certain slight risk of cracking.

In conclusion, the properties for SM support the structural use of these siderurgical mortars such as, for instance, inorganic matrixes in composites (i.e., textile reinforced mortars) for strengthening low-grade reinforced concrete elements.

5. CONCLUSIONS

Siderurgical mortars have been successfully prepared using black and white slags, following careful proportioning. The addition of LFS as a binder can reduce the amounts of Portland clinker that are needed (lower CO_2 emissions, energy consumptions, etc.).

Overall, the incorporation of these by-products in high-performance construction materials (self-compacting mortars, low contraction mortars, etc.) can enhance the mechanical behavior of masonry and structural mortars, giving them a promising future.

As a preventive measure, a limit is recommended on LFS content of between 20% and 30% in partial substitution of cement, so as to facilitate the bulk integrity of these siderurgical mixes.

Finally, the re-use of these by-products in cement mixes has obvious environmental advantages and, in terms of their performance, they might be said to behave as *"noble raw materials"*.

6. ACKNOWLEDGEMENTS

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