DESTRO APPROACH FOR UP-CYCLING THE AIR-COOLED BLAST FURNACE SLAG OLD DUMPS. CASE STUDY CĂLAN, ROMANIA

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Increasing recycled content in products, while ensuring their performance and safety, will make a decisive contribution to achieving climate neutrality by 2050 and helps decoupling the economic growth from genuine resources. In line with this aim, the authors propose a superior recycling (up-cycling) of the ferrous metallurgical waste laying in an old Romanian heap aiming four environmental positive effects: saving natural resources used in cement industry; mitigating CO_2 emission due to energy saving by reducing the mining work and of the amount of $CaCO_3$ that decomposes during clinker manufacturing; eradicate the waste dump, recover the landfilled soil. The paper emphasizes the DESTRO approach that facilitates the whole waste dump recycling and land rehabilitation. Also, the paper brings news on sampling and characterization of heterogeneous lumpy wastes.

Keywords: air-cooled blast furnace slag, dump, up-cycling, zero-waste approach

1. Introduction

Greenhouse gases (GHG) consisting of CO_2, CH_4, O_3, NO_x, CFCs and H_2O vapor caused by anthropogenic emissions must be mitigated to diminish their detrimental effects upon climate changes [1]. Total anthropogenic emissions were 11.1 Gt C yr^{-1} (40.7 Gt CO2 yr^{-1}) in 2022, with a similar preliminary estimate of 11.1 Gt C yr^{-1} (40.9 Gt CO2 yr^{-1}) for 2023 [2]. Industrial sectors like metallurgy, energy production based on caustobiolithe fuel, cement manufacturing etc., are responsible for more than a fifth of global GHG emissions [3].

The cement industry is the third largest industrial energy consumer and it contributes approximately 7% of global GHG emissions [3], with total process

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emissions estimated at 1.50 ± 0.12 Gt CO_2 in 2018 [4]. The direct CO_2 emissions intensity of cement production has been broadly flat over the 2018-2022 years and is estimated to have increased slightly (by 1%) in 2023 [5]. The Net Zero Emissions Scenario by 2050 requires a decreasing of the CO_2 intensity emission of 4% through to 2030 [5]. Seventy-seven percent of emissions from concrete come from cement production alone, which disaggregates into carbonate decomposition (~55%), fuel combustion (~31%), and electricity use (~14%) [6].

Global demand for cement is expected to increase, especially in developing countries, because of the key role concrete plays in urbanization and public infrastructure projects, as well as the need to build structures more resilient to climate change. Hence, the main challenge facing the cement industry is reducing CO_2 emissions at the same time as meeting global demand [3,5].

The studies reviewed in [3] identified seven strategies to reduce GHG emissions from the cement and concrete sector i.e.: (I) energy efficiency; (II) fuel switching; (III) carbon capture, use and storage); (IV) reducing clinker ratio; (V) alternative binders; (VI) material and construction efficiency and (VII) CO_2 uptake (carbonation by cement systems under appropriate moisture and atmospheric CO_2 conditions). As could be seen in [3], the replacing of clinker genuine precursors with secondary raw materials as a valuable approach for mitigating CO_2 emission is neglected. Also, the use of secondary raw materials as precursors to clinker contributes to the saving of genuine carbonate and dolomite resources whose intensive consumption of which will lead to the depletion of these resources in the foreseeable future. It is worth mentioning that not only cement precursors, but many raw materials are crucial to Europe’s economy as they form an unreplaceable industrial base for producing goods and applications used in everyday life and modern technologies [7]. Accordingly, the access to raw materials is at the forefront of the EU as the international supplying chains have become unsure [8, 9]. *The secondary resources are part of the solution for increasing EU resilience against disruption of the raw material supplying chain.* [10]. The paradigm of secondary raw material (SRM) resources relies mainly on turning the waste into useful materials. Moreover, recycling of the landfilled mineral waste increases their circularity, mitigate the environmental detriment caused by their dumping and facilitates the land reclamation. Thus, increasing recycled content in products, while ensuring their performance and safety, will make a decisive contribution to achieving climate neutrality by 2050 and partially decoupling of the economic growth from genuine resource usage, while ensuring the long-term competitiveness of the EU and leaving no one behind [11].

In line with above mentioned circumstances, the authors propose a superior recycling (up-cycling) of the ferrous metallurgical waste laying in an old Romanian heap aiming four environmental positive effects: i) saving natural resources used in cement industry; ii) mitigating CO_2 emission due to energy saving by reducing the mining work and of the amount of $CaCO_3$ that decomposes during clinker manufacturing iii) eliminating the pollution caused by waste dump (dust pollution, surface and underground water pollution and visual discomfort); iv) land reclamation by its retrieving to natural use or as brown terrain.

The paper addresses a composite waste stockpile consisting mainly of air-cooled blast furnace slag (ACBFS) produced by Călan Siderurgical Plant during a long period of time i.e. 1870-2006. The slag is mixed with used and braked refractory ceramic liner, waste foundry sand. Also, small quantities of coke lumps, waste soil, rocks and municipal waste were landfilled across the Călan dump.

Blast furnace slag (BFS) is the product of the thermo-chemical reduction of the iron in the blast furnace (Fig.1a). The physical properties of ACBS (Fig. 1b) depend on the chemistry as well as the cooling condition and can be influenced by processing once the slag is removed from the furnace. Slow cooling, for example, generates crystalline blast furnace and steel slag. Quenching the liquid slag (e.g., granulation) on the other hand will generate a vitreous slag with latent hydraulic properties [12, 13].

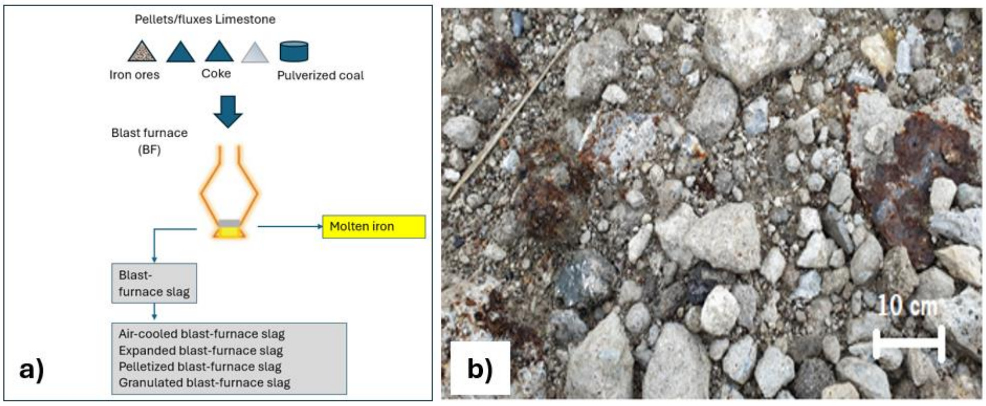

Fig. 1. a) BFS flow chart; b) characteristic aspect of the ACBFS deposited in Călan dump.

The typical compositional ranges of the BFS is given in Table 1.

Table 1.

Typical compositional ranges of BFS [17]

Constituent	CaO	SiO_2	Al_2O_3	MgO	FeO	MnO	P_2O_5	S_{total}	Cr_2O_3	Fe met
wt. %	35 - 42	33 - 38	10-15	7-12	≤ 1,0	≤ 1,0	<0.2	1 - 1,5	≤ 0,1	

The mineral composition of blast furnace slag generally consists of melilite ($Ca_2MgSi_2O_7$ - $Ca_2Al_2SiO_7$) and merwinite ($Ca_3MgSi_2O_8$) while steelmaking slag (BOF or EAF) mainly consist of dicalcium silicate (Ca_2SiO_4), dicalcium ferrite ($Ca_2Fe_2O_5$) and wustite ($(Fe_{1-x-y},Mg_x,Mn_y)O_z$) [14].

In the past, the liquid slag was sent to pits where it suffers an air-cooling in natural condition i.e. a slowly cooling process given rise to the so called ACBFS. In such conditions, the slag crystallize and finally it turns in a rock-like crystalline mass (Fig. 1b). Nowadays, liquid blast furnace slag is sent to a granulator where it is rapidly cooled by water jets or other processes as to turn it a glassy granular or sand-like product called granulated blast furnace slag (GBS). The present preferred route for BFS recycling is its transformation in GBS as Euroslag statistics underline (Fig. 2) [14].

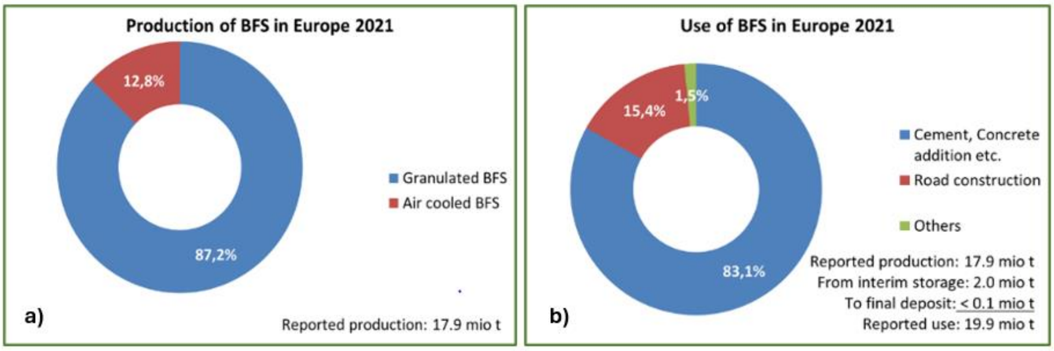

Fig. 2. Euroslag synthetical statistics: a) BFS production at EU level; b) Use of BFS in EU in 2021[14]

The Euroslag (Fig. 2 b) clearly shows that ACBFS is mainly recycled in road construction i.e. as aggregate in subbase layer and base layer, aggregates incorporated in road concrete and asphalt. Also, ACBS are used in railway construction for fortification/reinforcement of the base and for embankments. There are other low-value construction applications of the ACBFS in hydro construction (filler in banks, dykes, dams), manufacturing of finished products (aggregate in concrete blocks, bricks, bolts etc.) [15, 16].

The recycling of ACBFS as precursor of clinker was obscured by the GBFS, but some literature data on this topic exists [17-19].

Taking advantage of the possibilities of valorization of the ACBFS, the paper is focused on recovery of entire content of the old Călan deposit using a proper technology that separates the iron fraction from non-ferrous fraction through magnetic separation. The ACBFS size graded fraction is further separated from sand, soil and other particulate waste through sieving. Thus, the comminution technology applied to Călan waste pile generates five categories of still considered waste as: i) iron scraps; ii) 20-50 mm size graded ACBFS and ceramics; iii) 50-63 mm size graded ACBFS and ceramics; iv) fine particulate mixed waste (ACBFS, ceramics, sand, soil, coke), and v) municipal waste. The iron scraps are sent to metallurgical plants. The fine particulate mixed waste can be use as filler in road construction. The municipal waste, mainly plastics are sent to a specialized company in municipal waste treatment. The size graded ACBFS is sent to cement plants to be use as additive in clinker precursor. This up-cycling route of ACBFS is possible as it results from lime and silica-based melts, therefore calcium oxide (CaO) and silica (SiO_2) are their primary components. Other components in ACBFS include alumina (Al_2O_3) and magnesium oxide (MgO) that are not critical for a clinker precursor. The Fe content in BFS is critical for a clinker precursor and it must be lower than 0.5 wt. %. Thus, the ACBFS composition favorites its upcycling as additive in clinker precursor providing two main advantages CO_2 footprint reduction and saving genuine resources. The paper novelties consist in ACBFS up-cycling as clinker precursor and the DESTRO zero-waste approach which aim entire waste valorization as to facilitate the land rehabilitation.

2. Materials and Methods

2.1. Studied Area and Materials

The Călan slag dump is located near Old Town Călan, Hunedoara country and in the vicinity of the former Sidermet Călan metallurgical plant. (Fig. 3. a, b).

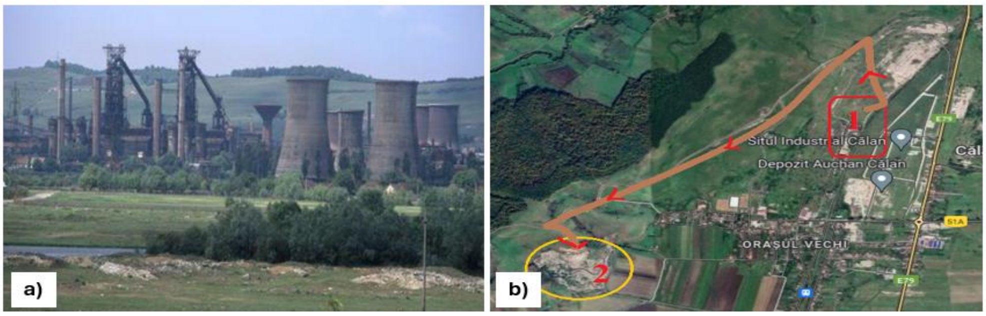

Fig. 3. Image of the Călan blast furnaces; b) the position of the Călan slag dump (2) relative to former Călan metallurgical plant (1) [https://csvCălan.wordpress.com/galerie-foto, accessed on 112.10.2023/ 11 h].

The beginning of the „Călan" plant is traced back to 1870 when was commissioned the furnace no. 1 of 82 m^3 capacity. During a hundred and half years (1870 -2012) in the so called Călan slag dump were deposited a part of the wastes coming out from cast iron production like: blast furnace slag, refractory ceramic linings, cast iron barks, ceramic molds, waste foundry sand etc. As some nearby inhabitants recall, approximately 2 million tons of solid waste were landfilled in Călan dump. Starting with 2012 the Călan dump was privatized, and the slag by-product and associated wastes were excavated and send to different application as filler in road and civil building construction up to 2021. Meantime, the owner has decided to up-cycle the remained content (fig. 4), assessed at a 1000.000 m^3.

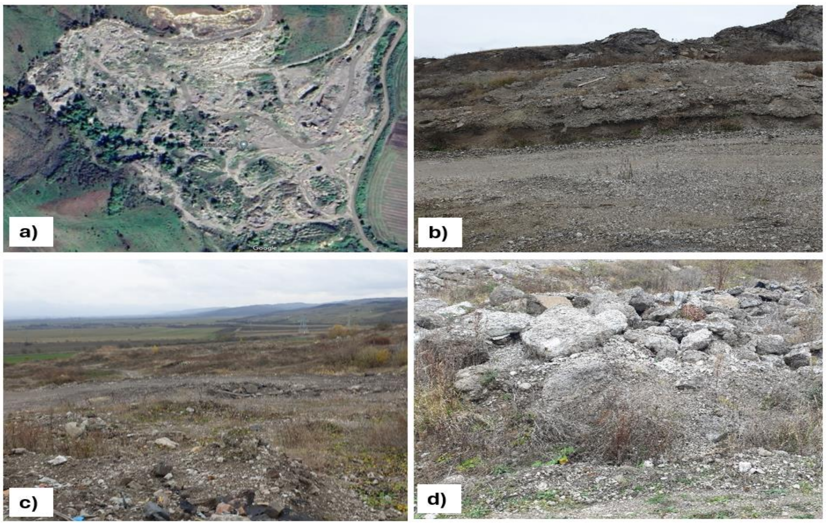

Fig. 4. Satellite images of the Călan slag dump and detailed images across the sit.

The Fig. 4 clearly shows the predominance of slag, but the composite nature of the materials discarded on the field. There are huge logs of slag, especially in the north part of the pile, great logs in the center and slag debrides mixed with ceramic waste of different provenience i.e. bricks, ceramic lining, plastics etc. It worth noticed that the wild flora has grown over the entire area of the site, which shows that the toxicity of the dumped waste is not significant.

The bulky slag lumps in Fig. 4.b), d) attest that the dumped slag was sent to air cooling. Hence, the dump contains for sure air-cooled blast furnace slag.

2.2. Waste comminution

The up-cycling of the ACBFS and of ceramic waste found in the Călan pile imposes a carefully crushing, deferring and sieving in 20-50, 50-63 and >63 mm of the composite waste. Also, a fine fraction is unavoidable produced during sieving. The flowchart of the applied technology is given in Fig. 5.

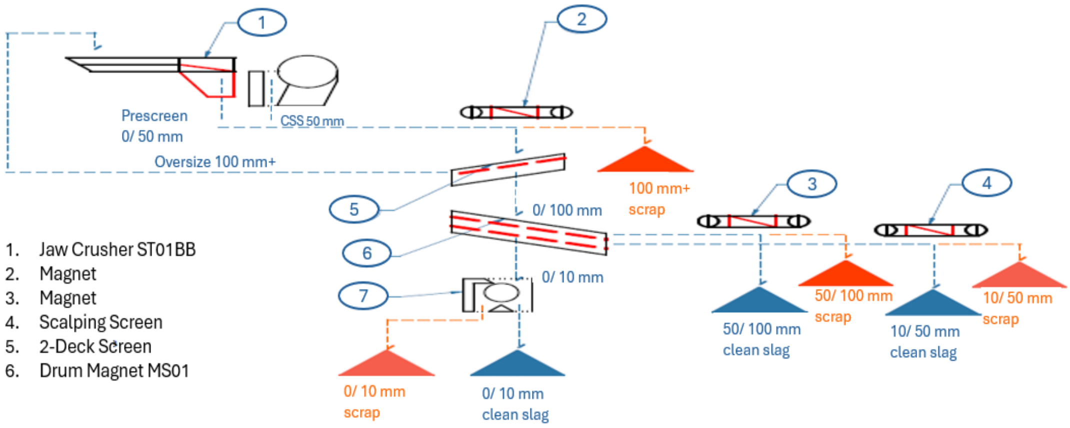

Fig. 5. Technological flow-chart applied to Călan old waste stockpile.

The DESTRO has developed technologies for extractive and metallurgical wastes valorization since 1991. Also, DESTRO has been an active player on Czech internal market for secondary raw materials. Thus, DESTRO company is engaged in sale and production of crushed slag aggregates and gravel. All products are certified and meet the CSN EN 13 242 standard. Based on DESTRO technologies portfolio an improved integrated solution for steel slag recycling in the frame of the zero-waste approach was advanced for a Călan old slag dump. Notwithstanding that the integrated solution presented schematically in Fig. 5 was designed based on sampling and a comprehensive characterization of the collected specimens [20, 21].

The comminution technology in Fig. 5 was implemented on Călan dump using Destro equipment. The hearth of the recycling slag process is a special JAW crusher with automated hydraulic release system of the non-crushable Fe or other particle that enter in the crushing chamber. In case where a non-crushable lump enters in the crushing chamber, the hydraulic system automatically opens the jaw up to 400 mm to allow the lump to go through the chamber to avoid harming the crusher and any interruption in the process. Such a protection process could not be done with a regular jaw crusher. The other equipment like sieving station, conveyor belts are of conventional type, and they can be adjusted depending on technological requirements.

2.3. Waste characterization

As was previously mentioned, the main aim of the paper is to demonstrate that the entire waste content of an old metallurgical waste pile can be retrieved to industrial use to eliminate its detrimental environmental effects. Besides, the major part of the as considered waste can be turned up into secondary raw materials. The secondary aim is to emphasize that ACBFS can be up-cycled as an additive in clinker precursor. In this regard, the cement industry sets up two prerequisites: 1) homogeneous mixture of granulated slag and ceramic waste ranging in 50- 63 mm in apparent diameter; 2) metallic iron and iron oxides content less than 2 %wt. The other waste categories coming out from waste commination at Călan have no prerequisite regarding their recycling. Therefore, only the fraction destined to up-cycling in clinker manufacturing must be adequate characterization to assess its bulk composition and its average mineralogical content. In this direction, macros-structural characterization was carried on by visual and optical microscopy.

An energy dispersive XEPOS spectrometer with 3D X-ray irradiation geometry, Spectro-AMETEK, was used to measure the elemental composition of the specimens. The software of the XEPOS can provide the results as elemental mass concentrations (%wt.) or as oxide (%wt.). XRFS technique is largely accepted as standard technique for waste characterization as oxide contents [20]. The oxides in ACBFS are found in complex compounds such as melilite ($Ca_2MgSi_2O_7$ - $Ca_2Al_2SiO_7$) and merwinite ($Ca_3MgSi_2O_8$), dicalcium silicate (Ca_2SiO_4), dicalcium ferrite ($Ca_2Fe_2O_5$) and wüstite (Fe_{1-x-y},Mg_x,Mn_y) O_z [14]. In this regard, the mineralogical composition of the sorted fractions was studied using a DRON M3 X-ray diffractometer and DIFFRAC plus BASIC – software package for qualitative and semi-quantitative phase analyses based on ICDD-PDF2 database.

3. Results and Discussion

The way of operating of the DESTRO slag comminution technology implemented at Călan dump is synthetically depicted in Fig. 6.

Fig. 6. The main steps of the DESTRO comminution technology applied at Călan landfill: a) wastes gathering; b) crushing step; c) magnetic deferrization; d) sieving and sorting stage.

The main outcomes of the DESTRO comminution technology consist of iron scrap (Fig. 7.a), and heterogeneous minerals sorted on three fractions (Fig 7b).

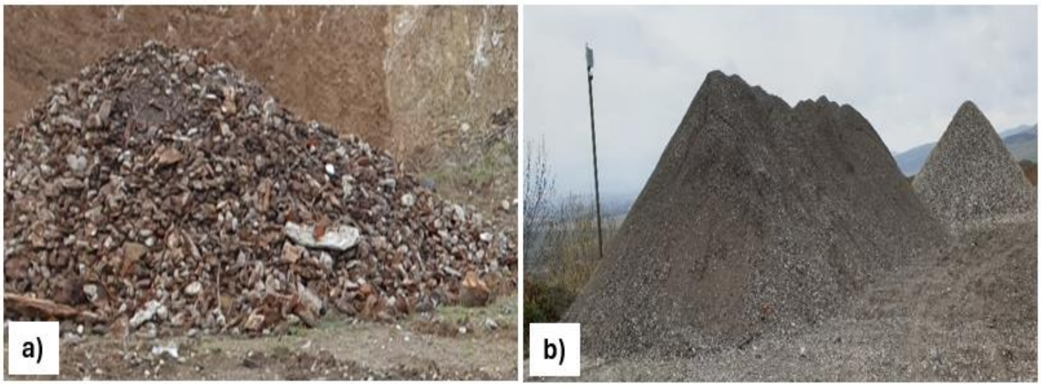

Fig. 7. The main outcomes of the DESTRO comminution technology a) iron scraps; b) heterogeneous minerals sorted on three fractions.

The visual observations of the size graded fractions (Fig 8. a, b, c) attest their higher heterogeneity as mineralogy and shape, independent on the size grade.

The image in Fig.8 d) shows an inner heterogeneity of a fragment of slag which underpin a higher heterogeneity at any scale level of the material under study. Accordingly, the characterization of the sorted fractions of 20-50 mm and 50-63 mm based on particle characterization is nonrealistic. Therefore, the elemental and mineralogical compositions of the fraction under study can be investigated as the mean composition. In this regard, representative samples must be obtained using reliable sampling and subsampling procedures. Hence, in the first stage, 3 samples of 20 kg were collected from a pile fraction of 50-63 mm located near base half-height and near top. In the same way, samples were collected from a pile fraction of 20-50 mm. The samples were crushed at laboratory scale and milled in a steel-ball mill until a powdery aspect was reached. 1 kg of powder was collected from 20 kg by taking subsamples with a sampling scoop from different locations of the primary sample. The 1 kg subsample was thoroughly mixed and subsequently conned and quartation. Two opposite quarters were selected and mixed to form a 250g sub specimen.

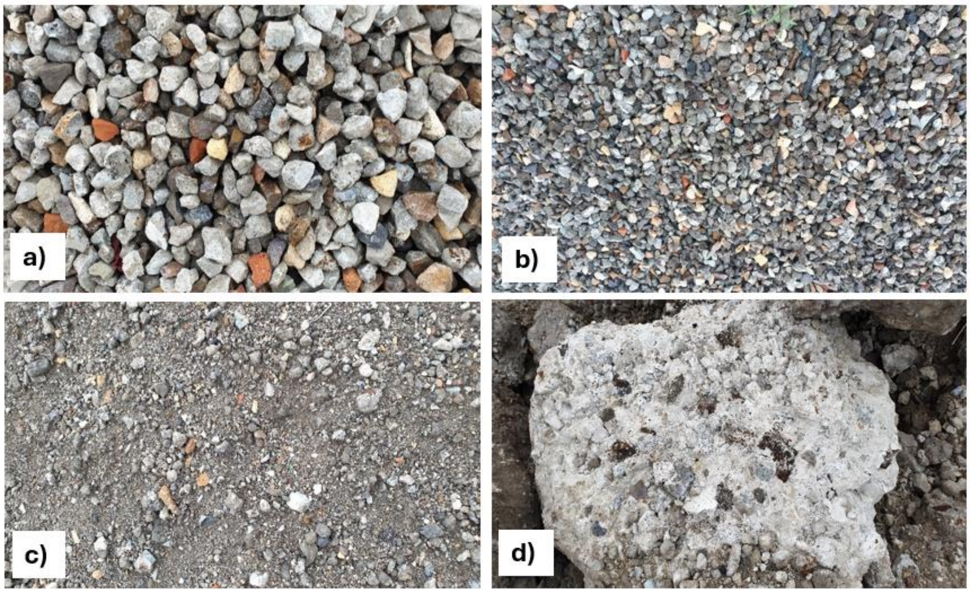

Fig. 8. Images of the sorted waste: a) grade size 50-63 mm; b) grade size 20-50 mm; c) less than 20 mm in size; detailed image of a heterogeneous fragment.

This sub specimen was sent to advanced milling to obtain a powder with particles less than 100 µm in diameter. The same procedure was applied to the all collected samples. Specimens of approximately 100 g were calcinated at 950 ^{o}C before undergone XRFS and XRD measurements. The loss of ignition (LOI) was estimated by weighing the specimen before and after calcination. The LOI measurement procedure is described in a previous published paper [21]

The composition of the representative samples collected from size graded waste 20-50 mm and 50-63 mm and the reference values of genuine clinker precursors are given in table 3. The specimens comminuted from 20-50 mm fraction are denoted S20_1, 2, 3 i.e. located at base, half-half and top of the heap. The similar notation S60_1, 2, 3 is used for specimens prepared from the 50-63 mm fraction.

Table 2.

Composition of the representative samples collected from size graded waste 20-50 mm and 50-63 mm

Characteristic [%wt]	**Călan ACBFS and accompanied waste**						**Genuine clinker precursors**		
	Determined values (measured)						Reference values		
	S20_1	S20_2	S20_3	S60_1	S60_2	S60_3	Limestone	Argyle	Sand
LOI	1.92	1.24	1.51	1.27	1.18	2.81	43.60	9.36	0.5
SiO_2	34.94	33.46	33.33	33.42	31.71	34.93	0.40	53.00	96.92
Al_2O_3	10.52	10.85	10.71	10.82	10.27	10.47	0.31	18.30	0.50
Fe_2O_3 total	0.56	0.43	0.55	0.56	0.55	0.56	0.10	7.07	0.39

CaO	41.3	42.26	41.76	43.29	44.77	42.35	54.98	6.07	0.83
MgO	5.03	5.13	6.08	4.14	4.71	5.21	0.20	1.98	0.40
SO_3	1.06	1.27	1.06	1.31	1.07	1.28	0.05	0.32	0.35
Na_2O	0.34	0.28	0.36	0.29	0.35	0.29	0.37	0.78	0.00
K_2O	0.34	0.5	0.35	0.48	0.36	0.49	0.02	2.39	0.10

The oxide composition of the two sorts is close as the values presented in the table 2 show up. The Xepos measurements indicated the presence of environmental detrimental heavy metals in the slag specimens like Pb, As, Hg, Cd, Zn, Cu at ppm level below and close to the its limits of quantification. The analytical uncertainty of the trace level concentration is higher therefore it was avoided to include them in Table 2.

The XRD analysis highlighted the crystallinity specific to non-granulated slags (Fig. 9. a,b).

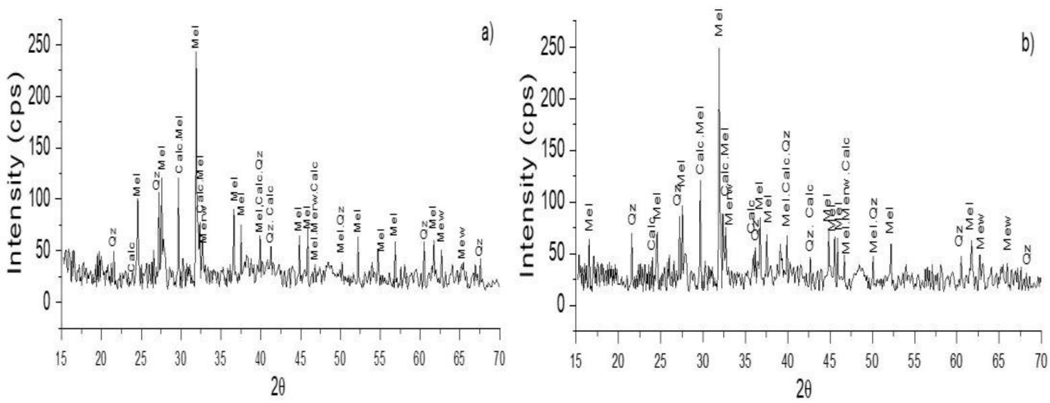

Fig. 9. a) XRD pattern of the 20-50 mm ACBFS sort; b) XRD pattern of the 50-63 mm ACBFS sort

The crystalline phases identified based on ICDD powder diffraction files (pdf) were melilite (pdf no. 79-2423), merwinite (pdf no.35-0591), larnite (card no. 33-0302)), quartz (pdf no. 46-1045) and calcite (pdf no. 05-0586). The peaks in diffractogram of Fig. 11.a) were marked as Mel for melilite, Merw for merwinite, Qz for quartz, Cal for calcite. Melilite is the major phase as its diffraction peaks are the most intense compared to those of quartz, calcite. Merwinite accompanies the melilite phase in ACBFS, therefore its presence is assumed even that there is no distinct merwinite peak in the diffractogram but overlapped diffraction lines. The phase content of the specimen prepared from 50-63 mm sort is similar to the one prepared from 20-50 ACBFS sort. The diffractograms in Figs. 11 a), b) do not show a background specific to the amorphous slag phase as is presented in the literature [22-24]. Hence, the crystallinity of the ACBFS in Călan dump is at least of the 75%.

4. Conclusions

The Destro technology applied to Călan metallurgical old dump for comminution of ACBFS and accompanied wastes (refractory ceramic linings, bricks, waste sand etc.) is effective as it avoids disruption of the crushing process based on intelligent jaw crusher.

The Destro comminution, deferrization and sieving ensure the turning of heterogeneous metallurgical waste into four recyclable by-products as: iron scrap and three size graded minerals. Also, a municipal waste fraction is collected and sent to a company that manages such a waste.

The size graded 50-63 mm is up-cycled as clinker precursor which contributes to saving the genuine resource and also to the CO_2 footprint reduction in the cement industry. The size grade 20-50 mm is recycled as aggregate in concrete for road and pavement manufacturing. The remaining sort, less than 20 mm in diameter, contains slag, ceramics, sand and soil. This barren composite can be used as filler in road construction or in mines and quarries rehabilitation.

The above solutions for recycling the entire content of the old Călan metallurgical waste dump finally lead to the elimination of the waste from Călan dump and facilitate the land reclamation, turning it into a proper biotope.

Future researchers will be carried on the topic of increasing ACBFS usage to the genuine resource replacement in cement industry.

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