WPI 3.4.3. Report on lessons learnt on site

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1. Introduction

The NEW-REGENERATIS project (Interreg North-West Europe), coordinated by SPAQUE, aims at demonstrating that resources (metal, minerals, and land) can be recovered from Past Metallurgical Sites and Deposits (PMSDs) by Urban Mining techniques.

CTP's involvement in the project essentially consists in assessing the metals recovery potential from the selected metallurgical site located in Belgium, i.e, the Duferco-La Louvière site, by applying mineral processing techniques.

The proposed flowsheet treatment mainly consists in crushing, screening and applying magnetic separation, in view of recovering valuable metal from the 3000 tons of metal gangue treated. Among the recovery routes foreseen for both the residual slag aggregates and the fines generated by the process, a valorisation in road construction is explored.

A previous experimental laboratory study carried out at the CTP (report D T2.1.2) showed that the use of the finest slag fraction generated by grinding and crushing (0-10mm) gives good performance as substitute of hydraulic binders for the mechanical stabilization of clayey/loamy soils. The valorization of the coarser aggregates fraction (10-30mm) has also been studied and one of the options considered is the use as road base layer provided that possible swelling problems have been ruled out.

In order to assess the performance of these two recovery routes at larger scale (pilot scale), different test slabs have been implemented on the Duferco's La Louvière site. This report summarizes the first results obtained.

2. Soil stabilization and road technology valorization

2.1 Introduction

Soil treatment with lime is a technique whereby fine moist loamy and clayey soils are mixed with quicklime in order to obtain a new material with enough improved geotechnical and engineering properties to build trafficable platforms for road. The use of quicklime-treated soil is then similar to aggregates use, and also contributes to the management of soils on earthworks projects. This technique fully embraces the logic of the circular economy. The addition of quicklime to soils can have two treatment objectives, improvement or stabilisation as depending on the lime quantity, the specific use and the performance level to be achieved. Stabilisation also improves resistance both to water and to frost damages.

However, quicklime is obtained by limestone firing at a temperature generally ranging between 900 and 1000°C. Consequently, quicklime is a high energy consuming material which in addition produces large CO_2 amounts due to decarbonation phenomenon. In addition, quicklime is an expensive material (its price in Belgium is around 180 € per ton) and this value is set to rise as a result of the increasing taxes to be paid for CO_2 quotas emitted.

Thus, any alternative material is welcome, especially if it consists in waste of by-product of low added value and contributes to the realization of eco-friendly products for road construction.

2.2 Laboratory tests

As a first step, the soil stabilization capabilities of fine slag particles was assessed at laboratory scale.

The selection and characterization of a suitable soil that complies with the Belgium Road Research Center recommendations (use for lime treatment of soils with at least 15% of particles having a size below 63 µm and a plasticity index equal or greater to 5%), the determination of the bearing capacity of this soil before and after stabilization and the description of the formulations and the treatment conditions have already been described in a previous report (c.f. D. T2. 1.2. preliminary report on mineral processing lab pilot scale test on samples from Duferco site (2021))

As shown in Fig 1, the results of the performance assessment show that a formulation incorporating both 0.5% quicklime and 10 to 15% of freshly crushed slag of less than 10mm leads to an improvement of the soil bearing capacity similar to that obtained through a treatment with commercial quicklime introduced at a rate of 2%. This treatment protocol will be the one chosen for the pilot scale testing phase. The drying or additional crushing of the slag gives better performances. However, the additional cost associated with those operations may be detrimental to the profitability of this recovery process.

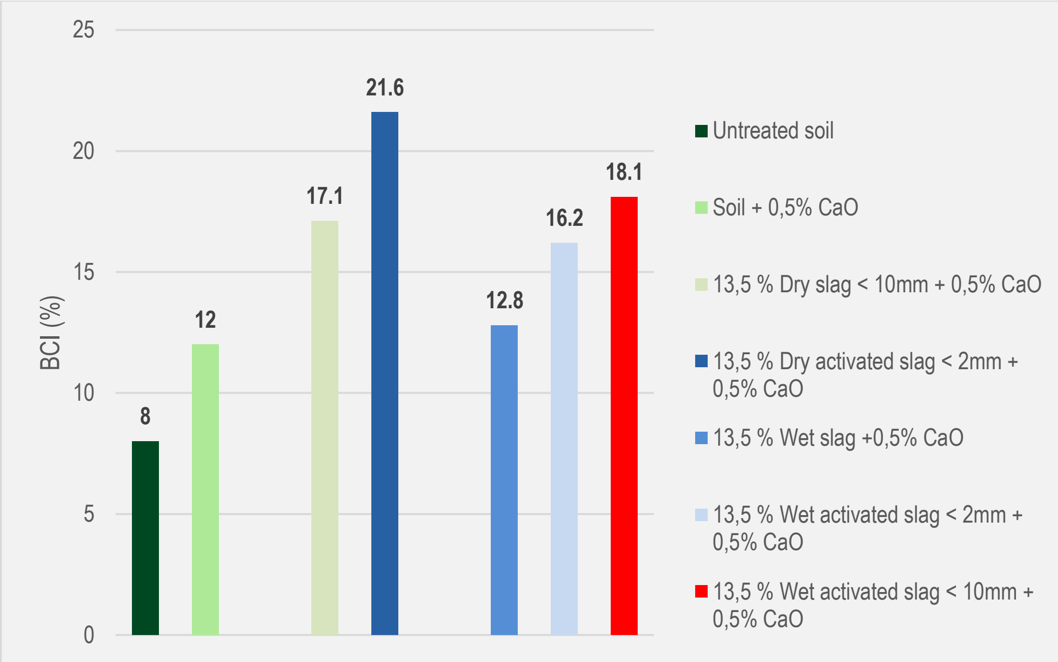


Figure 1 : Bearing Capacity Index of soils stabilized either with quicklime or slag containing compositions

2.3 Pilot scale test slabs (demonstrators)

From March to May 2023, the implementation at a large pilot scale of the slag process developed in CTP laboratory has been realized on the DUFERCO site based in La Louvière. A mobile processing unit allowed to treat over 3.000 tons of slag material with the purpose to refine the treatment conditions of metal gangue and to produce three experimental test slabs in view of performing plate bearing tests. Pictures 1A and 1B illustrate the operations associated with the test slabs implementation and compacting. Pictures 1C and 1D concern the layout of the test slabs and the layers thickness. Finally, picture 1E refers to the plate bearing test.
The three test slabs built on the Duferco - La Louvière host site are shown in Figure 2. The two former test slabs refer to soil stabilization using the 0/10 mm fraction and the last one to subbase or subgrade[1] road layers realization using the coarser aggregates fractions (10/20 mm + 20/32 mm).

- **First and second test slabs :**

Ninety m³ of loamy soil have been spread on the ground and compacted over a total area of 225m² (9m x 25m x 0.4m depth)

Before spreading, the soil has been previously mixed and homogenized with binding materials following three different formulations:

[1] Depending of the bearing capacity index as measured by plate bearing test.

- the selected laboratory conditions: 13.5% by mass of 0/10mm crushed fines slag and 0,5% quicklime (area 1a - tests n°1 to 3);
- 17% by mass of 0/10mm crushed fines slag and 0,5% quicklime (area 1b - tests n°4 to 6);
- 13.5% by mass of 0/10mm crushed fines slag and 1% quicklime (area 1c - tests n°7 to 9);

During the mixing operation, water content has been adjusted in order to comply with laboratory conditions.

A second testing platform has been realized in same conditions of surface and depth.

Three additional formulations have been tested using this time the screened 0/10 mm slag fraction:

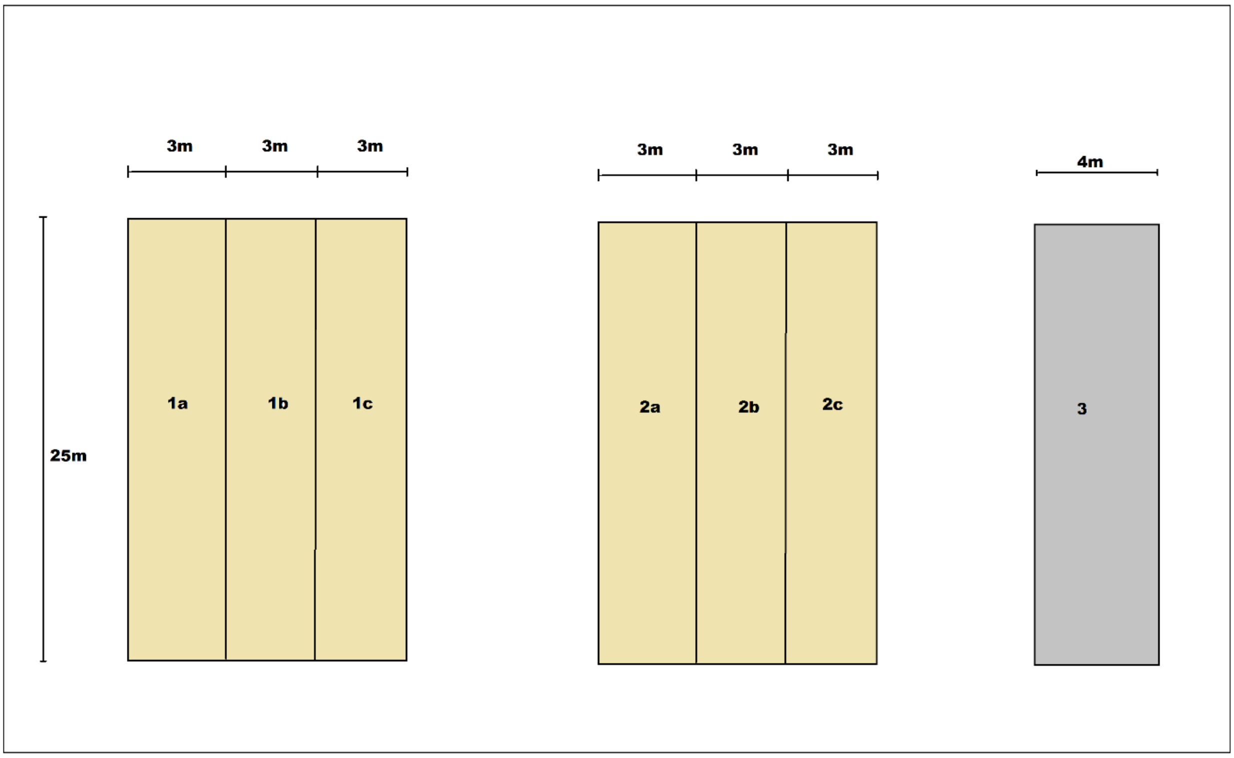
- 13.5% by mass of 0/10mm screened fines slag and 0,5% quicklime (area 2a - tests n°10 to 12);
- 17% by mass of 0/10mm screened fines slag and 0,5% quicklime (area 2b - tests n°13 to 15);
- 13.5% by mass of 0/10mm screened fines slag and 1% quicklime (area 2c - tests n°16 to 18).

It must be pointed out that in addition to the selected laboratory conditions based on the used of 13,5 % of the ground 0/10 mm, other compositions have been tested. First, the amount of this fine slag fraction has been enhanced and secondly the initial fine slag content has been kept but a larger amount of quicklime has been added. We believe that due to climatic conditions (rain), the freshly crushed fine slag has been submitted to weathering. Consequently, CaO and MgO oxides have been transformed into their respective hydroxydes and the hydraulic phase (Larnite) identified on the DRX pattern is likely hydrated. Thus, this fine slag fraction is presumably less reactive than the one used for laboratory tests in CTP. Based on these considerations, it is possible that slag obtained by simple sieving could give results quite similar to those obtained with crushed and then sieved slag. This would be particularly interesting, as it would considerably reduce process costs and make recovery more profitable.

- **Third test slab:**

As previously mentioned, this third test slab concerns road layers obtained by compacting the coarsest fractions. It consists of a 100m² surface composed of a 0.5m-thick layer of slag aggregates. This test slab is obtained by spreading and then compacting a suitable mixture of 10-20mm and 20-32mm aggregates (screened aggregates + crushed and screened aggregates) to comply with the particle size listed in section 4.4.1. of chapter C of Qualiroutes (area 3 - tests n°19 to 21). It must be emphasized that in order to ensure an enough bearing capacity and to allow a better compaction a little bit amount of the screened 0/10 mm slag fraction has been added.

Picture 1 : Illustration of the test slabs implementation (1A to 1D) and of the bearing test



(1E)

Figure 2 : Schematic representation of the test slabs layout

2.4 Results

According to Belgian legislation (Qualiroutes), the test reported in the technical standard applicable for road construction in Belgium and which allows to assess the material performance is the plate loading test CME50.01.

Test description : The vertical settlement is measured under a circular steel plate exerting a vertical pressure on the surface under examination increasing in increments. The result is represented by a load curve bringing together the experimental points with stabilization (pressure applied on the abscissa, associated settlement on the ordinate).

From the experimental data, the compressibility coefficients Mi can be calculated according to the general formula:

$$M_i = \frac{D.\Delta p}{\Delta s}$$

(in MPa)

where: D = theoretical diameter of the plate (in 0,1 mm);

Δp = pressure difference between two loading bearings (MPa);

Δs = difference in settlements, corresponding to Δp (mm).

2.4.1 Short term performance

On June 2, soil implementation was carried out according to the following steps : spreading, moisture adjustment, slag and lime incorporation by mixing using a road milling machine and compaction. Three days after the implementation, the bearing capacity of the test slabs has been measured to assess the performance trends. All the results are reported in Table 1. It must be pointed out that prior to the stabilization tests, bearing measurements were carried out on the raw soil to determine its bearing capacity before treatment. The bearing values will serve for comparison purpose.

Test référence		Area identif.	Binder formulation	Bearing coefficient M1 (MPa)	
Date	test n°			Indiv. values	Av. value
01-06-23	0A	Test realized on compacted raw material (soil without binder)		19	14,7
	0B			14	
	0C			11	
05-06-23	1	1A	13,5% of 0/10mm crushed slag + 0,5% quicklime	29	19,5
	2			26	
	3			18	
	4	1B	17% of 0/10mm crushed slag + 0,5% quicklime	43	37,3
	5			41	
	6			28	
	7	1C	13,5% of 0/10mm crushed slag + 1% quicklime	33	43,7
	8			46	
	9			52	
	10	2A	13,5% of 0/10mm screened slag + 0,5% quicklime	45	29,0
	11			27	
	12			15	
	13	2B	17% of 0/10mm screened slag + 0,5% quicklime	46	42,3
	14			38	
	15			43	
	16	2C	13,5% of 0/10mm crushed slag + 1% quicklime	45	35,0
	17			34	
	18			26	
	19	3	mixed crushed and screened aggregates :10/20 crushed, 20/32mm crushed, 10/20mm screened, 20/32 screened, 0/10mm screened	28	26,7
	20			25	
	21			27	

Table 1 : Bearing capacity test results (Belgium plate bearing test CME 50.01)

As we can see from Table 1 and Figure 2 examination, the initial bearing capacity of the compacted raw soil is about 15MPa which is just below the requirement values for a subgrade use (requirement of 17 MPa), but which could be accepted as an embankment (requirement of 11MPa).

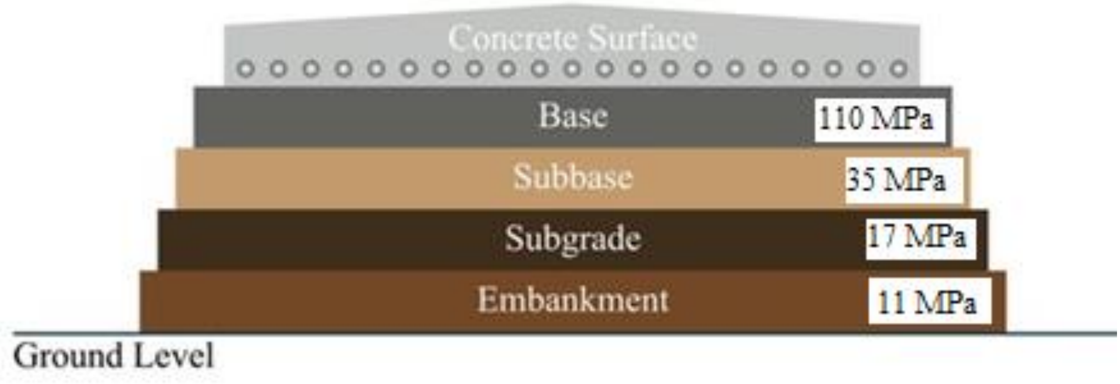


Figure 2: Rigid pavement profile

First of all, it is relevant to note that there is a significant variation in the individual values measured in each zone, which makes it difficult to interpret the influence of differences in formulations on the measured bearing capacity. Two phenomena could explain these large variations in measured values:

- Difficulties in homogenizing and mixing slag with the soil;
- Less energetic or less efficient compaction at the periphery of test platforms (side effect).

However, if we consider the results as a whole, we observe that:

The bearing results measured on the stabilized soil following laboratory conditions, incorporating 13.5% crushed slag and 0.5% quicklime (tests 1 to 3), and tests 10 to 12, integrating slag from a screening treatment in same quantities show a clear improvement in bearing capacity but without achieving the minimum values required for use in road subbase (35MPa). From table 1 examination, it is also interesting to emphasize that by increasing the 0/10 mm slag content (17% instead of 13,5%) or by increasing the quicklime content while maintaining the initial 0/10 mm slag fraction content, the 35 MPa criteria is met whatever the treatment applied (screening or crushing). Concerning test 19 to 21 which refer to layers obtained by compacting the coarser slag fractions, the corresponding bearing index shows that those layers can be used as subgrade in road construction.

So, as a conclusion, the bearing capacitiy index measured on test slabs proves that, despite the observation of heterogeneous values for slag stabilized soils, the overall performance in terms of bearing capacity is above the threshold criteria (35 MPa) and allows to meet the requirement for a subgrade use. The longer than expected storage and exposure to wet weather conditions of the prepared slag nevertheless led to a decrease in their binding properties and required either an adjustment in the incorporation rate from 13.5% (as planned in the laboratory study) to 17% or an increase in quicklime content (from 0.5 t0 2%) to achieve the minimum performance required after 3 days of treatment.

2.4.2 Long term performance

The laboratory analysis carried out on the slags showed a chemical composition that allows to expect a good reactivity. Their high content of alumina, silica and iron oxide (SiO_2+Al_2O_3+Fe_2O_3> 55%) suggests possible pozzolanic properties and long-term reactivity provided that part of those components is under amorphous state. In addition, presence of calcium (CaO) and magnesium (MgO) oxide generally allows to expect some short-term reactivity, but it is probable that, for the slag used for the test slabs, these phases having been in contact with wet weather for more than 3 months, they are under a hydrated form and consequently no longer offer the same reactivity.

When lime is added to soil, it happens:

- a quick change in moisture content (particularly when quicklime is used);
- short term modifications of the geotechnical properties of the soil;
- long term modifications of the soil properties.

The addition of lime having a basic character associated with the alkaline slag causes the pH of the soil to rise after mixing. The higher pH values promote dissolution of silica and alumina oxides from the clay minerals. These dissolved compounds react with Ca present both in the lime and the slag fraction to form calcium silicate hydrates (CSH) and calcium aluminate hydrates (CAH), which precipitate and bind the soil particles. The same phenomena may occur for silica and alumina contained in the amorphous slag phase therefore also leading to soil particles binding. This reaction is called pozzolanic and produces similar hydrated hydraulic phases as for composed cement even if their reaction kinetics is slower. The pozzolanic reaction if effective should improve the mechanical properties of the stabilized soils giving rise to a gradual increase of the bearing capacity with time (over months).

Considering this possible contribution to binding properties, we can expect an improvement of the bearing capacity of each of the stabilized soils composing the test slabs. Thus, in order to assess the performances variation over time, additional bearing capacity test have been realized on June the 12, i.e., after 10 days of treatment.

The results are reported in the following Table (Table 2).

Area identif.	Binder formulation	Curing time : 3 days		Curing time : 10 days	
		Bearing coefficient M1 (Mpa)		Bearing coefficient M1 (Mpa)	
		Indiv. Values	Av. value	Indiv. Values	Av. value
1A	13,5% of 0/10mm crushed slag + 0,5% quicklime	29	24,3	69	53,3
		26		65	
		18		26	
1B	17% of 0/10mm crushed slag + 0,5% quicklime	43	37,3	79	64,3
		41		59	
		28		55	
1C	13,5% of 0/10mm crushed slag + 1% quicklime	33	43,7	72	70,0
		46		69	
		52		69	
2A	13,5% of 0/10mm screened slag + 0,5% quicklime	45	29,0	59	51,0
		27		65	
		15		29	
2B	17% of 0/10mm screened slag + 0,5% quicklime	46	42,3	55	55,7
		38		58	
		43		54	
2C	13,5% of 0/10mm crushed slag + 1% quicklime	45	35,0	50	53,7
		34		64	
		26		47	
3	mixed crushed and screened aggregates (10/20 crushed, 20/32mm crushed, 10/20mm screened, 20/32 screened, 0/10mm screened)	28	26,7	40	42,3
		25		51	
		27		36	

Table 2 : Variation of the bearing capacity over time (after 3 days and 10 days)

Table 2 clearly emphasizes that the bearing capacity increases whatever the considered stabilizing composition therefore suggesting that pozzolanic reaction effectively occurred. Thus, the minimal value for subbase use is largely exceeded for the whole test slabs, also including the one stabilized with the coarser particle fractions (Area 3).

3 Conclusion

The plate bearing tests confirm that as depending on the considered grain size fraction the slag can be used either for soil stabilization or for road construction sublayers.

Concerning soil stabilization, either the 0/10 mm fine slag fraction content (17% instead of 13.5%) or the lime content (1% instead of 0.5%) must be increased due to a weathering effect associated with climatic conditions during processing (rain) which decreases its reactivity.

For those modified compositions, the minimal criteria for a subbase use is met.

Concerning sublayer for road construction composed of the coarser fractions (screened and ground 10/20mm and 20/32 mm fractions), the bearing capacity value is in accordance with a use as subgrade.

Long term assessment performance shows a large bearing capacity index improvement whatever the stabilizing composition used as the values of this parameter are well above the subbase minimal threshold (35 MPa). This undoubtedly proves that as expected, pozzolanic reactions contribute to soil binding.

Based of those results, it clearly appears that the screened as well as the ground fractions composing the slag stream can be valorised.