Recycling of Waste Materials Using Bitumen Emulsion for Road Pavement Stabilized Base Courses: a Laboratory Investigation

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Abstract. The valorisation and reuse of waste materials can enhance the environmental sustainability of road constructions, especially by means of cold recycling techniques, which, moreover, allow to reduce polluting emissions in atmosphere. Among the various technological approaches, the use of bitumen emulsion to stabilize waste materials is very common, especially in case of reclaimed asphalt pavement (RAP) aggregates. However, even other types of waste materials could be considered using a Cold Central Plant Recycling (CCPR) approach. The paper discusses the main results of a laboratory investigation aimed to evaluate the mechanical performance of bitumen emulsion stabilized mixtures for road pavements base courses, prepared with RAP, steel slag, coal ash and glass wastes, used with various percentages. In a first step of the laboratory study, both physical and toxicological properties of each waste material have been investigated, in order to assess their environmental compatibility. Subsequently, an extensive mechanical analysis of the bitumen emulsion stabilized mixtures has been carried out in the laboratory, in terms of indirect tensile strength, indirect tensile stiffness modulus at three temperatures (10°C, 25°C, 40°C) and repeated load axial tests at 30°C. The moisture resistance of the mixes has been also investigated by means of indirect tensile strength tests carried out on soaked specimens. Very good results have been observed, depending on the mix composition: indirect tensile strength at 25 °C on dry specimens up to 0.52 MPa and stiffness modulus up to 4,056 MPa (at 25 °C, for a rise time equal to 124 ms). Therefore, it has been verified that the waste materials considered in the study can be successfully reused to completely substitute conventional aggregates in bitumen emulsion stabilized mixtures for road pavements base courses.

1. Introduction

Economy and environmental sustainability are nowadays important principles to consider in civil engineering, including the road construction industry. In this context, the total or partial replacement of the quarry stone aggregate with a waste material that derives from local civil or industrial activities, which can ensure to the mixture the required mechanical performances and volumetric properties, still represents a technological challenge; nevertheless, it is also the most efficient approach to successfully combine both the aforementioned demands [1].
“Marginal materials” is the term increasingly used in the scientific literature with regard to different types of industrial by-products [2], as for example reclaimed asphalt pavement (RAP), electric arc furnace steel slag (EAF), coal fly ash (CF), and glass waste (GW). Previous studies, aimed at evaluating the effect of their individual use in road and airport flexible pavements, can be identified in the relevant literature [3-7]. In order to reuse simultaneously such marginal materials in road construction, a stabilization treatment with bitumen or cement is required. In pavement engineering, bitumen emulsion is mainly used as a stabilization technology for mixtures containing RAP [8-11], while it is less frequent for mixes prepared with the other marginal aggregates [12]. This cold recycling approach is to be preferred as it also ensures the environmental sustainability of the mixture production process by reducing the polluting emissions of hot technologies [13-20].

The current study presents the experimental results of a laboratory investigation aimed at verifying that the recycling of the above mentioned marginal materials into bitumen emulsion stabilized mixtures, for road pavements base layers, does not impair the mechanical response of the mixes itself. Both the physical and toxicological properties of each marginal material have been investigated. The subsequent mechanical characterization of the bitumen emulsion stabilized mixtures, prepared combining the marginal aggregates in different ratios, has been carried out in terms of indirect tensile strength, indirect tensile stiffness modulus and repeated load axial tests.

2. Materials
In this study, various marginal materials, from different industrial plants located in North-Eastern Italy, have been used for the aggregate structure of the bitumen emulsion stabilized mixtures:

1. Reclaimed Asphalt Pavement, a relatively fine aggregate made of the asphalt concrete recovered in road pavement milling operations;
2. Electric arc furnace steel slag (pH 9.6), a by-products resulting from steel production processes in plants which make use of the EAF technology;
3. Coal fly ash (pH 11.2), a waste derived from of the combustion of coal in controlled environments for the production of electricity;
4. Powdered glass waste (pH 8.2), obtained by crushing, shredding and grinding civic waste, such as bottles and containers primarily used for food and beverage storage.

In addition to such by-products and wastes, the CEM II/B LL 32.5R Portland cement has been used as a hydraulically active filler to improve the mechanical behaviour of the bitumen emulsion stabilized mixtures. Specifically, a commercial over-stabilized bitumen emulsion (C60B10), with producer-tested properties, was chosen for all mixtures to allow optimal mixing of the high content of fine materials, primarily derived from coal ash.

2.1. Toxicological properties of waste materials
For the proper management and exploitation of marginal materials, their environmental analysis is required. Tables 1 and 2 show, respectively, the results of the industrial by-products (i.e., EAF, CF, GW) characterization, in terms of initial heavy metal concentration, and their leaching behaviour. Differently, the RAP should be qualified as special waste pursuant to Art. 184 C.3 of the Italian Legislative Decree 152/2006 but, since it has already been treated for recovery in an authorized plant, according to the indications of the Italian Ministerial Decree 69/2018, it ceases this qualification to regain that of “product”: for this reason, no further analyses of heavy metals concentration and leaching were made.

Analysing the results reported in Tables 1 and 2, it can be observed that an aggregate structure given by EAF, CF, GW presents high initial concentrations (> 20 mg/kg) of copper, cadmium, lead, zinc, chromium (total), nickel, selenium, arsenic, beryllium, antimony and thallium; however, the leaching data are below the legal thresholds set by the Italian Legislative Decree 152/2006. Therefore, the marginal aggregates considered in this study do not pose specific toxicological problems for the
environment, according to Italian Laws, and are suitable for use in road construction. In addition, the non-toxicity and non-harmfulness properties of the individual marginal materials extend to the bituminous emulsion bound mixtures, making it unnecessary to perform further toxicological tests on mixture specimens.

### Table 1. By-products’ initial heavy metals concentration (mg/kg).

| Element                        | EAF  | CF  | GW   |
|--------------------------------|------|-----|------|
| Copper (Cu)                    | 219.0| 298.8| 955.0|
| Cadmium (Cd)                   | < 1.0| 2.1 | < 0.5|
| Lead (Pb)                      | < 1.0| 53.4| 217.3|
| Zinc (Zn)                      | 165  | 212.9| 884.6|
| Chromium total (Cr)            | 4275.0| 288.6| 244  |
| Chromium Hexavalent (Cr)       | < 5.0| 1.7 | < 5  |
| Nickel (Ni)                    | 9.1  | 139.1| 12.9 |
| Mercury (Hg)                   | < 1.0| < 1.0| < 0.5|
| Selenium (Se)                  | 8.8  | 61.2| < 2.0|
| Arsenic (As)                   | < 5.0| 22.4| < 2.0|
| Beryllium (Be)                 | 0.6  | 4.4 | 0.8  |
| Antimony (Sb)                  | 33.5 | 5.4 | 1.9  |
| Thallium (Ti)                  | 31.1 | < 1.0| < 0.5|

### Table 2. Leaching test results.

| Element                        | EAF          | CF             | GW             | Legal Thresholds     |
|--------------------------------|--------------|----------------|----------------|----------------------|
| Copper (Cu)                    | < 0.001 mg/l| < 0.05 mg/l    | 0.043 mg/l     | < 0.05 mg/l          |
| Cadmium (Cd)                   | < 1.0 µg/l   | < 5.0 µg/l     | < 1.0 µg/l     | < 5 µg/l             |
| Lead (Pb)                      | < 5.0 µg/l   | < 50.0 µg/l    | < 5.0 µg/l     | < 50 µg/l            |
| Zinc (Zn)                      | < 0.001 mg/l| < 3.0 µg/l     | < 0.001 mg/l   | < 3.0 mg/l           |
| Chromium (Cr)                  | 8.0 µg/l     | < 50.0 µg/l    | < 1.0 µg/l     | < 50 µg/l            |
| Nickel (Ni)                    | < 3.0 µg/l   | < 10.0 µg/l    | < 3.0 µg/l     | < 10 µg/l            |
| Mercury (Hg)                   | < 1.0 µg/l   | < 1.0 µg/l     | < 1.0 µg/l     | < 1 µg/l             |
| Selenium (Se)                  | < 10.0 µg/l  | < 10.0 µg/l    | < 5.0 µg/l     | < 10 µg/l            |
| Arsenic (As)                   | < 5.0 µg/l   | < 50.0 µg/l    | < 5.0 µg/l     | < 50 µg/l            |
| Barium (Ba)                    | 0.94 mg/l    | < 1.0 mg/l     | 0.01 mg/l      | < 1 mg/l             |

2.2. Physical properties and mechanical characteristics of waste materials

Figure 1 shows the grading curves (EN 933-1 Standard) of the marginal aggregates. As it would be expected, the gradations of EAF and CF are very different, being that of steel slag much coarser, whereas RAP and GW present intermediate particle size distributions.

From a qualitative point of view, the 2, 0.4 and 0.075 mm sieve passing data (ASTM 10, 40 and 200 sieve passing respectively) and the Plasticity Index values (according to EN ISO 17892-12) of each by-product, which are shown in Table 3, can be used for H.R.B. AASHTO M 145-2003 classification. This method, usually used for conventional soils classification, can also be applied to establish an equivalence between the physical properties (e.g. permeability, water sensitivity, as well as particle size) of conventional and marginal aggregates [12], and thus to assess their suitability for use in road
construction. The analysis showed that EAF and RAP have characteristics representative of an A1-a soil type, GW of an A1-b soil, and CF of an A2-4 soil, and thus of soils with excellent rather than good physical properties.

![Grading curves of marginal materials.](image)

**Figure 1.** Grading curves of marginal materials.

Table 3 shows the results of the physical-mechanical characterization of the marginal materials, which must be compared with the minimum acceptance thresholds set by ANAS, the Italian National Roads Authority: it establishes a minimum Los Angeles Coefficient of 25% and a minimum Equivalent in Sand of 50%. The Los Angeles test, performed on the coarsest marginal material, has verified the good and poor resistance to abrasion and friction of RAP and EAF slags, respectively. All the marginal aggregates exhibited a high Equivalent in Sand, well above the ANAS minimum cleaning level. As expected, EAF slags had the highest value of grain bulk (and dry) density while CF had the lowest.

**Table 3.** Physical-mechanical characterization of marginal materials.

| Property                        | RAP  | EAF  | CF   | GW   |
|---------------------------------|------|------|------|------|
| Los Angeles Coefficient (%) EN 1097-2 | 34   | 24   | -    | -    |
| Equivalent in Sand (%) EN 933-8 | 92   | 77   | -    | 89   |
| Grain Bulk Density (g/cm3) CNR 64/78 | 2.57 | 3.53 | 2.20 | 2.63 |
| Grain Dry Density (g/cm3) CNR 63/78 | 2.49 | 3.41 | 2.07 | 2.56 |
| Plasticity Index (-) EN ISO/TS 17892-12 | 0    | 0    | 0    | 0    |
| ASTM 10 sieve passing (%)      | 40.5 | 20.0 | 100.0| 70.4 |
| ASTM 40 sieve passing (%)      | 11.0 | 6.5  | 99.5 | 20.6 |
| ASTM 200 sieve passing (%)     | 0.9  | 0.9  | 26.2 | 3.2  |

3. Bitumen emulsion stabilized mixtures

3.1. Composition and gradation

Five mixtures, whose aggregate structure is composed entirely of the civil and industrial by-products under investigation, were prepared for this study by mixing the marginal aggregates with their original particle size distributions. The 15% and the 5% of the mixtures' lithic skeleton is made of GW and CF, respectively, whereas the remaining 80% consists of RAP and EAF according to five different complementary proportions, in which the 20% of steel slags is replaced at each composition by a 20% of RAP aggregate (Table 4). This approach would account for the possibility of temporary reduced...
availability of RAP or EAF, i.e., the two main marginal aggregates, in the mix production plant during the construction work.

### Table 4. Mixtures’ composition.

| Aggregate type | Mix 1 | Mix 2 | Mix 3 | Mix 4 | Mix 5 |
|----------------|-------|-------|-------|-------|-------|
| RAP            | 0     | 20    | 40    | 60    | 80    |
| EAF            | 80    | 60    | 40    | 20    | 0     |
| CF             | 5     | 5     | 5     | 5     | 5     |
| GW             | 15    | 15    | 15    | 15    | 15    |

The design grading curves, resulting from the mixing of marginal aggregates by the proportions in Table 3, were evaluated by comparison with the "ideal" and "less suitable" envelopes proposed by the Asphalt Academy (Figure 2), for bitumen emulsion stabilized mixtures [21]. Specifically, all mixtures were resulted suitable for use in road construction, although Mix 3, Mix 4, and Mix 5 fall within the less suitable (but still acceptable) envelope (Figure 2).

![Grading curves of mixes.](image)

**Figure 2. Grading curves of mixes.**

### 3.2. Mixes Design Procedure

The optimization of bitumen emulsion stabilized mixes, i.e., the identification of the optimal binder content, was performed by drawing inspiration from the Level 1 Mix Design discussed by the Asphalt Academy [21], but preparing 150 mm specimens compacted at 180 gyrations, according to the EN 12697-31 Standard. The Asphalt Academy procedure involves performing Indirect Tensile Strength tests (ITS test, EN 12697-23) on specimens cured for 72 hours at 40°C (curing condition are set by the Asphalt Academy and aimed at reaching a constant mass mix) and after immersion in water at 25°C for 24 hours to determine the parameters $\text{ITS}_{\text{dry}}$ and $\text{ITS}_{\text{wet}}$, respectively. The ratio (expressed in percentage terms) between these mechanical characteristics returns the Tensile Strength Retained (TSR), i.e., a measure of the mix’s susceptibility to moisture content, which is used along with the $\text{ITS}_{\text{dry}}$ to determine the optimum binder content. In fact, the ANAS specifications set the minimum acceptance threshold at 0.32 MPa for $\text{ITS}_{\text{dry}}$, while the relevant literature [21-23] suggests values higher than 50% for the TSR parameter: the minimum binder content meeting these requirements represents the optimum condition.
For each mixture presented in Table 4, 15 specimens were prepared in the laboratory with the same aggregate structure, water content (6% by weight of aggregates) and percentage of active filler (2% by weight of aggregates) but different bitumen content, varying from 3.0 to 5.0% by weight of aggregates in 0.5% increments (i.e., 3 specimens for each bitumen content and aggregate structure). The active filler, which in this study is the CEM II/B LL 32.5R Portland cement, helps to reduce the moisture content of the mixture improving the resistance to permanent deformations, but its addition has to be moderate to avoid brittleness [24]: as suggested in the scientific literature [13], the percentage of added cement (constant and equal to 2%) was kept lower to the bitumen content for each marginal aggregate combination [21].

Table 5. Indirect tensile strength test results.

| Parameter   | Mix 1 | Mix 2 | Mix 3 | Mix 4 | Mix 5 |
|-------------|-------|-------|-------|-------|-------|
| ITS\textsubscript{dry} (MPa) | 0.43  | 0.46  | 0.52  | 0.47  | 0.45  |
| ITS\textsubscript{wet} (MPa)  | 0.22  | 0.24  | 0.28  | 0.27  | 0.26  |
| TSR (%)     | 52    | 52    | 54    | 58    | 58    |

The minimum acceptance requirements on ITS\textsubscript{dry} and TSR were met for all mixes characterized by a bitumen emulsion content of 4% by weight of aggregates. Table 5 shows the results, averaged over specimen replicas, of the mix design process. In particular, the Mix 3, characterized by an equal percentage content of EAF slags and RAP (both at 40%, Table 4), had the highest ITS values, both in dry and soaked conditions, and a TSR value in-between that of the other mixes.

4. Performance characterization results

To assess the mechanical behavior under repeated load cycles and the permanent deformation resistance of the optimized mixtures, two additional tests were performed on 150 mm cylindrical specimens (EN 12697-31) as those prepared for the mix design process: the Indirect Tensile Stiffness Modulus test (ITSM test, Annex C, EN 12697-26) at temperatures of 10, 25, 40°C and the Repeated Load Axial Test (RLAT) with no confinement. The latter test involves the application of 1800 load pulses of 100 kPa with a loading/unloading time of 1s at a temperature defined as a function of the pavement layer considered and the climatic characteristics of the area under investigation (it was set at 30°C, because the research topic was focused on the study of road pavement base courses in North-East Italy). Figures 3 and 4 show, respectively, the stiffness modulus (MPa) and the axial deformation (microstrain) at the end of RLAT for each optimized mixture.

Figure 3. ITSM test results at different temperatures for each mixture.
Figure 4. Permanent deformation value at the end of RLAT for each mixture.

The analysis of Figure 3 reveals that all the mixes stabilized with bitumen emulsion at 4% by weight of the aggregate exhibit a reduction in stiffness as the test temperature increases reflecting the viscoelastic characteristic of the binder used. In particular, Mix 3 maintains the first-rank position also in terms of stiffness modulus (whatever the test temperature) and resistance to permanent deformation over all the other mixes: the ITSM value for Mix 3 varies from 4357 MPa (10°C) to 3348 MPa (40°C) as the temperature increases, remaining always higher than the values of the other mixes; on the contrary, the permanent deformation accumulated at the end of the RLAT test is small for Mix 3 and increases as the relative content of EAF steel slag and RAP varies. Differently, Mix 1 exhibits the highest percentage variation (i.e., -42.2%) in stiffness modulus passing from 10°C to 40°C (Figure 3), and consequently the highest axial creep strain among all other mixes (7582 microstrain, Figure 4).

5. Conclusions
In this paper, it has been verified via chemical, physical and mechanical analysis that civil and industrial waste materials, namely reclaimed asphalt pavement, electric arc furnace steel slag, coal fly ash, and glass waste, can be successfully reused to completely replace conventional stone aggregates in bitumen emulsion stabilized mixes for road pavement base courses. This outcome represents a further step towards the pursuit of cost-effectiveness and environmental sustainability of transportation infrastructure.

The Mix Design process based on the ITS test identified in the contents of bitumen emulsion at 4% and cement (as active filler) at 2% by weight of aggregate the optimal condition meeting the main mechanical acceptance requirements set by the Italian National Roads Authority (ANAS), whatever the marginal aggregate composition. However, the mixture characterized by an equal percentage content of EAF steel slag and RAP (both at 40%), along with CF and GW values set at 5% and 15% respectively, not only exhibited an excellent dry indirect tensile strength (ITS$_{\text{dry}}$ = 0.52 MPa > 0.32 MPa) and a satisfactory moisture resistance (TSR = 52% > 50%), but showed the best performance in terms of stiffness modulus (4357 - 3348 MPa passing from 10°C to 40°C) and permanent deformation resistance (5282 microstrain) compared to all other mixes. Based on the exposed results, this mixture is considered the most suitable for use as a base course in road pavements of Northern Italy.

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