ABSTRACT: Long term performance of secondary materials is becoming a challenging aspect in road construction since due to their benefits they are being used on a large scale, but on the other hand their future behaviors are difficult to estimate. In this study, aging is proposed as a means of exploring the long-term mechanical and physical performance of secondary materials. A Blast Furnace Slag (BFS) mixture which is routinely used in the Netherlands in road (sub-) base construction was selected as a reference material. The A32 motorway in the Netherlands was used as a source of field aged granulated BFS materials. The base layer of this motorway, suddenly experienced serious failure. In order to estimate future behavior of secondary materials and to prevent similar problems to occur an aging protocol was suggested to detect at an early stage potential poor material performance. Two types of aging approaches were chosen and applied to the field aged and fresh materials being steam aging and cyclic freezing and thawing. Both aging treatments have affected mechanical and chemical characteristics. The study of response variables showed there is a linkage between compressive strength, expansion, micro cracking and amount and type of binder.

Keywords: Blast Furnace Slag, aging, secondary materials, road base.
1. Introduction

The use of less traditional secondary materials in a road construction frequently requires the assessment of physical and mechanical performance to ensure durability and safety. Long term performance, especially the influence of the binding material under field conditions, is often difficult to predict. Therefore, there is a need to develop guidelines to estimate the long-term performance, particularly approaches that involve set of laboratory procedures, which can be done in relatively short period of time are relevant. This evaluation method can be helpful for practitioners and decision makers. Accelerated aging is one of the potential means of doing so for the application of secondary materials in a road (sub-) base construction.

BFS materials which is one of the major by-products of steel plants was chosen as a reference material. BFS materials can be considered as a rather heterogeneous materials with complex features. It essentially consists of glass with crystalline silicates and aluminosilicates of calcium. Rapid chilling of the slags results in preponderance of a glassy (amorphous) phase in the slag. The main reason behind selection of BFS material as reference material was this fact that BFS base courses are successfully used in the Netherlands for quite some years, but about 4 years ago unexpected poor behavior suddenly occurred on a particular highway which were potentially related to collapse of the BFS base course. Unexpected behavior was observed on a 10 km long stretch of the motorway A32 (constructed in 1986-1988). There, a mixture of slags was used as road base material. Some 10 years after construction of the pavement structure, the first transversal heaves occurred at the pavement surface causing unacceptable pavement roughness. Although these initial heaves were milled off and the riding quality was improved, such heaves continued to develop causing regular maintenance operations. Then, in 2008 and 2009 the pavement had to be fully reconstructed over a length of nearly 10 km because of sudden collapse of the BFS base course. Numerous failure mechanisms have been hypothesized, including chemical reaction, thermal deformation, increased stresses due to obstructed deformation. Additionally since the performance of material was very similar to concrete other failure mechanisms including, freeze-thaw damage and the appearance of ettringite \([\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12}\cdot26\text{H}_2\text{O}]\) in air voids were proposed.

Two types of accelerated aging methods were chosen for this project: steam aging or heat aging and freeze-thaw (FT) action. Steam aging was intended to accelerate chemical reactions leading to development and maturation of the BFS mixture and to accelerate possibly deleterious reactions between components, whereas FT was intended to rapidly stress the compacted material in manners similar to those experienced in the field. This approach allowed us to explore the effect of freezing temperature and time. Steam aging was done on laboratory cylindrical samples of mixtures of fresh materials and about 20 years old A32 granular materials. The FT investigations were done on the sealed cylindrical samples of mixtures of fresh materials compacted with optimum moisture content. During this experimental work different mixture components and proportions of BFS materials were studied.

2. Materials and Methods

2.1. Material

The main purpose of this experimental work was to study the potential effects of the composition of secondary materials on the long-term performance of the mixture. In this research, in order to ensure that within this study a range of potential failure mechanisms would be covered, fresh material consisting of air-cooled blast furnace slag, steel slag and granulated blast furnace slag (GBFS) sand was obtained from a well-known producer. The
selected materials were clean and free from detrimental levels of chemical impurities and harmful constituents. Also actual field aged material was needed and based on previous research (1), it was decided to collect actual field samples from the base layer of the A32 motorway near Wolvega, in the province of Friesland in the north of the Netherlands. As mentioned above a serious failure happened in the A32 motorway and this motorway experienced numerous heaves formation and finally complete failure. The role of BFS material, if any, in this failure was unknown. Thus it was arranged (with the Ministry of Transport, Public Works and Water Management) to collect base materials at different locations of the A32. Several slabs (maximum size was ~1×1.5 m) and about 50 kg loose granular materials were collected from the Eastern carriageway during its complete reconstruction. In general, the field aged BFS was found to be coarse, porous and rough. The particle size distribution curves of the A32 and the fresh materials are presented in Figure 1.

In order to obtain a homogeneous and representative sample of the aggregate population, a quartering method was used as suggested by Goodsall and Mathews (1970) (2). In this study, the samples of 50 kg of each type of aggregate (fresh and crushed field aged) were reduced to 5 kg samples using this method.

![Figure 1: Grading curves of the materials used in this study](image)

The physical properties of materials are given in Table 1. The apparent relative density values are determined after 3 trials with a gas pycnometer device. The tests are done according to the ASTM D5550 procedure (3). The water absorption of the aggregates was determined based on the principle of water saturation and in accordance to EN1097-6 (4).

<table>
<thead>
<tr>
<th></th>
<th>Apparent relative density ($10^3$ kg/m$^3$)</th>
<th>Water absorption(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Air cooled BFS</td>
<td>2.654</td>
<td>2.985</td>
</tr>
<tr>
<td>Steel slag</td>
<td>2.899</td>
<td>4.137</td>
</tr>
<tr>
<td>GBFS sand</td>
<td>2.901</td>
<td>4.867</td>
</tr>
<tr>
<td>A32 material (at chainage 33.38 km)</td>
<td>2.827</td>
<td>3.824</td>
</tr>
</tbody>
</table>

2.2. Mixture components
Fresh (un-hydrated) GBFS sand was selected as a material with binding properties and the other original materials that were used for making samples were air-cooled BFS and steel slag.
These three types of slags were mixed according to the Fuller curve using a power of 0.45 with a maximum grain size of 22.4 mm. Table 2 shows the relative proportions of the individual slags in the mixtures for fresh materials.

Loose granular materials which were collected from the A32 were sieved and they were mixed according to the Fuller curve using a power of 0.45 with a maximum grain size of 22.4 mm.

### Table 2: Proposed slag mixture for fresh materials

<table>
<thead>
<tr>
<th></th>
<th>Content (% by mass)</th>
</tr>
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<tbody>
<tr>
<td>Air-cooled BFS</td>
<td>91 86 81</td>
</tr>
<tr>
<td>Granulated BFS</td>
<td>0 5 10</td>
</tr>
<tr>
<td>Sand</td>
<td></td>
</tr>
<tr>
<td>Steel Slag</td>
<td>9 9 9</td>
</tr>
</tbody>
</table>

#### 2.3. Experimental work

This experiment was designed to obtain a better understanding of the long term performance of secondary materials with self-cementing properties which affects the mechanical and physical response of laboratory samples. In this experimental plan the simultaneous effect of three aging variables being temperature, moisture and time was taken into account. By using various levels of aging for each factor the influence of different aging conditions on the performance could be examined.

In the first treatment the samples were subjected to steam (at atmospheric pressure) and temperature. Another treatment was Freezing and Thawing (FT) in which materials were exposed to different number of FT cycles. Additionally several specimens were kept for different period of times in a controlled conditions (a fog room with ~ 20°C, 95% RH ± 5%).

The goal of these treatments was to induce degradation due to aging to the stabilized mixtures within a relatively short period of time scale (in the order of weeks versus years) in a manner that approximated typical aging of (sub-)base materials in a natural environment.

Steam aging was applied to allow for the development of a microstructure and then for potentially deleterious reactions due to the applied aging scenario. FT was applied to potentially produce internal stresses due to freezing and thawing and, as a result of that, some material disintegration and micro-cracking. Furthermore, the effects of all aging scenarios on the physical and mechanical performance were studied.

#### 2.3.1. Steam aging

The main purpose of the steam aging tests was to investigate the effects of aging of materials on the compressive strength and to measure amount of displacement that may happen during this aging. Cylindrical specimens with a diameter of 210 mm were made. After compaction, the specimens covered with damp cotton cloth were cured for 1 day in air at ~20°C and then were tested. The height of the compacted specimens was 100 mm. The compressive strength of specimens was measured immediately after it was aged under the different conditions.

The principle of the steam test is not complicated. The steam aging system is composed of two or more chambers, in the lower one water is heated up to its boiling point for the duration of the test. As shown in Figure 2 a compacted specimen is placed on a perforated base above the steam generating unit. The specimens have a grain size distribution between 0-22.4 mm. The specimen is subjected to a flow of steam at ambient pressure. In this way, the necessary moisture and temperature for the potential hydration reaction is continuously conveyed to the test sample.

It is important that the steam can evenly flow through the specimen. In order to prevent condensation building up on the inside of the cylinder due to the heat loss, the cylinder itself
can be heated up by a circular heating jacket fitted to the outside wall. Meanwhile the temperature of the sample is measured by a thermocouple with 100 mm length placed inside a hole located in the center and 20 mm depth from the upper surface of the sample. In this study, samples were preheated for 5 hr. at 65°C and this temperature was kept constant during the steam treatment.

Figure 2: View of the steam test equipment and its principle. The set-up may consist of more containers

Any change in the volume of sample caused by any type of chemical reaction is read off from two displacement gauges placed at the top of the specimen. The increase or decrease in volume can be measured as the result, calculated in % volume in relation to the original volume of the compacted specimen. After aging, the samples were demoulded and then the compressive strength was determined.

The steam test is accepted by European countries and has been incorporated into European aggregate standards as a test method for steel slags. It is part of EN 1744-1 "Tests for chemical properties of aggregates – chemical analysis" (5).

Additionally in order to compare the effect of steam aging conditions with normal aging conditions, a second group of test specimens were made. For this purpose, cylindrical samples with a diameter of 210 mm and a length of 100 mm were produced following the same mixture composition and grain size distribution used for steam test specimens. All specimens in this experimental work were compacted with an electrical vibrating hammer to reach a certain level of compaction. Reference cylindrical samples were also made for compressive strength testing. Samples were cured in a fog room for 90 days at 95% RH ± 5% relative humidity and about 20°C. Consequently, the samples were prepared for compressive testing.

2.3.2. Freezing and thawing action
The porous structure of BFS has raised questions about the performance of this type of material under FT action.
To investigate the effect of freezing and thawing, cylindrical samples of fresh material having a diameter of 100 mm and a height of 180 mm were produced. The specimens were prepared according the method proposed for making triaxial samples (Van Niekerk 2002) (6). Metal base and top plates were used at each end of the sample for the purpose of sealing (keeping optimum moisture content constant which is about 7.5%).
The specimens were cured for 7 days before testing. The samples were preheated for 5 hours to ensure that the whole sample (including its core) has a temperature of 30°C (starting air temperature).
During the freeze-thaw cycle the specimens were wrapped in two Polychloroprene membranes (high resistance to temperature variation) in order to be fully sure to keep the
moisture content stayed constant during the cycles. All FT samples were made at optimum moisture content.

The freezing and thawing procedure mainly followed RILEM TC 176 recommendations (7), where each test specimen was cooled from 30 to -10 °C in air with a cooling and warming rate of 4.0 °C/h (rate is meant the temperature change of air in the climate chamber). The cooling and warming procedure was repeated for 4, 8, 12 and 16 cycles. The number of FT cycles was selected based on the K. Fridh (2005) recommendations (8) and it was limited to a maximum of 16 cycles.

Reference samples were also made in order to be compared with the other samples which were to undergo FT cycles. During the test the axial displacement of the top surface was measured by three length measuring gauges, which were calibrated by cooling and warming a steel cylinder having a known coefficient of temperature expansion. As soon as the FT exposure was terminated, the compressive strength of specimen was measured.

On the similar specimens which have experienced the FT cycles, sampling was done by cutting slices from the middle portion of the specimen for optical microscopic studies. The observation area was arranged to be parallel to the side surface (perpendicular to the casting surface) with a depth of ~ 5 mm. Two plane sections with dimensions of 20×40×15 mm were prepared for each sample in accordance with the procedure developed by Gran (1995) (9). The optical microscopy investigation was performed mainly according to ASTM-C856 (10). The microscopic analysis was performed by means of PLM on standard petrographic thin sections prepared from different specimens.

3. Results and discussion

The results of the physical and mechanical performance of tests and also the microscopy test results are discussed in the following sections.

3.1. Influence of steam aging on compressive strength

Figure 3 shows the influence of steam aging on compressive strength of different mixtures at different ages. It can be seen that with increasing amount of GBFS sand content, the average compressive strength (two test repetitions) increased and further it looks the compressive strength decreases at longer exposure times, possibly due to expansion (Figure 3).

The effect of two different aging conditions on the compressive strength of specimens aged with the steam test at 14 days and specimens aged under normal condition (95% RH ± 5% and ~20°C) at 90 days is presented in Figure 4. It seems that the steam test is able to activate the material and improve the compressive strength. It can be seen that during normal aging the samples containing 5% GBFS sand did not show considerable changes in the compressive strength after 90 days aging while the same samples during steam aging show first an improvement and then a decrease in compressive strength (Figure 4).

The results of the deformation measurement (expansion) made during the steam aging tests are presented in Figure 5. As shown in Figure 5, during steam aging depending on the mixture composition materials may expand. This phenomenon may be due to the fact that at the high applied temperature (65°C) and with sufficient moisture available, materials hydrate rapidly. The expansion of the A32 material which is already hydrated (field aged materials collected after 20 years) is considerably lower than the fresh materials.
Figure 3: The influence of steam aging on compressive strength of different mixtures after 1, 7 and 14 days

Figure 4: Comparison between development in compressive during two aging regimes. Similar samples with 5% GBFS sand

Figure 5: Measured deformations during steam aging for different mixtures
The expansion which was happened in some of the mixture ingredients can lead to microcracking and consequently a decrease in compressive strength. The test results showed that during steam aging if the samples swell more than 0.2 % their compressive strength may decrease at the later ages.

The effectiveness of steam aging vs. normal aging for different ages is shown in Figure 4. This figure, proves that, if a system has cementitious properties and the hydration reaction may happen after a long period of time, steam aging may be of interest to study physical and mechanical performance within short period of time. The results show that application of steam aging can give an indication about performance of material especially in the case of slag materials which are hydraulically active and there is chance of expansion for this type of materials. Therefore, steam aging test can be beneficial in order to firstly realize if a certain type of material may swell and secondly how they will perform if a swelling reaction happens in a mixture.

3.2. The effect of GBFS sand content on expansion

The general chemical compositions of GBFS sand particles obtained with XRF tests are given in Table 3. GBFS sand can be considered as a material with binding properties and this may have a positive effect on the mechanical performance of mixtures. However, in this study, application of GBFS sand especially in percentages 5 and 10% caused an expansion in the steam test and consequently a reduction of compressive strength (Figures 3 and 5). Figure 5 shows that the slope of the curves depend on the mixture composition and mainly on the GBFS sand content. The mixture contains hydratable oxides (CaO) that may result in volumetric instability (expansion).

<table>
<thead>
<tr>
<th>Table 3: Chemical compositions of GBFS sand particles</th>
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<tr>
<td>SiO$_2$</td>
</tr>
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<td>Content (wt %)</td>
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</table>

3.3. Freezing and thawing test results

As previously mentioned, the specimens were subjected to different number of freeze-thaw cycles. They were chosen such to be able to analyze the effects of the composition on the behavior of the material under FT cycles and to study the effects on the microscopic structure.

Figure 6 shows the compressive strength test results as well as amount of its change as a function of number of FT cycles in relation to the composition of the mixtures. This figure was prepared based on the reference compressive strength of each mixture after 28 days curing. The reference compressive strength is the compressive strength of the samples before the FT test started.

A brief look at the results in Figure 6 shows that the FT cycles cause a significant amount of damage. Especially with the samples without GBFS sand show a large decrease in strength and almost behave like unbound material after 12 cycles. The specimens with 5 or 10 % GBFS sand were clearly less affected by FT cycles although not much strength is left after 16 FT cycles.

One can also observe that the samples which made of 5 or 10 % GBFS sand they performed more or less the same.

Figure 7 shows a typical series of FT cycles and the strain results. A length change curve for a specimen can be divided in 6 different parts; see Figure 7.

Part 1: Thermal contraction before nucleation (formation of solid crystals) of extremely cooled water.
Part 2: Rapid expansion caused by nucleation of extremely cooled water.
Part 3: Expansion caused by ice formation.
Part 4: Contraction before all ice is melted.
Part 5: Thermal contraction (caused by melting of ice).
Part 6: Thermal expansion of the thawed specimen.

**Figure 6:** Results from different mixtures which were exposed up to 16 FT cycles.

**Figure 7:** Different parts in a length change curve after 4 FT cycles

Three types of expansion were observed during a cycle:

A. Permanent expansion.
B. Rapid expansion when cooling terminates at some degrees below 0°C, whereby a considerable amount of ice is formed almost instantaneously.
C. Expansion at -10°C.
Permanent expansion indicates substantial internal micro-cracking and loss of cohesion of the specimen. Rapid expansion when cooling terminates indicates the occurrence of hydraulic pressure as a consequence of big and rapid ice formation and expansion of the frozen specimen at -10°C indicates that internal pressure is built up as a consequence of ice formation.

As mentioned earlier the microscopic analysis was also carried out to investigate the microstructure degradation of the material caused by FT action. In this part of the study, the resin impregnation technique in combination with the optical fluorescence microscopy and a computer image analysis technique were used to provide qualitative and quantitative determination of the crack system. The observation was carried out by means of an optical microscope at a magnification of 10× in order to detect fine cracks. On the generated photos a quantitative analysis was performed.

Two levels of damage were observed after the FT test. At the macroscopic level, large cracks were identified on the surface of the samples. In some cases, especially after 12 and 16 cycles, the samples were seriously damaged. Most of test cylinders had a crack pattern in which cracks were perpendicular to the axis and sometimes in the axial direction of the cylinder (Figure 8). The range of cracking varied across mixture types and number of cycles, for instance at 8 cycles a sample with 0% GBFS sand exhibited serious surface cracking and perpendicular cracks to the axis (Figure 8).

![Figure 8: Effect of FT action on a sample with 0 % GBFS sand](image)

On the microstructural level, the cracks were mainly observed in the samples that were subjected to more than 4 cycles. The crack formations were classified with the cracks density, $D_{cr}$, which was estimated by a point counting method. In this method, a two-dimensional grid was used and each crack which falls under a grid point was counted. $D_{cr}$ is defined as follows:

$$D_{cr} = \frac{\text{Area of cracks}}{\text{Total area}} \times 100\%$$

As it is shown in Figure 9, the higher number of FT cycles, the higher the crack density was. Figure 9 gives the crack characteristics of the samples sawn from the 100 mm diameter specimens subjected to the different number of FT cycles.

Optical microscopy test results show that in general two types of cracks occurred in the BFS mixture:

a) cohesive cracking, in which the texture of the mixture is disintegrated into several small internal cracks,

b) adhesive cracking in which loss of bond between the coarse slag particles and the paste took place and peripheral cracks formed around the slag particles. The distance between the cracks generally varies according to the size of the coarse aggregate which the cracks pass.
Figure 9: Estimation of crack density by means of the point counting method

As can be seen in Figure 10 it appeared that the cracks were mainly propagating through the paste (hydrated BFS aggregate) and around the boundary of slag aggregates. Figure 11 also shows a BFS mixture which has been exposed to the 12 FT cycles. Cracks are mainly parallel to the upper and lower surface of sample. Cracking here again happened through the paste matrix and in the interfaces between the coarse aggregate particles and the paste matrix.

The overall results suggest that the FT tests allow to recognize materials with a poor performance.

Figure 10: Cracks are present around coarse slag particles and through the paste (arrows show the cracks) [↔ L=2.87 mm].

Figure 11: Cracks pattern of BFS sample after 12 FT cycles made with 5% GBFS sand. The location of crack is specified [↔ L=2.87 mm].
4. Conclusions

Accelerated aging procedures were applied to study the interactive effects of temperature, moisture and time. The aging procedures had an impact on both the physical and mechanical properties of the tested specimens. Measurements done on laboratory and field aged samples, suggested that both aging methods (steam aging and FT action) did a reasonable job of producing distress phenomena. All response variables such as strength, displacement and microstructure show that there is a linkage between the performance of secondary materials and moisture, temperature and time. The results hold promise that a mixture with a poor performance can be detected by the aging protocol (e.g. from expansion and/or loss of integrity) even though a certain complex cementitious reaction as mineralization occur within the matrix of mixture. Further, the accelerated aging procedures can be used as a screening tool which shows that a certain mixture properties (e.g. GBFS sand content) can play a role in affecting future performance of the material and pavement layers made of it.

5. References