

Development of an evaluation protocol to estimate the long-term performance of secondary materials in road construction

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Abstract: Future performance of secondary materials in road construction is often difficult to estimate. In this study, aging is proposed as a means of exploring the long-term mechanical and physical performance of secondary materials. Blast Furnace Slag (BFS) material, routinely used in the Netherlands as a cementitious material was selected as a reference material in this aging approach. A motorway in the Netherlands (the A32 motorway) was used as a source of field aged BFS materials. The base layer of this motorway, suddenly experienced serious failures. Three types of aging treatments were chosen and applied to the cylinders made with the fresh materials and A32 field aged materials. These aging treatments are being normal aging, steam aging and freezing-thawing cycles. The aging methods affected the mechanical and chemical performance of material. Response variables measured in the laboratory and in the field were mostly similar and the acquired results from the laboratory aging methods suggesting that the aging methods produced a material of similar distress. Steam aging produced loss of strength which indicated that steam aging can contribute to the occurrence of deleterious reactions. Microscopic techniques were used to analyze this reaction. The study of response variables showed there is a linkage between compressive strength, expansion, micro cracking and amount and type of binder.

KEY WORDS: Evaluation protocol, slag, road base.

1 INTRODUCTION

Long term performance of such materials and pavement layers made of them and especially the influence of the binding material under field conditions, are often difficult to predict. Therefore, there is a need to develop guidelines to estimate the long-term performance. Particularly approaches that involve a set of laboratory tests which can be done in a relatively short period of time, say 1 week, are relevant.

Such an evaluation method will be helpful for practitioners and decision makers in determining the suitability of a secondary material to be used in (sub-)base courses in terms of sustainability. Accelerated aging is one of the potential means of doing so.

Slag materials consisting of Air-Cooled Blast Furnace Slag (ACBFS), steel slag and Granulated Blast Furnace Slag (GBFS) sand which are one of the major by-products of steel plants were chosen as study materials. A slag system can be considered as rather heterogeneous material 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 (1). In studying the slag system as a self-cementing material, the gain of strength was attributed to the hydration reaction which mainly happens at later ages (2).

From earlier research work it has been accepted that the hydraulic activity of ground GBFS slag materials is influenced by their properties such as chemical composition, mineralogical composition and fineness (2). Furthermore, slag materials contain less lime than Portland cement clinker which results in lower strength gain rate when compared to Portland cement (3).

The main reason behind the selection of slag material as study material was the fact that slag base courses are successfully used in the Netherlands for quite some years. About 4 years ago, however, unexpected poor structural pavement behavior occurred on a 10 km long stretch of the motorway A32 (constructed in 1986-1988) which was potentially related to collapse of the slag base course.

The original structure of the A32 motorway pavement structure consisted of 190 mm asphalt (dense asphalt concrete), 200 mm mixture of slags (GBFS sand, steel slag and ACBFS) as a base and a sand sub-base (varying in thickness) on the natural clay subgrade (5).

Some 10 years after construction of the pavement structure the first transversal heaves occurred at the pavement surface causing unacceptable pavement roughness. Then, in 2008 and 2009 the pavement had to be fully reconstructed over a length of nearly 10 km because of sudden collapse that could be partially attributed to the slag base course.

Numerous failure mechanisms have been hypothesized, including chemical reaction and increased stresses due to obstructed deformations. Additionally, since the performance of the material was very similar to concrete, other physical failure mechanisms including freeze-thaw damage was proposed.

In order to avoid similar problems as on the motorway A32 and to estimate the long term performance of self-cementing materials, three types of aging methods were chosen: freeze-thaw (FT) action, steam aging and normal aging. FT action was intended to rapidly impose thermal stress on the compacted materials in manners similar to those experienced in the field, whereas steam aging was intended to accelerate chemical reactions leading to development and maturation of the slag mixture and meanwhile to accelerate possibly deleterious reactions between components. Normal aging treatment is similar to a natural process that all (sub-)base materials may experience over their service life. With these approaches it was possible to explore the effect of temperature and moisture and time.

Aging treatments were done independently on different specimens. During the FT experimental besides slag mixtures, a loamy sand mixture stabilized with 65 kg/m^3 cement (type III/B) as a control material was also studied.

2 MATERIALS AND METHODS

2.1 Materials

In order to ensure that within this study a range of potential failure mechanisms would be covered, fresh material consisting of ACBFS, steel slag and GBFS sand was obtained from a well-known producer.

Also actual field aged material was needed and based on previous research (5) it was decided to collect actual field samples from the base layer of the A32 motorway in the north of the Netherlands. As mentioned above, the A32 motorway experienced numerous heaves formation and finally complete failure. The role of slag materials in this failure was not fully

clear. Thus it was arranged (with the Ministry of Transport) to collect base materials from different locations of the A32.

2.2 Freezing and Thawing (FT) Action

The porous structure of slag material has raised questions about the performance of this type of material under freezing and thawing condition.

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. Three types of slags were mixed to obtain a Fuller curve gradation using a power of 0.45 with a maximum grain size of 22.4 mm. This mixture consisted of air-cooled BFS, steel slag and GBFS sand. Three different mixtures containing 0, 5 and 10% by mass GBFS sand were studied. In all mixture the steel slag content was 9% by mass. The FT investigations were done on the sealed specimens compacted at optimum moisture content (Standard proctor). During the FT cycles no extra water was provided.

After removing the mold and sealing the sample, metal base and top plates were used at each end of the sample for the purpose of secure sealing (keeping the optimum moisture content constant which is about 7.5%). Before the FT test, the sealed samples were stored for 28 days in a temperature controlled room ($22 \pm 2^\circ\text{C}$). During this period the weight of the samples was measured in order to control the quality of sealing and avoid moisture loss.

The samples were preheated for 5 hours to ensure that the whole sample (including its core) had a temperature of 30°C (starting air temperature). During the FT cycles the specimens were wrapped in two Polychloroprene membranes (high resistance to temperature variation) in order to ensure that the moisture content stayed constant during the cycles.

The freezing and thawing procedure was mainly done according to the RILEM TC 176 recommendations (6) and each specimen was cooled from 30 to -10°C in air with a cooling and warming rate of 4.0°C/h (rate means 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 Fagerlund (1997) (7) study and it was assumed that for this type of material a maximum of 16 FT cycles is sufficient to possibly cause cracking and damage. Furthermore, the mechanical and microstructural performance of materials to the repeated freezing and thawing cycles while the moisture content was constant were studied.

Reference samples were also made in order to be compared with the other samples which were to undergo FT cycles.

For optical microscopic studies of the specimens which have experienced the FT cycles and the reference samples, sampling was done by cutting slices from the middle portion of the specimen. The observation area was arranged to be parallel to the side surface (perpendicular to the compaction surface), with a depth of ~ 5 mm. Two plane sections with dimensions of $20 \times 40 \times 15$ mm were prepared for each sample in accordance with the procedure developed by Gran (1995) (8). The optical microscopy investigation was performed according to ASTM C856 (9). The microscopic analysis was performed by means of PLM (polarized light microscopy) on standard petrographic thin sections prepared from different specimens.

2.3 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 the deformation that may happen during aging.

Cylindrical specimens with a diameter of 210 mm were made. The height of the compacted specimens was 100 mm. All specimens in this experimental work were compacted with an electrical vibrating hammer to degree of compaction of 100% of the standard Proctor density

at the optimum moisture content. After compaction, the specimens covered with damp cotton cloth were cured for 1 day in air at ~20°C and were then tested. Steam aging was done on laboratory cylindrical samples of mixtures of fresh materials and about 20 years old A32 granular materials.

The compressive strength of the specimens was measured immediately after they were aged. After stripping mold the compressive strength was measured in accordance with ASTM C39 (10). Reference cylindrical samples were also prepared.

The principle of the steam test is not complicated. In principle the steam aging system is composed of two chambers, in the lower one water is heated up to its boiling point for the duration of the test. A compacted specimen is placed on a perforated base above the steam generating unit. The specimens have grain sizes 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 are 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 (mold) due to the heat loss, the cylinder itself was heated up by a circular heating jacket fitted to the outside wall. Meanwhile the temperature of the sample was measured by a thermocouple with 100 mm length placed inside a hole located in the center and 20 mm below the upper surface of the sample.

Any change in the volume of the sample caused by any type of chemical reaction was read off from two displacement gauges placed at the top of the specimen while the specimen was inside the mold. The increase or decrease in volume can be measured in % volume in relation to the original volume of the compacted specimen.

The steam test is accepted by European countries and has been incorporated into the 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" (11).

2.4 Chemical Analysis - Electron Microprobe Technique

The purpose of this part of the research is to analyse the chemical composition of the components of the material as well as to get an impression of the microstructure before and after steam aging. For this purpose the electron microprobe analysis (EPMA) technique was used.

In this study the measurements with the microprobe were made on flat, polished sections, immersed in resin. Per specimen, about 50 points were analysed. Quantitative analyses were carried out in the Wavelength Dispersive Mode, with LDE-1, TAP, LIF and PET analyzing crystals. Instrumental conditions were 15 kV accelerating voltage, 2.0×10^{-8} amps beam current and focused spot. The matrix correction method was PhiRhoZ-method and calibration was done in accordance with certified analytical standards.

2.5 Normal Aging

In order to compare the effect of steam aging conditions with similar field aging conditions, a third 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 the steam test specimens. Samples were stored in a fog room for 90 days at $95\% \pm 5\%$ relative humidity and about 20°C. Consequently, the samples were prepared for compressive testing.

3 RESULTS AND DISCUSSION

3.1 Freezing and Thawing Resistance

As previously mentioned, specimens were subjected to different numbers 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 of FT cycles on the microscopic structure.

Freezing and thawing resistance of the samples made with 0% GBFS sand (containing 9% steel slag and 91% rest Air-cooled BFS) was quite poor, although the amount of moisture was limited (optimum content). Figure 2 illustrates the loss of the static modulus of elasticity of the control sample (cement stabilized loamy sand) and the samples incorporating different GBFS sand content during freezing and thawing exposure.

The experimental results indicate that the static modulus of elasticity of the samples without GBFS sand, decreased more than 90% at 12 cycles. In some cases these specimens were divided into pieces. The specimens with 5 or 10 % GBFS sand were clearly less affected by FT cycles although not much stiffness is left after 16 FT cycles. It seems that if GBFS sand particles are not present in the mixture, serious durability loss under frost attack can be expected. On the other hand, the loamy sand samples stabilized with 65 kg/m^3 cement were remarkably frost resistant. The overall performance of slag materials containing more than 5% GBFS sand at the end of 16 freezing and thawing cycles was superior to that of the mixture without GBFS sand, although the slag mixtures have noticeably the same value for the volume of pores (the voids content was kept between 18 and 22%). It is obvious that even a limited amount of binder (GBFS sand) in the form of fine aggregates (5%) or cement (2.5%) is necessary to avoid a sharp decrease in freezing and thawing resistance of the secondary materials (Figure 1).

It can be concluded that GBFS sand in the BFS mixtures should not be completely excluded if the mixture will be exposed to freezing and thawing.

As another important result, in general the freezing and thawing resistance of BFS samples was poor and better freezing and thawing performance could be achieved by enhancing the matrix with GBFS sand. However, further studies show that a GBFS sand content more than 5% can be problematic by causing expansion. As Figure 1 shows, only the mixture containing more than 5% GBFS sand performed relatively well during FT cycles.

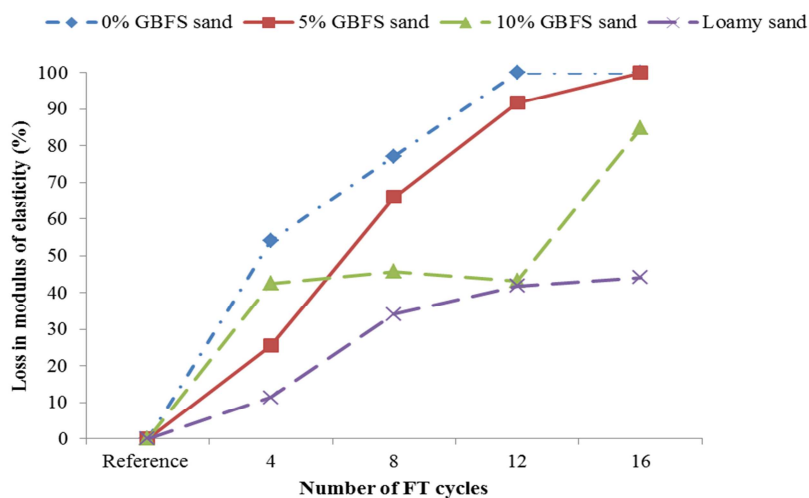


Figure 1: Results from different mixtures which were exposed to FT cycles.

The axial strain on the outer surface of the samples was measured by three LVDTs, which were calibrated by cooling and warming a steel cylinder having a known thermal coefficient of expansion. Three types of expansion were measured: a) Permanent expansion, b) Rapid expansion at some degrees below 0°C, whereby a considerable amount of ice is formed almost instantaneously and 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. The expansion of the frozen specimen at about -10°C indicates that internal pressure is built up as a consequence of ice formation.

The microscopic analysis was carried out to investigate the internal microstructure degradation of the material caused by FT action. In this part, 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 the test cylinders had a crack pattern in which cracks were perpendicular to the axis. The range of cracking varied across mixture types and number of FT cycles.

On the microstructural level, the cracks were mainly observed in the samples that were subjected to more than 8 cycles. The crack formations were classified with the cracks density, which was estimated by the point counting method (ASTM C457-Procedure A) (12). The results showed that the higher the number of FT cycles, the higher the crack density is.

Optical microscopy test results show that mainly adhesive cracking occurred in the slag mixtures 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.

As can be seen in Figure 2 it appeared that the cracks were mainly propagating through the paste (hydrated slag aggregate) and around the boundary of slag aggregates. Figure 3 right also shows a slag mixture which has been exposed to 12 FT cycles. 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.

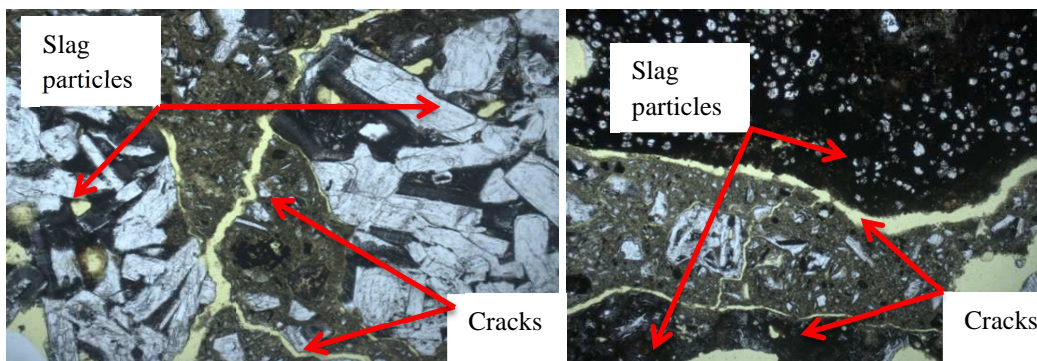


Figure 2: Cracks are present around coarse slag particles and through the paste. Both photos after 12 FT cycles on specimens made with 5% (left) and 10% (right) GBFS sand [\leftrightarrow L=2.87 mm].

3.2 Influence of Steam Aging on Compressive Strength

Figure 3 shows the influence of steam aging at 65°C on the compressive strength of different mixtures at different ages. It can be seen that with increasing GBFS sand content, the average compressive strength (two test repetitions) increased and furthermore the compressive strength decreases at longer exposure times, possibly due to expansion. It also appears that the 20 years old field aged A32 materials behaves more or less similar to the fresh materials with 0% GBFS sand. However, application of high temperature and moisture (steam) increased the compressive strength of all samples after 1-day aging, which demonstrates that it is possible to detect level of activity of materials after 1 day steam aging.

The effectiveness of steam aging vs. normal aging (standard curing) for different ages for all mixtures is shown in Figure 3. It can be seen that the steam-aged mixtures containing 10% GBFS sand showed on average 2.84 MPa compressive strength after 1 day while the normal aged mixture with the same composition exhibited a strength of only 0.89 MPa. This figure clearly proves that, with slag systems containing GBFS sand (or other binder), steam-curing may be of interest to study performance of materials.

Further test results indicated that during the steam aging the increase of the compressive strength is very well dependent on the existence of some active chemical components.

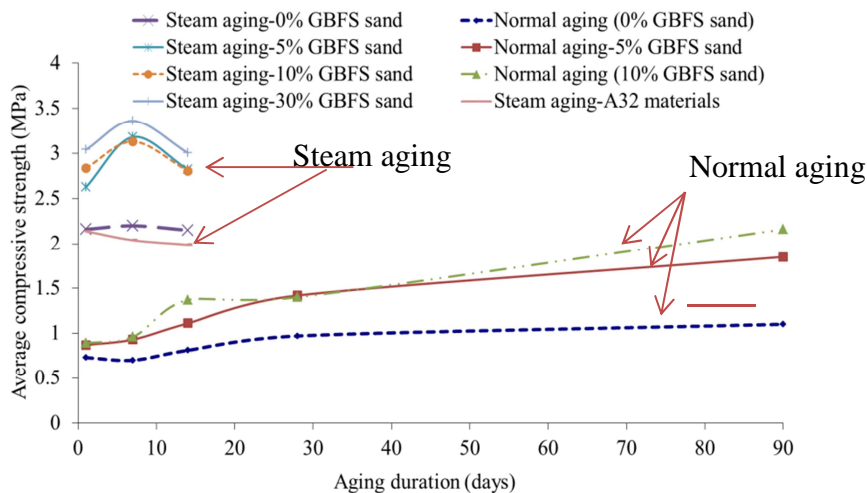


Figure 3: General comparison between development in compressive during two aging regimes.

3.3 Volume Stability

The results of the deformation measurement (volume expansion) made during the steam aging tests are presented in Figure 4. Due to the presence of a significant amount of CaO, MgO in GBFS sand and steel slag, it is possible that the combination of these compositions may influence the volume stability of mixtures adversely, especially with high GBFS sand percentages. Besides the expansion of the A32 material which is already hydrated (field aged materials collected after 20 years) was measured and it was considerably lower than that of the fresh materials.

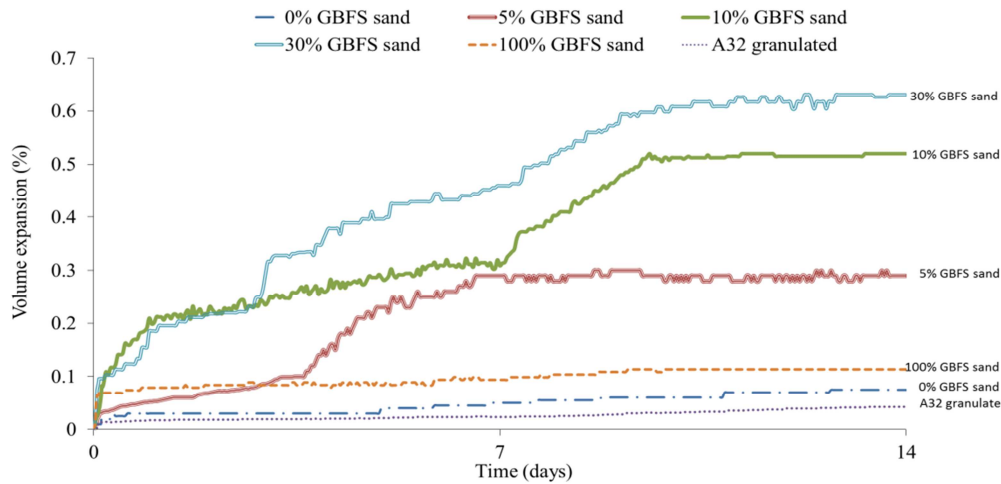


Figure 4: Measured deformations during steam aging for different mixtures.

After the failure of the A32 pavement structure there was a hypothesis that a high GBFS sand content was one of the reasons of heaves formation. Accordingly mixtures incorporating 100% GBFS sand were tested. To determine the effect of each type of slag aggregate on the volume expansion, also specimens were prepared with 100% steel slag and 100% air cooled BFS and they were steam aged up to 14 days.

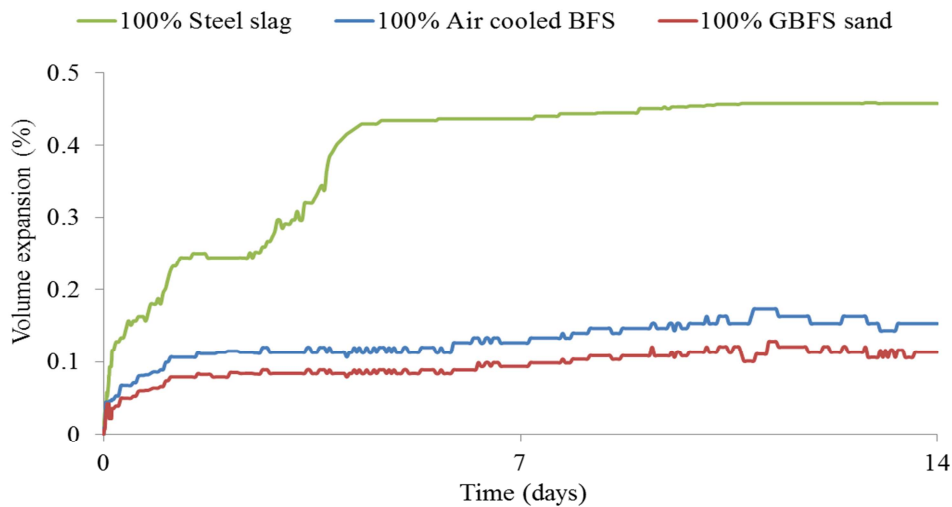


Figure 5: Measured deformations during steam aging.

The volume expansion test was repeated for three samples and as shown in Figures 4 and 5, with increasing GBFS sand content in the mixture, the expansion of cylinders during steam aging increased especially in the case of mixtures containing 30% GBFS sand. But interestingly, when purely GBFS sand was aged with steam, the expansion is not considerable. This phenomenon can be related to the chemical composition of the mixture. Micro probe images confirm this result and (see Figure 6) in case of 10% GBFS sand rims were formed around slag grains while in case of pure GBFS sand only cracks were formed.

The development in compressive strength and expansion may happen only if a certain level of influential chemical compositions exist in the mixture. It means there would be very limited expansion if for instance a certain amount of CaO and MgO does not exist in the mixture. More chemical study is presented in the next section.

Although there is no universally accepted limit for the volume expansion of slag materials under steam aging regimes, ASTM D 2940 (13) states that the volume expansion value of the steel slags should not be greater than 0.5 %. If this criterion is accepted as the maximum expansion limit, most of the studied mixtures (except mixtures containing 30% GBFS sand) will satisfy this limit. However, previous studies (5) show that an expansion more than 0.1% for the road (sub-)base application can be problematic.

3.4 Influence of Steam Aging on the Microstructure

The Microprobe analysis gives a reliable estimate of the real degree of hydration of the slag. Backscattered electron imaging provides a means of directly examining the relative densities of different phases of the microstructure. The intensity of electrons backscattered from a point on the surface of a specimen depends on the mean atomic number of the material at that point. In this investigation it was possible to distinguish three general phases: unhydrated slag grains appear as bright, hydration products appear as dark gray and pores (filled with resin) appear as black.

The comparison of the slag particles after different aging times with the original slag suggests that in presence of moisture and high temperature (65°C), the slag particles react with each other.

After 14 days of steam aging, hydration products formed around the slag particles in the case of using more than 5% GBFS sand, whilst none or very little hydrates could be observed in the mixtures without GBFS sand. GBFS sand plays a role as an activator and apparently has binding properties. In the matrix of high MgO and CaO content slag hydration products can be observed extensively after steam aging (see Figure 6).

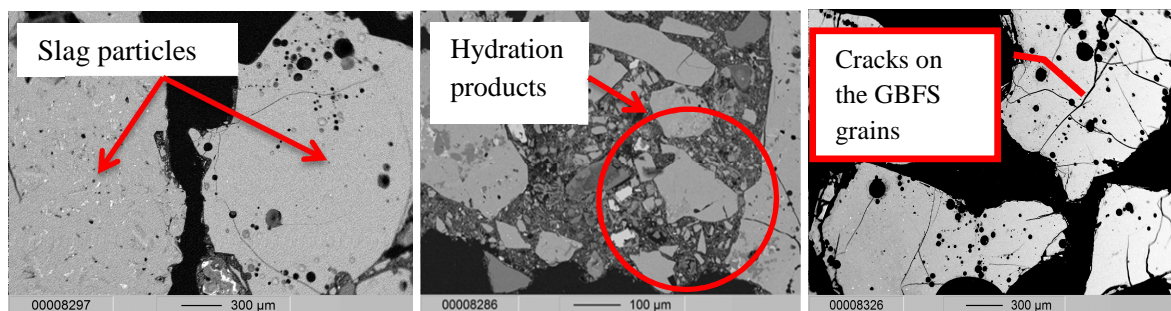


Figure 6: Backscattered electron images of a mixture with 10% GBFS sand exhibiting effects of steam aging. Left image is before aging and right image shows formation of hydration products after aging.

4 CONCLUSION

Three types of aging treatments were applied to study the interactive effects of temperature, moisture and time on the performance of the materials. Measurements done on laboratory and field aged samples suggested that two aging methods: steam aging and FT action, did a reasonable job of producing distress phenomena. All response variables such as strength, deformation, microstructure and chemical analyses show that there is a clear linkage between the performance of slag materials and moisture, temperature and time. The failure mechanisms such as material expansion and strength loss during laboratory aging were similar to the A32 motorway collapse.

It appears that heaves formation and cracking in the A32 motorway were occurred because of chemical alternation of materials. At the same time the FT cycles contributed to the failure.

Steam and FT aging methods appeared successful in recreating phenomena that can promote the initiation of deleterious mechanisms.

The results hold a promise that a slag mixture with a poor performance can be detected by the aging protocol (from expansion and/or loss of integrity). Furthermore, the aging procedures can be used as an accelerated screening tool which shows that a certain mixture property (e.g. GBFS sand content) can play a role in affecting future performance of the material and pavement layers made of it.

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