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Long-term performance of MSWI bottom ash in a test road-construction

Paul Frogner Kockum ^{1,*} and Jan Erik Lindqvist ²

¹ Swedish Geotechnical Institute, Linköping, Sweden

² Swedish Cement and Concrete Research Institute, Lund, Sweden

* Corresponding author E-mail: paul.frogner-kockum@swedgeo.se

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Abstract

The study focuses on long-term performances of MSWI bottom ash used as a reinforcement layer in an 8 years old road-construction. Long term properties may change under the combined effects of loading, climate- and chemical conditions. Characterization of the chemical changes in aged MSWI bottom ash is thus of prime interest as secondary alteration is a key process for the ageing of these kind of materials. The MSWI bottom ash in this study comprises a 60 meter-long segment of a test road, which was sampled eight years after construction. The objective of the sampling was to obtain a very low degree of disturbance to the application's in-situ properties. Access to the sub-base was achieved by removing the surface course and unbound base course, leaving the top surface of the unbound sub-base reachable. Epoxy impregnated slabs were also used for a micro textural and chemical characterization by SEM/EDS of the bottom ash sub-base layer. No cracks that imply movements or rotation of particles in the road construction or other disturbances as due to the sampling process were found. This undisturbed material made it possible to study chemical processes and structural changes that have been ongoing in the test road since it was constructed. The SEM/EDS analysis showed that most particles had reacted to some extent and that reaction-products surrounding aluminum particles were undisturbed. Partly decomposed particles indicate that the reaction (that has been ongoing since the road was constructed) has been slow and incomplete because of the coexistence of metallic aluminum and aluminum hydroxide. It also shows that the material not has been subjected to any physical influence during these 8 years that otherwise would have moved the reaction products from the particles that originally have reacted. Clay mineralization that indicates long-term ageing of the ash material was also detected by XRPD. The pH of the material was lower than 8.5, indicating a mature degree of carbonization. It is also concluded from the study that chemical reactions consistent with this maturity have been taking place in the road construction as indicated by textural relationships.

Keywords: MSWI bottom ash, aged, chemical alteration, microstructure, (long-term) stability

1.0 Introduction

1.1 Background

Contemporary community development is accompanied with a growing awareness of costs; e.g. regarding environmental and economic aspects of non-renewable resources or as a shared responsibility between industry and the end user to maintain an acceptable standard of living. One expression of this awareness is to look upon "waste" as a resource, fit for various use within the growing community. Thus "waste" or rather residues such as crushed concrete, various steel slags and ash materials, are increasingly looked upon as a material source comparable to natural resources. Many kinds of residues have been tested and proved as construction materials within the infrastructural sector, particularly true within pavement design. Although recycling technology has now been in use for many years, there are still technical issues that need to be studied in further detail. One of the more important issues is the technical and environmental long-term performance of residues [1-2]. The current understanding of the long-term properties and ageing effects is insufficient in spite of several years of research on residues and their applicability [3-4]. Material properties for a wide range of residues are not constant over the full service life. In the short term they will change during production, storage, transport and laying. In

the long term, they will change under the combined effects of loading conditions, ageing, climate and additives (e.g. de-icing products). Research has so far been devoted to mechanical or environmental properties, each evolving separately. It has been shown that residues are generally more sensitive to degradation and more prone to leaching than the natural aggregates that are normally used [5]. The prime technical factors influencing use in roads are material stiffness and stability, which are determined by particle size and microstructure. Residues age in roads and this ageing affects pH, particle size and microstructure of the materials. These ageing reactions may be predicted through study of pH, gas fluxes, redox conditions, climate, mineralogy and the matrix surrounding particles together with leaching and stability.

Long-term changes in this context are considered to correlate with a road construction's technical service life, e.g. for an unbound application up to 40 years. It is therefore understood that to quantify changes related to material properties – and in particular to understand any changes in technical performance – that are active over a road's service life, a sampling technique that acknowledges microstructural characteristics is of uttermost importance. Characterization of the macrostructure and microstructure is of prime interest as secondary alteration is a key process to understand more about ageing of MSWI in this environment [6]. This requires a sampling technique that ensures undisturbed samples for a thorough analysis of the macrostructure and microstructure of the MSWI bottom ash sub-base layer. Any changes in a long-term perspective are taking place in situ, a fact that underlines the importance of acquiring undisturbed samples. Changes in material properties may include a wide array of ageing effects such as influences on pH, pore solution, particle size, mineralogy and microstructure. Efforts have previously been made in Sweden as part of the Vändöra project to extract undisturbed samples from a test road in the Linköping area of Sweden constructed with MSWI bottom ash. In the Vändöra project drilling without water cooling was used to obtain undisturbed samples. It was reported from the Vändöra project that disturbance of the samples as due to the drilling could not be excluded [7].

In the present study a new stabilizing sampling technique was applied and used for the first time to acquire undisturbed in-situ samples of MSWI bottom ash used in a sub-base of a 60 m test road section. Sampling provided stabilized pillars of bottom ash that made it possible to obtain samples without structural disturbances. Bottom ash is the slag-like material produced by the incineration of municipal waste and is predominantly composed of a glassy constituent, which includes inherited manufactured glasses and glasses formed during incineration [8]. It has in earlier studies been shown that during combustion carbonates such as calcite, dolomite, anchorite and siderite decompose with the release of carbon dioxide (g) and this will eventually lead to the formation of lime or periclase. In the combustion chamber, pyrites oxidize and lose SO₂ (g) forming sulphates, and iron oxides such as hematite and magnetite. Aluminium silicates may melt or decompose to form glass or mullite [6]. Bottom ash from municipal waste plants has a complex and potentially reactive matrix and some high-temperature phases formed in the combustion chamber are metastable and may become transformed in to more thermodynamically stable phases at ambient conditions [9]. Studies have also shown that the alteration processes of bottom ash may involve the formation of calcium hydrate phases [10]. The reactions are exothermic, which means that they are reactive under ambient conditions. The following alteration reactions may occur in fresh bottom ash: Dissolution/precipitation of salts, glass corrosion, hydration and oxidation, slaking of lime, and hardening reactions like cementation, carbonation and pozzolanic reactions. Some of these processes may involve the formation of crystalline phases [11-13].

1.2 Objectives

The present paper is one of three papers from a project aiming at the development of a multi-scale, multi-method approach to the assessment of long-term properties of residues used in road constructions [1-2]. The two previous papers concerned the development of methods for an accelerated ageing and the second paper was giving an overview of the project methodology for different kind of industrial residues. This study provides the basic results from the material characterization of MSWI bottom ash aiming at to identify the crucial processes of ageing related

to the usefulness of MSWI bottom ash in a road construction. The novelty of the study results is mainly due the innovative sampling method that made it possible to relate micro structure and chemical processes to the meso- and macro- structure of the road base. This in turn made it possible to study and evaluates the long term chemical and physical behavior of MSWI bottom ash in a 10 years old pilot road construction with the aim to investigate the possibility to use and recycle MSWI bottom ash materials in road constructions.

2.0 Materials and Methods

2.1 Material

The bottom ash in this investigation originally came from Sysavs municipal waste plant in Malmö, southern Sweden, and had been piled outdoors before being used in the test road construction. It was not possible to pinpoint the accurate age of the ash before use as a sub-base layer in the road. Based on the properties of the ash it was assumed that it must have been stored at least six months to reach that degree of maturity, but it could have been stored for up to four years. After storage, in 1998, the ash was used as sub-base in a 60-metre stretch of the Törringe road in the Malmö area. The pH of the bottom ash used as a road material was within the range of 7.5 - 8 and the water content was around 0.5 liters / kg ash material [2,14]. The sub-base thickness was 465 mm and the total road pavement 745 mm.

2.2 Sampling and sample preparation

Sampling was performed in the Törringe road in September 2006. A pillar measuring approximately 300x500x460 mm³ (LxWxH in relation to the orientation of the road) of the MSWI bottom ash sub-base layer was stabilized in field using epoxy glue and a coating of wire net and gypsum plaster (Fig. 1). This sample covered the depth of the sub-base except for approximately 5 cm at the top and bottom. Prior to laboratory preparation, the specimen was further stabilized by freezing to -25°C. The frozen sample was cut into three slabs about 5 cm thick, measuring 34x17 cm² which were dry-sawn with a diamond saw from the sample. These slabs were vacuum-impregnated using epoxy glue containing a fluorescent dye. The impregnated slabs were glued to a glass slice and polished flat. This resulted in an epoxy-impregnated flat polished slab with a size close to that of the depth of the bottom ash that enabled the undisturbed ash to be studied. The samples used for SEM and XRD analysis were selected from one of these samples. The samples were then selected in order to identify variation in relation to the depth in sub-base. However no difference was documented. The drying process may cause shrinkage cracks but very few such cracks were observed. The preparation technique used has been developed for brittle materials and causes no cracking in the sample due to mechanical damage.



Figure 1. Sampling of in-situ specimens of bottom ash using mesh and gypsum.

2.3 SEM/EDS - analyses

The samples were analyzed using Scanning Electron Microscopy – Energy Dispersive Spectroscopy, (SEM/EDS). In this study we were using a low-vacuum scanning electron microscopy (Jeol LV5310) with Link Inca EDS equipment for micro-chemical analyses and element mapping. It is possible by this method to define a very small area for the analyses. The major constituents identified on the plane polished slabs were selected for analysis. Mineral reactions and zonation were investigated through element mapping.

2.4 XRPD -analyses

Samples were analyzed with X-ray powder diffraction (XRPD). In this study analyses were performed with a Philips PW 1710 diffractometer, equipped with a monochromator and coupled to a computer to obtain the X-ray data. Cu Ka radiation (50kV, 25mA; $\lambda = 1.5418\text{\AA}$) was used. All samples were mounted with ethanol on a Si plate to minimize background interference. The phases were identified from X-ray diffractograms obtained between 5 and 70 2-theta, at a scan-speed of 0.5 2-theta min⁻¹. The diffractograms were thereafter compared with published 2-theta values of known minerals.

3.0 Results

3.1 Quantitative macro analysis

The composition of the three plane polished samples was determined through point counting on a macroscopic scale using a stereo microscope for particle identification. Particles with a diameter larger than 2 mm were counted. Observation in stereo microscope revealed no significant differences in composition or amount of different types of particles between the three different slabs or different levels in the slabs (Fig. 2). No macro-scale cracks or movements were observed under the stereo microscope nor during the field sampling on site. The shapes of the macro-pores were irregular although not elongated.

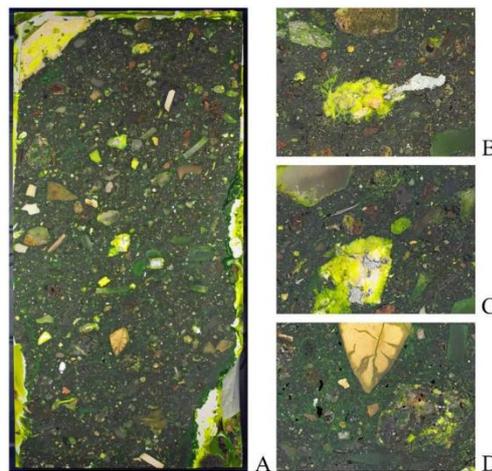


Figure 2. Epoxy-impregnated flat-polished slab measuring approximately 34x17 cm². The figures in A, B, C and D confirm undisturbed ash samples that could be studied from micro to decimeter scale. In B and C it can be seen how aluminum metal reacts to form white aluminum hydroxides. The yellow color comes from the fluorescent dye in the epoxy. These are both examples of ageing reactions that have occurred around solid phases. It can be noted that they have not been deformed due to mechanical loads from the traffic. D shows the initial reaction of a beige sulphur-containing particle. B, C and D can be identified at the corresponding levels in figure A.

3.2 SEM/EDS analyses

Subsamples were selected from the undisturbed polished slabs. The emphasis of this study is on particles larger than approximately 10 microns and their reaction products. The reason for this is that the smaller particles which constitute a partly solidified bulk of the matrix are too small for chemical characterization using EDS technique. Reactions where the reaction products and reaction structure remain undisturbed are assumed to have taken place in the road construction.

3.2.1 Particles of used glass

Glass particles occur as angular, up to centimeter-large particles. The glass particles generally show reaction rims where the glass cracks and the rims decompose into a less dense structure. The chemistry in the rims is depleted in calcium compared to the glass (Fig. 3). This decomposition includes a volume change that leads to formation of cracks in the glass. Chemical alteration and formation of cracks has been also continued after the construction of the road, as can be seen by new cracks running from the glass into the matrix (Fig. 4). This shows that internal expansion continues in the glass after deposition in the road construction.

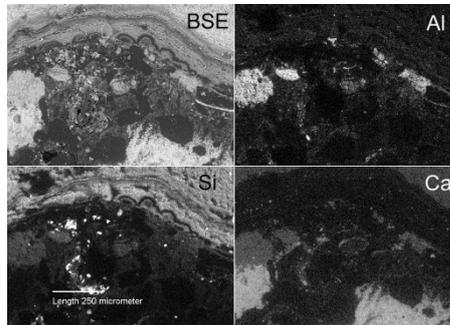


Figure 3. EDS element maps that show that the rim of the glass particle is depleted in calcium while silica and aluminum contents are unchanged. The instrument magnification is 200 times.

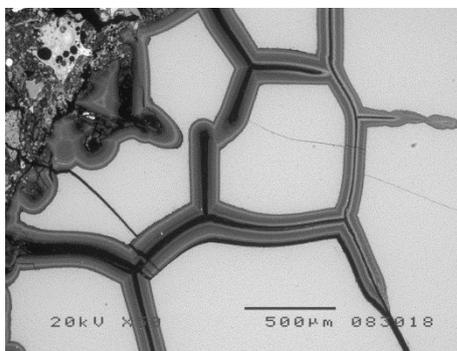


Figure 4. A BSE image that shows the glass weathering reactions in the glass rims. The crack running through the glass into the surrounding matrix shows that expansion reactions have taken place in the glass in its present position. This type of cracks are not formed in the used preparation process.

3.2.2 Particles of porous vitreous silicate glass

Porous silicate slag particles with irregular shape occur as micrometer- to centimeter-large particles. These particles show complex chemical patterns. These particles are formed through partial melting or sintering of minerals and other siliceous material during combustion. An example is given in Fig. 5. The BSE image shows a sharp boundary between the porous particle and the matrix. The element maps show diffuse boundaries with no distinct change between particle and matrix. This implies that chemical exchange between particle and matrix has been going on in the road construction.

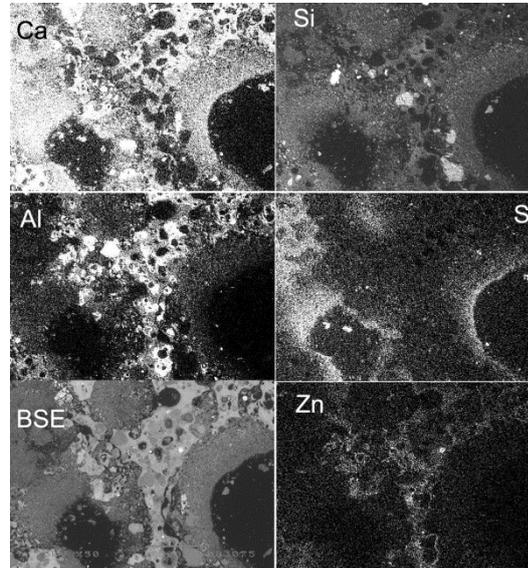


Figure 5. BSE and EDS mapping images showing a porous glassy calcium silicate particle with large pores and surrounded by a very fine-grained matrix. The length of the image is approximately 2.5 mm.

3.2.3 Silicates and porcelain

Porcelain and tiles occur as up to centimeter-large angular fragments. The silicate minerals and porcelain do not show signs of reactions or chemical dissolution in the present SEM/EDS investigation. There is no difference in the composition of the rims of the particles compared to the cores, nor is there any chemical zonation in the matrix close to the silicate particles.

3.2.4 Carbon particles

Carbon particles occur as up to centimetre-large rounded porous particles with a high amount of small mineral inclusions (Figure 6). The EDS method did not identify hydrogen and it is consequently not possible to differentiate between pure carbon and carbon hydrogen compounds. The carbon particles generally have inclusions with a complex chemistry in the EDS spectra, containing phosphorus, chlorine, sulphur, zinc and copper, in addition to carbon and the main elements in the silicate inclusions. It is not possible from the EDS analyses to determine if the elements are to some extent bound to the carbon or to inclusions in the carbon particles. These inclusions may be smaller than the size of the interaction volume that the spectra are obtained from.

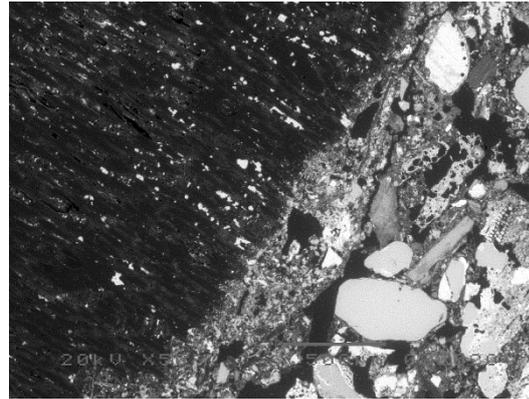


Figure 6. SEM/BSE image that shows a carbon particle with inclusion of calcium, silica and aluminum minerals.

3.2.5 Sulphur-rich particles

Sulphur occurs both as particles enriched in sulphur and in low contents spread in the fine-grained matrix. The sulphur-rich particles generally show a complex chemical pattern (Fig. 7). The element maps show no clear correlation between sulphur and calcium. Near the open continuous cracks the particle is depleted in sulphur and enriched in silica. The texture of undisturbed reaction product adjacent to this particle implies that these reactions have continued in the road construction. This can be demonstrated for most of the sulphate particles.

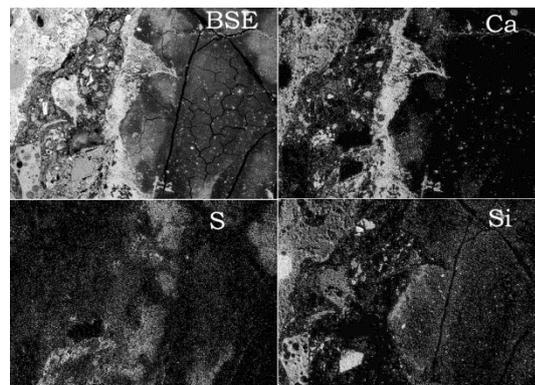


Figure 7. EDS map over the rim of a sulphur-enriched particle. The length of the image is approximately 2.5 mm.

3.2.6 Metals, metal oxides and hydroxides

Iron and aluminum are the most common metals. The larger iron particles mostly retain their original shape while small fragments vary in shape from rounded particles to irregularly shaped crusts of iron hydroxides that are formed in-situ as seen in Fig. 8. Particles of stainless steel are chemically stable. There is however a slight increase in iron and alloying elements in the matrix near these particles.

The aluminium metal occurs as particles ranging in size from micrometers to centimeters. These particles have very irregular shapes and are surrounded by an irregular halo of reaction products composed of aluminium hydroxides and often small amounts of copper (Fig. 9). The reaction products in Fig. 8-9 show no disturbance due to deformation, which implies that the reactions have taken place in the road construction.

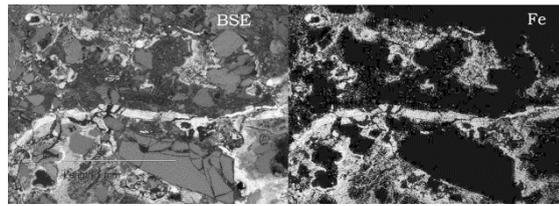


Figure 8. BSE image and EDS map of iron hydroxide in an area that can be seen as a colored area in the sample. The instrument magnification is 50 times.

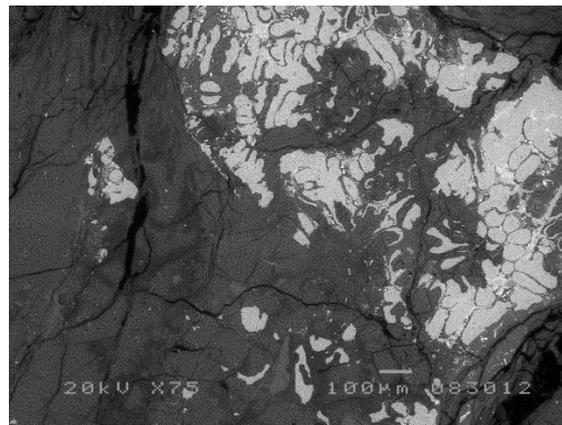


Figure 9. BSE image showing an irregular metallic aluminium particle in bright grey surrounded by hydroxides in darker grey.

3.2.7 Fine-grained particles in the matrix

The larger particles are embedded in a matrix composed of fine particles with a wide range in chemical composition and with a typical size of a few microns and finer. The particles are composed of calcium, silica, alumina, potassium and sodium and are to a large extent silicates. Sieve analysis of materials from the Törringe road gave 45% finer than 2 mm while the optical analysis gave 75% finer than 2 mm [15]. The reason for this is that the bottom ash in the road is a partly solidified composite that does not fully disintegrate into individual grains during the preparation for sieving.

3.3 XRPD analyses

Samples of bottom ash were selected from the undisturbed polished epoxy impregnated slabs. Four different samples consisting of various amounts of matrix and 1–2 mm grains were picked with a nail-shaped spatula from different parts of the slab. The various samples were then ground separately in ethanol using an agate mortar and subsequently analysed by XRPD (see section 2.4).

The indication of glass by SEM/EDS chemical analysis was confirmed by the XRPD mineralogical investigation. A higher background in all diffractograms with a maximum around 30° 2-theta indicates the presence of modified depolymerised glasses (Stutzman PE, Centeno RL., 1995). Diffuse and broad peaks may also indicate a low crystallinity. Clay minerals were also found through the diffuse indication as better-defined peaks around 7° 2-theta as well around 7° 2-theta in several diffractograms (Fig. 10). The analysis furthermore showed that quartz, hematite, calcite and plagioclase among other minerals were present in the sample. It can be noted that gypsum and ettringite were not identified in the samples.

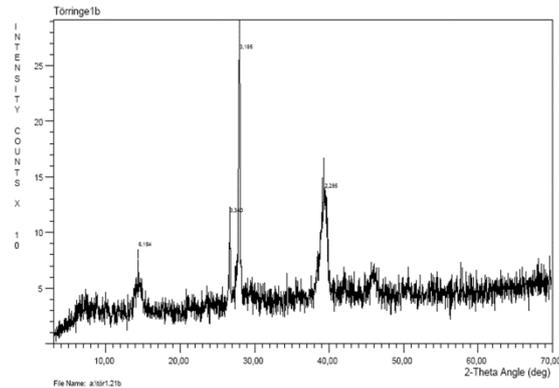


Figure 10. XRPD analysis of bottom ash from Törringe. The broad peak at 7.5-2-theta implies the presence of clay minerals in the sample.

4.0 DISCUSSION

The SEM study of the microstructure of the bottom ash showed no cracks that imply movements in the material or rotation of particles. It can be noted that all reaction products surrounding partly decomposed particles were undisturbed. The microtexture shows that the relationship between the original phases and reaction products was intact. This showed that the sample was undisturbed by the sampling process, but also that there had been no movements in the road construction. This made it possible to study the chemical and structural processes that had been active in the road construction, from micro to macro scale.

The SEM/EDS analyses showed that most particles had reacted to some extent in the road construction. This ranged from strongly decomposed sulphur-containing particles to the particles of stainless steel that had a halo with a weak increase of iron and chromium. Particles of ceramics and rocks were exceptions and showed no signs of reactions. The observed reactions involve an increase in volume. The microstructural analyses demonstrated no disturbances such as cracks or physical redistribution of the material. There were however exceptions, such as micro-cracks related to glass particles. These few cracks had no influence on the structure of the road construction or function. The interpretation was that the expansion had been taken up as plastic deformation and a decrease in pore volume without changes in the volume or shape of the road construction. The decomposition of aluminum was of special interest and hydrogen gas is one of the reaction products that may reach critical concentrations. The SEM results showed the coexistence of metallic aluminum and aluminum hydroxide. This showed that the reaction had been incomplete.

Earlier studies have shown that MSWI bottom ash, which has been stored in the open air, has similar weathering products as usually found in volcanic ash. Zevenbergen [13] found evidence of formation of clay minerals such as illite from glasses in MSWI bottom ash after years of natural weathering. In volcanic ashes, ageing results in the formation of secondary phases such as clay minerals and precipitation of iron and aluminium hydroxides. Clay minerals were also detected in the bottom ash from the Törringe road even though this road is asphalt-paved. However the water exposure of the bottom ash in the Törringe road is calculated at 0.1 – 0.5 liters per kg [2,14]. Due to the lack of textural observations the clay mineralization process could not be restricted only to a process ongoing in the road construction and it may already have been initiated in the pile material in the storage site at the Sysav municipal waste plant in Malmö.

The bottom ash from Sysavs municipal waste plant in Malmö contains iron mineral phases such as hematite and pyrite that are usually found in other studies of bottom ash as well. It has been shown that rock-forming minerals usually found in soils, such as pyroxene, quartz and feldspars are also usually found in bottom ash [16].

The results of the present study compare with the three major stages in the weathering of bottom ash that are generally identified. Each stage has a characteristic pH that is controlled

largely by Ca minerals and CO₂, but also by soluble Al and SO₄; (1) unweathered bottom ash, with pH > 12 (grate-sifting and unquenched bottom samples); (2) quenched/non-carbonated bottom ash, with pH 10–10.5 (freshly quenched and 6-week-old samples); and (3) carbonated bottom ash with pH 8–8.5 (1.5- and 12-year-old samples).

The weathering of bottom ash may further be divided [12] into weathering products that have been waterlogged (ettringite, Ca zeolites, sepiolite, analcime and brucite) and weathering products that have been open to the air (calcite, dolomite, basaluminite, 2:1 clays, halloysite, gypsum and analcime). Quartz was present in all samples that were analysed in this study and calcite and hematite were present in three of four samples. Considering the age of the Törringe road this was expected for stage 3 in the model. Stage 3 also agrees with the pH of slightly below 8.5 on average for the material in the Törringe road [14,15] and with the formation of clay minerals. The mineral samples that contained calcite and hematite were sampled with more matrix besides the single mineral phases that were visible to the eye. In the two samples from the Sysav bottom ash, analysis of the matrix using XRPD indicated that calcite and hematite are common mineral phases. From results of earlier studies [12,16,17] it was found that hematite most likely is formed during the combustion process of bottom ash. Calcium oxide, lime, is usually formed in the combustion chamber. In stage 1 of the model this is converted to calcium hydroxide which in stage 3 has been further transformed to calcite. This reaction has probably occurred before it was used as sub-base in the Törringe road. However, percolation tests of the road material from the Törringe road compared with the pile material [2] showed that bottom ash of the road was more aged than the bottom ash in the pile.

Pozzolanic reactions are likely the reason for the formation of aggregations in the fine-grained matrix of the bottom ash as the carbonation reaction is considered as a minor contribution to this observed increased stability of the ash in the road. It is also very likely that any further weathering processes of the bottom ash in the road have been restricted due to a limited transport of water beneath the asphalt as it could also be seen that the edge material in the road was more aged than the material in the center [2]. As the transport is restricted in the center, the pore solution most likely becomes saturated with respect to weathering products and diffusion will restrict the weathering reaction. The ageing differences were concluded as caused by differences in water exposure [2].

5.0 Conclusions

The successful sampling of undisturbed samples made it possible to study the textural relationship between original phases and reaction products and thus which reactions that had been ongoing in the road construction since it had been constructed.

The microstructure in the bottom ash showed no cracks or physical redistribution of the material that implies movements in the material or rotation of particles. For example, aluminum reaction products surrounding partly decomposed particles were undisturbed. Clay mineralization that indicates a long-term ageing of the ash material was also detected by XRPD. The clay mineralization could not be restricted to an ongoing process only in the road construction.

It was furthermore concluded that the ash had been carbonized as due to the relatively low pH and the presence of calcite. It is also concluded that the observed chemical reactions are in consistence with the degree of maturity of the studied MSWI bottom ash. This study thus showed that bottom ash seems to be a good candidate among other alternate materials for a sustainable use in road constructions. This statement is made due to the observation of no cracks that implies movements or rotation of particles in the road construction was found and the fact that the bottom ash is protected under the pavement.

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