

ABSTRACT

In order to bring understanding to deterioration of pavements in Finland, prematurely failing pavement of Ring Road II in Espoo (Finland) was investigated. Documentation of mix design (MD), construction and material quality analysis, prior to and after construction, was reviewed and compared to the samples collected from the road. Traditional quality assessment of the pavement by means of binder content and aggregate gradation analysis, air voids content (V_a), Indirect Tensile Strength (ITSR), as well as binder tests (Penetration, Ring and Ball Softening Point, Dynamic Shear Rheometer) did not provide information on the possible reasons of failure. Discrepancy was then found in the filler composition and the discovery was made by applying standard analytical procedure of hydrochloric acid solubility, coupled with scenario simulations. This technique was positively evaluated by means of Thermogravimetric Analysis (TGA), X-Ray Diffraction (XRD), and Scanning Electron Microscopy (SEM), supported by BET-adsorption surface area (BET-SA) measurements. Composition of the filler used was established on the basis of results, which suggested that, between the stage of MD and construction, limestone filler was substituted with a mixture of limestone and fly ash. Blends of filler were reconstructed both according to MD reports and established forensic laboratory findings. Comparative analysis, of reconstructed fillers, by means of Fourier Transform Infrared (FT-IR) spectrometry, proved almost an ideal match to the latter. Alteration of construction material is suggested as the main reason for discrepancies between the MD and actual properties of the analyzed wearing course. It can be concluded that the main reason for the pavement failure was due to this substitution and the consequent problems that followed in the construction and thereafter in the pavement performance.

Key words: HMA, field study, forensic investigation, quality assurance

INTRODUCTION

Over the last two decades asphalt pavement performance has steadily declined in Finland leading to major arterial roads with frequent maintenance needs. Multiple factors under discussion, that could influence this fact, include increasing traffic (both passenger cars and heavy trucks), use of recycling and thinner asphalt layers, alterations of Mix Design (MD) practices and specifications (*1*), or changes in construction materials available on the market. Since 1995, when MD was outsourced to the contractors, the Finnish Transport Agency (FTA) has focused on development of procurement methods and documentation and quality assurance (QA) of the executed constructions relies heavily on contractor's own quality control (QC) documentation. However, no methodology existed to analyze asphalt mixture composition in detail for the purpose of comparing actual quality against the design and specification requirements. This paper presents findings of forensic case study on Ring-road II's (RRII) asphalt pavement deterioration commissioned by the FTA, focusing on construction material analysis post-mortem.

Initial forensic testing was conducted according to best QA practices, which included visual inspection (VIS), ITS, ITR, V_a , layer thickness, aggregate gradation and binder content. This was extended into extracted binders' analysis by means of Penetration (Pen), Ring and Ball Softening Point (R&B), Dynamic Shear Rheometer (DSR) and generic fractions (SARA).

Afterwards construction quality control and assessment (QC/QAS) records were reviewed and compared to the forensic results and clear deficiencies in densification and mechanical properties were observed. In order to quantify effect of materials on poor performance and origin of failure, more advanced methods were used. The role of the filler material used in asphalt mixture was found of special interest. Methodology (including techniques such as Hydrochloric Acid Solubility Test, BET-SA, TGA, XRD and SEM), necessary to deduce filler composition from field samples was thereafter proposed and is presented in this paper. For continuation both reported and deduced fillers were reconstructed. Their composition was analyzed with FT-IR spectrometer and results compared to the spectra of the field extracted sample, which then proved the proposed methodology to be correct.

Based on findings presented in this paper, filler material was changed between the stages of MD and construction, against the best practices given in specifications (*1*). Limestone (LS) filler was substituted with a filler mixture of LS and fly ash (FA). Daily fluctuations in material characteristics of the latter are proposed as an explanation for the otherwise random distribution of failure locations within the surface layer of the pavement. Multiple reports on the subject of mastic composition (*2, 3, 4, 5, 6, 7, 8, 9, and 10*) explain vastly the potential effects filler substitution could have on mastic stiffening and workability of the mixture, but also its moisture susceptibility (*11*). Therefore, a detailed quantification of the substitution effect was commissioned as the second phase of this case study and will be presented in near future by our research team.

Case description

RRII is an important 2-lane arterial road located in metropolitan area of Helsinki. Road section is 6.8 km long and has ca. 49 000 vehicles/day of which ca. 2000 are heavy vehicles. The speed limit is 80 km/h. Due to large areas of patching, road suffered from poor ride quality

1 before overlay rehabilitation in 2011. Forensic investigation comprised of road condition
 2 measurements, distress inventories, analysis of construction and maintenance records and
 3 laboratory testing. Sampling, majority of laboratory testing, and data analysis have been
 4 conducted by Aalto University.

5 Stage one of RRII's construction was completed in 2000. Pavement design included 250
 6 mm thick rock bed base course of blasted and crushed rock, bound base course of 70 mm thick
 7 asphalt concrete base layer, and top of that 40 mm thick binder course layer of SMA20 (1, 12).
 8 In stage two, 40 mm thick wearing course SMA16 was placed down in autumn 2002 (SMA16).
 9 Despite predictions of good performance over 20 years of design life, distresses such as potholes,
 10 cracking and raveling started to emerge early on. Sections of the road surface were rehabilitated
 11 using Remix technique in 2007, 2008 and 2009 by mixing 16 to 19 kg/m² fresh SMA16 with
 12 existing SMA16 mixture. The multiple attempts of surface rehabilitation were ineffective and, in
 13 2011, road was overlaid with 40 mm thick SMA16 layer.

14 Construction records and air temperature records from the Finnish Meteorological
 15 Institute allowed estimating that air temperature during paving work in fall 2002 was close to
 16 zero degrees. This cold temperature is possible origin for the poor compaction, in addition of
 17 possible deficiencies in raw materials and mixture composition.

18 In spring 2011 before the new overlay, 50 cores were drilled from different locations.
 19 Based on VIS, sampled areas were selected to be taken from "bad" and "good" areas of road.
 20 However, VIS of cores revealed that in some "good" areas pavement was severely deteriorated
 21 underneath the surface. Table 1 lists investigated core locations separated to original (2002) and
 22 rehabilitated surfaces.

23
 24 **TABLE 1 Core locations, assessment of pavement surface age and condition based**
 25 **on visual inspection of cores**

Core location	Surface mixture	Surface condition vs. condition of cores
A,B,C,E, K, L	SMA16 (2002)	BAD - samples broke during coring and layers were not bonded (except K)
G,J	SMA16-REM (2007)	GOOD - full samples obtained, layers were bonded

26
 27 As Table and Figure 1 show, sampling revealed that original asphalt layers were not
 28 bonded together, which indicates severe water infiltration to the pavement structure. Also, what
 29 was observed visually was the odd color of the SMA16 pavement layer; instead of being shiny
 30 black in color it was brown and matt.

31 Although conventional paving industry quality assessment (QA) methods revealed clear
 32 deficiencies in densification and mechanical properties, to establish origin of failure, more
 33 advanced methods were used. Therefore, focus of this paper is in mixture composition of
 34 SMA16. Rheological and chemical analyses were carried out to compare actual materials to the
 35 materials reported in MD.

37 FORENSIC TESTING METHODOLOGY

38
 39 Samples after collection were separated into layers either by means of self-detachment
 40 (bad samples, see Fig. 1 (c)) or cutting by diamond saw (good samples). Mechanical and
 41 chemical test were performed to explain differences in "good" vs. "bad" pavement locations.

1 Tests included conventional QA/QC testing, more advanced rheological testing for binders and
 2 chemical analysis for fillers separated from extracted aggregate. Selected set of samples were
 3 subjected to ITS (SFS EN 12697-26) and ITSR (SFS EN 12697-23) testing. Measurement failed
 4 on few samples collected from bad locations, mostly because of cracking during handling and
 5 preparation (see Fig. 1 (d-f)).
 6



12 **FIGURE 1 (a) Core collected from good area K, (b) core collected from remixed**
 13 **area G, (c) SMA16 separation from supporting bound layers in area L, (d) cracking of**
 14 **collected samples during collection – SMA16, area C, (e, f) and manual handling – SMA16,**
 15 **area E**
 16

17 All samples were then subjected to measurement of bulk density (EN 12697-6, method A
 18 and B). Maximum density of mixture (EN 12697-5) of each core location was obtained by
 19 combining samples from the same layers to obtain large enough specimens for testing. On the
 20 basis of the above results, V_a was calculated, according to EN 12697-8.

21 Samples were extracted according to EN 12697-1, where dichloromethane was used as a
 22 solvent. Bitumen was recovered by rotary evaporation method according to EN 12697-3 and
 23 bitumen content was calculated according to EN 12697-1. Centrifugation did not reveal any
 24 residue; investigation of binder by means of optical microscopy did not reveal any solid cluster
 25 formations. Additional binder content testing by thermal decomposition EN 12697-1:2005
 26 (Annex C) conducted on recovered binder resulted in 100% mass loss reading in temperature of
 27 575°C.

28 Extracted binder was investigated by means of Penetration (EN 1426) and Ring and Ball
 29 Softening Point (EN 1427) tests. Further analysis of binder was conducted by separation into
 30 SARA fractions and rheological investigation by DSR.

31 Determination of aggregate particle size distribution was conducted according to EN 933-
 32 2. Afterwards each fraction was held separately and representative samples processed in further
 33 study by TGA, XRD, SEM and hydrochloric acid solubility tests (PANK 2405). Filler surface
 34 area was determined according to DIN 66131, (PANK 2401).
 35

36 **RESULTS FOR CONVENTIONAL QA ASSESSMENT**

37 **Air voids content (V_a) vs. mechanical properties**

38 In the V_a QC report conducted by NDT method, an average value of 2.7% and very high
 39 standard deviation of 2.42% was reported for SMA16. All the bad areas were found to have 5-
 40 8% V_a and layer thickness less than required minimum of 40 mm, though. The good area K had
 41 V_a of 4%.
 42
 43

44 A correlation was found between ITSR values at 10°C and V_a , with bad areas of high V_a

1 giving predicted response of lowest strength. Values ranged between 1.21 – 2.56 MPa. ITS at
2 10°C was measured only for good areas, as bad area samples were not sound for the
3 measurement. However, for good area samples, pavement stiffness was between 7000 and 9000
4 MPa, with lower values in the samples with higher V_a .

6 **Mixture Composition**

8 Examination of construction quality assurance/quality control (QAS/QC) records for
9 SMA16 revealed discrepancies between design and constitution. In addition, MD data and
10 information of raw materials was scarce.

11 SMA16 wearing course was designed as a mixture of 3 components, namely sand (#0-2),
12 gravel (#5-8) and crushed rock (#8-16), in ratio 15:10:66. Additionally, 9% limestone filler was
13 to be added. The total amount of fines passing 0.063 mm was 9.9% constituting of limestone
14 filler and baghouse fines, mainly from the sand fraction. Design bitumen (grade 70/100) content
15 was selected at 6.1% and 0.33% cellulose fiber was to be added to prevent binder drain-down.
16 However, cost charge was found in documentation for SMA16 with 8% of unspecified filler.

17 Average binder content in QC records was 5.97% with standard deviation of 0.11%.
18 Forensic testing revealed that for the bad areas, the average binder content was 6.0%, while the
19 only good area (K) had binder content of 6.3%. Amount of fines (<0.063 mm) in QC reports
20 were well within design limits; only percent passing 4 mm was 5% and passing 8 mm was 7%
21 coarser than specified. Our testing indicated some segregation, i.e., lower binder content
22 corresponds to the coarser gradation.

24 **Observations**

26 Magnetic round particles were found on the walls of the extractor after binder content
27 evaluation, in the fraction of 0.063-0.5 mm. Even more, fines after extraction, expressed
28 increased hydrophobicity (14% floated), despite no apparent remains of methyl chloride soluble
29 organic residue. This prevented performing standard measurements of particle density of filler in
30 water (EN 1097-7) and gradation curve of minus 0.063 mm fraction via hydrometer technique
31 (EN 1687). Similar behavior was observed in our laboratory previously for fines of extracted
32 samples containing FA as filler. Particle density of filler was measured in toluene (EN 1097-7) at
33 2.640 Mg/m^3 , which was lower than MD values for aggregate (2.700 Mg/m^3) or chalk filler
34 (2.770 Mg/m^3).

36 **Bitumen properties**

38 *DSR results and conventional properties*

40 Frequency sweep tests were performed with a stress-controlled Reologica StressTech
41 rheometer in the controlled-strain measurement mode. Measurements were done in the
42 temperature range of 2-100°C starting from the lowest temperature. Parallel plate geometries
43 having diameters of 8 mm and 25 mm and measurement gaps of 2 mm and 1 mm were used in
44 the temperature ranges of 2-40°C and 50-100°C, respectively. In all of the temperatures a
45 frequency sweep from 0.01 Hz to 10 Hz was performed within the region of linear viscoelastic

(LVE) response of the studied binders. These limits for LVE response were determined by strain sweep tests prior to the frequency sweep measurements.

In Figure 2 (a), a semi-logarithmic plot of phase angle δ against complex shear modulus G^* , commonly known as Black diagram, is presented for the studied binders. From the plot it can be seen that extracted RRII binders exhibited relatively more elastic behavior compared to the reference 70/100 binder. However, no significant differences between the Black curves of aged RRII binders could be seen. Susceptibility to fatigue cracking can be predicted with the Superpave fatigue parameter $G^* \sin \delta$, for which a critical temperature is defined to be the temperature at which $G^* \sin \delta = 5000$ kPa (13). From Figure 2 (b) it can be seen that RRII extracted aged binders had considerably higher critical temperatures compared to 70/100 binder indicating vulnerability to fatigue cracking.

Excluding the reference 70/100 binder, the Superpave PG-grading rutting parameter $G^* / \sin \delta$ at 10 rad/s given in Figure 2 (b) suggests that the softest binder was AB followed by E, G and J grouped quite closely together. Area C deviated from this group significantly having the stiffest binder properties. Interpreting results according to Superpave criteria for rutting (13), $G^* / \sin \delta$ at 10 rad/s must exceed 1 kPa to meet the specification. Binders extracted from RRII were thus vastly altered and hardened in comparison to the reference 70/100 binder. However, these findings do not completely agree with the conventional test results, and need to be studied further.

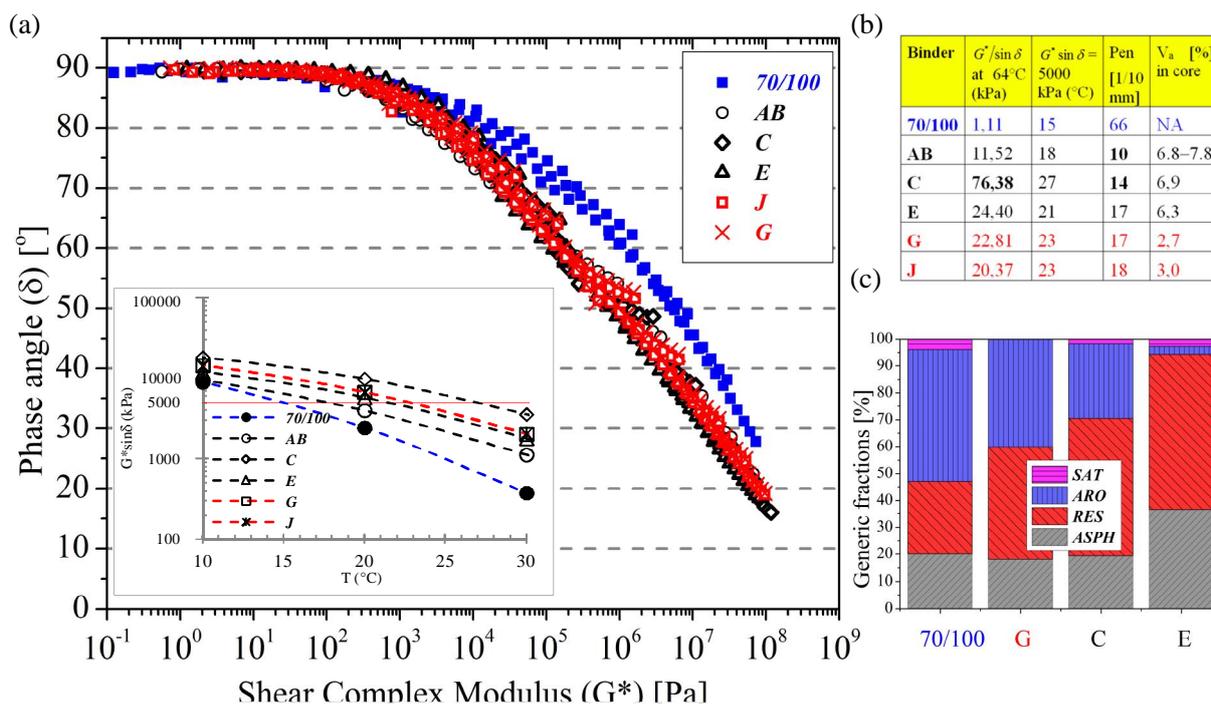


FIGURE 2 Binder properties: (a) Black curves and SHRP fatigue cracking parameters $G^* \sin \delta$, (b) Comparison between fatigue cracking parameter (13), penetration values and air voids' content from representative areas, (c) generic fraction composition of extracted bitumens

1 Pen values and R&B values (65-74°C) are consistent with aged binder extracted from
2 pavements of similar age and similar V_a , although some variation existed (14).

3 4 *SARA fractions*

5
6 The generic fractions of the extracted bitumens were determined by thin-layer
7 chromatographic (TLC) method with flame-ionization detector (IATROSCAN MK-6s) –
8 methodology described elsewhere (15) and results are presented in Figure 2 (c).

9 Transformation of aromatic fraction into resin fraction stays in agreement with
10 knowledge of bitumen aging (16, 17). However, area G and C express similar levels of
11 asphaltenes compared to the original bitumen. Similar observations were not found from
12 literature dealing with aging of binders. Rehabilitated area G may contain a supplementary
13 amount of softer binder and thus reading of asphaltene on the lower scale (15). The same cannot
14 be stated for area C and this is currently under further investigation. Samples collected from area
15 E resulted in extraordinary readings because there was no saturates or aromatics. Similar reports
16 were not found in literature for laboratory aged binders, analyzed by TLC technique. Results
17 indicate extreme degree of aging, though. Preliminary hypotheses based on above results and the
18 brown color of asphalt mixture are that either bitumen was overheated during preparation or
19 addition of aging promoting modifying agent caused instability and separation of bitumen into
20 subcomponents. No clear correlation between DSR, SARA fractions or air voids content was
21 obtained, as seen in Figure 2.

22 23 **RESULTS FOR ADVANCED AGGREGATE TESTING**

24 25 **Test on solubility in hydrochloric acid (HAST)**

26
27 After gradation analysis of extracted aggregates, a fraction <0.063 from samples
28 collected of area E, C and G were subjected to tests of hydrochloric acid (HCl_{aq}) solubility
29 (PANK 2405), standard measurement required as supplemental information of any limestone
30 fillers used in road construction in Finland. Method can be described as followed: 10 g of a dry
31 material is weighted in beakers and dried in oven at 110°C, allowed to cool down for 1 hour in
32 desiccator and weighted again following addition of 50 ml of deionized water and 25 ml of
33 concentrated HCl_{aq} (37%). Such prepared samples are conditioned in boiling water bath for 30
34 min. Beaker residues are filtered on a medium filter paper, previously dried in the oven at 110°C,
35 allowed to cool down in desiccator and weighted. Filter with residue is dried in the oven at
36 110°C for one hour after which samples are allowed to cool down to room temperature in
37 desiccator and weighted again. **Mass loss**, recalculated into percents, provides a value of total
38 solubility in HCl_{aq} . Reference tests for FA ($\text{FA}_{\text{HAST}}=7.78\%$) originated from biomass and crushed
39 aggregate ($\text{AGG}_{\text{HAST}}=4.91\%$) were conducted on fraction passing 0.063 mm sieve.

40 In addition of testing the presence of limestone filler, a scenario simulation (SSIM) was
41 conducted in which the solubility was calculated based on the known or assumed fractions of
42 each material. The SSIMs were calculated for variables discussed below, and both the calculated
43 and measured results are gathered and presented in Table 2. First, calculations were conducted on
44 minus 0.063 mm fraction with an assumption that 78.1% limestone is passing 0.063 mm sieve,
45 and 94.5% - 0.125 mm sieve (QC report). The calculated solubility i.e, HAST results for minus
46 0.063 fraction were found significantly lower for all the cases formulated based on the MD

report ($LS_{HAST}=93.7\%$). According to construction specifications (1), the LS filler needed to meet criterion of HAST over 75%, so-called CC75. The second step was to investigate if filler with 75% solubility was used. Alternatively some other type of LS filler or filler diluted with other non-soluble solid material (referred here as **additive**) so that it still met the criteria of CC75 could have been used. Table 2 shows that measured solubility values met the minimum HAST criterion for fillers but differed vastly from the actual MD formulation. The LS filler composition was investigated further as follows.

TABLE 2 Presentation of hydrochloric acid solubility test results in comparison with SSIM values, row with green background highlights the best match

Scenario Simulations (SSIM), HAST%				Measured HAST%		
Composition of minus 0,063 mm	LS filler to aggr.	HAST% for LS ¹	Calc.	C	E	G
Min. spec requirement for MD: 9% LS filler	71.0:29.0	CC75 ²	54.57	51.86	48.30	47.58
Max. MD requirement: 9% LS filler	71.0:29.0	CC93.7 ³	67.95			
8% LS filler	63.1:36.9	CC93.7 ³	60.93			
Min. spec req. if 8% LS filler	63.1:36.9	CC75 ²	49.14			

¹) LS = limestone, ²) from (1), ³) from QC measurement

Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA) confirmed by X-Ray Diffraction

The thermogravimetric analyses were done in a thermobalance (Perkin Elmer Pyris 1 TGA) in air atmosphere (40 ml/min) by heating powders, passing through sieve size of 0.063 mm, up to 1000°C with a relatively slow heating rate (5°C/min).

Samples from area E prior to and after thermal treatment were analyzed with XRD. Phase identification of the samples was confirmed with a laboratory X-ray powder diffractometer (XRPD; PanAnalytical X'Pert PRO MPD, CuK α 1 radiation) at room temperature in the 2 θ range 10°–100°.

The largest mass change observed occurred in the region of 556-779°C, with minimum in DTA expressed at 755°C. Mass loss equaled 21,641% and was assigned to complete carbonate decomposition, both calcite and dolomite (18). This mass loss corresponds to **47,45-49,98%** of carbonates in initial sample, and stays in agreement with results obtained through HAST (48,30%, area E).

Surface Area (SA) analysis

Due to the fact that limestone, FA and aggregate dust (baghouse fines) are having different surface areas, this path was followed during investigation and surface area (N₂-BET, Flowsorb II 2300, N₂/He 30:70) was measured for fractions <0.063 mm. Figure 3 (b) represents obtained data against the SSIM. Unfortunately, data for the surface area of the **additive** investigated was not available in the documentation, yet Loss on Ignition (LOI) values, for unspecified **fly ash**, were found from documentation for September and October 2002. The LOI

utilized in the Finnish specifications is the amount of matter lost on heating up to $1000\pm 50^\circ\text{C}$. We plotted the reported LOI values correlating them to the surface area (given in Figure 3 a) by utilizing correlation derived from literature data (19).

As presented in Figure 3 b, there exist a clear difference between content of binder and fines fraction among collected samples. The conventional dust/asphalt ratio (i.e. fines passing 0.063 mm) for the MD was $9.9\%/6.1\% = 1.62$ and for the SA (surface area)/binder $1.65/6.1 = 0.27$. As all measured values were higher, this indicates that mixture was dry and did not have enough binder. The SA/binder ratio correlated better with the performance than the actual dust/binder ratio.

Nevertheless, sample K containing more of fines of similar surface area to those investigated from areas A and B, was observed to originate from the good performing area. The origin of good mechanical behavior of the sample was thought to be assigned to its increased binder content in comparison with those found for all the bad samples (6.0%).

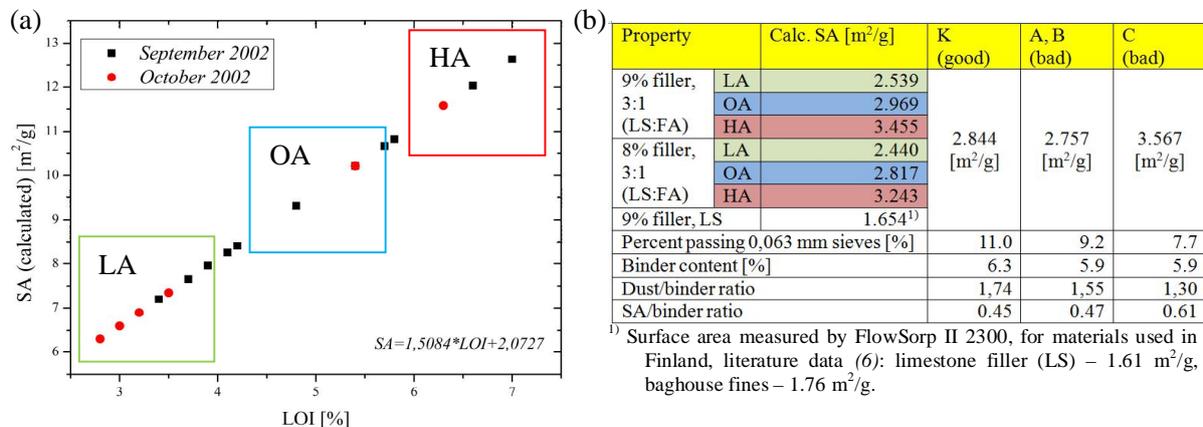


FIGURE 3 (a) Correlation between LOI values to surface area (squares mark the range of values used in (b). LA – low average, OA – overall average, HA – high average), (b) SA results (samples K, AB, C) compared against simulated scenarios, in which SA of the filler blend depends on the amount of FA and it's the foreseen value of LOI

Scanning Electron Microscopy (SEM) analysis

With a prediction of FA presence in the filler, we set off to verify findings by investigating minus 0.063 mm fractions size distribution. As mentioned before, increased hydrophobicity of the samples and partial floatation prevented hydrometer analysis. Minus 0.063 mm fraction after extraction was additionally exhausted in vacuum (2.5 kPa) at room temperature, for a period of 30 minutes. Such prepared powder was mounted on the microscope stub using copper-carbon tape. Image analysis was conducted with JEOL JSM-840 SEM.

Dominating shape within particle size of 100 μm was assigned (20) to the coarse aggregate (Figure 4 (a)). As magnification increased (Figure 4 (b)), the dominant particle size became spherical, ovoid, amorphous or fractured spherical, which was assigned to the particulates originating from the FA.

Exclusion of hydrated lime as an additive

As discussed above, observations from binder extraction, supported by review of QA documentation, suggested that SMA16 mixture may contain FA instead of LS filler specified by the MD. Hydrated lime (HL), which is a high surface area powder commonly used in road construction, was a second possible additive that might have been used. This hypothesis was investigated as follows. First, the presence of limestone filler or lack of thereof was established via solubility testing and verified by TGA and XRD. Surface area measurements were conducted to exclude the overflow of low surface area aggregate fines and to confirm the addition of high surface area component. CaO content could not be established according to EN459-2 due to presence of heavy metals. A methodology adapted from SFS-EN 196-2 indicated CaO content at 19.49%, which is lower than expected for the MD (32.43%) or the SSIM in which HL would be used. XRD was applied and it confirmed lack of calcium hydroxide in fines. Additional optical investigation of particle shape by SEM was conducted and it confirmed the presence of FA rather than HL.

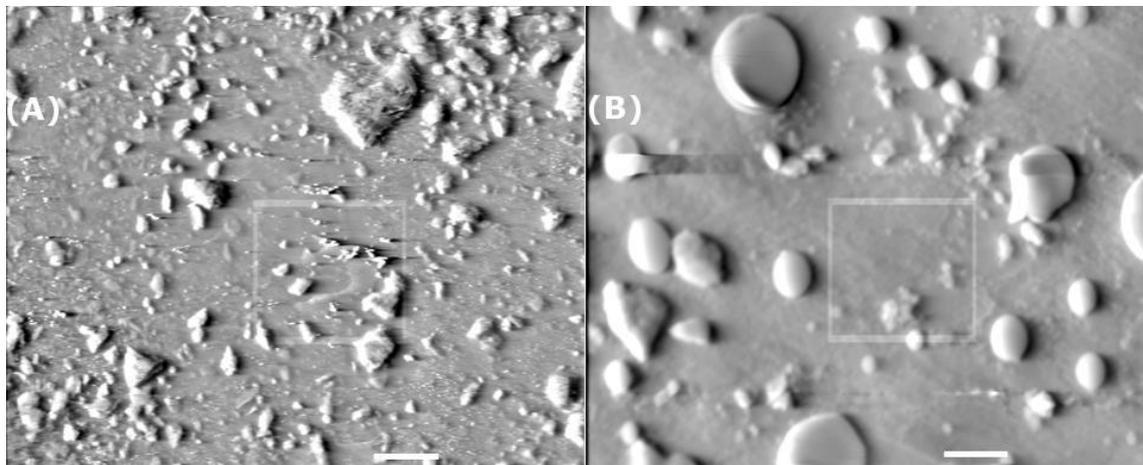


FIGURE 4 Images of Ring-road fines, area E after extraction, fraction <0,063 mm. Legend bar indicates distance of (A) 100 µm and (B) 10 µm respectively

FT-IR analysis of reconstructed fines passing 0,125 mm sieves

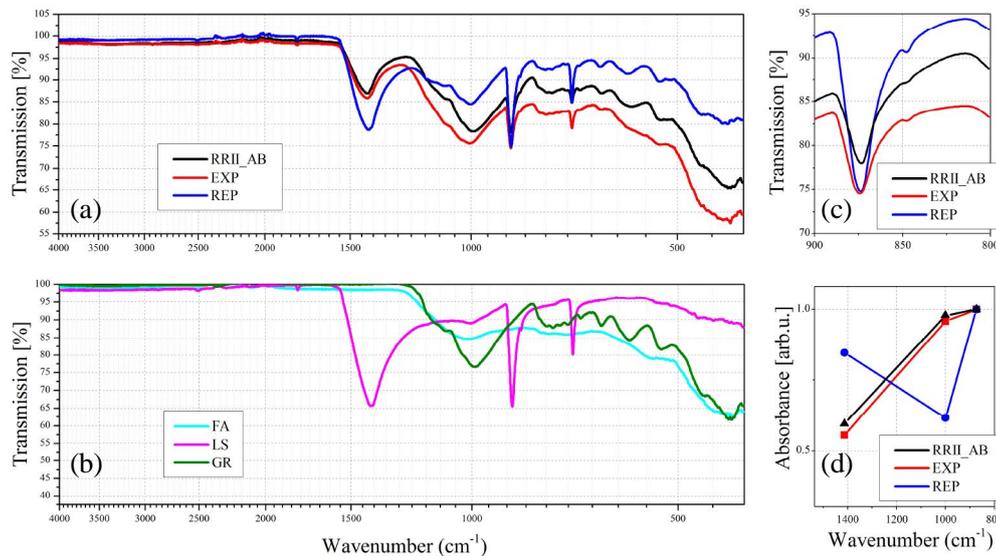
In order to provide reference database, raw materials used constituting of granite (GR) specified in the MD, limestone filler (LS, HAST=91.4%) and fly ash (FA, LOI=5.2%), all passing 0.125 mm sieve, were analyzed. These raw materials were then blended in order to reconstruct the actual composition of material passing 0.125 mm sieve. A filler blend according to QA reports (proportion of 71.74 : 28.26 by weight of LS:KK) designated as **REP** and a filler blend according to findings of forensic study (52.04 : 28.01 : 19.96 by weight LS:GR:FA) designated as **EXP** were prepared. Results were compared to fines of RRII from location AB (RRII_AB).

Fourier Transform Infra-Red spectrometer (Nicolet iS50 Analytical equipped with GladiATR Attenuated Total Reflectance accessory, diamond crystal) was used to collect spectra of the dried powders in ATR mode, between the wavelength $4000-400\text{ cm}^{-1}$, with 32 scans per sample. No correction was used during measurements (temperature was at $25-30^{\circ}\text{C}$) and results are presented in Figure 5.

Analysis of LS stayed in agreement with literature (21) and characteristic carbonate's peaks were found at $711, 872, 1005$ and 1404 cm^{-1} . For the GR, wide absorption band with three

1 distinctive peaks at 989, 1091 and 1134 cm^{-1} , along 646 cm^{-1} weak peak was assigned to quartz
 2 (22) and other silicates. The peaks at 722, 743, 760 and 776 cm^{-1} were found characteristic for
 3 albite (23) mineral. The far-IR region, defined in here as 700-400 cm^{-1} , still captured strong
 4 peaks at 419, 527, 587 and 693 cm^{-1} , which are assigned in this work to the presence of iron
 5 oxides (24, 25). All of the above was consistent with known mineralogy of GR(26). The FA
 6 expressed absorbance in similar regions as the GR with less distinctive peaks for silica (1166,
 7 1008, 664), albite (727, 750, 772, 793) and heavy metal oxides (421, 529, 541, 550, along
 8 additional multiple peaks in the region 400-520 cm^{-1}). Smoother spectra are observed for silicon
 9 oxide in glass form (amorphous) as opposed to crystal form (23). The FA region characteristic
 10 for silica can be convoluted with signals coming from unburnt carbon residue and carbonates,
 11 thus clear assignment is not achieved in this work. However, spectra collected for the FA stays in
 12 agreement with literature (20).

13 The spectra collected for mixtures were consistent with the raw material spectra, yet
 14 slightly shifted (REP: 712, 873, 1001, 1412; EXP: 712, 874, 1003, 1415; AB: 712, 873, 996,
 15 1414). Spectra were compared in terms of relative ratios of peak heights, at 874, 1000 and 1414
 16 cm^{-1} , by normalization in absorbance mode to the value at 874 cm^{-1} .



17
 18 **FIGURE 5 FT-IR spectra of fines passing 0.125 mm sieve, where (a) are mixtures**
 19 **and (b) are raw materials; window (c) increases resolution on the wavelength characteristic**
 20 **for carbonates in mixtures and (d) normalized absorbance for mixtures at characteristic**
 21 **wavelength**

22
 23 Fines reconstructed according to the forensic study findings discussed in this article
 24 (EXP) matched with the extracted construction material's FT-IR spectra (sample AB) better than
 25 the REP of mix design. Normalized spectra of the EXP and RRII_AB in respect of chosen
 26 wavelengths are almost identical (see Figure 5 (d)).

27 28 DISCUSSION

29
 30 Hydrochloric acid solubility tests indicated lower than designed content of LS filler in
 31 fines fraction (<0.063 mm). Calculations indicated the use of limestone filler conforming to filler

1 specification of calcium carbonate content above 75%, CC75 (1). Surface area measurements
2 indicated the use of filler component of vast surface area. Exclusions by XRD and TGA, along
3 measurement by SEM confirmed the presence of FA in the fines. We confirmed the findings by
4 analyzing reconstructed fine material passing 0.125 according to both construction records and
5 above results. Material was analyzed by FT-IR with ATR, proving that proposed methodology
6 was adequate.

7 The proposed methodology, in which filler is analyzed from the point of view of multiple
8 properties – chemical composition (HAST, TGA, XRD, FT-IR), surface area, particle shape
9 (SEM), is an advance in pavement engineering. Preceding practices have involved staff
10 interviews and construction site inspections (8). Both become problematic when investigating an
11 over decade old pavement. The data obtained from analytical testing in the forensic stage
12 compared against the composition simulations were sufficient enough to provide us with exact
13 information on fines' composition, allowing us to move on to the reconstruction stage.

14 In terms of fault, it must be stated that the construction contemporary specifications were
15 not clearly defined for upcoming materials, such as blends of LS and FA. However, use of filler
16 other than the one denoted in the MD requires additional material characterization for fines (1) or
17 alternatively demonstration of suitability by performance-based MD, this being an override
18 clause. Neither of the above was found from documentation.

19 Furthermore, results of this investigation indicate that, on top of filler substitution, 8% or
20 less of altered filler was used (MD was optimized for 9% LS filler.). Found binder content was
21 on average 0.1% lower than that of the MD and gradation analysis indicated problems with
22 dust/binder ratio.

23 Additionally, better performing parts of the road were found to have slightly higher
24 binder content (0.2-0.4%) than specified in the MD or established for the bad areas, respectively.
25 As reported by (7), use of higher surface area filler in the same MD increases rut resistance but
26 decreased fatigue resistance. However, use of overly fine filler in place of designed material (8),
27 along other alterations to the MD during construction, may cause premature failures in asphalt
28 surface layer. Substituting (9) LS for other materials changes mixtures cracking characteristics.
29 Additionally, increase of the silica content in fines increased mixture moisture susceptibility (11,
30 27). It was proven for RRII that a change not only in chemical composition but also in shape
31 characteristics of the filler occurred. This additionally affects mastic and mixture properties (28).

32 Observations of binder stay in agreement with aforementioned. Authors strongly believe,
33 supported by literature, that insufficient amount of binder was used to meet the criteria of free
34 bitumen (2, 3, 4). It is obvious that contractor failed to take this account. As surface area of fines
35 affects to mastics hardening (10) by reducing free bitumen content, the needed temperature for
36 adequate compaction is expected to rise for higher surface area filler (2, 3, 4). Problems in
37 workability may have caused the higher air voids content (29) due to handling issues, leading to
38 more pronounced aging (14) manifested in the SARA fractioning results and DSR. The observed
39 brown color of mastic can be explained by bitumen constituent separation hypothesis, when
40 fractions responsible for lubrication (saturates and aromatics) are found missing (14).

41 Further warranted research will focus on quantifying the effect of such substitution in
42 terms of mixture workability and aging, as well as developing a new MD framework based on a
43 fingerprinting material characterization, which better takes account the origin and nature of raw
44 materials in the paving industry.

45 46 CONCLUSIONS

1
2 A reason of pavement inconsistency was found to originate in alteration of filler
3 composition between the step of mix design and construction. A foreign element added into the
4 limestone filler was found to be fly ash. Abandonment of adjustment stage led to increased
5 mastic viscosity, resulting in higher air voids content in laid pavement surface layer. This led to
6 increased susceptibility to moisture damage, potholes and fatigue cracking.

7 Further investigations presented in this article found concentration of limestone filler in
8 the range between 4.7% and 6.9%, which supports an assumption that dosing of particular mix
9 components during production was cyclical and subject to error.

10 Hydrochloric acid solubility test (HAST) as a cheap and easily executable technique,
11 proved to be an attractive preliminary stage test in forensic analysis of failed pavements. Data
12 collected with it, provided a hypothesis necessary in the troubleshooting to be verified by more
13 expensive analysis techniques such as XRD, TGA, BET-SA and SEM. Results from those
14 techniques were compared against scenarios and allowed us to reconstruct the fines passing
15 0.125 mm. Comparative analysis of reconstructed and field samples, using FT-IR with ATR,
16 found a filler match and verified results.

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