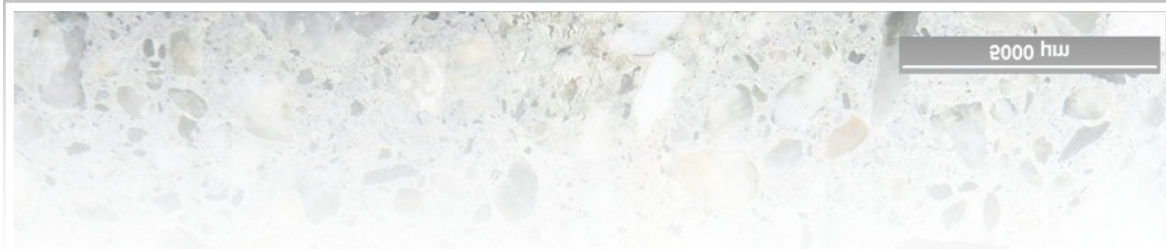
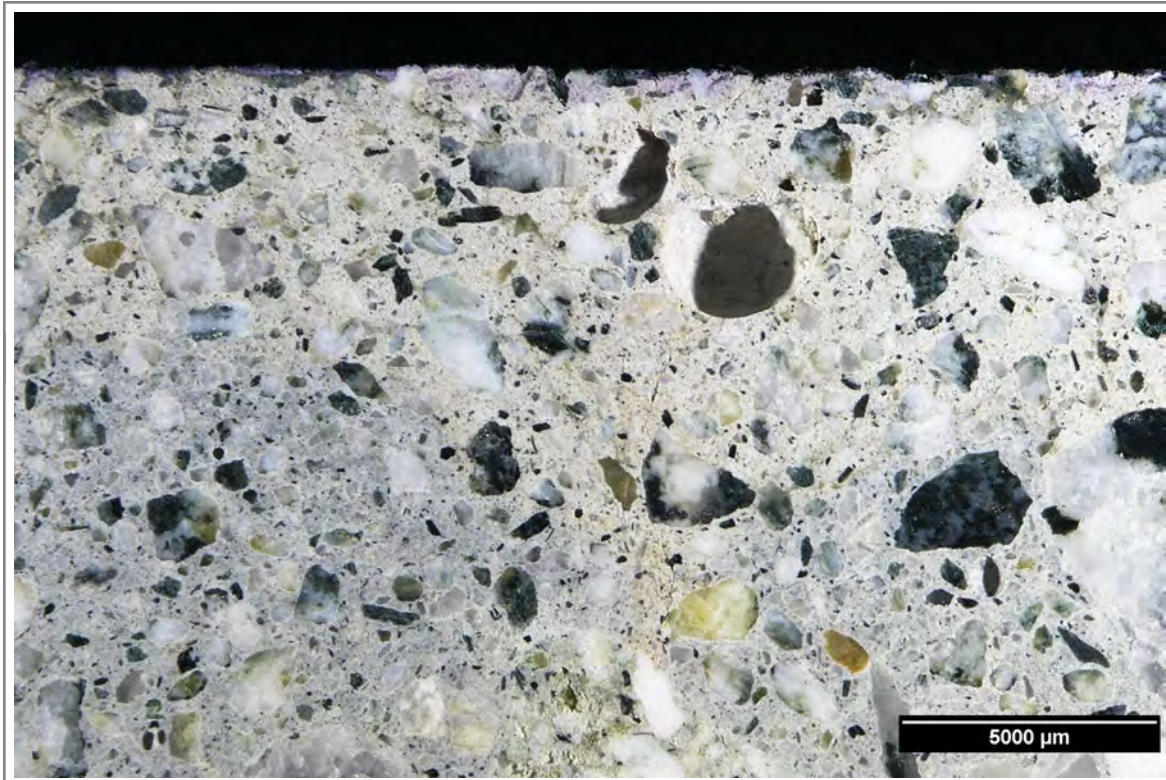


Petrographic Investigation of One Concrete Core Extracted from a Slab-on-Grade near the Moss Landing Harbor located in Moss Landing, California



Prepared for

Craig Hargis
Arelac, Inc. d.b.a. Fortera.
251 East Hacienda Avenue, Suite B
Campbell, California 95008

Prepared by

Meredith Strow
Report No. 207527.d
9 October 2020

EXECUTIVE SUMMARY

One (1) concrete core extracted vertically from a slab-on-grade near the Moss Landing Harbor in Moss Landing, California is subject of petrographic examination per ASTM C856, quantitative x-ray diffraction (QXRD), and scanning electron microscopy with energy dispersive x-ray spectroscopy (SEM/EDS) per ASTM C1723. The purpose of the investigation is to characterize the composition and condition of the concrete represented by the core as well as to quantify the amount of vaterite (CaCO_3) in the concrete.

The findings from this scope of work indicate that the concrete is of good quality and is in good condition. The concrete core was received hard and intact. No cracks are observed. The core contains a couple vertical shrinkage microcracks in the top 5-12 mm ($\frac{1}{8}$ - $\frac{1}{2}$ in.) of the core. Such microcracks are common in concrete and do not affect the performance of the slab.

The concrete consists of crushed quarried stone coarse aggregate with a 12.5 mm ($\frac{1}{2}$ in.) nominal top size and natural sand fine aggregate bound in a non-air-entrained portland cement and fly ash paste. Both the coarse aggregate and fine aggregate are siliceous in composition. Total air content is less than 3%. The paste exhibits low capillary porosity and the estimated water-cementitious materials ratio (w/cm) is 0.40 ± 0.05 . The depth of paste carbonation is 4-6 mm ($\frac{1}{8}$ - $\frac{1}{4}$ in.) from the top surface. Other than paste carbonation, no secondary deposits are observed.

No evidence of finishing issues, inadequate curing, or deterioration associated with service conditions or progressive deterioration mechanisms related to durability was observed. No cracks or notable microcracks are present within the core. No evidence of alkali-aggregate reactions, such as alkali-silica reaction (ASR) or alkali-carbonate reaction (ACR), is observed.

Quantitative x-ray diffraction (QXRD) analysis was conducted on a $1 \frac{1}{4} \times \frac{1}{2}$ in. subsample taken from the core. Results show that the concrete is comprised of 30% quartz, 40% feldspar (Ca-Na), 20% calcite, 5% kaolin, 3% gypsum, 1-2% vaterite, and 2% amphibole and ettringite; percentages are in weight percent (wt. %). The quartz, feldspar, kaolin, gypsum and amphibole fractions are associated with the aggregates. The calcite, vaterite, and ettringite fractions are associated with the paste fraction.

SEM/EDS analyses performed on a polished coupon and a fractured surface did not reveal the presence of distinct vaterite particles at magnifications up to 12,000x. Although particles of calcium carbonate were detected by EDS analysis, the particles did not show the orbicular or raspberry-like morphology typical of vaterite particles before they are used in the production of portland cement concrete.



1.0 INTRODUCTION

Mr. Craig Hargis, of Arelac, Inc. d.b.a. Fortera (**AF**) located in Campbell, California, requested **DRP**, A Twining Company (**DRP**) to perform petrographic examination per ASTM C856, quantitative x-ray diffraction (QXRD), and scanning electron microscopy with energy dispersive x-ray spectroscopy (SEM/EDS) per ASTM C1723. A subsample of the core was also tested by quantitative x-ray diffraction (QXRD) to quantify the amount of vaterite in the concrete. The purpose of the investigation is to characterize the composition and condition of the concrete represented by the core as well as to quantify the amount of vaterite (CaCO_3) in the concrete.

On 28 August 2020 **DRP** received one (1) core from **AF** designated as Moss Landing Slab Core A6-28. The core was assigned **DRP** sample number 24YD10810.

AF reported the following information to **DRP**: The slab-on-grade was placed approximately 10 years ago and is located approximately 300 feet from the Moss Landing Harbor in the Moss Landing Commercial Park. The concrete reportedly contains a cementitious mixture of 70% portland cement, 15% fly ash, and 15% vaterite. The vaterite portion consisted of 90% vaterite and 10% calcite. Vaterite can react with calcium and alumina in the portland cement and fly ash to form hemicarboaluminate and monocarboaluminate. It can undergo a through-solution dissolution-precipitation process to form a new calcium carbonate phases like calcite or aragonite. Provided secondary EDS images of vaterite particles show an orbicular or raspberry-like shape; these images were of vaterite particles, not vaterite within concrete, and were taken with field emission microscope.

2.0 SCOPE OF WORK

The testing performed at **DRP** involved petrographic examination per ASTM C856, quantitative x-ray diffraction (QXRD), and scanning electron microscopy with energy dispersive x-ray spectroscopy (SEM/EDS) per ASTM C1723. This report contains the general findings from the scope of work described above. *Appendix A* contains the notes, photographs and micrographs from the petrographic examination. *Appendix B* contains the results of SEM/EDS analyses. *Appendix C* contains the results of QXRD testing from another laboratory. *Appendix D* describes the procedures used to perform this scope of work.

3.0 FINDINGS

The following findings are relevant to the concrete represented by the core. The referenced figures are in *Appendix A*.

- 3.1 The core is vertical in orientation and measures ~44 mm (1 ¾ in.) in diameter and 152 mm (6 in.) in length (**Figures A1 and A2**). The top surface is a flat, worn concrete surface with fine aggregate exposed in low relief. The bottom surface is a rough, uneven concrete surface with adhered gravelly sub-base such that the core represents a full thickness of the slab.
- 3.2 No reinforcement is present within the core.
- 3.3 No cracks are present in the core. No notable microcracks are observed. Only few randomly-oriented 25-50 µm (1-2 mil) wide microcracks are observed within the paste in thin section.
- 3.4 The concrete is non-air-entrained; total air content is estimated at less than 3%. Few spherical voids are observed throughout the core (**Figure A3**). The concrete is well consolidated with no notable entrapped voids in the body of the core.
- 3.5 The coarse aggregate is a crushed quarried stone that has a 12.5 mm (½ in.) nominal top size (**Figure A4**). The coarse aggregate is siliceous in composition and consists of granodiorite with few other igneous and metamorphic rock types.
- 3.6 The fine aggregate is siliceous in composition and consists of a natural sand that contains feldspar, quartz, pyroxene, amphibole, granite, quartzite, chert, biotite, chlorite and few other rock and mineral particles (**Figure A5**).
- 3.7 Paste is light gray (Munsell Soil Color GLEY 1 8/N) in the top 4-6 mm (⅛ - ¼ in.) and gray (Munsell Soil Color GLEY 1 6.5/N) in the remainder of the core (**Figures A6 and A7**). Paste throughout the core is hard (Mohs 3.5-4.0). The paste exhibits a smooth texture on the polished surface. The paste-aggregate bond is moderately tight; when struck with a geology hammer in the petrographic laboratory, the concrete fractures through ~75% of coarse aggregate particles (**Figure A8**). As observed in thin section, the paste consists of portland cement and fly ash (**Figure A9**). The paste exhibits moderately low capillary porosity and an estimated water-cementitious materials ratio (w/cm) is 0.40 ± 0.05 (**Figure A10**).
- 3.8 The paste is carbonated in the top 4-6 mm (⅛ - ¼ in.) of the core (**Figure A11**). Other than carbonation, no other secondary deposits are observed.
- 3.9 QXRD results show that the concrete is comprised of 30% quartz, 40% feldspar (Ca-Na), 20% calcite, 5% kaolin, 3% gypsum, 1-2% vaterite, and 2% amphibole and ettringite. Percentages are in weight percent (wt. %) (see *Appendix B*).
- 3.10 SEM/EDS analyses performed on a polished coupon and a fractured surface did not reveal the presence of distinct vaterite particles at magnifications up to 12,000x. Although particles of calcium carbonate were detected by EDS analysis, the particles did not show the orbicular or raspberry-like morphology typical of vaterite particles before they are used in the production of portland cement concrete (see *Appendix C*).

4.0 CONCLUSIONS

The findings above indicate that the concrete is of good quality and is in good condition. The concrete core was received hard and intact. No cracks are observed. The core contains a couple vertical shrinkage microcracks in the top 5-12 mm ($\frac{1}{8}$ - $\frac{1}{2}$ in.) of the core. Such microcracks are common in concrete and do not affect the performance of the slab.

The concrete consists of crushed quarried stone coarse aggregate with a 12.5 mm ($\frac{1}{2}$ in.) nominal top size and natural sand fine aggregate bound in a non-air-entrained portland cement and fly ash paste. Both the coarse aggregate and fine aggregate are siliceous in composition. Total air content is less than 3%. The paste exhibits low capillary porosity and the estimated water-cementitious materials ratio (w/cm) is 0.40 ± 0.05 . The depth of paste carbonation is 4-6 mm ($\frac{1}{8}$ - $\frac{1}{4}$ in.) from the top surface. Other than paste carbonation, no secondary deposits are observed.

No evidence of finishing issues, inadequate curing, or deterioration associated with service conditions or progressive deterioration mechanisms related to durability was observed. No cracks or notable microcracks are present within the core. No evidence of alkali-aggregate reactions, such as alkali-silica reaction (ASR) or alkali-carbonate reaction (ACR), is observed.

Quantitative x-ray diffraction (QXRD) analysis was conducted on a $1 \frac{1}{4} \times \frac{1}{2}$ in. subsample taken from the core. Results show that the concrete is comprised of 30% quartz, 40% feldspar (Ca-Na), 20% calcite, 5% kaolin, 3% gypsum, 1-2% vaterite, and 2% amphibole and ettringite; percentages are in weight percent (wt. %). The quartz, feldspar, kaolin, gypsum and amphibole fractions are associated with the aggregates. The calcite, vaterite, and ettringite fractions are associated with the paste fraction.

SEM/EDS analyses performed on a polished coupon and a fractured surface did not reveal the presence of distinct vaterite particles at magnifications up to 12,000x. Although particles of calcium carbonate were detected by EDS analysis, the particles did not show the orbicular or raspberry-like morphology typical of vaterite particles before they are used in the production of portland cement concrete.

This concludes work performed on this project to date.



Meredith Strow
Senior Petrographer

Moss Landing Core Petrography

Appendices

Appendix A	Moss Landing Slab Core A6-28 Petrography (ASTM C856)
Appendix B	Quantitative X-ray Diffraction (QXRD) Report
Appendix C	Scanning Electron Microscopy and Energy Dispersive X-ray Spectroscopy (SEM/EDS) (ASTM C1723)
Appendix D	Procedures

1. RECEIVED CONDITION	
ORIENTATION & DIMENSIONS	Sample is a vertical core measuring ~44 mm (1 3/4 in.) in diameter and ~152 mm (6 in.) in length (Figure A1).
SURFACES	The top surface is a flat, worn concrete surface with fine aggregate is exposed in low relief; no surface finish is observed. The bottom surface is a rough, uneven concrete surface with adhered gravelly sub-base such that the core represents a full thickness of the slab. (see Figure A1)
GENERAL CONDITION	The core was received hard and intact in one piece (Figures A1 and A2).

2. EMBEDDED OBJECTS	
GENERAL	None present.

3. CRACKING	
MACROSCOPIC	None present.
MICROSCOPIC	A couple vertical 50-75 μm (2-3 mil) wide microcracks extend down from the top surface to depths of 5 to 12 mm (1/8 - 1/2 in.) into the core (Figure A3). Only few randomly-oriented 25-50 μm (1-2 mil) wide microcracks are observed within the paste in thin section.

4. VOIDS	
VOID SYSTEM	The concrete is non-air-entrained; total air content is estimated at less than 3%. Few spherical voids are observed throughout the core (Figure A4). The concrete is well consolidated with no notable entrapped voids in the body of the core.
VOID FILLINGS	None present, as observed on the polished surface of the core.

5. COARSE AGGREGATE	
PHYSICAL PROPERTIES	The coarse aggregate is a crushed quarried stone with a 12.5 mm (1/2 in.) nominal top size. Particles are hard and dense, well graded, equant in shape, angular to sub-angular in texture, and uniformly distributed throughout the core (Figure A5).
ROCK TYPES	The coarse aggregate is siliceous in composition. The aggregate consists predominantly of granodiorite with few gabbro particles and metamorphic rocks. No particles are potentially susceptible to alkali-silica reaction (ASR). No carbonate particles are observed; no particles are potentially susceptible to alkali-carbonate reaction (ACR).
OTHER FEATURES	No evidence of ASR or ACR is observed. No coatings are observed.

6. FINE AGGREGATE	
PHYSICAL PROPERTIES	The fine aggregate is a natural sand. Particles are hard and dense, equant to elongated in shape, angular to sub-angular in texture, and uniformly distributed throughout the core (Figure A6).
ROCK TYPES	The fine aggregate consists predominantly of feldspar, quartz, pyroxene, amphibole, granite, quartzite, chert, biotite, chlorite and few other rock and mineral particles. Quartzite and chert are potentially susceptible to alkali-silica reaction (ASR). No carbonate particles are observed; no particles are potentially susceptible to alkali-carbonate reaction (ACR).
OTHER FEATURES	No evidence of ASR or ACR is observed. No coatings are observed.

7. PASTE OBSERVATIONS	
POLISHED SURFACE	Paste in the top 4-6 mm (1/8 - 1/4 in.) is light beige-gray (Munsell Soil Color GLEY 1 8/N). Paste in the remainder of the core is gray (Munsell Soil Color GLEY 1 6.5/N). Paste throughout the core is hard (Mohs 3.5-4.0). The paste exhibits a smooth texture on the polished surface. (see Figure A7 and Figure A8).
FRESH FRACTURE	Freshly fractured surfaces exhibit the same coloration as the polished surface, a granular texture, and a sub-vitreous luster. The paste-aggregate bond is moderately tight; when struck with a geology hammer in the petrographic laboratory, the concrete fractures through ~75% of coarse aggregate particles (Figure A9).
THIN SECTION	The paste contains portland cement and fly ash (Figure A10). The amount of residual portland cement is estimated at 5-10% (by volume of paste). The amount of calcium hydroxide is estimated at less than 5% (by volume of paste). Calcium hydroxide is fine grained. Paste is carbonated to a depth of 4-6 mm (1/8 - 1/4 in.) from the top surface.
ESTIMATED W/CM	The paste exhibits moderately low capillary porosity and the w/cm is estimated at 0.40 ± 0.05 (Figure A11).

8. SECONDARY DEPOSITS	
PHENOLPHTHALEIN	After phenolphthalein was applied to a cross-sectional saw-cut surface of the core, the paste throughout the core stained dark purple except for the top 4-6 mm (1/8-1/4 in.). (Figure A12); this testing is performed to determine general depth of paste carbonation.
SECONDARY DEPOSITS	Other than carbonation, no additional secondary deposits are observed.

FIGURES

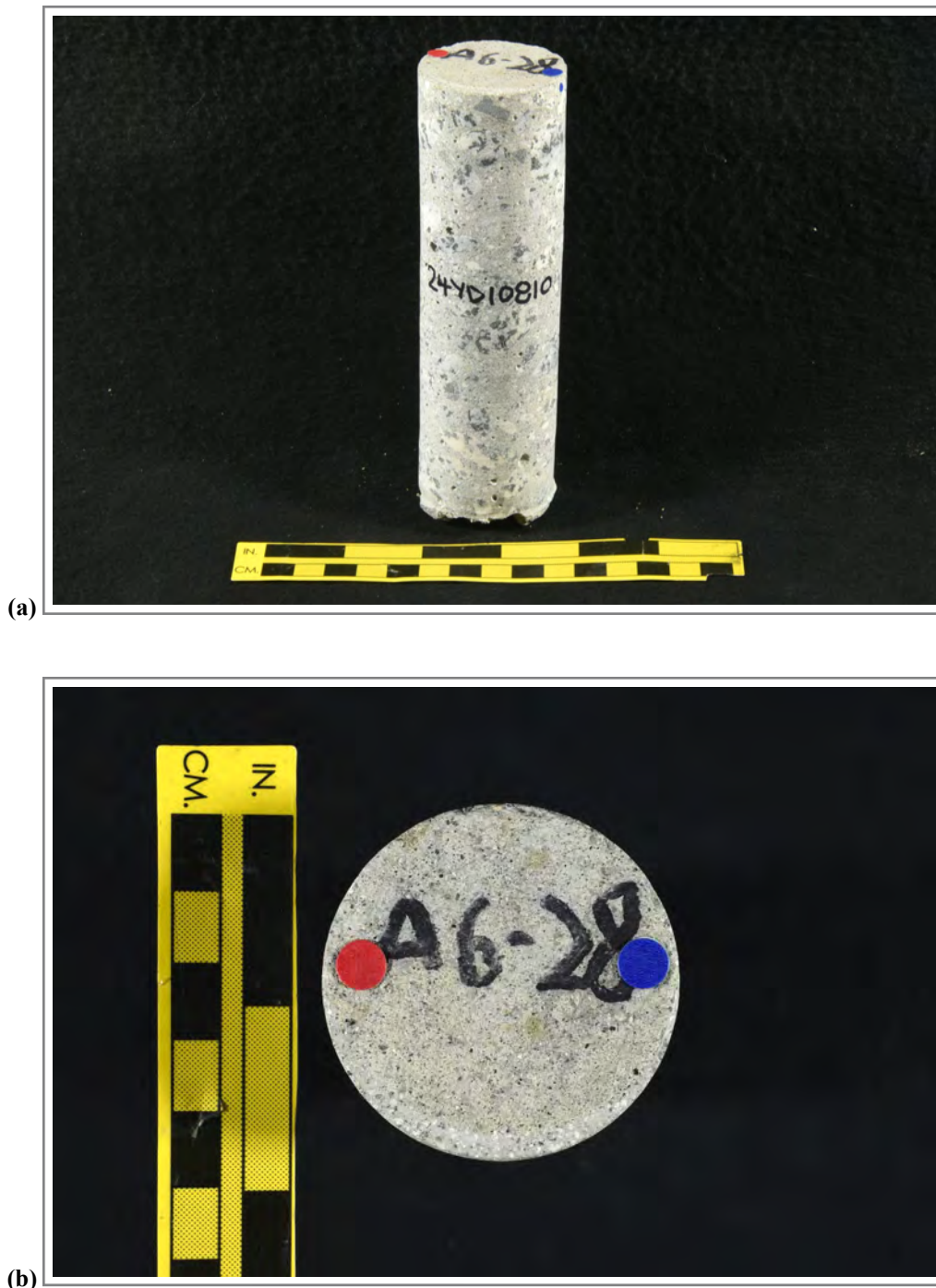


Figure A1. Photographs of the core in as-received condition showing (a) an oblique view of the top surface and side of the core with identification labels and (b) the top surface of the core. The red and blue dots show the orientation of the saw cuts used to prepare the core. The yellow scale is ~ 150 mm (6 in.) long; the small and large divisions are in centimeters and inches, respectively.



(c)



(d)

Figure A1 (cont'd). Photographs of the core in as-received condition showing (c) the side of the core and (d) the bottom surface of the core. The yellow scale is ~ 150 mm (6 in.) long; the small and large divisions are in centimeters and inches, respectively.

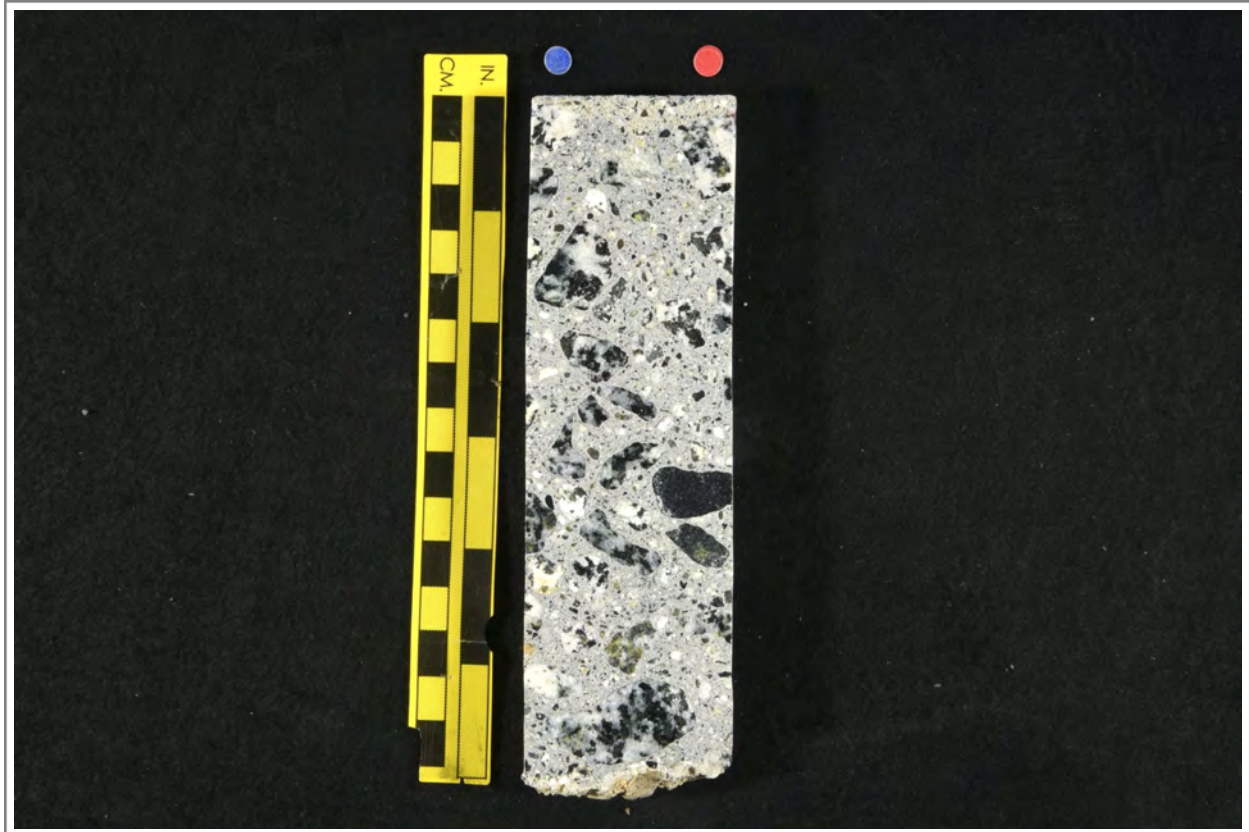


Figure A2. Photograph showing the polished surface of the core. The small and large divisions on the yellow scale are in centimeters and inches, respectively.

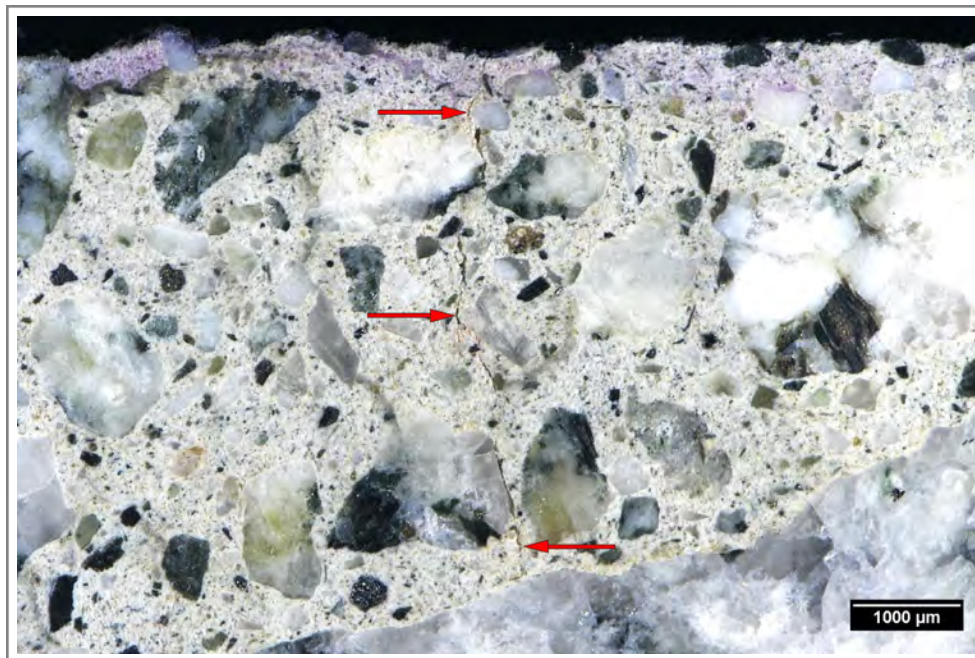


Figure A3. Reflected light photomicrograph showing vertical microcrack (red arrows) in top portion of core.

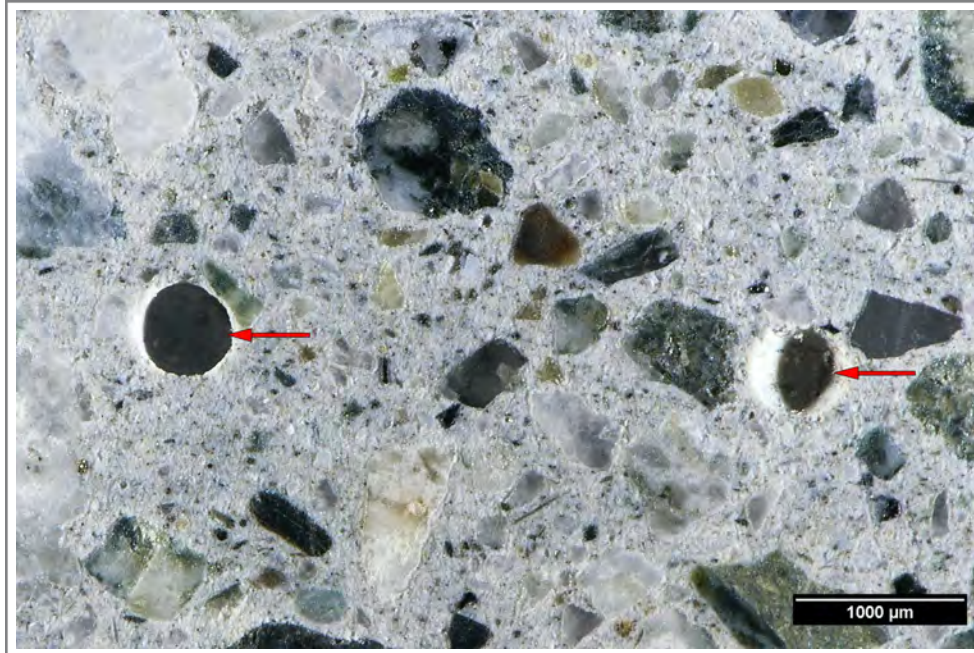


Figure A4. Reflected light photomicrograph showing numerous air voids (red arrows) throughout the paste.

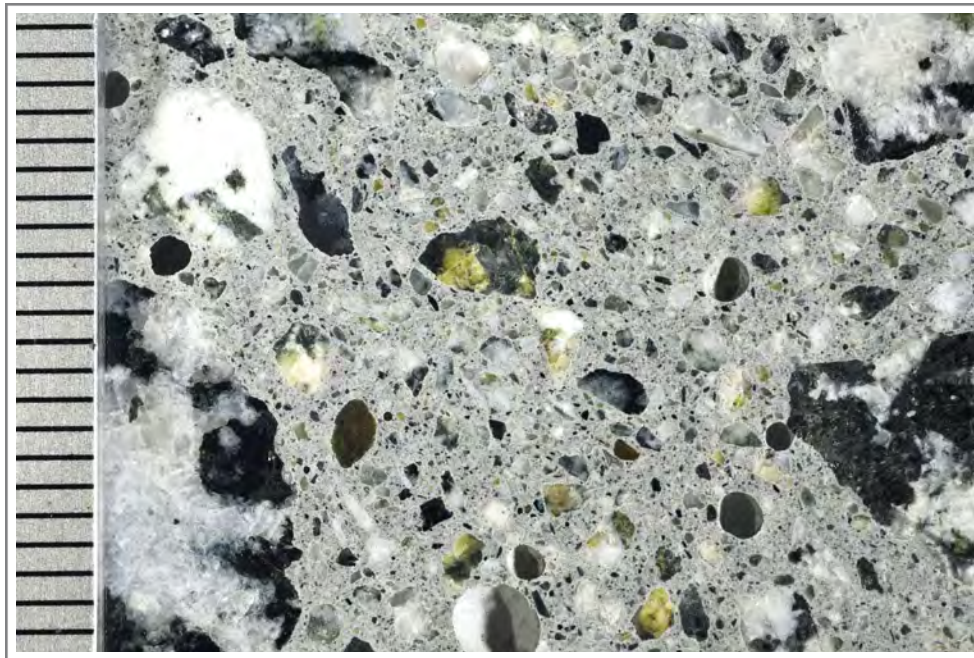


Figure A5. Photograph of the polished surface showing detail of the coarse aggregate. Scale is in millimeters.

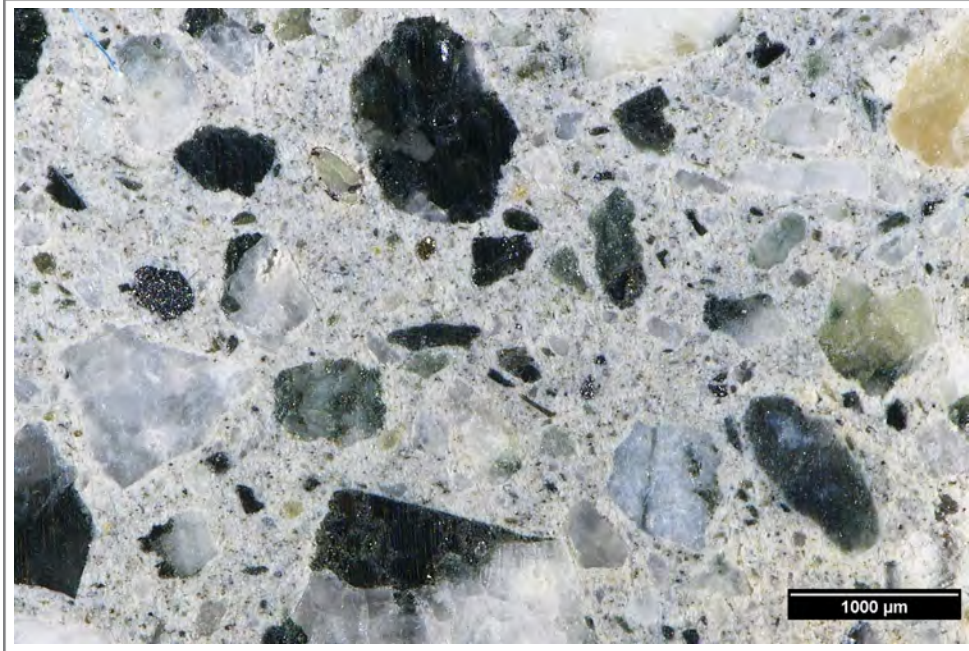


Figure A6. Reflected light photomicrograph of the polished surface showing the fine aggregate.

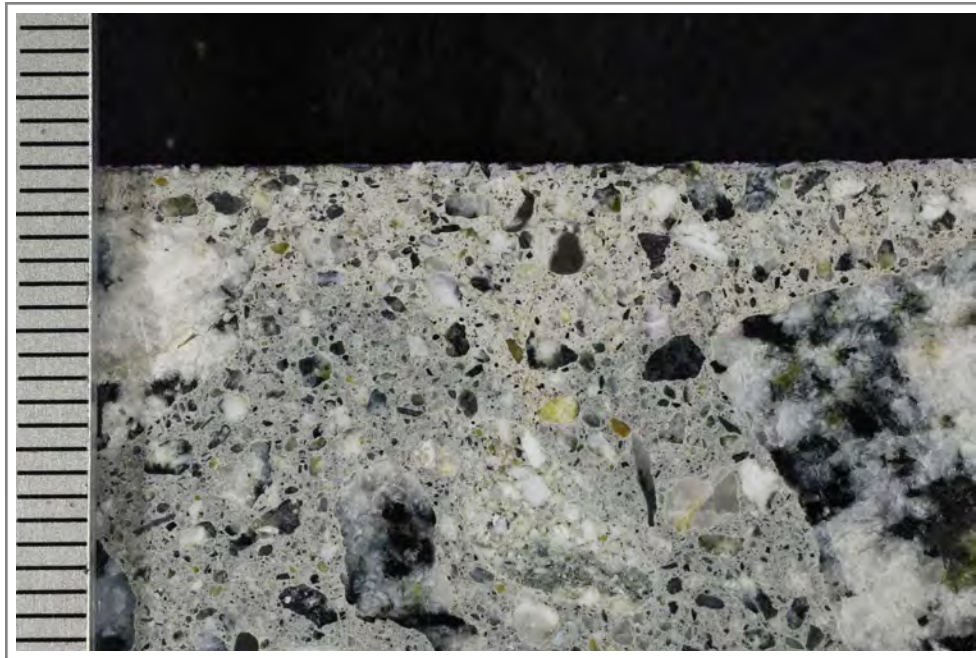


Figure A7. Photograph showing detail of the paste on the polished surface in the top portion of the core. Scale is in millimeters.

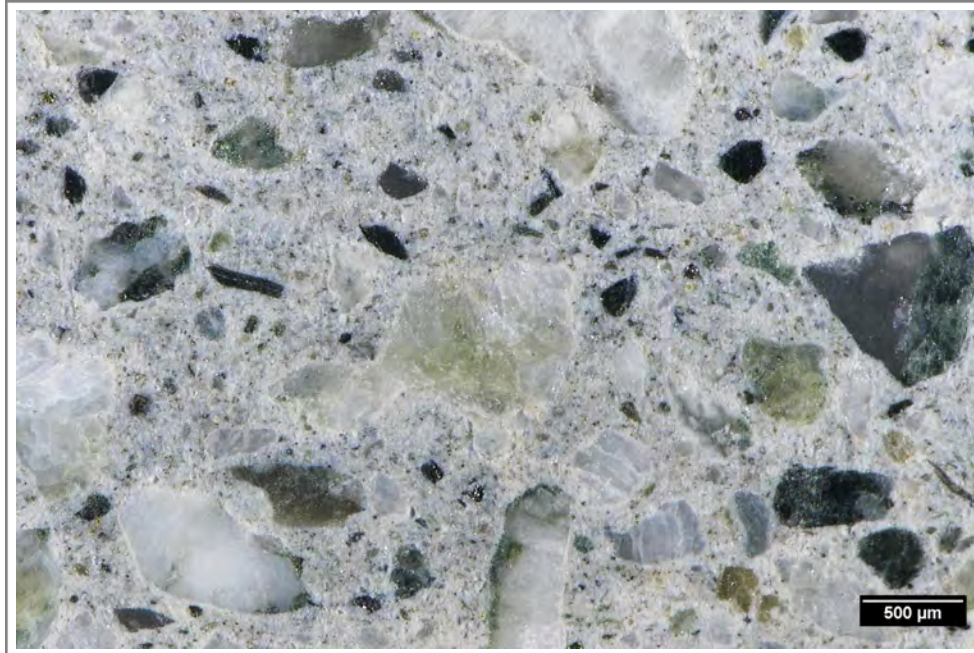


Figure A8. Reflected light photomicrograph showing detail of the paste in the body of the core on the polished surface.



Figure A9. Photograph showing detail of the paste on freshly fractured surfaces. Scale is in millimeters.

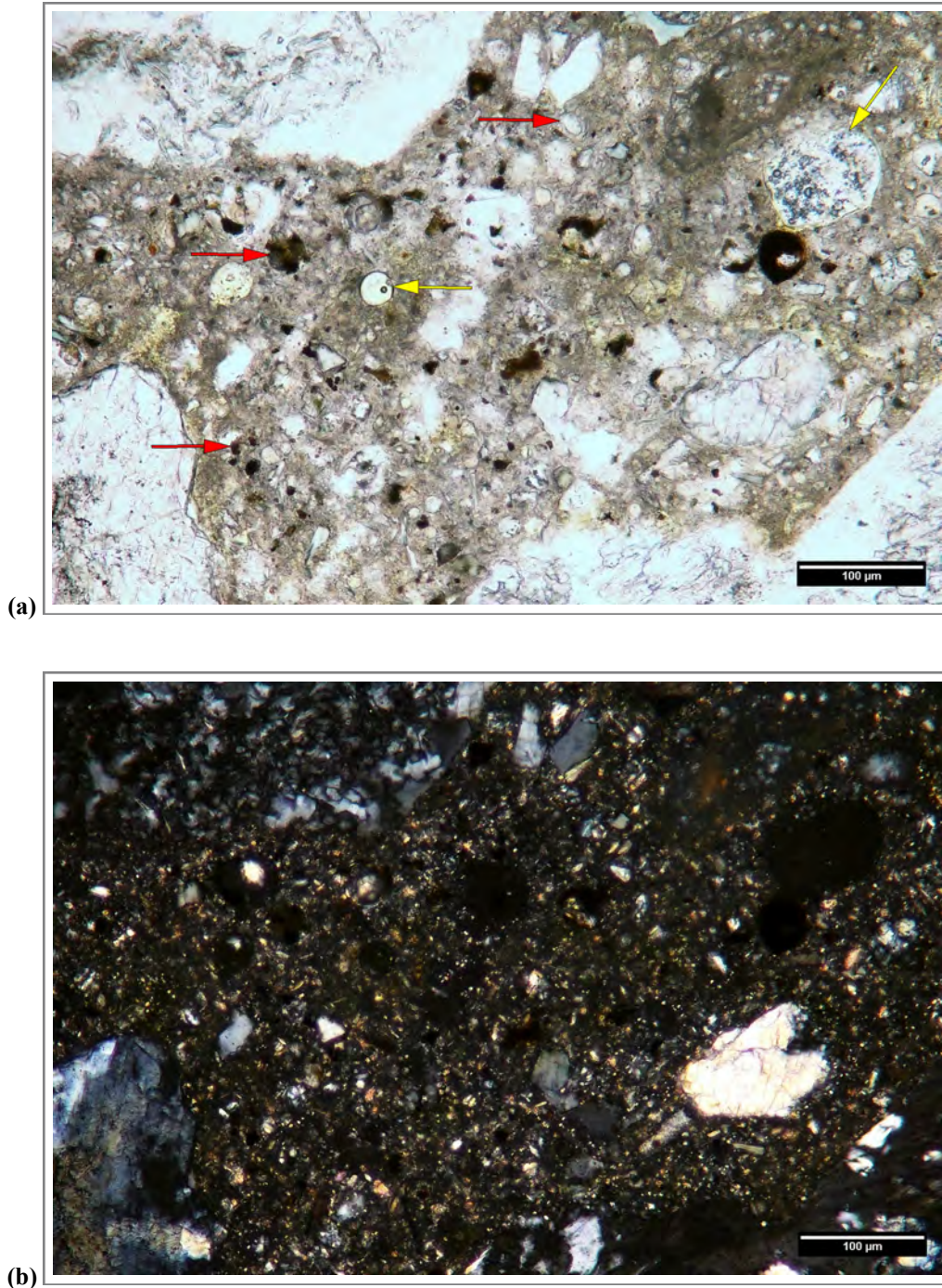


Figure A10. Transmitted light photomicrographs of thin section showing detail of paste in (a) plane-polarized and (b) cross-polarized light. Red arrows indicate portland cement particles and yellow arrows indicate fly ash particles in (a).

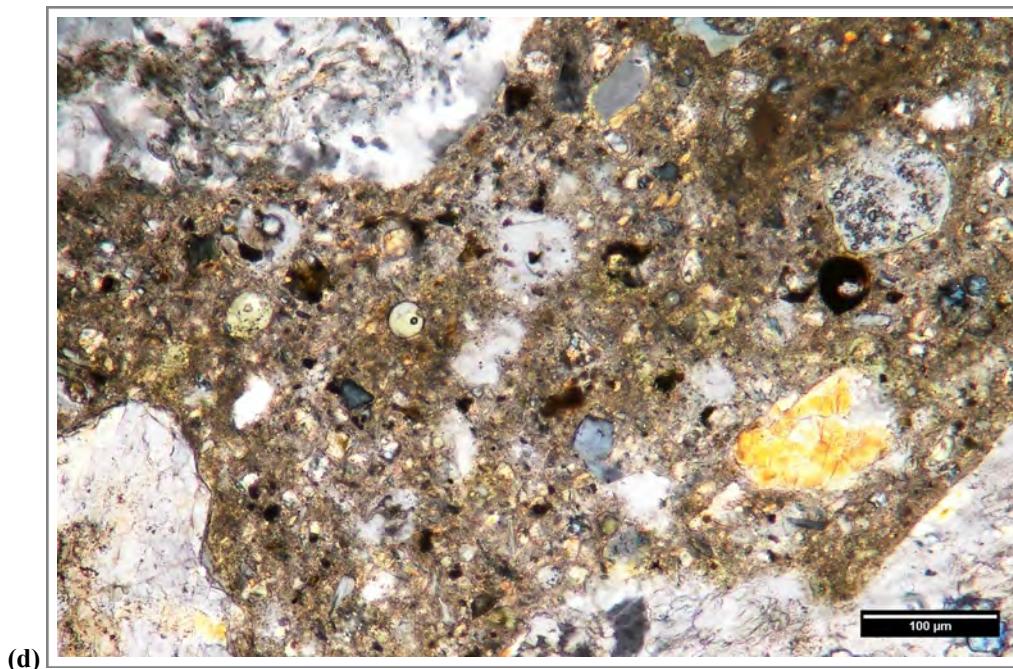
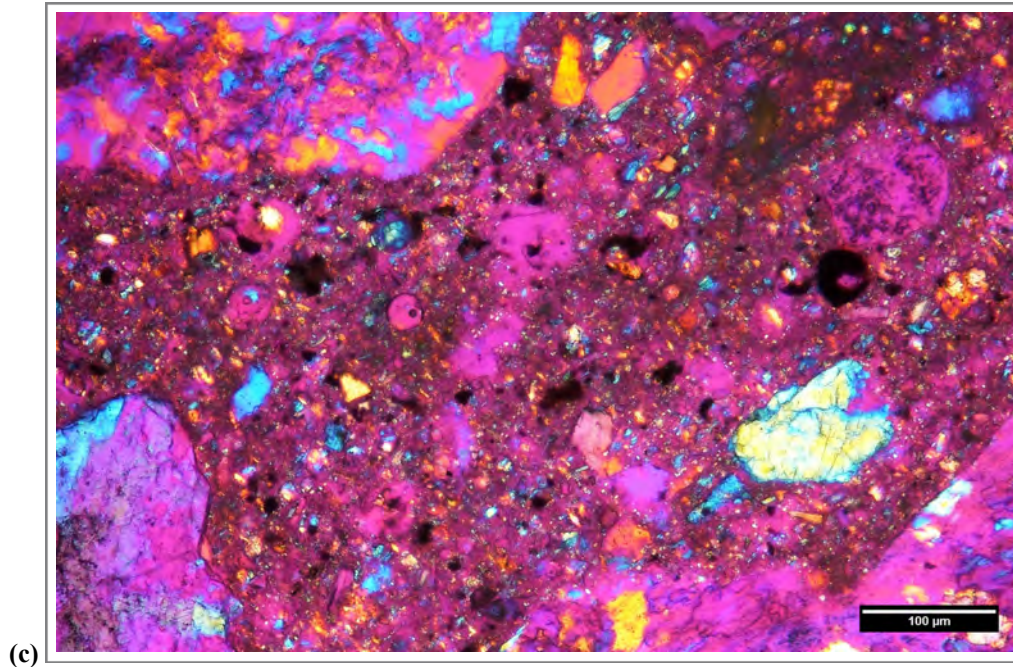


Figure A10 (cont'd). Transmitted light photomicrographs of thin section showing detail of paste in (c) cross-polarized light with the gypsum plate inserted and (d) cross-polarized light with the quarter wavelength plate inserted.

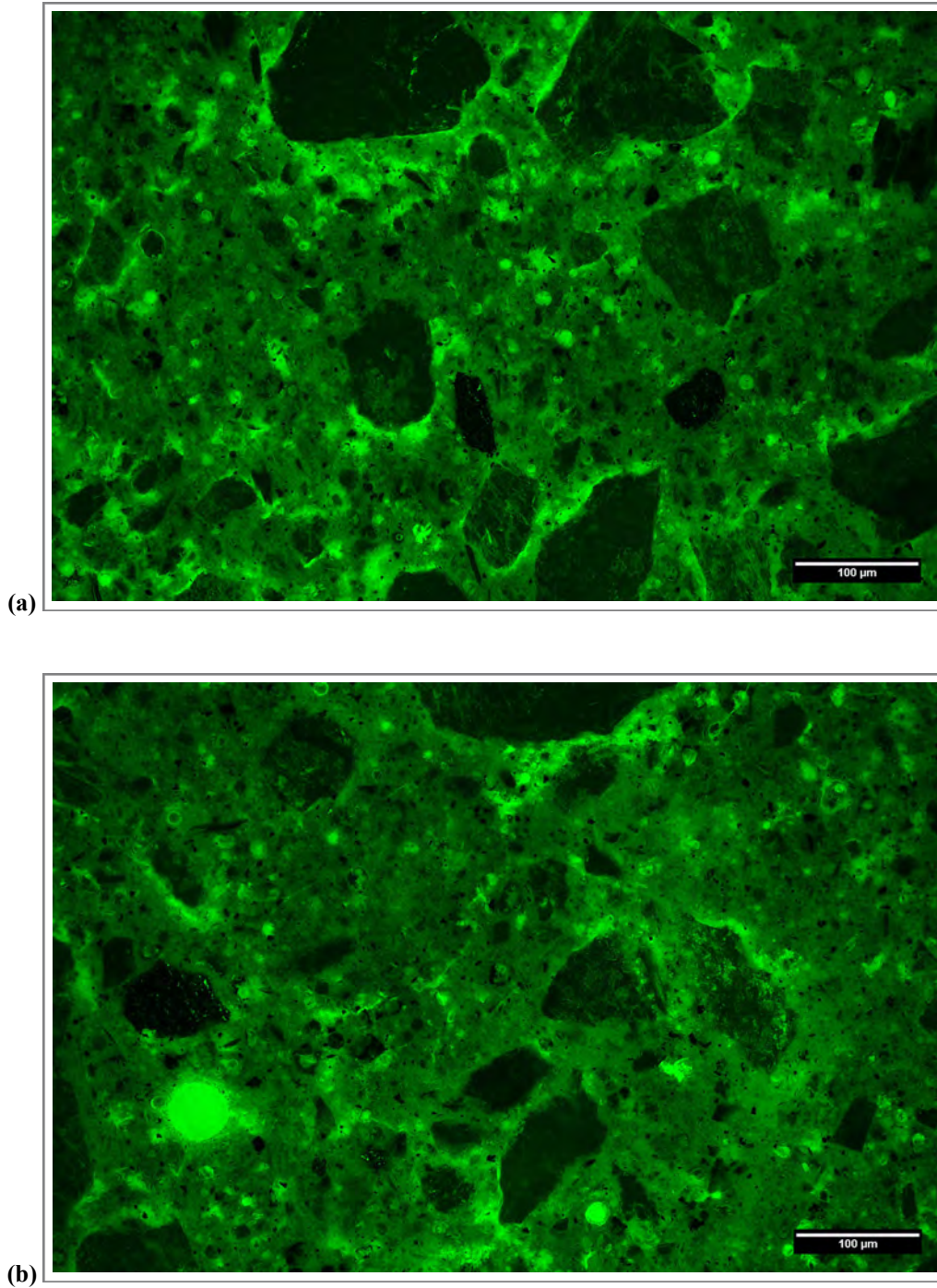


Figure A11. Transmitted light photomicrographs of thin section showing detail of paste in fluorescent light in two different regions of the core. The bright circular areas are voids that have 100% porosity and the black regions are aggregate particles with effectively zero porosity. The variations in green tone between these two end-members reflect variations in the capillary porosity of the paste.

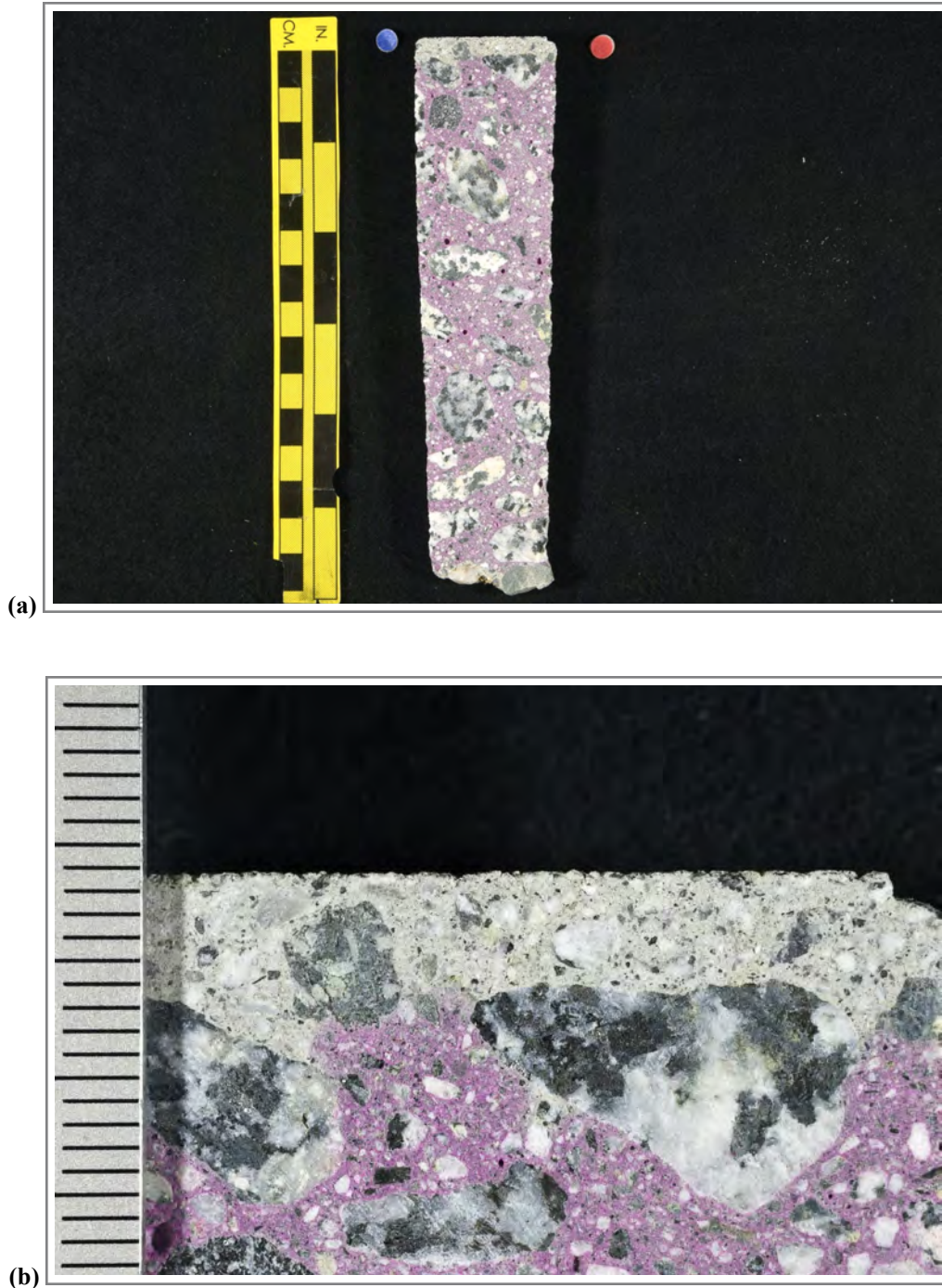
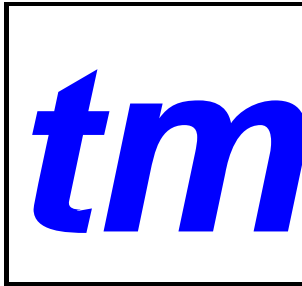


Figure A12. Photographs showing (a) overview of phenolphthalein-stained cross-sectional surface of the core, and (b) detail of stained surface at the top of the core. The yellow scale in (a) is ~150 mm (6 in.) long; the small and large divisions are in centimeters and inches, respectively. The scale in (b) is in millimeters.



TECHNOLOGY OF MATERIALS

4020 N. Palm Street, # 202
Fullerton, CA 92835

Sam Iyengar Ph.D.
Technical Director

Ms. Meredith Strow
DRP, Twining Company
3200 Carbon Place Ste. 104
Boulder, CO 80301

September 30, 2020

Dear Meredith:

Enclosed please find a report on the XRD analysis of a sample. Please call me if you have any questions or concerns.

Sincerely,

Sam Iyengar

Phone: (714) 446-9227

www.xraydiffrac.com

Cell: (951) 852-4463

X-ray Diffraction Analysis of a Powder Sample

Introduction:

A powder sample was received at the laboratory for analysis. It was requested that the samples be analyzed by X-ray powder diffraction (XRD). It was analyzed by XRD to determine the presence of crystalline components, especially Vaterite. This report summarizes the findings

Materials and Method:

The following sample was analyzed:

1) 24YD10810

X-ray Diffraction (XRD):

Analysis was carried out on a Phillips Diffractometer at 30 Kv and 20 ma using Cu K-alpha radiation and a scintillation detector. Bulk soil sample was run after grinding it to pass through a 325 mesh (44 um) sieve and it was scanned from 2 to 50 degrees two-theta. The resulting patterns collected on a computer were matched with the reference standards for various inorganic minerals stored in the JCPDS database. Semi-quantitative estimation of mineral components was carried out from the peak intensities.

Quantitative analysis was carried out using the **Rietveld method** developed by Young and Wiles (2000). The powder data was refined using the Rietveld Method. The method is a whole pattern fitting least squares technique and uses the entire pattern rather than a limited number of reflections to extract required information. In this method, the observed pattern for each phase is compared with the calculated one (modeled using single crystal structural data), and any differences between the two are minimized by refining structural as well as profile related parameters. Since the method uses all lines, severely overlapping reflections are not a problem.

Results and Discussion:

XRD patterns are shown in the attached Figures 1 and 2. Stick patterns for reference mineral standards from the Powder Diffraction File (PDF) database are also shown. Rietveld plot is shown in Figure 3

Overall Mineralogy:

The mineralogy is shown in Table 1. A semi-quantitative estimation of various minerals is shown. It is accurate to +/- 5 -7 wt. %.

Table 1: Mineralogical Composition (wt. %)

Sample ID	Quartz (SiO ₂)	Ca-Na Feldspars	Calcite (CaCO ₃)	Vaterite (CaCO ₃)	Gypsum	Kaolin	Amphibole & Ettringite
24YD 10810	~30	~40	~20	~1-2	~3	~5	~2

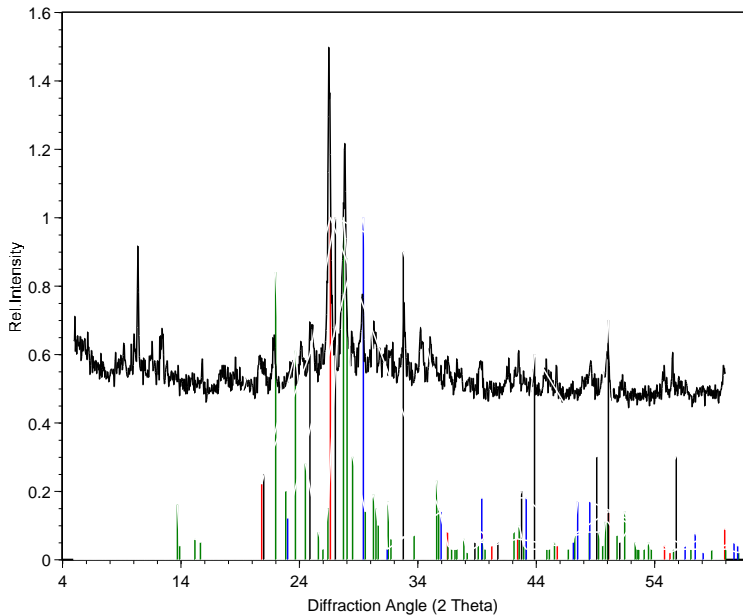


Figure 1: XRD pattern for the **concrete sample** with stick patterns for reference phases: **quartz (red)**, **feldspars (green)**, **calcite (blue)**, and **vaterite (black)**

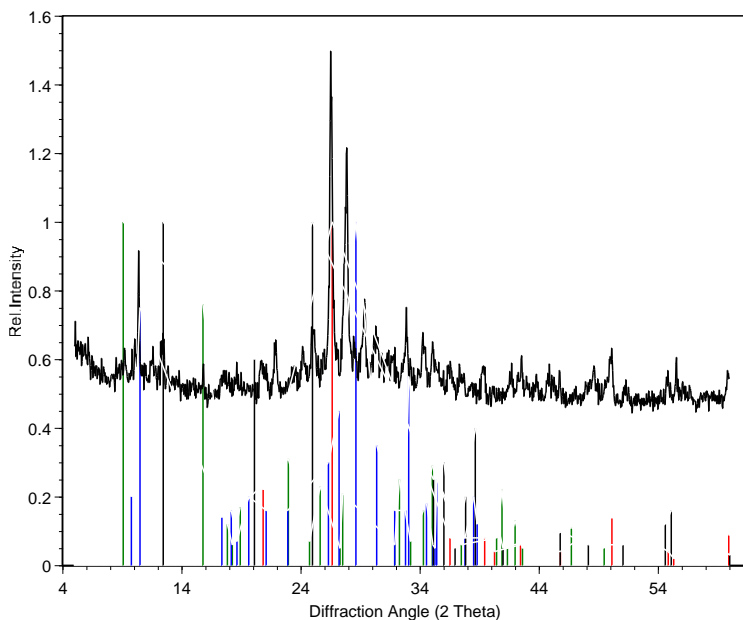


Figure 2: XRD pattern for the **concrete sample** with stick patterns for reference phases: **quartz (red)**, **ettringite (green)**, **actinolite (blue)**, and **kaolinite (black)**

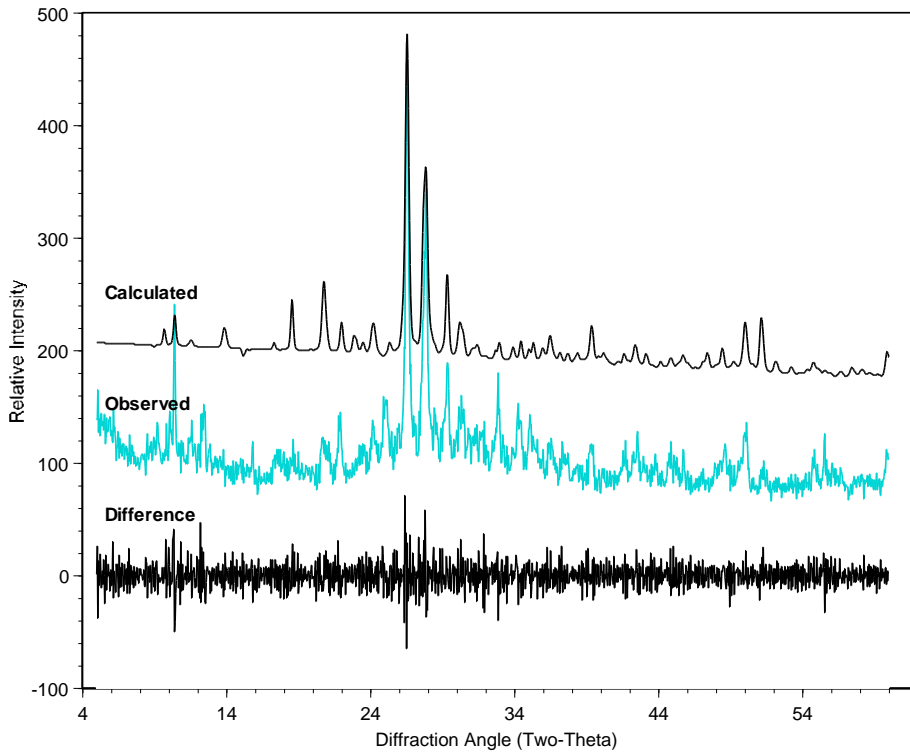
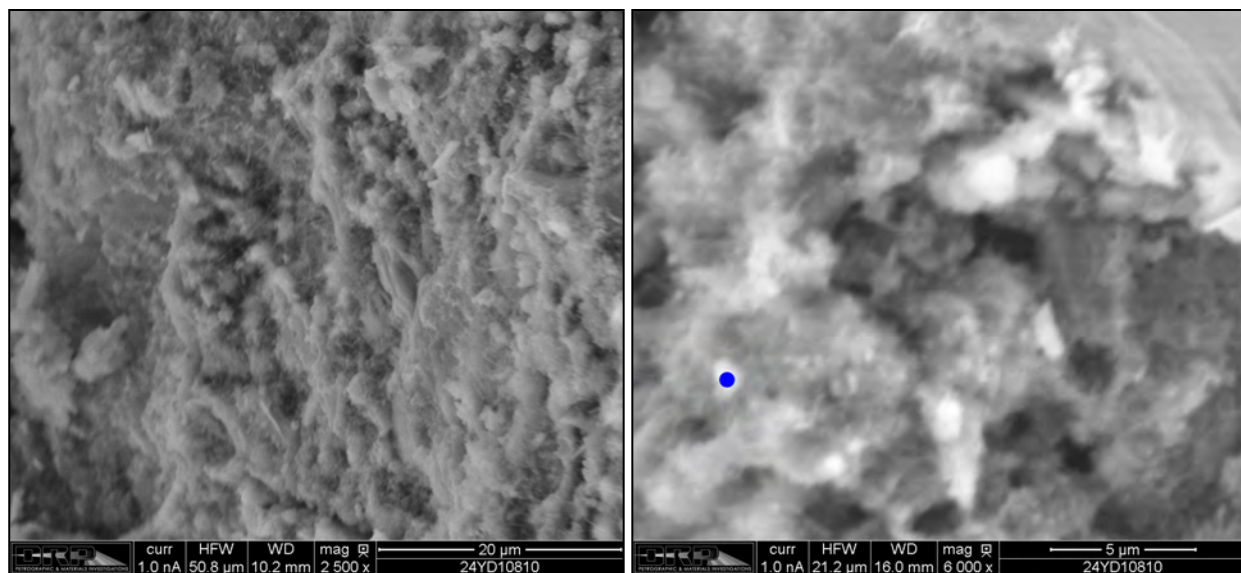


Fig 3: Calculated and observed XRD patterns for **the concrete sample**

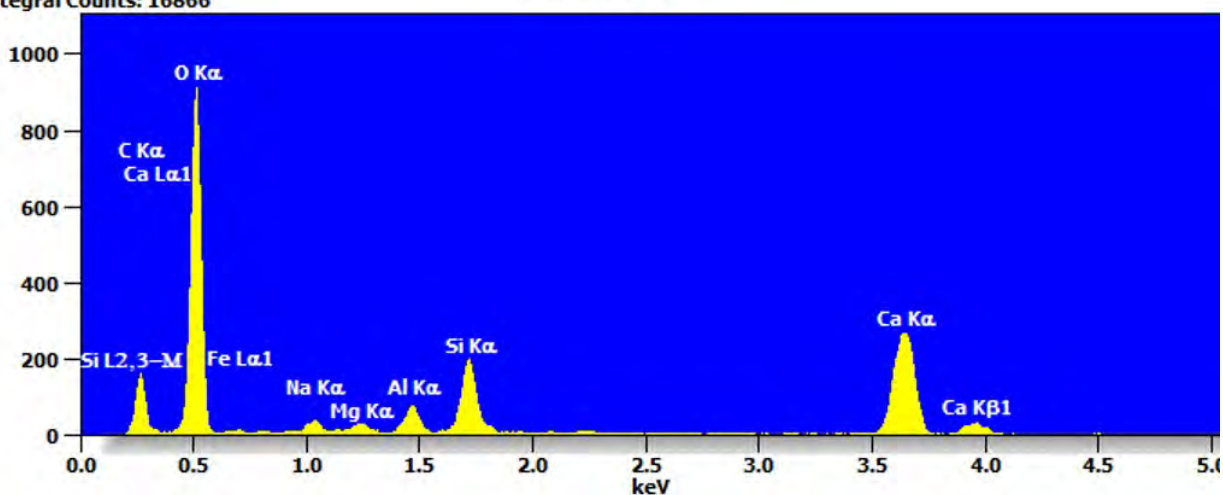


(a)

(b)

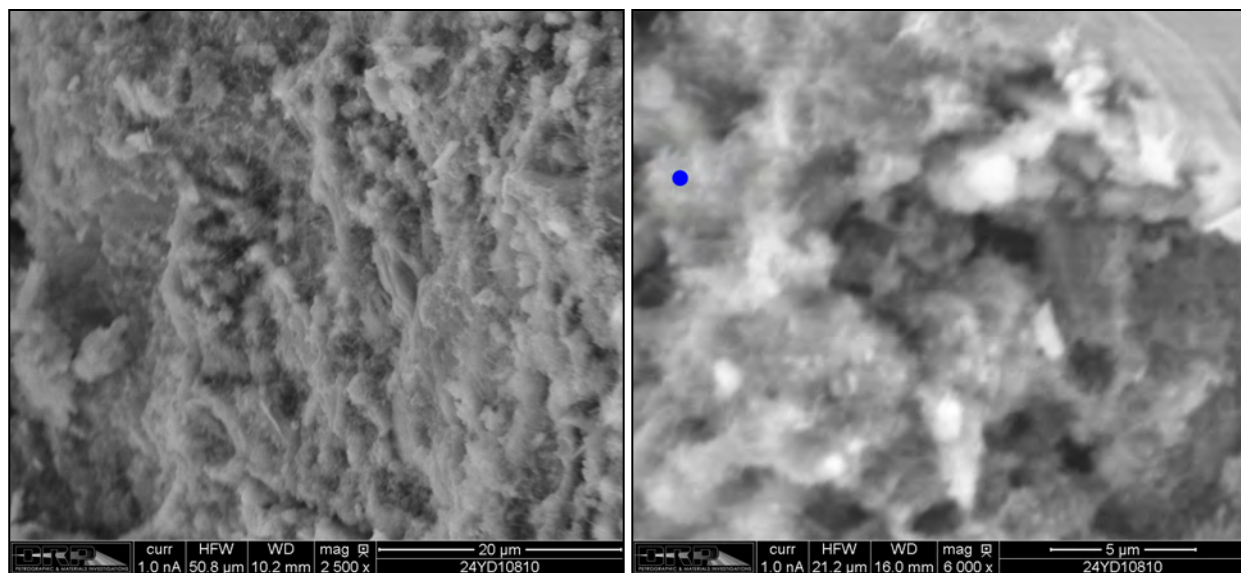
Full scale counts: 920
Integral Counts: 16866

Vaterite(7)_pt1



(c)

Figure C1. (a) Secondary electron micrograph of a fractured concrete surface at 2500x magnification. (b) Secondary electron micrograph of the same region of fractured surface shown in (a) at 6000x magnification with EDS point analysis location indicated by blue circle. (c) Corresponding spectrum from EDS point analysis shown in (b). Note carbon K α peak at ~0.3 keV, oxygen K α peak at ~0.5 keV, and calcium K α peak at ~3.7 keV.

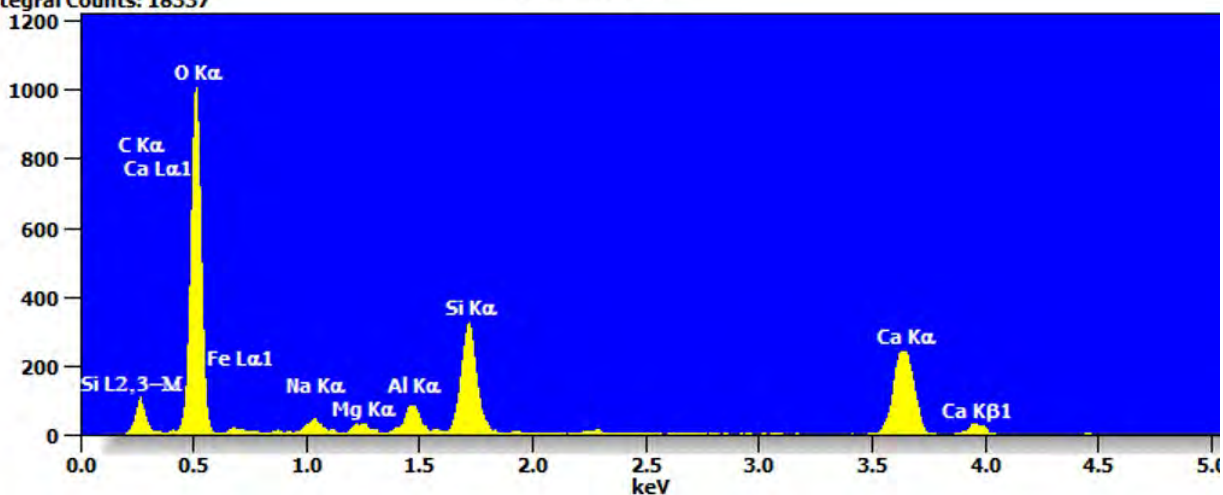


(a)

(b)

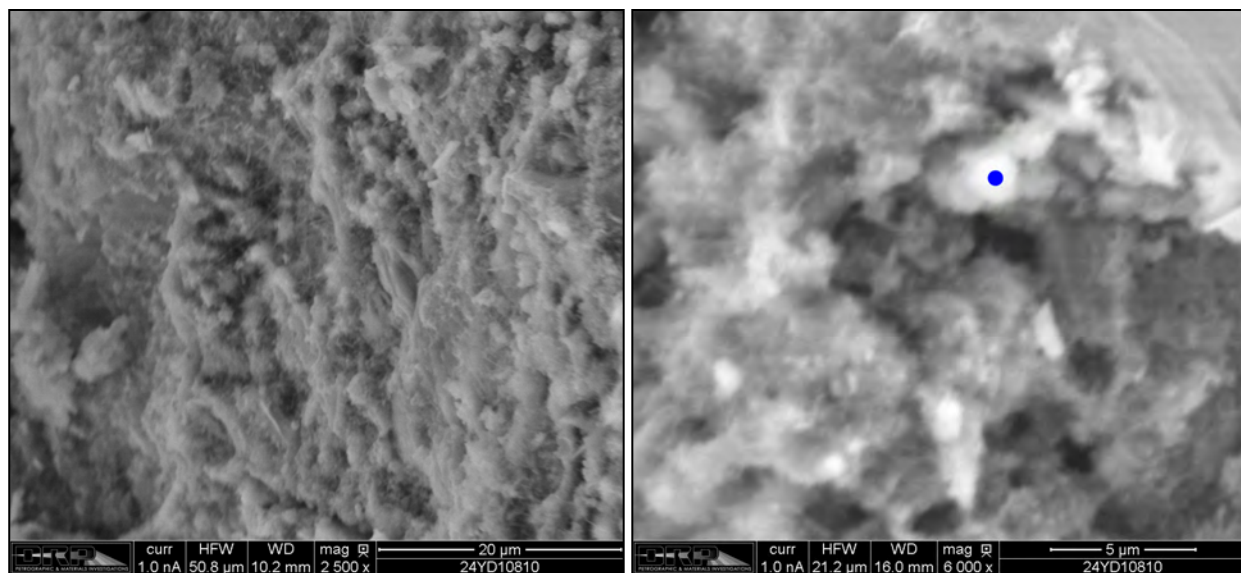
Full scale counts: 1013
Integral Counts: 18337

Vaterite(7)_pt3



(c)

Figure C2. (a) Secondary electron micrograph of a fractured concrete surface at 2500x magnification. (b) Secondary electron micrograph of the same region of fractured surface shown in (a) at 6000x magnification with EDS point analysis location indicated by blue circle. (c) Corresponding spectrum from EDS point analysis shown in (b). Note carbon K α peak at ~0.3 keV, oxygen K α peak at ~0.5 keV, and calcium K α peak at ~3.7 keV.

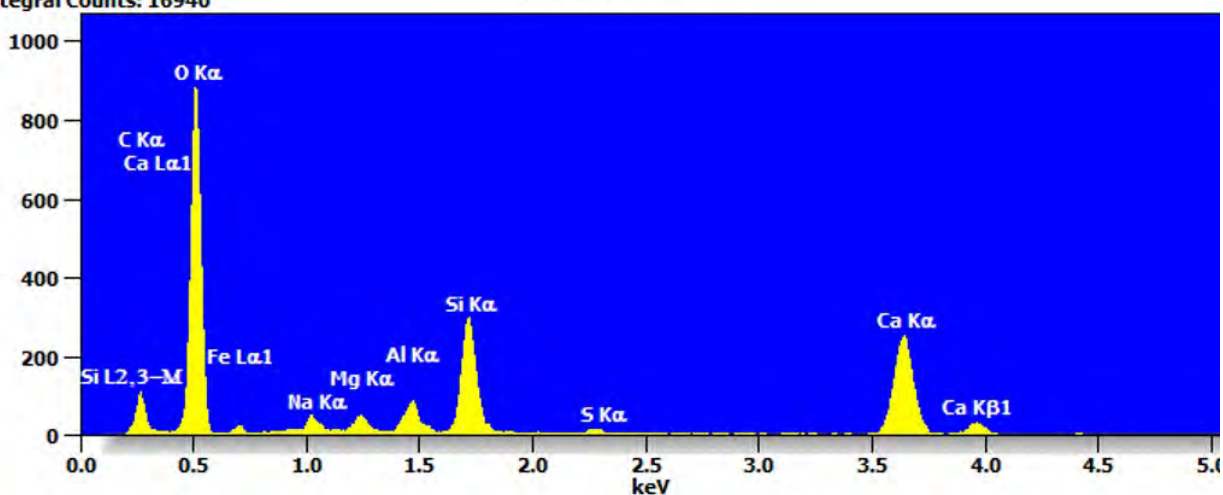


(a)

(b)

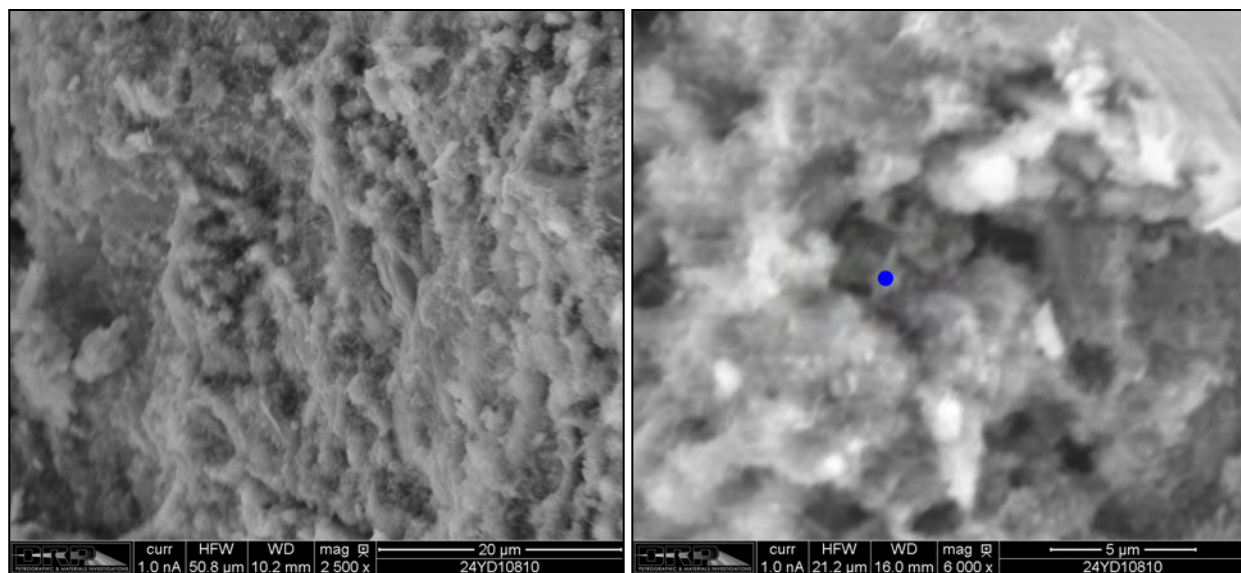
Full scale counts: 890
Integral Counts: 16940

Vaterite(7)_pt4



(c)

Figure C3. (a) Secondary electron micrograph of a fractured concrete surface at 2500x magnification. (b) Secondary electron micrograph of the same region of fractured surface shown in (a) at 6000x magnification with EDS point analysis location indicated by blue circle. (c) Corresponding spectrum from EDS point analysis shown in (b). Note carbon K α peak at ~0.3 keV, oxygen K α peak at ~0.5 keV, and calcium K α peak at ~3.7 keV.

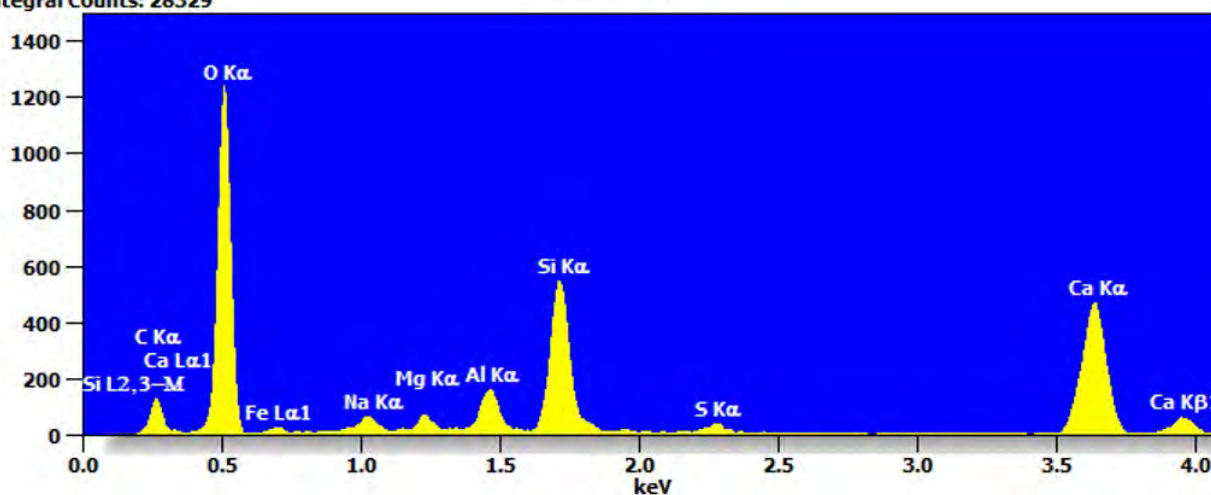


(a)

(b)

Full scale counts: 1243
Integral Counts: 28329

Vaterite(9)_pt1



(c)

Figure C4. (a) Secondary electron micrograph of a fractured concrete surface at 2500x magnification. (b) Secondary electron micrograph of the same region of fractured surface shown in (a) at 6000x magnification with EDS point analysis location indicated by blue circle. (c) Corresponding spectrum from EDS point analysis shown in (b). Note carbon K α peak at ~0.3 keV, oxygen K α peak at ~0.5 keV, and calcium K α peak at ~3.7 keV.

PROCEDURES

Petrographic Examination (ASTM C856) The petrographic work was done following ASTM C856 [1] with sample preparation done at **DRP** in the following manner. After writing the unique **DRP** sample number on each sample near the received label, the samples were measured and inspected visually and with a hand lens. The orientation of the saw cuts used to prepare the samples was then indicated on each sample with blue and red dots. The samples were then photographed in their as-received condition.

A slab representing a longitudinal cross section of each sample was cut from the central portion of the core using a Raimondini Zipper-105®, a 14-inch diameter water-cooled saw. This produced three (3) longitudinal sections for each core. These sections were rinsed and oven dried in a Gilson® Bench Top laboratory oven at ~ 40°C (~ 105°F). After drying, each piece was labelled with the appropriate **DRP** sample number. One piece was set aside for phenolphthalein staining and the other was set aside for thin section preparation.

The central slab was then lapped and polished on a Diamond Pacific® RL-18 Flat Lap machine. This machine employs an 18-inch diameter cast iron plate onto which Diamond Pacific® Magnetic Nova Lap discs with progressively finer grits are fixed. The Nova Lap discs consist of a 1/16 in. backing of solid rubber containing magnetized iron particles that is coated with a proprietary Nova resin-bond formula embedded with industrial diamonds of specific grit. The slab preparation involved the use of progressively finer wheels to a 3000 grit (~4 µm) final polish following procedures outlined in ASTM C457 [2]. An aqueous lubricant is used in the lapping and polishing process. The polished slab from each sample was examined visually and with a Nikon® SMZ-25 stereomicroscope with 3-158x magnification capability following to the standard practice set forth in ASTM C856.

Phenolphthalein was applied to a freshly saw-cut surface from each sample to assess the extent of carbonation, along with thin section analysis. Phenolphthalein is an organic stain that colors materials with pH of greater than or equal to ~ 9.5 purple. Portland cement concrete generally has a pH of ~ 12.5. Carbonation lowers the pH of the paste below 9.5, so areas not stained by phenolphthalein are an indicator of carbonation. The depth of paste not stained by phenolphthalein was measured from each exposed surface.

A coupon measuring approximately 12 x 25 x 25 mm (½ x 1 x 1 in.) was cut from the core and submitted to an external laboratory for quantitative x-ray diffraction (QXRD) testing.

1 *Standard Practice for Petrographic Examination of Hardened Concrete*. Annual Book of ASTM Standards, Vol. 4.02., ASTM C856-18.

2 *Standard Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete*, Annual Book of ASTM Standards, Vol. 4.02, ASTM C457-16.

Scanning Electron Microscopy & Energy Dispersive X-ray Analysis (ASTM C1723) A FEI™ Quanta 250 Environmental Scanning Electron Microscope (ESEM) was used to supplement the petrographic methods described above (ASTM C1723 [3]). The Quanta 250 is capable of operation in three different vacuum modes: high vacuum ($< 6 \times 10^{-4}$ Pa) for conductive or conventionally prepared specimens; low vacuum (10-130 Pa) for non-conductive specimens without preparation; and ESEM™ mode (10-2600 Pa) for specimens such as hydrous materials (cement paste) that are incompatible with high vacuum. The instrument is equipped with several detectors for imaging: an Everhardt Thornley secondary electron detector (SED), a Large Field, Low vacuum SED, a high-sensitivity, low kV solid state backscatter electron detector (BSED), and gaseous SED and BSED for ESEM conditions.

The instrument uses a Tungsten hairpin filament mounted within a tetrode gun assembly and operates with an accelerating voltage of 200 V to 30 kV with beam currents up to 2 μ A. The instrument has a magnification range from 6 to more than 1,000,000 x. The resolution of the instrument is as follows (measured as particle separation on a carbon substrate):

High vacuum mode: 3.0 nm at 30 kV and 8.0 nm at 3 kV for SED
4.0 nm at 30 kV for BSED

Low vacuum mode: 3.0 nm at 30 kV and 10.0 nm at 3 kV for SED
4.0 nm at 30 kV for BSED

Extended vacuum mode (ESEM): 3.0 nm at 30 kV for SED

The Quanta is also equipped with a Thermo Scientific® UltraDry Energy Dispersive X-ray (EDX) Detector. The detector is equipped with a 30 mm² window and has a resolution of 133 eV. The detector is capable of handling input count rates greater than 1,000,000 cps. The detector has a ± 5 eV peak shift (± 3 eV typical between 1% and 60% deadtime) from minimum to maximum count rate at a given analyzer time constant. The detector is capable of detecting all chemical elements down to Beryllium. The EDX has a take-off angle of 35° at a 10 mm working distance.

Sections are prepared for the ESEM/EDX work in the following manner. After visual and microscopic examination of the polished surface used for stereomicroscopy, a coupon measuring ~ 50 mm x 50 mm (2 x 2 in.) in area and ~ 15 mm ($\frac{5}{8}$ in.) in thickness is cut out of one of the hemicylindrical sections from the core. The sample is then lapped and polished using rigid polishing wheels (Buehler Meta-Di Supreme™) embedded with the following sequence of progressively finer grits of industrial diamonds: 165 μ m, 45 μ m, 15 μ m, 9 μ m, 3 μ m and 0.5 μ m. A 0.05 μ m diamond suspension and polishing cloth is used for the final polishing step. None of the abrasive wheels or suspensions contain silicon, aluminum or cesium.

3 *Standard Guide for Examination of Hardened Concrete Using Scanning Electron Microscopy*, Annual Book of ASTM Standards, Vol. 4.02, ASTM C1723-16.