1 **Fiber reinforced mortar affected by alkali-silica reaction: A study by synchrotron**
2 **microtomography**

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18

19 Abstract

20 Alkali-Silica reaction (ASR) is a physicochemical process that can deteriorate concrete and
21 is a recurring engineering problem. In this study three different cylindrical samples affected
22 by ASR were prepared: a plain mortar and two composite mortars containing fibers
23 (polypropylene and a polymer hybrid), which were analyzed at the microtomography
24 (μ CT) beamline 8.3.2 at the Advanced Light Source (ALS). In general, three different
25 features were observed during the 136 day observation period: (1) aggregate dissolution,
26 (2) crack propagation from inside the aggregate, through the cement matrix, and at the ITZ,
27 and (3) the alkali-silica gel filling cracks and voids. In addition, accelerated mortar bar tests
28 were utilized to observe ASR's expansive effect in the plain and composite mortars, and the
29 fibers' ability to restrain expansion due to ASR.

30 **Keywords:** ASR, alkali-silica reaction, synchrotron tomography, polymer fiber, mortar,
31 crack formation

32

33 1. Introduction

34 Alkali-silica reaction (ASR) is a physiochemical deterioration mechanism in
35 Portland cement (PC) based concretes that contain reactive siliceous aggregates. During PC
36 hydration, alkalis are released and enter the concrete pore solution, raising the pH. Silica
37 networks in the reactive siliceous aggregates then begin to depolymerize and form alkali-
38 silica gel. With time and ion migration, the alkali-silica gel begins to produce expansive
39 forces within the concrete matrix. When the expansive forces exceed the tensile strength of
40 the surrounding aggregate and/or PC paste, cracks develop in the concrete. The cracking
41 not only results in reduced strength of the concrete, but opens up pathways for other
42 deleterious agents. For instance, CO_2 can follow the crack path and carbonate the PC
43 matrix, lowering the concrete pH, and chloride ions can migrate along the crack to the
44 imbedded steel reinforcement. Both of which destroy the steel's ability to form a passive
45 layer and protect the reinforcement steel from corrosion. Likewise, oxygen can diffuse
46 along the crack to the reinforcement steel, which facilitates the oxidation of the steel. As
47 the steel corrodes, the reinforced concrete composite loses its ability to carry tensile forces,

48 which leads to repairs being necessary or a reduced service life for the reinforced concrete
49 structure.

50 Many chemical approaches have been developed to mitigate ASR. One of the
51 earliest found solutions was to limit the alkali content in the cement; however, some
52 aggregates are prone to react at very low alkali contents [1, 2]. Another solution is to use
53 pozzolan in the concrete mix, such as fly ash, silica fume, natural pozzolan, or rice husk ash
54 [3-7]. Pozzolans, which contain reactive silica by nature, react with the calcium hydroxide
55 in the PC paste to form additional calcium silicate hydrates (C-S-H). This lowers the
56 porosity of the paste, which hinders ion mobility, slowing ASR's kinetics. Ground
57 granulated blast furnace slag (GGBFS) can also be utilized to mitigate ASR. GGBFS acts
58 to dilute the PC (as do all supplementary cementing materials), thus lowering the calcium
59 and alkali hydroxides released to the hydrated paste from the PC, and it reduces the
60 porosity [8]. Lithium salts have been shown to be very effective at mitigating ASR [9].
61 They can be added to the initial concrete mix to prevent ASR, driven by a potential into an
62 existing structure suffering from ASR, or used to form aggregate passivation layers on
63 aggregates susceptible to ASR [9-12].

64 In addition to the chemical mitigation strategies, physical mitigation strategies have
65 also been developed. ASR needs water to proceed; therefore, one strategy is to limit the
66 water in the concrete pores. Silane coatings have been shown to repel water while allowing
67 vapor to escape from concrete, which lowers the relative humidity or water in the concrete
68 pores [7, 13]. Fibers can be added to the concrete to increase its tensile strength, which can
69 work to keep cracks from forming and to aid in keeping any cracks that do form closed [14-
70 18]. Fiber reinforced polymer (FRP) wraps can be applied to damaged concrete to help
71 restore load bearing capacity.

72 Various models have been proposed for ASR's effect on concrete [19]. Stark et al.
73 [20] give an explanation of the ASR mechanism considering: chemical reaction
74 preferences, crack propagation, stressed aggregate, osmotic pressure, pore distribution,
75 reaction rim formation timing, and silica gel formation. Pignatelli et al. [21] developed a
76 poro-mechanical model with two isotropic internal variables: a) a chemical damage caused
77 by the pressure generated by the expanding ASR gel on the solid concrete skeleton and b) a

mechanical damage associated to the strength and stiffness degradation induced by the external load. Ben Haha et al. [22] utilized image analysis to measure the void and crack content of aggregates affected by ASR, and Dunant and Scrivener [23] built a finite element model linking microscopic damage observations with macroscopic free expansion. Their simulations showed that macroscopic damage and expansion were strongly linked to the microstructural localization of ASR (i.e. aggregate surface, pockets in the aggregate, or homogeneous aggregate expansion). Additionally, they suggest ASR damage is produced in three different regimes: a) the expansion is caused by the cracking of the aggregate and the elastic deformation of the paste, b) both aggregate and paste cracking drive the expansion, and c) the expansion is driven by paste cracking and the exhaustion of reactive materials [24].

The previously proposed models have been supported by experimental work, and most of them used conventional microscopy techniques to investigate features and validate their observations. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are invasive and destructive techniques because the samples have to follow a preparation process before investigating them. Such preparation can modify the morphology and the microstructure of the sample, which can mislead the interpretation. Lately, synchrotron microtomography (μ CT) has become an important non-destructive method of observation in material science. Few investigations on concrete have been carried out, but they demonstrate the capabilities of μ CT for understanding the internal physical behaviors and chemical reactions. For instance, Burlion et al. [25] investigated the distribution and variation of porosity in leached mortar. Gallucci et al. [26] investigated the microstructure of cement paste phases by identifying the grey scale differences at different dates of curing. Finally, Voltolini et al. [27] performed a quantitative characterization of the microstructural features found in ASR affected mortars. Three main kinds of voids due to three different mechanisms were found: a) cracks from ASR expansion, b) irregular shaped voids due to the aggregate particle dissolution and c) bubbles due to the mortar preparation. Using morphological parameters a volume fraction calculation was performed for each kind of void in order to separate each mechanism. However, this work only shows results for samples cured at 27 days, making it difficult to understand how ASR initiates and evolves with time.

This investigation studied at the microscale ASR's effect in plain and fiber reinforced mortars. A plain mortar and two fiber reinforced mortars, using two types of fibers, were observed by μ CT from 2 through 136 days. Crack formation and propagation, aggregate dissolution, and silica gel formation are some of the observed features, which confirm the capabilities of μ CT as non-invasive technique for materials inspection. Additionally, mortar bars were utilized to evaluate the fibers' ability to control ASR induced expansion [28].

2. Materials and Methods

2.1. Materials

A monofilament polypropylene (PP) fiber (approx. 50 μ m in diameter and 13 mm long) and a relatively new hybrid (bi-component) fiber [29] (approx. 17 μ m in diameter and 18 mm long), were used. The hybrid fiber is composed of an ethylene acrylic acid copolymer sheath (EAA $\sim$ 1.7 μ m thick), coaxially extruded with PP fiber core (15 μ m in diameter). Because of its carboxyl functional groups, the EAA sheath is expected to act as a reactive layer, enhancing the adhesion or bonding between the reinforcing PP fiber and the cement matrix [30]. Both fibers were provided by The Dow Chemical Company (DOW®). ASTM Type I PC and a reactive sand, siliceous fine aggregate selected from natural sand deposits with mixed mineralogy, were utilized. In a previous study, the sand was characterized by X-ray fluorescence, X-ray diffraction, and SEM and had been shown to be susceptible to ASR [12]. In bulk the sand, contains 3.3% Na₂O and 4.1% K₂O. The sand is composed of 37% quartz (SiO₂), 1% calcite (CaCO₃), 44% albite (NaAlSi₃O₈), 2% anorthite (CaAl₂Si₂O₈), 2% microcline (KAlSi₃O₈), and 14% orthoclase (KAlSi₃O₈).

2.2. Methods

Two types of experimental approaches were implemented during this investigation. The first was an expansion bar test and the second was the observation of the microstructural effect of ASR by means of μ CT imaging and rendered 3D volumes.

For the expansion test, three specimens of size $2.54 \times 2.54 \times 27.94$ cm (1 in. $\times$ 1 in. $\times$ 11 in.) were prepared for each of the three mixes (a control sample without fibers, a second sample containing PP fibers, and the third sample composed of hybrid fibers) and kept in an accelerated ASR environment, a modified version of ASTM C 1260 was utilized [28]. The test was modified by using a finer sand gradation (38.5% 0.60-1.18 mm, 38.5% 0.30-0.60 mm, and 23% 0.15-0.30 mm) corresponding to the smallest three aggregate sizes specified by ASTM C 1260 renormalized to 100% and lower temperature (50°C). This curing temperature was selected to accelerate the ASR effect without approaching the polymer fiber melting point. The sand gradation was mainly chosen to better accommodate the small μ CT sample molds and to not dominate the μ CT field of view with aggregates. The w/c ratio was set to 0.47, a sand-to-cement ratio of 2.25, and a fiber volume of 1% when mixes contained fibers. The sealed samples were demolded after curing for 24 hours at 21°C, and then stored in 1 M NaOH solution at 50°C for 126 days. The mortar bars were measured three times per week for the entire duration of the study. The differences in length change measurements between the smallest and largest length change for a set of 3 bars steadily increased from near zero initially to 0.01, 0.05, and 0.03% for the plain, PP fiber, and hybrid fiber mortars, respectively, at 126 days.

For the μ CT studies, composite mortar cylinders (7 mm diameter and 3 cm long) were fabricated to accommodate the transmitted x-rays and to facilitate mounting into the μ CT's three-jaw chuck. Three cylinders were prepared, the control (without fibers), one containing PP fibers, and one containing hybrid fibers. The fibers were cut to ~5mm so they could fit along the cylinder's transverse direction. Samples utilized the same mix proportions and curing conditions (3 cylinders stored in 50 ml of 1 M NaOH at 50°C) as the accelerated mortar bars. The plain and hybrid fiber mortars samples were imaged at 2, 61, 74 and 136 days, and the PP mortar samples were imaged at 9, 22 and 84 days. The samples were imaged using hard X-ray μ CT at beamline 8.3.2 of the Advanced Light Source (ALS), utilizing an X-ray beam with an energy of 38 keV at a ring current of 500 mA. A PCO.edge sCMOS camera (2560x2160 pixels) was used with a 2x lens, giving a 3.32 μ m/pixel size with up to 8.3 mm field of view. This resolution facilitated the study of the microstructural behavior and the ASR effect. A total of 1025 images were captured (~1.8 mm-thick section), reconstructed using the software Octopus®, and visualized with

168 FIJI. The final 3D rendering and segmentation was performed with Avizo. All images
169 presented have a pixel or voxel size of 3.32 μm .

170 After reconstruction, the images were segmented manually based on grayscale to
171 differentiate the anhydrous inorganic components (cement and aggregate), cement
172 hydration products, ASR gel, polymer fibers, and voids. Known features were selected and
173 maximum and minimum values were selected that properly covered the known areas for the
174 features. Intact aggregates, polymer fibers and voids were segmented in this manner. Air
175 voids that became filled over time were utilized to facilitate the segmentation of cement
176 hydration products from ASR gel. Additionally, locations of aggregate dissolution where
177 ASR gel was expected to form and not cement hydration products was used to confirm that
178 the proper thresholding had been achieved. The ASR gel in this study was more water rich
179 (absorbs less X-rays) than the cement hydration products, such as C-S-H.

180 **3. Results**

181 **3.1. Expansion bar tests**

182 The expansion bar test results are shown in Fig. 1. After 125 days, both polymer
183 fibers reduced expansion by ~15% compared to the mortar without fibers. However, no
184 significant performance difference was observed between the expansion bars with hybrid
185 fibers [6] compared to those samples containing PP fibers. It is important to mention again
186 that a finer sand gradation and lower temperature were utilized in this study than are
187 specified by ASTM C 1260, so the criterion for reactive aggregate given in the standard
188 cannot be applied to the test results presented in this study.

189

190 **3.2. Microtomography analysis**

191 Tomographic images of the mortars are shown in Fig. 2. Early images show the
192 intact mortars. The interfacial transition zone (ITZ) between the cement paste and
193 aggregate often shows a region less densely populated by hydration products at early ages.
194 As time progresses, the ITZ becomes densified and the gap between the cement paste and
195 aggregate closes. Additionally, the hybrid fibers tend to agglomerate or bundle (B),

preferring to localize in less dense areas where finer aggregates are found [30]. With time, ASR attack occurs: reactive aggregates dissolve (D) and nearby pores are filled with ASR gel (G). At later ages, microcracks can be observed usually filled with ASR gel (C). Ettringite needles (indicated as N) can be seen growing in air voids.

Additionally, the μ CT results (Figs. 2-3) show significant amounts of entrapped air in the mixes containing fibers. Air void systems have been shown to reduce, or at least delay, expansion caused by ASR due to the gels escaping to the air voids and relieving or avoiding the pressures that can develop from ASR gel formation [31]. Fig. 2 confirms that ASR gel is able to travel from dissolving aggregates to pressure relief voids in-situ and confirms prior research (such as polished sections, fracture surfaces, etc.) showing gel in pores and entrained air. As seen in Fig. 3, the air voids (grey spherical shapes) tend to be more numerous in the hybrid fiber mortar compared to the plain mortar or the PP fiber mortar. Additionally, the maximum entrapped air bubble size tends to be larger with the larger PP fibers compared to the smaller hybrid fiber mortars. In order to better appreciate some of the mentioned features, subvolumes from the reconstructed volume mortar cylinders were extracted and their tomographic and volume rendered images are shown in Figs. 4 to 9. Figs. 4, 6, 8 and 9 show tomographic images at different dates and at different depths in the volume, while Figs. 5 and 7 show 3D rendered images.

3.2.1. Plain mortar

In Fig. 4, starting at 61 days, one can observe the presence of crack propagation, aggregate dissolution, and voids filled with ASR gel. In this figure, the first column shows an image (tomographic slice) from the top of the subvolume, while the second column shows an image from the middle and finally the third column shows an image close to the bottom of the subvolume. As expected, the progressive deterioration of the mortar can be followed along the different measured dates. The cracks (C) initiate within the aggregate, then extend and propagate through the paste and to neighbor aggregates, followed by the ASR gel (G) filling the cracks as well as a nearby pore. After 136 days, the aggregate which first showed cracking has totally disintegrated. It is important to notice that most aggregates are not reactive, even after 136 days.

Figure 5 gives the 3D reconstruction from the rendered images shown in Fig. 4. Fig. 5 segments out the empty space and ASR gel based on grayscale differences and gives additional insight into the dissolution of aggregates and crack propagation. At 2 days, one can observe the empty voids and pores (in black). At 61 days, ASR has progressed significantly. Voids are beginning to be filled with gel (G) and aggregate dissolution (D) is taking place. Additionally, the first few cracks (C) have started to appear. The cracks generally originate within some defect, such as pores in the aggregates, the ITZ, and in air gaps. At 74 days, the aggregate dissolution and gel formation have progressed, and gel can be observed in cracks extending away from the dissolving aggregate. At 136 days, the aggregate has completely dissolved and filled with gel, and the cracks become more pronounced and filled with gel.

3.2.2. Composite mortar

3.2.2.1 Polypropylene fibers

Tomographic images of a subvolume (2.5 x 1.2 x 1.6 mm) extracted from the PP composite mortar cylinder, taken at 9, 22 and 84 days, are shown in Fig. 6. Similar features as in the control mortar were observed. Ettringite needles can be found inside some voids, which by 136 days are completely filled with ASR gel (G). Again, aggregate dissolution (D) appears to initiate internally in an aggregate pore or crack, and cracks (C) propagate out from the dissolution site as ASR progresses.

Fig. 7 gives the 3D reconstruction from the rendered images shown in Fig. 6. At 9 days, the porosity, voids, and the less densely populated portion of the ITZ (black) around the PP fibers (yellow) can be observed after segmenting the image based on grayscale. At 22 days, some cracks and ASR gel (pink, G) have started to appear. In addition, the ASR initiation sites within the aggregate can be identified. At 84 days, ASR has fully dissolved a few aggregates, voids are filling with ASR gel, and cracks (C) are propagating away from the aggregate. Although, there are many fibers surrounding the dissolving aggregate on the right, the PP fibers with their low elastic modulus are unable to restrain the cracks from widening. Additionally, due to the fiber's Poisson's ratio as the fibers are stretched, they

255 become narrower opening gaps between the fiber and the matrix. At 84 days, these gaps
 256 can be seen filling with ASR gel as well.

257 Fig. 8 shows a tomographic slice from the PP composite mortar at the edge of the
 258 cylinder. Between 22 and 84 days, a crack (C) develops along the aggregate ITZ and
 259 propagates to the boundary of the cylinder.

260

261 3.2.2.2 Hybrid fibers

262 Fig. 9 shows tomographic images for the hybrid fiber composite mortar. Observing
 263 the images in the first column, the aggregates progressively dissolve (D), and a crack (C)
 264 develops near or at the surface of the aggregate next to the entrapped air bubble which is
 265 filled with ASR gel (G) as the aggregate dissolves. Meanwhile, in the middle and last
 266 columns of images, cracks propagate through the matrix, between the ITZs and through
 267 other surrounding aggregates. It can be determined that the cracks are propagating through
 268 these surrounding aggregates and not initiating in them because they are not showing
 269 deterioration prior to cracking. Although crack initiation wasn't observed due to the gap in
 270 observation times, it is plausible that cracking originated between the aggregate and the
 271 adjacent air void in first column due to this flaw in the composite. In addition, this
 272 subvolume contains some areas where hybrid fiber can be observed to agglomerate such as
 273 below the large dissolving aggregate in the first column, a tendency found due to the nature
 274 of this kind of fiber [30].

275

276 4. Discussion

277 The μ CT results, tomographic slices and 3D reconstructed images show that significant
 278 amounts of air get entrapped in the mixes containing fibers. As mentioned, it has been
 279 shown that air void systems reduce, or at least delay, the expansion caused by ASR due to
 280 the gel migrating to the air voids and relieving or avoiding the pressures that can develop
 281 from ASR gel formation. Here, air voids can be observed filling with ASR gel as nearby
 282 aggregates dissolve. However, it is difficult to describe the reduction in expansion observed

in Fig. 1 for the mortars containing fibers being restrained as a result of the incorporation of the fibers or attributed to a reduction in the swelling pressures that result from gel escaping into the distributed air voids. Future studies that investigate the use of fibers to ameliorate ASR expansion should test the mixes for air content to determine if any reduction in expansion could be attributed to the inclusion of fibers or is in fact a result of differences in air content.

The presented images depict some of the principal effects of ASR in the microstructure of three different mortar specimens analyzed: crack initiated in an aggregate, cracks propagating from the aggregates through the matrix and through other aggregates, cracks propagating through the ITZs, voids and gaps filled with ASR gel, and to a lesser extent cracks initiating at the aggregate ITZ or boundary between a defect, such as an air void, and the aggregate. It was observed that few aggregates tend to be highly reactive, as also shown by Dunant et al. [24]. Prior research has shown that for the aggregate utilized in this study the sand grains that contain alkali (e.g. $\text{NaAlSi}_3\text{O}_8$ or KAlSi_3O_8) dissolve more readily than the grains that do not (e.g. SiO_2), which explains why some aggregates are much more reactive than others in this study [12].

Crack initiation more often begins internally within the aggregate in the center of the specimens; however, near the edge of the specimens or when a defect (air void) is adjacent to the aggregate, the propensity for cracks to occur at the aggregate ITZ appears to be increased. Since tomography samples are of a small size, the ratio of boundary affected cracking habits to boundary free cracking habits would be expected to be increased compared to actual concretes, which have a much higher volume to surface area ratio. Some of these effects have been extensively studied. As Stark et al. [20] summarize, the ASR gel formation takes place in the reactive aggregate and promotes microcracking and its propagation. Hence, the osmotic pressure due to the concentration difference between the gel and the pore solution in the surrounding hydrated cement paste, promotes the crack expansion and therefore the progressive deterioration. Theoretical approaches can be used in order to quantify the stress, displacement and expansive cracking under different expansive conditions as proposed by Garboczi [32]. The observations found in this μCT

study are consistent with Votolini et al. [27] but further the understanding of crack initiation in ASR affected mortars.

A different, but important observation is the preferred distribution for each type of fiber, a bundled distribution for the hybrid fiber and the more uniform distribution of PP fiber. This bundling behavior can be related to the aspect ratio of the fibers and to the fact that the hydrophilic coated polymers may prefer the less dense, water rich areas, to the denser regions. The carboxylate groups of the EAA sheath may be helping bridge the bonding problems between hydrophobic polymers and cement paste, since the EAA is more hydrophilic [30]. Future studies to evaluate hydrophilic polymer fibers with different aspect ratios can be performed, in order to determine if the hydrophilic fibers are aiding or hindering dispersion, this would be valuable in interpreting these findings. Here, one can observe the effect of using different types of fiber, on the size and distribution of air-bubbles in the mortar composites, as shown in Fig. 3. This characteristic could give important insights as to how polymer fibers aid in reducing expansion due to ASR.

5. Conclusion

Microtomography (μ CT) studies were carried out on three different cylindrical samples: a plain mortar and two different composite mortars containing polymer fiber (PP and PP/EAA hybrid). Using air voids that filled with ASR gel proved to be a reliable way to manually select minimum and maximum grayscale values to segment the ASR gel from the cement hydration products in this study. The samples were exposed to ASR attack for different dates, ranging from 2 to 136 days. The μ CT studies allowed, in general, the observation of the preferred fiber distribution, the ASR gel formation, preferred crack initiation sites, crack propagation paths and the aggregate dissolution. It was observed that few aggregates tend to be highly reactive, and that crack initiation more often begins internally within the aggregate in the center of the specimens. When the reacting aggregate is near the edge of the specimen or adjacent to defects (air voids), the propensity for cracks to occur at the aggregate ITZ appears to increase. The reaction and expansion originating at isolated aggregates drives the entire deterioration in the mortar. Microcracks propagate

along the path of least energy through weakness in the mortar such as partially dissolved aggregates, air gaps and aggregate ITZs. In addition, the use of two different polymer fibers and the air-bubble distribution in the mortar composites seem to show an important expansion reduction effect, as evidenced by the mortar bar expansion tests and the many air voids filled with ASR gel near reacting aggregates.

Acknowledgments

C.W. Hargis was supported by the Berkeley Fellowship for Graduate Study and the Carlson-Polivka Fellowship. The authors want to acknowledge The Dow Chemical Company (DOW®) for their financial support and Dula Parkinson for all his support at the Advanced Light Source. The Advanced Light Source is supported by the Director, Office of Science, Office of Basic Energy Sciences, of the U.S. Department of Energy under Contract No. DE-AC02-05CH11231.

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429 **List of Figures**

430

431 Fig. 1: Expansion of mortar bars due to ASR for three types of specimens: the control with
 432 no fibers, hybrid fibers, and PP fibers.

433

434

435 Fig. 2: Tomographic images taken from the control, PP and hybrid fiber mortar cylinders.

436 Each cylinder is 7mm in diameter.

437

438 Fig. 3: 3D images showing air voids and fiber distributions in a cement mortar: (a) control,
439 taken at 2 days, (b) hybrid fiber, taken at 9 days, and (c) PP fiber, taken at 2 days. The
440 block volume is 4.875mm x 4.875 x 1.5mm.

441

442 Fig. 4: Tomographic images (2.7 x 1.5 mm) taken from the plain mortar sample, after 2, 61,
 443 74 and 136 days in a 1M of NaOH solution at 50°C. The rows show the days when they
 444 were measured. The columns show three different slices from the tomographic stack.

445

446 Fig. 5: 3D images showing the progression of the ASR effect in a plain mortar sample, after
 447 2, 61, 74 and 136 days in a 1M NaOH solution at 50°C. Subvolume sizes are 2.7 x 1.5 x 1.5
 448 mm. Initial air gaps and voids (bubbles) are represented in black, while pink shows the
 449 ASR gel.

450

451

452 Fig. 6: Tomographic images (2.5 x 1.2 mm) taken from the composite mortar sample,
 453 containing PP fibers, after 9, 22 and 84 days 1M of NaOH solution at 50°C. The rows show
 454 the days when they were measured. The columns show two different slices in the
 455 tomographic stack.

456

457 Fig. 7: 3D images showing the progression of the ASR effect in a composite mortar,
 458 containing PP fibers, after 9, 22, and 84 days in a 1M NaOH solution. Subvolume size 2.5 x
 459 1.2 x 1.6mm. Initial air gaps and voids (black), PP fibers (yellow) and ASR gel (pink).

460

461 Fig. 8: Tomographic images (1 x 1 mm) taken from the composite mortar sample,
462 containing PP fibers, after 22 and 84 days 1M of NaOH solution at 50°C.

463

464

465 Fig. 9: Tomographic images (1.7 x 2.1 mm) taken from the hybrid fiber composite mortar
 466 sample after 2, 61, 74 and 136 days in a 1M of NaOH solution at 50°C. The rows indicate
 467 the days when they were measured. The columns show three different slices in the
 468 tomographic stack.