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Mitigation of alkali-silica expansion in pulp fiber-mortar composites

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ABSTRACT

The physical and microstructural effects of kraft pulp, thermomechanical pulp, and polypropylene fibers for restraining expansion due to alkali–silica reaction are investigated through expansion measurements, environmental scanning electron microscopy, and energy dispersive spectroscopy. This preliminary research revealed that thermomechanical pulp fibers are more effective at reducing expansion due to alkali–silica reaction than kraft pulp and polypropylene fibers. It is shown that the open lumen of the TMP fibers, and to some degree, the kraft pulp fibers, serves as a reservoir for alkali–silica gel, potentially minimizing internal expansive pressures. Furthermore, increases in the fiber volume fraction and fibercement bond strength (accomplished through extended initial moist curing) decreased expansion for all composites.

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1. Introduction

Various methods have been used to mitigate ASR including the reduction in alkali loading by the use of low alkali cement (Na $_2$ Oeq < 0.60%) and by limiting the cement content, the use of non-reactive aggregate, the use of low water-to-cement ratio concrete, the addition of supplementary cementing materials, the addition of lithium, and the incorporation of fibers to physically restrain expansion and limit subsequent cracking. The latter method has been previously investigated with steel, brass-coated steel and polypropylene fibers.

Using brass-coated steel with a fiber aspect ratio (fiber length/ fiber diameter or l/d) of 40 and polypropylene (l/d > 40) fibers, Haddad and Smadi [1] evaluated ASR expansion in concrete. Specimens were soaked in a 0.5 N NaOH solution at 40 °C, after 7 or 28 days of water curing. It was found that specimens with both types of fibers exhibited higher expansive strains during the exposure period, compared to the control specimens. Concretes with polypropylene fibers appeared to resist cracking better than those with brass-coated steel fibers, possibly owing to the higher aspect ratio of the polypropylene fibers. However, different results were reported by Park and Lee [2] in mortars, where steel microfibers (l/d = 60) minimized mortar bar expansion more than polypropylene fibers (l/d = 250).

Turanli et al. [3] have established that the introduction of steel microfibers to mortar reduced the overall mortar bar expansion due to ASR. Steel microfibers ($l/d \sim 30$) were used at fiber volume

fractions of 1%, 3%, 5%, and 7%. It was found that expansion due to ASR decreases with increasing steel fiber volume fractions. With the 7% fiber volume fraction specimens, an expansion reduction of 62.5% was observed, as compared to the control specimens, after 30 days.

Results by Yi and Ostertag [4,5] have also shown that the addition of steel microfibers to mortar reduced expansion and microcracking due to ASR. It was found that the fibers were extremely effective in reducing microcrack widths by fiber bridging which induces closure stresses on the crack. Thus, by reducing the overall crack width and length, alkali–silica gel (the expansive product of ASR) is constrained to the ASR reaction site, leading to higher free Na (alkali) and Si (silica) concentrations in the alkali–silica gel. The higher concentrations may be the result of decreased rate of reaction. In other words, extrusion of the silica gel from the reaction site is minimized due to microcrack control, thus controlling widespread expansion in microcracks.

Pulp fibers offer a low-cost, renewable alternative to the steel and polypropylene fibers previously used for reduction of ASR-induced expansion. Two general types of pulp fibers are commonly available and differ in the processing methods used to extract the respective fibers. Chemical pulp processes are also referred to as kraft pulping and constitute the largest portion of pulps manufactured. Mechanical pulps are made without chemical processing and thus only require mechanical energy, and often heat (i.e., thermomechanical pulp), to produce pulps with distinct properties, which are often used in newsprint paper.

Kraft pulped and thermomechanically pulped (TMP) fibers have been shown to provide ductility and toughness to cement-based materials while having a negligible to minimal negative impact

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Table 1Oxide analysis and Bogue potential composition of ASTM Type I/II portland cement.

Oxide	Type I/II portland cement
SiO ₂	21.26
Al_2O_3	4.79
Fe ₂ O ₃	3.14
CaO	64.10
MgO	2.35
Na ₂ O	0.02
K ₂ O	0.36
TiO ₂	0.19
Mn_2O_3	0.04
P_2O_5	0.03
SrO	0.03
BaO	0.04
SO ₃	2.63
Loss on ignition	1.04
Insoluble residue	0.11
C ₃ S	55.24
C_2S	19.28
C ₃ A	7.38
C₄AF	9.54

on compressive strength (similar to polypropylene fibers) [6–17] and may provide some mechanical restraint to internal expansion. Furthermore, kraft pulp and TMP fiber have an open cavity or "lumen" surrounded by their hygroscopic cell wall structure; water held within the fiber structure has been shown to provide internal curing in high-performance pastes and mortars [17–20]. However, in this application, it may be possible for alkali–silica gel to accumulate in the fiber, lessening expansive pressures. Therefore, the application of two types of pulp fibers for mitigation of ASR expansion is examined, as is the mechanism(s) by which expansion control may occur. Southern Slash pine softwood kraft pulp fibers and southern Loblolly pine TMP fibers are investigated in this research. Companion studies with polypropylene fibers were performed for comparison.

2. Experimental study

2.1. Materials

Composites were prepared according to ASTM C 1260 [21] with a water-to-cement ratio of 0.47 and a sand-to-cement ratio of 2.25. Commercially available ASTM Type I/II portland cement, highly reactive cherty sand (Jobe sand from Texas, USA), and deionized water (resistivity of 18.2 $M\Omega$ m) were used. Oxide analysis and Bogue potential composition for the cement are listed in Table 1. ADVA Flow superplasticizer, obtained from Grace Construction Products, was used, as needed, up to a maximum dosage rate of 6.15 μL per gram of cement to aid workability.

Softwood kraft pulp and thermomechanical pulp (TMP) fibers were used at fiber volume fractions of 1% and 3%. Polypropylene fibers with a $\frac{1}{4}$ inch (6.35 mm) length were also used at the same fiber volume fractions. The kraft pulp, TMP, and polypropylene fibers had average fiber aspect ratios ($l_{\rm avg}/d_{\rm avg}$) of 275, 30, and 158.75, respectively. The physical properties of these fibers are listed in Table 2. The TMP southern Loblolly pine fibers were

obtained from Augusta Newsprint Company in Augusta, Georgia, USA. The TMP fibers were collected from the secondary refiner. The southern Slash pine softwood kraft pulp fibers (bleached, unbeaten, once-dried) were obtained from Buckeye Technologies in Plant City, Florida, USA. The kraft pulp fibers were treated by a process described in [14,15], where the fibers are treated with cationic starch and fly ash to improve their dispersion during mixing in cement-based materials. The polypropylene fibers were obtained from Propex in Chattanooga, Tennessee, USA.

2.2. Mortar bar sample preparation and conditioning

Mortars were prepared by mixing the fibers, fine aggregate and approximately 50% of the water for 3 min at 60 rpm in a 1.5 L-capacity Hobart mixer to ensure separation of the fibers. Subsequently, the cement was added, followed by the remaining water. Superplasticizer (W.R. Grace ADVA Flow) was added, as needed, after all water was added. Mixing continued at 120 rpm for another 5 min to allow for uniform fiber dispersion.

Samples were demolded 24 h after casting and stored in deionized water at 80 ± 2 °C for an additional 24 h. After removal from the hot water curing, initial length measurements were taken and specimens were subsequently stored in a 1 N NaOH solution at 80 ± 2 °C for the remainder of the testing. Measurements were taken periodically, up to 28 days. Additional samples were cured for 7 days prior to ASTM C 1260 exposure to evaluate the effect of fiber–cement bonding on the expansion behavior.

2.3. Microstructural characterization

Microstructural observations were conducted on a FEI Quanta 200 environmental scanning electron microscope (ESEM) in a gaseous (water vapor) environment. Energy dispersive spectroscopy (EDS) was accomplished using an EDAX SUTW detector. All EDS spectra and chemical maps were collected at an accelerating voltage of 10.0 kV and a water vapor pressure of 93.3 Pa (0.70 Torr).

3. Results and discussion

In this research, the mitigation of expansion due to alkali-silica reaction was assessed for three fiber types. All fibers were investigated at fiber volume fractions of 1% and 3%. Due to differences in fiber physical properties (Table 2), the fiber surface area and number of fibers per unit volume were used as means to elucidate the effectiveness of the three fiber types. In addition, the influence of fiber-cement bond strength was evaluated by increasing the initial curing period. Environmental scanning electron microscopy was conducted to observe if the pulp fibers (i.e., kraft pulp and TMP fibers) provide a reservoir for alkali-silica gel, potentially minimizing expansion.

3.1. Expansion results

As seen in Fig. 1, the control mortar experienced $0.462 \pm 0.007\%$ and $0.623 \pm 0.010\%$ expansion at 14 and 28 days, respectively, indicting the high alkali-reactivity of the aggregate used in this

Physical properties of fibers.

Fiber type	Average fiber diameter, $d_{ m avg}~(\mu{ m m})$	Average fiber length, $l_{ m avg}$ (mm)	l _{avg} /d _{avg} ratio	Average specific gravity	Average tensile strength (MPa)	Average modulus of elasticity (GPa)
Kraft pulp TMP	20 50	5.5	275 30	1.5 0.5	800-1000 500-700	18–40 N/A
Polypropylene	40	6.35	158.75	0.91	300-700	3.5-5.0

Note: N/A = not available.

study. The three fibers examined (kraft pulp, TMP, and polypropylene) varied in average fiber length, average fiber diameter, specific gravity, and average fiber tensile strength (Table 2). The effects of fiber volume fraction are shown in Fig. 1. It can be seen that 1% kraft pulp, TMP, and polypropylene fibers reduced ASR expansion after 28 days of exposure by 27.0%, 70.9%, and 49.8%, respectively. At a 3% fiber volume fraction, expansion was reduced by 56.7%, 83.5%, 56.5%, respectively. As expected, increasing fiber volume fractions led to decreased expansion regardless of fiber type. However, it can be seen that the TMP fibers reduced expansion more than the kraft pulp and polypropylene fibers, at both fiber dosage rates examined. At a 3% fiber volume fraction, the longer fibers (i.e., kraft pulp and polypropylene) reduced expansion by similar amounts.

Thus, as there are more shorter (i.e., TMP) fibers per unit volume, $N_{\rm v}$ (fibers/mm³), compared to the longer fibers (Fig. 2), the distributed nature of the TMP fibers is proposed to be a contributing factor in making them more effective at controlling microcrack growth and reducing internal stresses. To further investigate the influence of fiber distribution, Fig. 3 illustrates the influence of total outer fiber surface area, $S_{\rm v}$ (mm²/mm³) – as related to the number of fibers and fiber volume fraction – on the measured mortar bar expansion at 28 days. As the total outer fiber surface area increases, expansion decreases.

The effect of fiber–cement bond strength was also investigated by increasing the initial ASTM C 1260 curing period from 1 to 7 days to increase the bond strength. As seen in Fig. 4, the control sample cured for 7 days prior to alkali exposure exhibited 0.446 \pm 0.022% and 0.596 \pm 0.012% expansion after 14 and 28 days of exposure, respectively. It can be seen that there are negligible differences in the expansion of the control samples (i.e., no fibers) with prolonged initial curing time.

Compared to the control mortar cured for 7 days, the kraft pulp and TMP composites reduced expansion by 36.7% and 78.0%, respectively, after 28 days. Thus, after 28 days of exposure, the kraft pulp and TMP composites cured for 7 days exhibited 17.1% and 27.6% reductions in expansion, respectively, compared to the corresponding composites cured for only one day. This suggests that prolonged curing to increase fiber–cement bonding improves microcrack deflection within the cementitious composite. Furthermore, the longer initial curing may better reflect actual field performance.

4. Microstructural observation

Fig. 5 illustrates ESEM BSE chemical mapping of a 3% kraft pulp fiber-mortar polished fracture surface examined after 28 days of

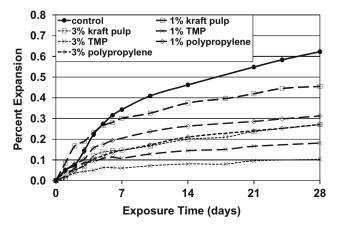


Fig. 1. Expansion results - effect of fiber type and volume fraction.

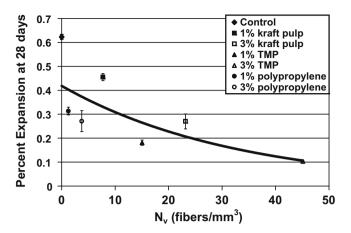


Fig. 2. Influence of number of fibers per unit composite volume, N_v , on ASTM C 1260 expansion after 28 day exposure.

exposure with 1 day initial curing. As seen, Fig. 5 reveals that the area within the kraft pulp fiber lumen and cell wall and approximately 5 µm from the outer fiber cell wall exhibited higher levels of Na and Si compared to the surrounding bulk paste, but similar levels as the ASR gel surrounding the reactive aggregate and within microcracks. Though the kraft pulp fibers are treated in an alkali solution during the pulping process, it has previously been shown that there is a lack of residual alkalis on the kraft pulp fiber surface [15]. Thus, the increased presence of sodium, with increased amounts of silicon, in the fiber lumen, fiber cell wall, and nearby paste indicates the presence of ASR gel.

This further suggests that, in addition to gel collecting around the fiber-cement interface, the lumen (open interior) of the pulp fibers may act as a reservoir for ASR gel. This may be viewed as analogous to the protection afforded by air entrainment during freeze/thaw. Previous research has illustrated that the lumen of these pulp fibers tends to fill with reprecipitated calcium hydroxide during wet/dry cycling [11–13]. However, for the exposure conditions in this research, the samples remained relatively saturated. Thus, the lack of calcium within the fiber lumen and in the fiber-cement interface in Fig. 5 would be expected due to the lack of sample drying. Also worth noting is the lack of elevated potassium levels in this regions, which would normally be associated with ASR in petrography of field-exposed materials. Here, the accelerated exposure conditions (high concentration of sodium in solution) likely overwhelmed the concentration of potassium in

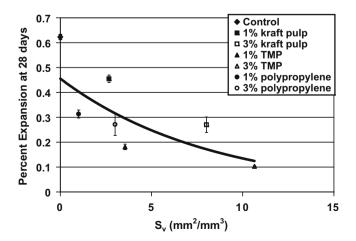


Fig. 3. Influence of outer fiber surface area per unit composite volume, S_v , on ASTM C 1260 expansion after 28 day exposure.

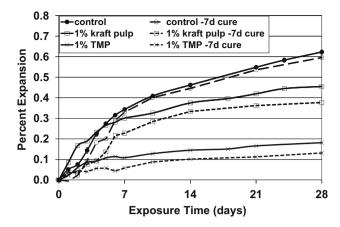


Fig. 4. Expansion results - effect of initial curing time.

the ASR gel and bulk paste and, as a result, the higher concentration of Na and Si in this region serve as the best markers for ASR.

Furthermore, Figs. 5 and 6 illustrate the microcrack deflection capabilities of the kraft pulp and TMP fibers. Microcracks were observed to initiate at the reactive siliceous aggregate and propagate through the cementitious matrix until a fiber was encountered. At the fibers, cracks were deflected around the fiber, leading to apparent fiber–cement debonding (Figs. 5 and 6a). In addition, some cracks were seen to stop at fibers (Fig. 6b). This observed behavior may be due to the cracks propagating along the longitudinal axis of the fiber and/or crack tip termination due to blunting and the redistribution of internal stresses.

Further research is ongoing to validate the mechanism(s) underlying the improved performance of the kraft pulp and TMP fibers, relative to polypropylene fibers, at reducing expansion due

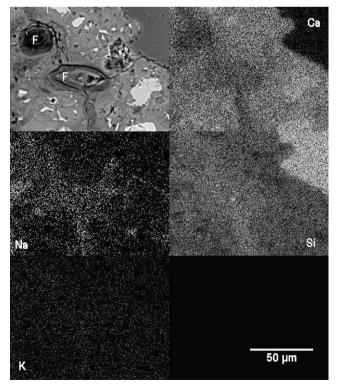
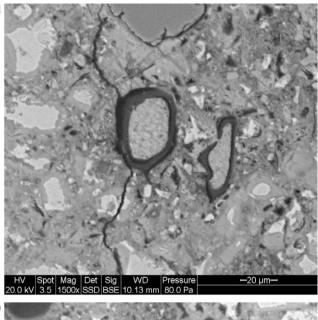


Fig. 5. ESEM BSE chemical mapping of polished kraft pulp fiber–mortar composite containing 3% fibers by volume after 28 day exposure (traditional ASTM C 1260 curing), Note: "F" indicates fiber.





 $\begin{tabular}{ll} \textbf{Fig. 6.} ESEM BSE micrographs of TMP fiber-mortar composites illustrating crack deflection. \\ \end{tabular}$

to ASR. In addition to the restraint and crack arrestment/deflection capabilities of all fibers investigated, it is proposed, based upon microstructural evidence, that both types of pulp fibers allow for the accumulation of ASR gel within their hollow lumen. This sequestration of the gel may minimize bulk internal stresses in the mortar. In comparison, polypropylene fibers reduce expansion by mechanical restraint and crack deflection only.

5. Conclusions

In this preliminary research, kraft pulp, thermomechanical pulp, and polypropylene fibers were used as reinforcement in mortars containing alkali-reactive aggregate, and their effect on mortar expansion during standard accelerated testing for alkali-silica reaction was assessed. The effect of fiber type, fiber volume fraction, and fiber-cement bond strength were evaluated for their ability at mitigating expansion due to alkali-silica reaction. Environmental scanning electron microscopy and energy dispersive spectroscopy were used to elucidate microstructural and chemical effects due to fiber addition. From mechanical testing

and microstructural observations, the following conclusions may be drawn:

- All three fibers examined reduced expansion by ASR during ASTM C 1260 exposure.
- At 1% and 3% fiber volume fractions, the TMP fibers were more effective at reducing ASR expansion than the kraft pulp or polypropylene fibers.
- At a 1% fiber volume fraction, the kraft pulp fibers reduced expansion more so than the polypropylene fibers. However, at the 3% fiber volume fraction, both fiber types exhibited similar results
- As the total outer fiber surface area and number of fibers per unit composite volume increased, the observed expansion after 28 days of exposure decreased for all fiber types examined.
- Increasing the pulp fiber-cement bond strength by prolonged initial curing prior to alkali exposure decreased the observed expansion due to ASR compared to the control sample without fibers.
- As compared to polypropylene fibers, the improved performance
 of the TMP and, to a lesser degree, the kraft pulp fibers is
 believed to be due to the open lumen (interior) of these fibers.
 As observed by ESEM chemical mapping, ASR gel accumulates
 within the lumen, minimizing expansive pressures.

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