After the sol-gel reaction, nanosilica particles were clearly seen on both the treated micro and macro PP fibers compared to the untreated (**Fig. 4**). Further, treated PP fibers had nanosilica particles (white colored spots) on their surface regardless of drying temperature. **Table 3** presents the mass change results of the micro PP fibers. Both of the treated micro PP fibers gained mass after sol-gel reaction in the same amount; $15.2 \pm 0.15\%$ for RmPP and $15.1 \pm 0.22\%$ for OmPP. This shows that the total amount of nanosilica to adhere onto the fiber surface was not affected by drying condition. To compare, it was previously measured that treated macro PP fibers also gained mass after sol-gel reaction; $12.5 \pm 0.08\%$ for RMPP and $8.75 \pm 0.12\%$ for OmPP.²⁴ It is apparent that the mass change values were lower for the macro PP fibers compared to the micro PP fibers. This may be attributed to the micro PP fiber having a relatively high specific surface area compared to the macro PP fiber, which can provide more deposition sites for nanosilica particles during the sol-gel process.

From the mass change results, all treated fibers gained mass after coating, indicating nanosilica was effectively adhering onto the fiber surface. To provide further insight, the results of the water absorption and solubility matter loss tests will be discussed, which are presented in **Table 4**. As expected, the untreated PP fiber did not absorb any water. For treated micro PP fibers, RmPP and OmPP absorbed $9.38\pm1.07\%$ and $9.46\pm0.60\%$, respectively. And for treated macro PP, RMPP and OMPP absorbed $2.04\pm0.73\%$ and $2.26\pm0.55\%$, respectively. This is very similar to previously reported values for RMPP and OMPP (1.713% and 2.101%, respectively).²⁴ Slight differences may be due to the higher sample size tested in the present study (approx. 1 g vs 0.1 g (0.0022 lb) vs 0.00022 lb)), but the trend is the same.

Comparing the micro vs macro PP fibers coated with nanosilica, it is apparent that the measured absorption was much higher for micro PP. As mentioned prior, this can be attributed to the relatively high specific surface area and resultant higher concentration of nanosilica. For both micro and macro PP fibers, a slightly higher value in absorption was measured for the fibers that were oven dried at 50°C (122°F). This can be attributed to differences in the moisture status of the nanosilica due to drying temperature, and thereby likely not representative of any difference in the quantity or morphology of the nanosilica themselves. Finally, all treated fibers showed zero water solubility matter loss, which indicates that the nanosilica is adhered onto the fiber surface and not lost upon contact with water.

The key findings here are that the sol-gel process is effective in coating both micro PP and macro PP fibers, with higher concentrations for micro PP due to their higher specific surface area. Drying temperature did not have any apparent effect on the amount of nanosilica formed to coat the PP fibers. And the sol-gel process was effective in producing a coating of nanosilica that is not soluble. Next it will be discussed how this translates to flexural strength and recovery.

Flexural strength and recovery

The flexural strength and recovery results are presented in **Fig. 5** for macro PP and **Fig. 6** for micro PP, as well as concisely summarized in **Table 5**. It should be noted that the mechanical performance of the cement-composites reinforced with micro PP fibers and macro PP fibers can not be compared directly due to differences in their reinforcing mechanism. The fracture surface of a sample reinforced with macro PP is shown in **Fig. 7(a)**. Due to their lower specific surface area, there were much fewer macro PP fibers at the site of the crack, even though the dosage was higher than that of micro PP fibers by volume. In contrast, micro PP fibers were able to bridge the fractured area effectively with a uniform distribution (**Fig. 7(b**)). Therefore the effect of the micro PP and macro PP fibers will be discussed separately herein.

<u>Macro PP fibers</u> – The results of macro PP cement-composites are shown in **Fig. 5**. The flexural strength of the plain system without fibers was set at 100% to serve as a reference. It is observed that the initial flexural strength increases with the introduction of macro PP fibers, both treated and untreated (**Fig. 5(a)**). Polymeric fibers have poor bonding with cement matrices due to their hydrophobicity. As a result, the sites of poor bonding at the fiber-matrix interface can act as points of entry for water during curing,^{41,42} which can promote cement hydration and subsequent pozzolanic reaction by the nanosilica. Through this mechanism, the cement-composites with macro PP can develop higher flexural strength at early ages.⁴³

Looking at the effect of fiber treatment, there was no notable effect of nanosilica, i.e. no difference between uMPP versus RMPP and OMPP. However, a previous study by the authors indicated improved bonding strength through nanosilica coating through pull-out tests, so the present flexural strength results were unexpected. A potential explanation is that the nanosilica coating applied through the sol-gel process is not completely uniform, as observed previously.²⁴ Therefore the macro PP fibers may still have sites of poor bonding with the cement matrix, which govern and cause them to perform similar to untreated fibers.

Fig. 5(b) shows the flexural strength recovery results of the macro PP fiber cement-composites. Macro PP fibers did not lead to any recovery in flexural strength – 85.3% for uMPP, 51.9% for RMPP, and 51.8% for OMPP. In fact, the results indicate that nanosilica-coated macro PP fibers decreased the recovered flexural strength ($f_{L,healing}$) compared to the untreated fiber. Recall that the 28day flexural strength of cement-composites reinforced with macro PP fibers were higher than that of the plain due to potential water entry at the fiber-matrix

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interface, which promoted cement hydration and pozzolanic reaction during water curing (**Fig. 5(b**)). As a result, the amount of unhydrated cement particles was reduced, which did not lead to sufficient strength recovery during additional curing in air.^{20,22} This effect may have been even more pronounced with the introduction of nanosilica, which explains why RMPP and OMPP exhibited less strength recovery than uMPP. It is noted that the fiber diameter was relatively large compared to the specimen size and the systems were limited to pastes. The performance may differ in concrete systems, which was out of the scope of the current study but is the topic of ongoing work.

<u>Micro PP fibers</u> – The flexural strength and recovery results for micro PP fiber reinforced cement-composites are shown in **Fig. 6**. Generally, it was observed that flexural strength increases with the introduction of micro PP fibers (**Fig. 6(a)**). The flexural strength of umPP and RmPP were comparable, indicating that nanosilica coating dried at room temperature did not have a measurable effect on flexural strength at 28 days. On the other hand, OmPP exhibited an increase in strength of 129.3% compared to umPP. Through this, it could be surmised that the nanosilica exhibited different reaction mechanisms to affect strength depending on drying temperature. Main parameters influencing the mechanical properties of cement-composite have been reported as the specific surface area, the micropore volume and the average size of the primary particles of silica.^{8,44-46} Nanosilica with higher surface area have higher reactivity, which can affect early-age strength.^{5,31} In the sol-gel process, the elimination of water during drying causes the concentration of the sol to increase and also creates fluid drag, which can cause the particles to aggregate.⁴⁷ Since drying occurs more slowly at room temperature than 50°C (122°F) in an oven, nanosilica particles can be expected to be more aggregated in RmPP than OmPP. Therefore the higher flexural strength of OmPP compared to RmPP can be attributed to the nanosilica on the OmPP fibers exhibiting higher surface area and subsequently higher reactivity.

Flexural strength recovery of cement-composites with micro PP are shown in **Fig. 6(b)** and detailed in **Table 5(b)**. First looking at the flexural strength recovery of plain and umPP, they were 78.8% and 64.3%, respectively, which indicate that autogenic self-healing was not enough to lead to full strength recovery. In contrast, when comparing umPP to RmPP and OmPP (in particular, RmPP, which had similar flexural strength (f_L) as umPP), development of strength through nanosilica was clearly observed. Both RmPP and OmPP recovered flexural strength completely at 112.8% and 99.0%, respectively.

Results indicate that the nanosilica dried at room temperature after sol-gel led to the highest strength recovery. This can be tied to a dominant pozzolanic effect (versus seeding effect) by the nanosilica, which would promote the formation of hydration products at later ages and contribute to strength recovery (versus initial strength). This is supported by the results of 28 day flexural strength (**Fig. 6(a)**), which showed RmPP did not have any measurable effect compared to umPP. In contrast, OmPP exhibited the highest 28 day flexural strength (**Fig. 6(a)**) but led to less significant recovery than RmPP (**Fig. 6(b**)). This can be attributed to the nanosilica dried at 50° C (122°F) having a dominant seeding effect, which leads to earlier strength development versus sustained. To add, the absolute recovered flexural strength of OmPP was higher than that of RmPP. This also explains why OmPP exhibits higher CH content than RmPP, indicating a seeding effect by the nanosilica dried at 50° C (122°F) and pozzolanic effect dried at room temperature.

In summary, results indicate that drying condition had an impact on the reactivity of the nanosilica, and subsequently initial and recovered flexural strength. Nanosilica on the OmPP fiber appeared to react faster, acting dominantly as a nucleation agent, which accelerated hydration at early age and led to higher initial strength. On the other hand, nanosilica on the RmPP fiber appeared to react later on, acting dominantly as a pozzolan, which did not affect initial strength but went on to enhance strength recovery.

Analysis of hydration products

From the flexural strength results, it was found that treated micro PP fibers led to the most significant strength recovery. To better understand the mechanisms underlying this and resolve the contribution of the nanosilica, XRD, TGA, and SEM imaging were performed on samples reinforced with treated micro PP fibers.

The results of XRD are shown in **Fig. 8**. There were no apparent differences between the XRD patterns overall. However, there were some changes in the peaks related to CH (2-theta= 18.1° , 28.7° , 34.2° , 47.2° , and 50.9°). To further analyze the CH phase, TGA was performed, which can quantify calcium hydroxide content (CH, %) to further obtain a measure of pozzolanic reaction due to the nanosilica. Generally, the decomposition of hydration products can be divided into three ranges, as shown as **Fig. 9**. The first range below 400°C represents the evaporable water and the hydrates, i.e. hydrated silicate or aluminates decomposition. Weight loss between 440 and 520° C (824 and 968° F) corresponds to the decomposition of CH. And calcium carbonate (CaCO₃) decomposes at high temperatures.⁴⁸⁻⁵²

It should be noted that for the calculation of CH, the temperature range considered is typically from 400 to 600°C (752 to 1,112°F). However, here, it was calculated based on weight loss from 400 to 800°C (752 to 1,472°F) since the samples for TGA were 140 days old and CH may produce CaCO₃ due to carbonation.⁵³ Therefore the final CH content values reported in **Table 6** take this into consideration.

First, comparing CH content, TGA results show that RmPP and OmPP exhibited lower CH content than plain, which agrees with the results of XRD. This can explain the enhanced recovery in flexural strength exhibited by both RmPP and OmPP, where the pozzolanic reactivity of the nanosilica facilitated self-healing (**Fig. 6(b**)).

To better understand the influence of drying condition, i.e. RmPP vs OmPP, further analysis was performed for temperatures below 400°C (752°F), which relate to decomposition of C-S-H, C-A-H and ettringite. Within this range, weight loss from 115 to 125°C (239 to 257°F) is due to chemically bound water evaporation of C-S-H and cement gel,⁴⁸ from 135~140°C (275~284°F) by decomposition of ettringite, and at 185~200°C (365~392°F) by iron-substituted ettringite (Ca₆Al₂(SO₄)₃(OH)₁₂·26H₂O) and hematite (Fe₂O₃) solid solution – the reaction products of tetracalcium aluminoferrite (4CaO·Al₂O₃·Fe₂O₃).⁴⁸⁻⁵⁰ Finally aluminate hydration products, i.e. Ca₃Al₂O₆·6H₂O (C₃AH₆), Ca₂Al₂SiO₇·8H₂O (C₂ASH₈), decompose at 200~400°C (392~752°F).⁵⁰ Based on these temperature ranges, the distribution of weight loss for each cement system is shown in **Fig. 9(b)**. Total weight loss was very similar for all cement-composites when temperatures reached 125°C (257°F), approximately 30%. However, it is apparent that the decrease in OmPP was higher in the range of 115 to 125°C (239 to 257°F), which relates to chemically bound water in C-S-H, than plain and RmPP. This can help explain why OmPP exhibited higher flexural strength overall, i.e. both initial and recovered (**Fig. 6**). This can again be attributed to differences in formation and morphology of the nanosilica due to drying conditions. More investigation is needed to elucidate this.

Through SEM, the surface of micro PP fibers (treated and untreated) and the fiber-matrix interface were observed to identify the effect of nanosilica on microstructure. The site of interest was a fractured surface where fibers were either pulled out or fractured, and they are shown in **Fig. 10**. In the case of umPP, it is apparent that the fibers were pulled out from the cement matrix. There are some friction marks visible on the fiber surface, but the interface between the fiber and cement matrix appears not to exhibit any bonding phase (**Fig. 10(a)**). There was no evidence of additional hydration product forming after initial fracture, which agrees with umPP's low strength recovery. Untreated micro PP fibers generally result in poor bonding with the cement matrix and the fiber surface. There was also visible fracturing of the fiber, which can be attributed to enhanced bonding by the nanosilica coating.²⁴ And the cement matrix showed more cracking near the interface in RmPP and OmPP than umPP, which could also be evidence that the fiber surface, which could be hydration products and other constituents of the cement matrix, provide some evidence of how the treated micro PP fibers led to strength recovery (**Fig. 10(c)**).

CONCLUSIONS

In this study, nanosilica was applied to the surface of polypropylene fibers to introduce self-healing abilities when incorporated into cement-composites. Based on the results of this experimental investigation, the following conclusions are drawn:

- 1. Optical microscopy and mass change results confirmed the existence of nanosilica particles on the surface of both the micro and macro PP fibers applied through a sol-gel process at both drying conditions. Solubility matter loss results also indicated that the nanosilica remains adhered even after exposure to water, which has positive implications for mixing.
- 2. The flexural strength recovery of cement-composites reinforced with nanosilica-coated PP fiber (loaded to 60% of peak load) was evaluated after additional curing for 28days in air. It was found that the nanosilica-coated micro PP fibers led to strength recovery (up to 112.8%), especially when the nanosilica was dried at room temperature after the sol-gel process. None of the other systems exhibited strength recovery.
- 3. Through XRD and TGA, calcium hydroxide content was found to decrease in cement-composites reinforced with coated micro PP fibers, which indicates promoted pozzolanic reaction by the nanosilica.
- 4. Nanosilica-coated micro PP fiber surfaces and interfaces with the cement matrix were observed through SEM. Cement-composites with coated micro PP were observed to not only have a denser interface but also hydration products on the fiber surface, likely due to the reactivity of the nanosilica.

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Fig. 1 Polypropylene fibers

(a) micro type

(b) macro type



Fig. 2 Sol-gel process



Fig. 3 Flexural strength test setup



(dried at room temp.) Fig. 4 Surface of nanosilica-coated PP fibers

(dried at 50°C)



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Fig. 5 Flexural strength and recovery of cement-composites reinforced with nanosilica-coated macro PP fibers



Fig. 6 Flexural strength and recovery of cement-composites reinforced with nanosilica-coated micro PP fibers