PERMEABILITY CHARACTERISTICS OF CEMENTITIOUS MATERIALS REINFORCED WITH KRAFT PULP FIBRES

Emad Booya\textsuperscript{1,2}; Karla Gorospe\textsuperscript{1,3}, Adeyemi Adesina\textsuperscript{1,4}, and Sreekanta Das\textsuperscript{1,5}
\textsuperscript{1} University of Windsor, Canada
\textsuperscript{2} booya@uwindsor.ca
\textsuperscript{3} gorospek@uwindsor.ca
\textsuperscript{4} adesina1@uwindsor.ca
\textsuperscript{5} sdas@uwindsor.ca

Abstract: In the current study, an experimental program was conducted to investigate the mechanical and durability characteristics of cementitious composites reinforced with Kraft pulp fibres. Mechanically modified fibres, chemically treated fibres, and unmodified pulp fibres were used to reinforce concrete and mortar specimens. An unreinforced specimen was used as the control. The compressive strength, chloride permeability, and drying shrinkage of the fibre composites were examined. Experimental test results showed that pulp fibre composites exhibited lower strength and higher chloride ion penetration compared to the unreinforced concrete. However, a reduction in drying shrinkage strain was observed for cementitious mortars containing 1\% by weight of chemically treated fibres. The fibre interfacial characteristics of the reinforced composites were also observed via scanning electron microscopy (SEM) and it was found that fibre rupture was the governing mode of composite failure.

1. INTRODUCTION

Cementitious composites are one of the most widely used building material in the world. Nonetheless, as the construction industry becomes more environmentally conscious, initiatives towards the development of durable, eco-friendly cementitious materials have arisen. The use of fibres in cementitious composites can greatly improve the material properties of the composite. However, sustainability issues associated with some types of fibres, such as steel fibres, have compelled researchers to find and develop alternative eco-friendly fibres. Cellulosic fibres, which are obtained from plants, are green fibre alternatives that can be used to reinforce and improve the properties of cementitious composites (Faruk et al., 2012, Biagiotti et al. 2004). In addition to its sustainable advantages, cellulosic fibres are also cheaper and are often locally available compared to conventional fibres (Wambua et al. 2003). Furthermore, the morphology of cellulosic fibres can be easily engineered to enhance desired properties (Ardanuy et al. 2015). However, the limited knowledge on the durability of cementitious materials reinforced with virgin and modified cellulosic fibres have limited its large-scale application and acceptance by the construction industry.

A large percentage of the research on cementitious materials reinforced with cellulosic fibres are focused on mechanical properties, such as compressive, tensile, and flexural strength. Some durability studies have been undertaken and are available in the open literature. Nevertheless, these studies mainly examined the effect of cellulosic fibres on shrinkage (Toledo et al. 2005). Therefore, there is a need for more research on the durability of cementitious materials reinforced with these types of fibres. The durability assessment of newly developed modified cellulosic fibres, such as Kraft pulp fibres, will propel more applications of these eco-friendly fibres and will also boost the confidence of its users.
This study was focused on assessing the effect of modified pulp fibres, which is a type of cellulosic fibre, on the durability properties of cementitious composites. The pulp fibres herein are referred to as modified fibres because its physical properties have been engineered to enhance its bonding and adhesion capability with the cementitious matrix. The durability properties assessed were compressive strength, chloride ion permeability, and drying shrinkage. The results presented in this study aims to provide a source of valuable reference to stakeholders in the construction industry who are looking for sustainable ways to improve the durability of cementitious composites.

2. Experimental Program

2.1. Materials

The binder used for all mixtures is general use limestone (GUL) cement conforming to CSA A3001. The fine aggregate employed has a maximum particle size of 5 mm, fineness modulus of 2.63 and specific gravity of 2.51. The coarse aggregate used has a maximum nominal size of 19 mm and specific gravity of 2.55. A poly-carboxylate high-range water reducing admixture (HRWRA), conforming to type A and F of ASTM C494, was used to adjust the workability of the mixtures. Two types of modified pulp fibres, similar to those used by Booya et al. (2018), alongside an unmodified fibre (UF) with 0.8 mm length were used in this study. The modified fibres are mechanically modified fibres (MF) and chemically treated fibres (CF) with lengths of 1.8 mm and 2.1 mm, respectively.

2.2. Mixture Proportioning

The experimental procedure in this research was conducted in two different phases. In phase I, one unreinforced concrete mixture and three reinforced concrete mixtures incorporating UF, MF and CF were made with a water-to-cement ratio of 0.35. The fibre content in all mixtures was 2%. To enhance the dispersion of the pulp fibres, the fibres were first mixed with water (and HRWRA when needed) using a blender. Then, the cement and aggregates were dry mixed in a high-speed mixer before the premixed solution of water and fibre were slowly added. The fractured cementitious composite in Figure 1 shows the effectiveness of the mixing procedure in dispersing the pulp fibres. Phase II consisted of 10 mortar mixtures having water-to-cement ratio of 0.5 and sand-to-cement ratio of 1:1. The control mixture had no fiber in it while the remaining mixtures were reinforced with three different Kraft pulp fibres (UF, MF or CF). The fibre amount was 0.5%, 1.0%, and 2.0% by weight of cement for each fibre type. These mixtures were used to determine the influence of pulp fibres on free drying shrinkage. The test matrix and mixture details for both phases are presented in Table 1. In this table, the mixture ID was defined to reflect the fibre type and the fibre content. For example, phase I mixture MF-02 refers to a concrete mixture that contains 2 percent by weight of cement of mechanically modified fibre (MF).
Table 1: Test Matrix and Mixture Details

<table>
<thead>
<tr>
<th>Phase I (Concrete)</th>
<th>Concrete Mixture ID</th>
<th>Control</th>
<th>UF-02</th>
<th>MF-02</th>
<th>CF-02</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fiber Content</td>
<td>No fibre added</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Type of cellulose fibre</td>
<td>Bleached cellulose pulp</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Unmodified fibre (UF)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Mechanically modified fibre (MF)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Chemically treated fibre (CF)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Concrete Proportions (kg/m³)
Cement = 485; Sand = 765; Gravel = 925; Water = 170

<table>
<thead>
<tr>
<th>Phase 2 (Mortar)</th>
<th>Mortar Mixture ID</th>
<th>Control</th>
<th>UF-0.5</th>
<th>UF-01</th>
<th>UF-02</th>
<th>MF-0.5</th>
<th>MF-01</th>
<th>MF-02</th>
<th>CF-0.5</th>
<th>CF-01</th>
<th>CF-02</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fibre Content</td>
<td>No fibre added</td>
<td>0</td>
<td>0.5</td>
<td>1</td>
<td>2</td>
<td>0.5</td>
<td>1</td>
<td>2</td>
<td>0.5</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>Type of cellulose fiber</td>
<td>Bleached cellulose pulp</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Unmodified fibre (UF)</td>
<td></td>
<td></td>
<td>Unmodified fibre (UF)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Mechanically modified fibre (MF)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Chemically treated fibre (CF)</td>
<td></td>
<td></td>
<td>Chemically treated fibre (CF)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 1: Fibre dispersion in a fractured fibre reinforced composite
2.3. Test Methods

The compressive strength test of the mixtures was conducted as recommended by ASTM C39 (ASTM C39 2016) and the average of five capped cylinders were reported at 28 days. The samples were first removed from a curing chamber and kept out exposed to air dry in an ambient temperature for two days until testing. Scanning electron microscopy (SEM) was used to study the fibre-matrix interface and the microstructural properties of the reinforced cementitious mixtures.

The chloride ion permeability in terms of charge passed through the concrete was conducted at testing ages of 28 days and 90 days. For each concrete mixture and at each testing age, two 50 mm disk specimens were cut from the mid-portion of Ø100 × 200 mm cylinders and were conditioned as per ASTM C1202 (ASTM 2012). A computerized data acquisition system recorded the direct current (DC) of 60 ± 0.1 volts that passed through the concrete over a period of six hours. The total charge (coulombs) passed through the specimen was computed using the current and time history.

Drying shrinkage tests were performed as per ASTM C596 (ASTM 2009). Mortar bar specimens with dimensions of 25 x 25 x 250 mm were cast then cured in its molds for 24 hours. Afterwards, the specimens were cured in water for 48 hours. Upon removing from the water, the mortar bar specimens were surface dried and initial length readings were taken. A room with a set temperature of 22°C (± 1°C) and relative humidity of 40% (± 2%) was used to store the mortar bar specimens. After 25 days of storage (28 days after casting), length readings were obtained and the average shrinkage of four specimens was reported.

3. Results and Discussion

3.1. Compressive Strength

The compressive strength test results are presented in Table 2. It was found that the addition of fibres decreases the compressive strength of the concretes. Hence, the control (unreinforced) concrete had the highest compressive strength of 52.5 MPa and 54.8 MPa at 28 and 90 days, respectively. Concretes incorporating UF marked the highest reduction in compressive strength of about 28%. These findings contradict that reports by Bhargava and Banthia (2008) for mixtures containing 0.1% and 0.3% by volume of virgin fully purified cellulose fibre. This could be attributed to the fact that proper compaction was difficult to achieve since large amounts of fibres were used in this study (2% by weight of cement). On the other hand, concretes reinforced with modified fibres (MF and CF) had less compressive strength reduction compared to concrete reinforced with UF. Hence, this study found that modified fibres (MF and CF) enhanced the axial load transfer by providing a better fibre-matrix bond. Moreover, it is also possible that both MF and CF induced less voids in the concretes (Booya et al. 2018). The compressive strength of MF and CF concretes were 44.4 MPa and 44.1 MPa at 28 days, respectively.

Table 2: Test results for phase I mixtures

<table>
<thead>
<tr>
<th>Mixture ID</th>
<th>Age</th>
<th>Compressive Strength (MPa)</th>
<th>Chloride Ion Charge (Coulombs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>28</td>
<td>52.5 (2.3)</td>
<td>3374 (75)</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>54.8 (1.4)</td>
<td>2362 (85)</td>
</tr>
<tr>
<td>UF</td>
<td>28</td>
<td>37.0 (1.6)</td>
<td>5560 (250)</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>45.1 (2.5)</td>
<td>3799 (9)</td>
</tr>
<tr>
<td>MF</td>
<td>28</td>
<td>44.4 (3.9)</td>
<td>4682 (22)</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>54.3 (1.1)</td>
<td>3052 (5)</td>
</tr>
<tr>
<td>CF</td>
<td>28</td>
<td>44.1 (2.9)</td>
<td>4356 (214)</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>47.6 (1.0)</td>
<td>3102 (88)</td>
</tr>
</tbody>
</table>

Note: Values in parentheses are the standard deviation of the mean.
3.2. Chloride Ion Permeability

The rapid chloride penetration test is a good assessment for checking the durability of cementitious composites. The ease of chloride ion movement through the mixtures was assessed in accordance with ASTM C1202 (ASTM 2012), which gives the total charge passed at the end of a 6-hour test. The higher the total charge passed, the more permeable the composite. Table 2 shows the total charge passed for each mixture at 28 and 90 days. It was observed that at all ages, the total charge passed for all mixtures containing fibres is greater than that of the control. However, at 90 days, the charges reduced by approximately 40%, indicating hydration progression. Mixtures containing CF and MF outperformed mixtures containing UF as CF and MF mixtures exhibited lower charges at both ages. Moreover, in accordance with ASTM C1202 (ASTM 2012), the total charge passed at 90 days for the mixtures containing modified fibres is still acceptable as they are considered moderate, having less than 4000 coulombs. The higher chloride ion permeability of the composites containing fibres might be due to the porous nature of the pulp fibre which eased the transfer of the ions. Hence, the permeability properties of cementitious composites highly depends on its pore structure (Guneyisi et al. 2015). Sappakittipakorn and Banthia (2012) studied the influence of unmodified pulp fibre (virgin pulp) on the chloride permeability of concretes. The study found that the interfacial porosity at the fibre matrix zones and the existence of connected pores are the main factors that contribute to the increase in chloride permeability.

3.3. Drying Shrinkage

Figure 2 presents the free drying shrinkage strain values determined at the end of eight weeks. Generally, the addition of pulp fibres has insignificant influence on the drying shrinkage and the volume stability of the mortar mixtures, regardless of the fibre type. At a fibre content of 0.5%, the drying shrinkage strains for mixtures UF-0.5, MF-0.5, and CF-0.5 were comparable to the control mixture. However, increasing the amount of CF to 1% (CF-01) reduced the shrinkage by about 5%. Similar observations were previously reported by Kawashima and Shah (2011), based on their study on conventional cellulose fibre concrete. However, higher fibre amounts of 2% did not show any reduction in drying shrinkage strain. This is likely due to the fact that mixtures with larger amounts of fibres required higher amounts of superplasticizer, which resulted in an increase in drying shrinkage strain. Hence, the current study concludes that incorporating modified fibres reduces the drying shrinkage at fibre contents of less than 1%.

![Figure 2: Free drying shrinkage test](image-url)
3.4. Scanning Electron Microscopy

Scanning electron microscopy (SEM) of MF and CF embedded in the cementitious matrix, at the age of 90 days, are presented in Figure 4. From these images, it is evident that a good bond exists between the fibres and the cementitious matrix. Moreover, the figures show a densified cementitious matrix. These observations are further confirmed by the fact that fibre rupture occurred as opposed to debonding. Though SEM investigation shows no ettringite formation around the fibre, some ettringite was observed in the pores of the specimen made with MF, as shown in Figure 5. This might be a result of supplementary sulphate in the composite (Mohr et al. 2006).

![Figure 4: SEM images of fibre reinforced mortars with Kraft pulp fibres](image1)

![Figure 5: Needle shaped ettringite in composites containing MF at a magnification of x4000](image2)

4. Conclusions

This study explored the effects of modified pulp fibres on the durability and mechanical properties of cementitious materials. Based on the test results, the following conclusions can be drawn.

1. The incorporation of pulp fibres led to a decrease in the compressive strength of the cementitious composites. However, composites containing mechanically modified pulp fibre (MF) exhibited comparable compressive strength as that of the control at 90 days.
2. The addition of pulp fibres generally increases the chloride permeability of concrete. Nonetheless, it was found that concretes containing modified fibres (MF and CF) exhibited lower charges passed compared to concretes containing unmodified pulp fibres (UF).

3. Modified fibres can be successfully incorporated into cementitious composites to reduce drying shrinkage strains when fibre amounts of 1% or less are used.

References


