

## Technical Note

## Properties of nanofibre reinforced cement composites

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## ABSTRACT

**Objective:** This paper investigates the potential and limitations of using relatively low volume fractions of cellulose nanofibre as reinforcement in cement pastes.

**Materials and methods:** General use limestone (GUL) cement and cellulose nanofibre gel suspension were used in the preparation of cement paste. Mixture conductivity, hydration, flexural strength, energy absorption and fibre dispersion in hardened cement matrix were evaluated.

**Results:** Cellulose nanofibre reduced the conductivity and the early age hydration of mixtures. However, at 28 days, the cumulative heat of hydration and the degree of hydration of cement pastes containing cellulose nanofibre were higher than those of the unreinforced reference mixture. Optimum mechanical properties were observed for paste containing 0.1% nanofibre. The decrease in mechanical properties at higher nanofibre content is as a result of fibre agglomeration. In comparison to the reference paste, the flexural strength and energy absorption of 0.1% nanofibre paste increased by approximately 106% and 184%, respectively.

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## 1. Introduction

Steel and polymer fibres have been shown to enhance the ductility, tensile strength, toughness, fatigue strength, impact resistance and energy absorption capacity of cement composites [1–4]. However, these aforementioned properties of cement composites could also be improved with vegetable fibres. The use of vegetable fibres as reinforcement for cement composites is particularly attractive since they are procured from renewable sources and are available for a relatively lower cost in comparison to synthetic fibres. However, chemical constituents and soluble sugars contained in vegetable fibres have been reported to delay the hydration of cement-based mixtures [5–9]. This drawback could be reduced through fibre processing and the addition of chemical accelerators to mixtures.

Previous studies have reported improved mechanical strength of vegetable fibre reinforced cement composites. Research findings by Ramakrishna and Sundararajan [10] showed that the impact resistance of vegetable fibre reinforced mortar slabs was 3–18 times greater than those of the unreinforced slabs. A 9–60% improvement in the flexural strength of concrete mixtures reinforced with different vegetable fibres has also been reported [11–13]. In comparison to unreinforced concrete, Li et al. [14]

showed that the flexural toughness and flexural toughness index of hemp fibre reinforced concrete mixtures increased by 144% and 214%, respectively.

Presently, nano-manufacturing is generating interest across many industries. Substantial improvements in the mechanical properties of polymer composites reinforced with 5–16.5% by weight of cellulose nanofibres have been reported [15–17]. Wang and Sain [15] reported that relative to pure polyvinyl alcohol (PVA), the tensile strength and stiffness of PVA composite reinforced with 5% soybean nanofibre increased by 58% and 170%, respectively. Compared to pure polyurethane (PU), approximately 500% increase in strength and 3000% increase in stiffness were observed in a PU matrix reinforced with 16.5% by weight of wood cellulose nanofibril [16]. A recent study by Xu et al. [17] showed that 7% by weight addition of cellulose nanofibre improved the flexural strength and toughness of polyethylene oxide (PEO) matrix by 92% and 732%, respectively. Hence, given that cracking and load induced deformations of cement composites originate at the nanoscale, the use of nano-sized cellulose fibres with very high specific surface area as reinforcement could enhance the mechanical strength of cement-based materials significantly.

This study investigates the hydration and mechanical properties of cement pastes reinforced with cellulose nanofibre. The aim is to evaluate the potential and limitations of using relatively low volume fractions of cellulose nanofibres as reinforcement in cement pastes.

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## 2. Experimental works

### 2.1. Materials

The binder used in preparing paste mixtures in this study was general use limestone (GUL) cement. The nanocellulose fibre gel was prepared by mechanical defibrillation of bleached softwood pulp procured from Domtar Canada. To ensure complete defibrillation, the softwood pulp was fed through a super fine disc grinder for 10–15 number of times. Thereafter, distilled water was added to the ground pulp to form a viscous translucent nanofibre gel suspension.

#### 2.1.1. Physical and chemical properties of constituent materials

The physical properties and chemical composition of the cement is shown in Table 1. The average length of the nanofibres ranged from 1.0 to 2.5  $\mu\text{m}$ , and the moisture content was 98.1%. Visual images of the nanofibres are shown in Figs. 1a–1c. Fig. 1a shows a highly viscous nanofibre gel. Fig. 1b shows the resultant translucent mix water containing dispersed nanofibres. The transmission electron microscope (TEM) image shown in Fig. 1c indicates that nanofibre suspension consists of a complex network of high aspect ratio interwoven nanofibres. The agglomerates shown in Fig. 1c are irregularly distributed, depending on nanofibre concentration and the degree of entanglement.

#### 2.1.2. Mixture proportions

At a constant water-to-cement (w/c) ratio of 0.5, paste mixtures incorporating nanofibre at 0%, 0.05%, 0.1%, 0.2% and 0.4% by mass of cement were prepared, and are identified as reference, 0.05 N, 0.1 N, 0.2 N and 0.4 N, respectively. For each mixture, nanofibre gel and the mixing water were pre-mixed for 5 min in a beaker using a hand mixer. To aid the dispersion of fibres, a polycarboxylate based superplasticizer was added to the mixing water at a maximum dosage rate of 1.6% by mass of cement. The fibre-mixing water suspension was then agitated for 10 min in an ultrasonic bath. Thereafter, cement paste was mixed in two stages (140 and 285 revolutions/min) with a Hobart mixer, each mixing stage was 2.5 min.

#### 2.1.3. Casting and curing

Several 40 mm  $\times$  40 mm  $\times$  160 mm moulds were used and filled in two layers, with each layer compacted for 30 s using a vibrating table. Upon completion of casting operations, samples demoulded at 24 h and were then stored at a 95% relative humidity and 23.0  $\pm$  2.0  $^{\circ}\text{C}$  for 28 days.

### 2.2. Test methods

#### 2.2.1. Cement paste conductivity

Due to equipment tolerance limit, only the conductivity of the reference mixture, 0.05 N and 0.1 N mixtures was measured using a YSI conductivity meter. The first measurement was taken 5 min after mixing operation commenced. Thereafter, subsequent measurements were taken after 30 min, 60 min, 90 min and 120 min after mixing. Two samples from each mixture were tested.

#### 2.2.2. Cement paste hydration

Heat evolution in cement pastes was monitored with a TAM Air isothermal calorimeter. For each mixture, two samples weighing 6 g each were used, and the heat evolution was measured at a constant temperature of 23  $^{\circ}\text{C}$ . From the heat-time plots, the time to reach the peak silicate heat of hydration of each mixture was determined.

**Table 1**  
Chemical and physical properties of GUL cement.

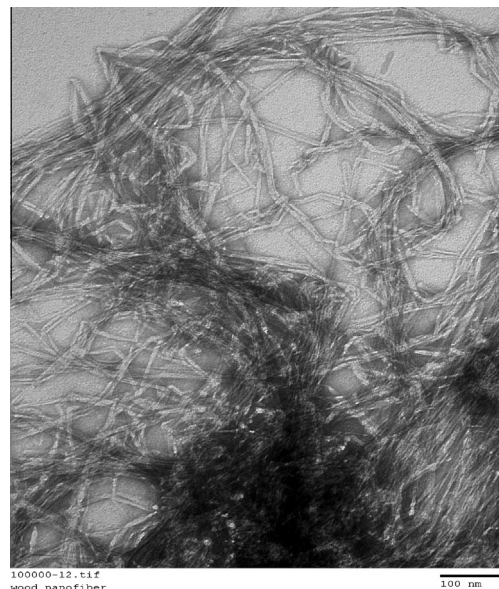
Component	
<i>Chemical composition (%)</i>	
SiO <sub>2</sub>	17.92
Al <sub>2</sub> O <sub>3</sub>	4.94
Fe <sub>2</sub> O <sub>3</sub>	2.24
CaO	60.70
MgO	2.24
CaCO <sub>3</sub>	–
SO <sub>3</sub>	4.14
Cl	–
Total alkali	0.90
Free lime	1.21
Loss-on-ignition	5.90
Insoluble residue	–
<i>Physical properties</i>	
Blaine specific surface area (m <sup>2</sup> /kg)	491.00
BET specific surface area (m <sup>2</sup> /kg)	–
Fineness, retained on 45 $\mu\text{m}$ sieve (%)	4.98



**Fig. 1a.** Stock nanofibre gel.



**Fig. 1b.** Nanofibre-mix water suspension.



**Fig. 1c.** Transmission electron microscope image of diluted nanofibre gel in aqueous solution.

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