Use of NanoFibrillated Cellulose for Improved Strength and Reduced Cement Content in Cementitious Paste Backfill

Keith Gourlay[1], Mehrdad Kermani[2], Agus Sasmito[2], Gurminder Minhas[1] and Ferri Hassani[2]

[1]Performance BioFilaments Inc.; [2]Department of Mining and Materials Engineering, McGill University

Abstract

The mechanical performance of cemented paste backfill (CPB) can be enhanced by incorporating very low amounts of nano-fibrillated Cellulose (NFC), a novel and sustainable biomaterial derived from wood pulp. This study demonstrates that even a minimal addition of 0.1 wt% NFC significantly accelerates the strength development in CPB, while simultaneously enhancing the ultimate strength of the cured material. In a CPB system containing 5% cement, the addition of 0.1% NFC resulted in nearly doubling of the 7 day compressive strength. This achievement led to a 7 day compressive strength in the NFC-supplemented sample equivalent to the 14 day compressive strength of the control sample. These results indicate that low addition levels of NFC have the potential to significantly reduce the required cement content to achieve desired mechanical performance. Furthermore, it is hypothesized that incorporating NFC into the plug can play a pivotal role in continuous fill operations, where high yield stress and rapid curing of the plug are advantageous. Additionally, NFC could be used to reduce the overall cement content and therefore contribute to a reduced carbon footprint in CPB.

Introduction

As mining operations seek new strategies for reducing greenhouse gas emissions, one target is the cement content used in CPB. Recent developments in wood pulp processing have led to the development of new biomaterials with the ability to significantly reinforce cementitious materials such as concrete (Onuaguluchi et al., 2021, 2023).

NFC is a new high-performance biomaterial produced through mechanical refining of wood pulp. This refining process promotes the fibrillation of the wood pulp fibers, resulting in the conversion of individual pulp fibres (approx 20–30 µm width and 2–3 mm length), into hundreds of very high-aspect ratio nanofibrils (approx 30–500 nm width and 100–500 µm length). The resulting nano-fibrillated material is insoluble in water and has a very high number count (fibrils/g of material). In addition, NFC has an exceptionally high surface area (approx 120–150 m^2/g) and aspect ratio (length:width ~ 1000–1200). These unique properties impart NFC with exceptional reinforcing capabilities in concrete (Onuaguluchi et al., 2021, 2023).

For this research NFCs were integrated into CPB and the rheological and mechanical properties were assessed, to our knowledge for the first time. The rheological properties of the resulting NFC-reinforced CPB formulations were assessed, followed by an assessment of the mechanical performance of these materials. In order to develop a lower-carbon solution for CPB, the cement used in this study was a blend of 90% blast furnace slag and 10% General Use (GU) cement.

Methodology

Materials

The tailings used in this study were initially obtained from a gold mine located in northern Ontario. These tailings were partially dried and subsequently reconstituted in water for the purpose of our investigation. To ensure uniformity, tailings underwent homogenization using a ribbon drill mixer. Characterization of the tailings involved measuring specific gravity using a pycnometer using ASTM D 854-10. Particle size distribution analysis was performed employing laser diffraction particle sizing. For particle size distribution testing, two 50 g representative samples were carefully selected and prepared using a riffle sample splitter. The coefficient of uniformity was calculated, along with the percentage of sand, silt, and clay based on the MIT soil classification system. Tailings mineralogy was also determined by X-ray Diffraction (XRD).

NFC was provided by Performance BioFilaments Inc (PBI). The NFC from PBI is produced from Canadian softwood Kraft pulp using a proprietary, high-consistency mechanical refining process. This process converts individual wood pulp fibers (approx 20–30 µm wide and 1.5–2.5 mm long) into hundreds of nanofibrils (approx 20–500 nm wide and 100–800 µm long). These exceptionally high-aspect ratio fibrils have a specific gravity of approx $1.5 g/cm^3$, exhibit shear-thinning behaviour in aqueous dispersions, and have high water-holding ability. After refining, the resulting fibrillated NFC was then redispersed in water to the desired consistency for dosing into the final application (approx 1–10% solids in water).

Rheology Testing
A 90:10 mixture of blast furnace slag:general use cement provided by Lafarge Canada was used for this work. A desktop shear rheometer was used to measure the rheology of tailings samples and specifications were obtained (Table 1).

Table 1: Specifications of desktop shear rheometer.

Description	Details
Rheometer	HAAKE Viscotester 550
Rotation Rate (←/min)	75
Rate of Shear (1/s)	0.04355
Cup and bob	ISO 3219-1993
Bob radius (mm)	19.36
Cup radius (mm)	21.00
Bob height (mm)	58.08
Room Temperature (°C)	22

A bob and cup attachment complying with ISO3219-1993 was used to monitor the shear stress developed in the backfill over a range of applied torque and angular viscosity. The rheometer results were exported to an Excel sheet for curve-fitting on to the non-Newtonian Bingham plastic equation (Equation 1).

$$\tau = \tau_B + \mu_P \gamma \qquad \text{Equation 1}$$

For rheology testing, the cement composition was varied from 0 to 5%, and NFC loading levels of 0, 0.1, and 0.2 wt% (relative to total dry solids) were used. Samples were tested over a range of moisture contents, and at least three replicates of each condition were measured. All tests were conducted at 15°C and were performed after the backfill had been fully sheared for accurate analysis.

Uniaxial compressive strength testing (UCS)

Tailings, binder and water were measured separately. The tailings and binder were first added into a 5-quart stainless steel bowl and mixed for one minute at a stir rate of 75 RPM. Water was then introduced and the materials were mixed for a further 5 mins. Mixtures were poured into cylindrical polyvinyl chloride (PVC) molds. The molds used were 10 cm long with a 5 cm diameter. Molds were capped and sealed after casting. For each batch, 7 PVC molds were cast. The capped samples were stored in a humidity chamber at 90 ± 4% RH, and temperature was adjusted to 23 ± 2 °C. Before conducting compressive strength testing, samples were demolded and the top and bottom of the samples were trimmed flat to reach a ratio of 2:1 (±5%) height:diameter.

The UCS testing was conducted on a 50 kN compression testing machine according to ASTM D 2166-06, specifically the standard test method for unconfined compressive strength of cohesive soil (ASTM 2006D). The press was equipped with a 50 kN load cell and a Linear Variable Displacement Transducer sensor (LVDT). A normal loading rate under displacement control of 1 mm/min was applied and data were collected digitally.

Results

Tailings Characterization

The specific gravity of the tailings was measured at 2.5 g/cm^3 using a pycnometer adhering to ASTM D 854-10. The particle size distribution of the tailings was determined through laser diffraction particle sizing (Figure 1). Particle size distribution fractions and coefficients of uniformity and curvature were also calculated (Table 2).

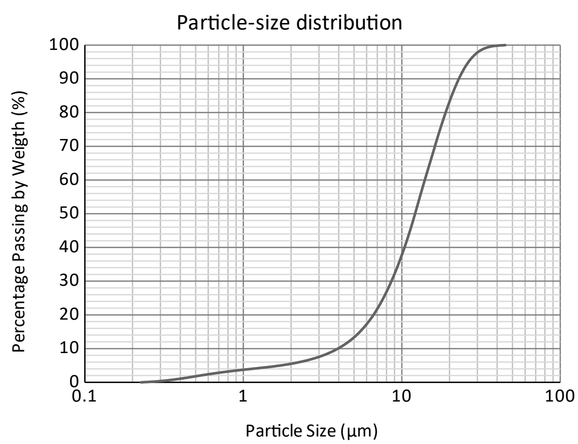

Figure 1. Particle size distribution of tailings

Table 2: Coefficients of uniformity and curvature for the tested tailings.

Particle size	Tailings (%)
D_{10} (μm)	3.95
D_{30} (μm)	8.6
D_{50} (μm)	12.1
D_{60} (μm)	14
D_{90} (μm)	23
Cu $C_u = \frac{D_{60}}{D_{10}}$	3.5
Cc $C_c = \frac{(D_{30})^2}{D_{10} \times D_{60}}$	1.3

Table 3. Percentage of sand, silt and clay for tested tailings.

Description	Tailings (%)
Sand (% passing 2 mm - % passing 0.06 mm)	27
Silt (% passing 0.06 mm - % passing 0.002 mm)	56
Clay (% passing 0.002 mm)	17

XRD results are summarized in Table 4. The results indicate that the tailing samples consisted predominantly of quartz, dolomite, ankerite, and muscovite. The elemental compositions of the tailings were analysed using the X-ray fluorescence (XRF) method (Table 5).

Table 4: Summary of semi-quantitative XRD results for tailings.

	Major (>30% Wt)	**Moderate (10% -30% Wt)**	**Minor (2% -10% Wt)**	**Trace (<2% Wt)**
Tailings	-	quartz, dolomite, ankerite, muscovite	biotite, chlorite, albite, orthoclase	calcite, magnetite, pyrite

Table 5: Tailings XRF result

Formula	Concentration (%)
SiO_2	44.6
Al_2O_3	9.18
Fe_2O_3	5.07
MgO	7.89
CaO	11.2
Na_2O	0.26
K_2O	2.32
TiO_2	0.51
P_2O_5	0.17
MnO	0.15
$C_{r2}O_3$	0.08
V_2O_5	0.03
LOI	18.2
Sum	99.7

Rheology
A rheometer with cup and bob attachment was used to measure the Bingham Yield Stress at 5% cement for 0, 0.1, and 0.2% NFC addition across a range of solids contents (Figure 2). The same testing was done at a fixed NFC loading of 0.2 wt% using 0, 2.5, and 5% cement over a range of solids contents (Figure 3). It was found that formulations containing 5% cement exhibited an increase in yield stress with increasing NFC loading. It was also found that at constant NFC loading of 0.2 wt%, increasing the cement content also increased the yield stress, although this effect only became pronounced at the higher (5%) cement loading.

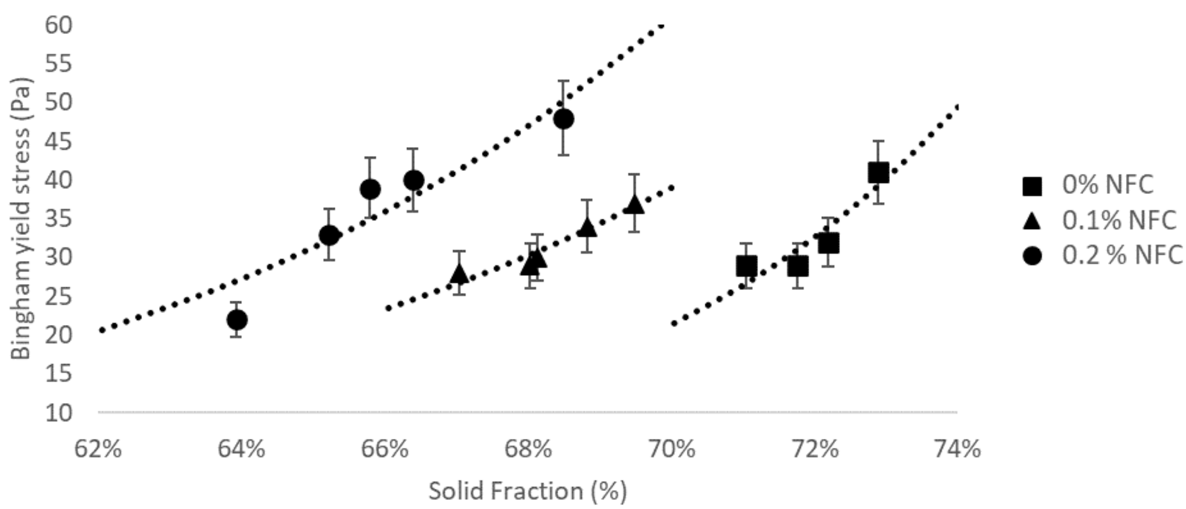

Figure 2. Bingham Yield Stress at 5% cement for 0, 0.1, and 0.2 wt% NFC at various solids contents.

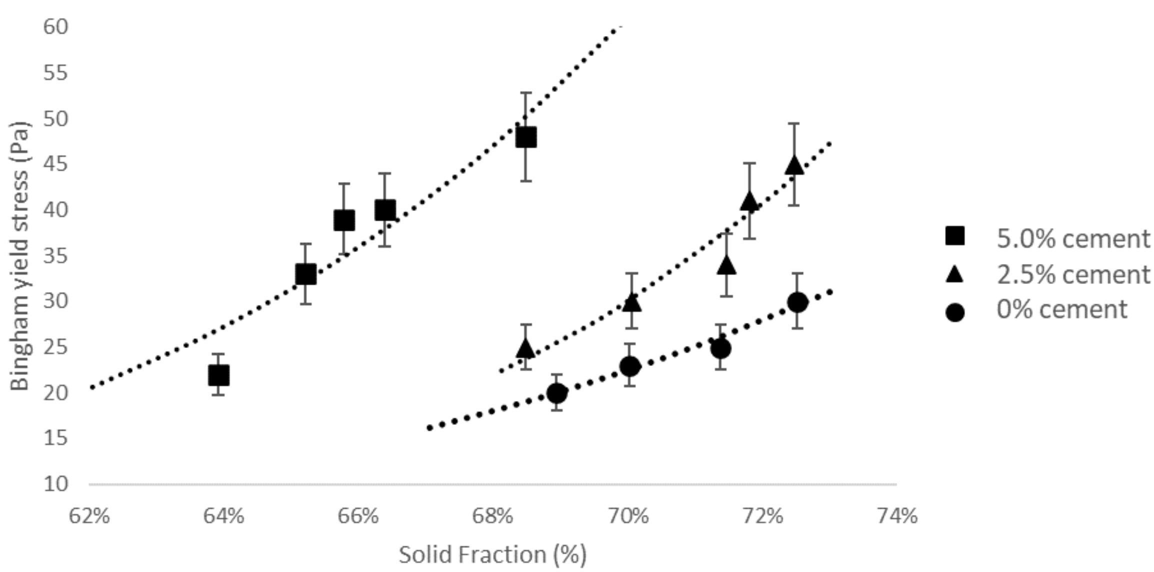

Figure 3. Bingham Yield Stress at 0.2 wt% NFC for 0, 2.5, and 5.0% cement at various solids contents.

UCS Results

For all UCS testing, two replicates of each sample type were tested, and average of the two are presented here. UCS measurements were taken at 7, 14, and 28 days of curing under controlled temperature and humidity. Table 6 shows the formulations of the tested mixes, as well as the resulting UCS data.

Table 6. Formulations, rheology results, and UCS test results for NFC-treated tailings containing 2% or 5% 90:10 Slag:Cement (S:C).

Batch	**Test Mix Design**								
	Tailings (%)	**90:10 Slag:Cement (%)**	**NFC (%)**	**Solid Conc. (%)**	**Req. Yield Stress (Pa)**	**Actual Yield Stress (Pa)**	**7 days (MPa)**	**14 days (MPa)**	**28 days (MPa)**
0.1% NFC 5% S:C	94.9	5	0.1	73.2	250	700+*	0.44	0.61	1
0% NFC 5% S:C	95	5	0	73.2	250	280	0.25	0.48	0.8
0.1% NFC 2% S:C	97.9	2	0.1	73.2	250	700+*	0.08	0.24	0.31
0% NFC 2% S:C	98	2	0	73.2	250	200	0	0.06	0.14

UCS results were plotted for 0 and 0.1 wt% NFC at 2 and 5% cement (Figure 4). These results demonstrate that addition of 0.1 wt% NFC promoted a rapid and dramatic increase in UCS.

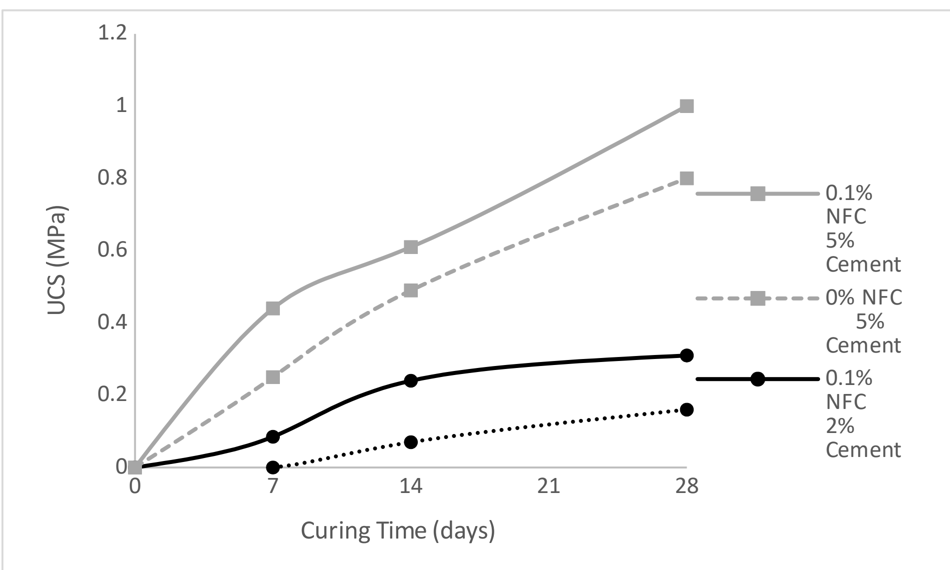

Figure 4. UCS results after 7, 14, and 28 days curing for 0 and 0.1 wt% NFC in formulations containing 2% and 5% cement.

Discussion

Our results demonstrate that NFC addition to CPB has a dramatic effect on expediting early-age strength gain. This rapid increase in strength gain can reduce delays associated with waiting for CPB to set prior to carrying out adjacent excavations. Specifically, the results for both the 2 and 5% cement samples (Figure 4) demonstrate that addition of NFC reduces time to a given strength by approximately 50% (ie, strength achieved after 14 days without NFC can be achieved in just 7 days with 0.1 wt% NFC addition). Additionally, NFC is expected to play a beneficial role in the plug portion of backfill operations which utilize a cemented plug followed by bulk backfill addition. Due to its ability to facilitate rapid strength gain, NFC is expected to significantly decrease the setting time requirement for the plug to attain the desired strength prior to continuation of filling. Further research into even earlier setting timepoints is required, particularly those occurring within the first seven days, to evaluate the extent of NFC advantages in the minutes and hours following the pouring of the plug. If a demonstration of strength gain in NFC-containing samples at very early timepoints is achieved, then it seems likely that NFC addition could be used to enable continuous-fill backfill operations, where bulk backfill is added immediately after pouring of the plug.

Due to previous work demonstrating NFC ability to retain water and gradually release it throughout the cement curing process, it is thought that use of NFC in mine backfill is likely to minimize water runoff while simultaneously reducing the self-heating of the backfill during curing (Gwon et al., 2024, Haque et al., 2022). One drawback to the use of NFC is the viscosity build presented in Figure 2. These results show a significant viscosity increase upon NFC addition. However, prior work has demonstrated that this NFC-induced rheology build can be offset through addition of low dose superplasticizer (Peters et al., 2010). Additionally, it may be possible to optimize NFC loading level to achieve desired strength gains without pushing the viscosity above required limits.

Finally, although NFC is a bio-based material with potential for long-term degradation over time, the data presented here suggest that the primary effect of NFC use is to expedite the time to a given strength gain in CPB systems. Specifically, if NFC is used to achieve faster strength gain and is not relied upon to increase the ultimate strength of the CPB, then it appears that NFC could make a valuable addition to CPB operations. Accelerated aging studies may be required to confirm this hypothesis. Given the very significant increases in early-age and ultimate strength of NFC-supplemented CPB, it appears likely that the costs of NFC plus superplasticizer could be offset by the gains in process efficiency (eg, faster fill time, enabling of continuous-fill operations) and potential cement reduction (cement cost savings). Additionally, the low-carbon nature of NFC will lead to carbon savings when used to offset cement use in CPB operations.

Next steps in this research should involve selection of specific operation sites where rapid strength gain is of particular interest. Dose optimization of NFC for specific strength gain targets in tailings from these sites should then be carried out. Finally, a cost-benefit analysis for this optimized NFC loading level in these specific operational environments should be completed.

Conclusions

NFC is a recently-developed high-performance sustainable biomaterial produced from wood pulp. In this paper we describe how very low loading levels of NFC can be used to dramatically increase the early age strength of CPB. This strengthening effect can be exploited to improve operational efficiency in mining operations. Specifically, faster strength gain can reduce the time required prior to excavating material adjacent to or below the CPB fill site. Additionally, rapid strength gain can enable the switch to continuous-fill backfill operations, where a plug containing NFC is poured and is immediately followed by filling with the bulk cemented paste backfill material. Overall, it is expected that the additional costs of NFC addition into a CPB will be offset by efficiencies in strength gains.

References

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