



# Use of pulp and paper mill residual solids in production of cellucrete

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## Abstract

Fibrous residuals generated from pulp and paper mills were included in concrete. Carefully proportioned concrete mixtures containing some of the residuals showed higher compressive and splitting-tensile strengths than concrete without the residuals. Overall, a high correlation was observed between density and strength of concrete containing the residuals. By achieving equivalent density, concrete containing the residuals may be produced equivalent in strength to the concrete without the residuals.

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## 1. Introduction

Pulp and paper mill wastewater-treatment plant residuals are the solid residue removed from mill wastewater before the water is discharged into the environment or reused in the mill. Residuals are removed via a two-step process of treating the wastewater [1–4].

The primary residual is the solid residue removed from the primary clarifier. The primary clarification is usually carried out by sedimentation and sometimes by dissolved air flotation. The primary residual consists mainly of cellulose fibers, papermaking fillers (kaolinitic clay, calcium carbonate, and/or titanium dioxide), and water. In some cases, ash generated at mills and inert solids rejected during chemical recovery processes become part of the primary residual. The water clarified by the primary treatment is passed on to the secondary treatment.

The secondary treatment is usually a biological process in which microorganisms convert soluble organic matter to carbon dioxide and water while consuming oxygen. The secondary residual is mainly microbial biomass (also called biosolids) grown during this process and removed through clarification.

Many times, primary and secondary residuals are combined to facilitate handling. In most cases, the residuals are dewatered before they are being used or disposed of.

In 1995, the U.S. pulp and paper industry generated about 5.3 million metric tons of mill wastewater-treatment residuals (on oven-dry basis), which is equivalent to about 15 million metric tons of dewatered (moist) residuals. About half of this was disposed in landfills/lagoons, a quarter was burned, one-eighth was applied on farmland/forest, one-sixteenth was reused/recycled in mills, and the rest, one-sixteenth, was used in other ways [5].

Due to increasing cost of landfilling, increasingly stringent environmental regulations, and potential long-term environmental liabilities, the percentage of the residuals that are disposed of in landfills has decreased considerably over the past several decades [1,2,5]. However, a significant amount of residuals still need to be diverted from landfilling.

Numerous projects have been conducted on the use of cellulose fibers as reinforcement in pressed cement and/or mortar sheets. Cellulose fiber-reinforced cement-based sheet composites showed considerably higher flexural toughness than plain cement-based sheets [6,7]. Use of cellulose fibers reduced the extent of shrinkage cracking of mortar [8] and improved the resistance of cement-based composites to freezing and thawing [9].

Being very fine and slender, cellulose fibers can be placed uniformly in concrete and can effectively bridge, suppress, and stabilize microcracks [10]. It has been

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reported that the use of proprietary (processed) virgin cellulose fibers is effective in reducing plastic-shrinkage cracking [11] as well as drying-shrinkage cracking [12].

Concrete is weak in tension (3–9 MPa). Wood cellulose fiber is strong in tension (300–900 MPa) [13]. Therefore, the use of wood fibers in concrete should improve the usefulness of concrete. The fibrous residuals from pulp and paper mills could become an economical source of wood fibers for microfiber reinforcement of concrete.

No research results have been reported in any technical journals or conference proceedings, on the use of pulp and paper mill residuals in structural-grade concrete. This report summarizes the results of a small-scale research project conducted on the use of the residuals in concrete. Results of an extensive research project, which was conducted later, are available elsewhere [14,15].

## 2. Materials and deflocculation of the residuals

### 2.1. Cement, fine and coarse aggregates, and chemical admixture

Type I portland cement, fine aggregate (sand), and coarse aggregate (pea gravel with a 9.5-mm maximum size) supplied by a local ready-mixed concrete producer were used in this research. The cement and the aggregates met the requirements of ASTM C 150 and C 33, respectively. The high-range water-reducing admixture (HRWRA) used in this research was an aqueous solution of a modified naphthalene sulfonate and met the requirements of ASTM C 494 for Type F (HRWRA). The manufacturer's recommended dosage rate for the HRWRA was 0.39–1.3 liter/100 kg of cement.

### 2.2. Pulp and paper mill residuals

A total of three sources of fibrous residuals from pulp and paper mills were used—one each from a de-ink paper mill in the USA, a pulp mill in the USA, and a pulp mill in Canada. Properties of the residuals are presented in Tables 1 and 2. In the research [14,15]

Table 2

Oxides composition of ash left after ignition of residuals at 1000 °C

Residuals	N	F	K	Average
Ash (%) <sup>a</sup>	36.1	24.7	17.7	26.2
Oxides (%) <sup>b</sup>				
SiO <sub>2</sub>	47.6	33.4	53.3	44.8
Al <sub>2</sub> O <sub>3</sub>	37.0	2.5	9.5	16.3
CaO	9.4	57.5	13.2	26.7
MgO	2.0	1.6	11.0	4.9
Fe <sub>2</sub> O <sub>3</sub>	0.8	1.6	4.7	2.4
TiO <sub>2</sub>	4.9	0.1	0.4	1.8
K <sub>2</sub> O	0.4	0.7	4.4	1.8
Na <sub>2</sub> O	0.7	1.4	2.4	1.5

<sup>a</sup> Percent of oven-dry (105 °C) mass of residuals.

<sup>b</sup> Percent of ash (1000 °C) by mass.

conducted later using other various sources of residuals, the properties of most of the residuals were relatively uniform and consistent.

### 2.3. Deflocculation (or “repulping”) of residuals

Due to flocculation and dewatering, the as-received residuals contained moist fibrous clumps that consisted of wood fibers, kaolin-type clay (residual N), and other particles—calcium carbonates, silica (residuals F and K), and carbon (residual F). These clumps may be considered as weaker spots in concrete compared with well-dispersed individual fibers and particles. In addition, in order for the fibers to function efficiently as fibers, they must be separated into individual fibers as much as possible.

Therefore, the fibrous residuals were deflocculated, or “repulped,” into separated wood fibers and particulates before they were introduced into a concrete mixture. The “pulper” used for this purpose in the laboratory consisted of a 19-liter plastic bucket and a high-speed mixer with a rotor positioned above the bottom of the bucket. Mechanical repulping was performed by immersing the fibrous residuals in room-temperature water with a prescribed amount of HRWRA in the bucket and subjecting the mixture to a high-speed rotation by the rotor blades for not less than 20 min. HRWRA was used with the assumption that it would help “repulp” the residuals. However, in the research conducted later [14,15], it was shown that the

Table 1

Types, physical properties, loss on ignition at 590 °C, and wood fiber content of residuals

Residuals	Type of residual	Type of mill	Fiber origin	Moisture content (%) <sup>a</sup>	Average fiber length <sup>b</sup> (mm)	Loss on ignition at 590 °C (%) <sup>a</sup>	Wood fiber content (%) <sup>a</sup>
N	primary	paper	recycled	87	1.46	42	46
F	primary	pulp	virgin	125	— <sup>c</sup>	41	40
K	primary	pulp	virgin	138	1.81	78	83
Average	—	—	—	117	1.64	54	56

<sup>a</sup> Percent of oven-dry (105 °C) mass of residuals.

<sup>b</sup> Length-weighted average fiber length,  $L_L = \sum_{i=1}^N n_i l_i^2 / \sum_{i=1}^N n_i l_i$ , using Kajaani FS-200.

<sup>c</sup> Not available. “Could not run... due to shives and/or large particles.”

Table 3  
Mixture proportions and fresh concrete properties (control [C], N)

Mixture name	C	N1	N2	N3	N4	N5	N6	N7
Residuals, as-received (moist) (% of concrete by mass)	0	0.2	0.4	0.5	0.6	0.7	0.8	1.2
Wood fibers from residuals (kg/m <sup>3</sup> ) <sup>a</sup>	0	1.1	2.3	2.6	3.0	3.4	3.9	6.1
HRWRA (l/100 kg cement)	0.6	1.0	1.0	1.6	2.2	2.0	2.4	4.1
Residuals, as-received (moist) (kg/m <sup>3</sup> )	0	4.5	9.2	10.5	12.3	13.8	16.0	24.7
HRWRA (l/m <sup>3</sup> )	2.3	3.4	3.5	4.9	6.9	6.0	7.3	12.2
Cement (kg/m <sup>3</sup> )	357	334	340	313	307	295	300	296
Sand, SSD (kg/m <sup>3</sup> )	848	790	801	743	718	697	710	691
Pea gravel, 9.5-mm maximum, SSD (kg/m <sup>3</sup> )	1050	978	994	921	899	866	877	866
Water (kg/m <sup>3</sup> )	166	138	149	128	132	122	125	134
Water/cement, w/c	0.46	0.41	0.44	0.41	0.43	0.41	0.42	0.45
Slump (mm)	235	185	85	165	125	135	110	70
Air content (%)	1.1	8.6	5.2	>10	>10	>10	–	>10
Density (kg/m <sup>3</sup> )	2420	2250	2300	2120	2070	2000	2030	2020

<sup>a</sup> As-received moist fibers were used. The quantities of the fibers shown are on oven-dry basis.

HRWRA was not helpful in repulping the residuals; a high-speed mixing of residuals in room-temperature water was shown to be the most practical and satisfactory means of repulping most of the residuals that were used in the second research.

### 3. Mixture proportions, test results, and discussions

#### 3.1. Mixture proportions

Mixture proportions and fresh properties of the concrete mixtures produced in the laboratory are shown in Tables 3 and 4. The amount of as-received residuals in concrete

ranged from 0.2% to 1.2% (by mass) for residual N and from 0.2% to 0.8% for residuals F and K. The amount of wood fibers in concrete ranged from 1.1 to 6.1 kg/m<sup>3</sup> for residual N, from 0.9 to 3.3 kg/m<sup>3</sup> for residuals F, and from 1.7 to 6.3 kg/m<sup>3</sup> for residual K (when converted to oven-dry mass).

Overall, water–cement ratio (w/c), slump, air content, and density were in the ranges of 0.40–0.52, 65–260 mm, 1.1–>10%, and 2000–2420 kg/m<sup>3</sup>, respectively. Control mixture C and mixtures F1 and F2 showed very high slump (235, 255, and 260 mm, respectively). Mixtures N1 and N3 showed high slump (185 and 165 mm). If the quantities of the mixing water in mixtures C, F1, F2, N1, and N3 had been reduced, the slump would have been lower and the

Table 4  
Mixture proportions and fresh concrete properties (F, K)

Mixture name	F1	F2	F3	F4	K1	K2	K3	K4
Residuals, as-received (moist) (% of concrete by mass)	0.2	0.4	0.6	0.8	0.2	0.4	0.6	0.8
Wood fibers from residuals (kg/m <sup>3</sup> ) <sup>a</sup>	0.9	1.7	2.5	3.3	1.7	3.3	4.8	6.3
HRWRA (l/100 kg cement)	1.0	1.0	1.0	1.0	1.7	1.6	1.8	1.9
Residuals, as-received (moist) (kg/m <sup>3</sup> )	4.8	9.5	14.1	18.4	4.8	9.6	13.8	18.1
HRWRA (l/m <sup>3</sup> )	3.6	3.6	3.6	3.5	5.9	5.7	6.2	6.6
Cement (kg/m <sup>3</sup> )	356	353	352	346	359	356	344	339
Sand, SSD (kg/m <sup>3</sup> )	829	802	822	796	829	824	797	771
Pea gravel, 9.5-mm maximum, SSD (kg/m <sup>3</sup> )	1047	1033	1033	1006	1049	1040	1003	994
Water (kg/m <sup>3</sup> )	161	183	141	159	169	167	163	172
Water/cement, w/c	0.45	0.52	0.40	0.46	0.47	0.47	0.47	0.51
Slump (mm)	255	260	140	145	110	65	100	140
Air content (%)	1.2	1.4	3.5	4.7	2.2	2.0	3.7	3.5
Density (kg/m <sup>3</sup> )	2400	2380	2360	2330	2410	2400	2320	2300

<sup>a</sup> As-received moist fibers were used. The quantities of the fibers shown are on oven-dry basis.

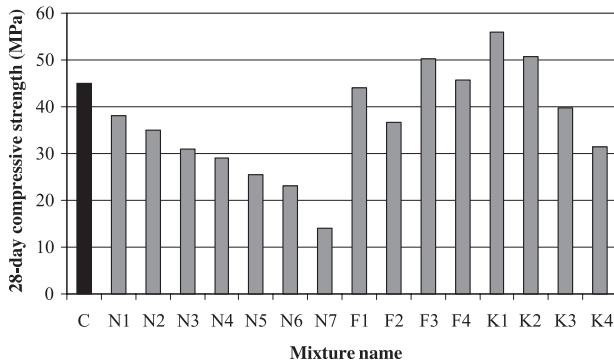


Fig. 1. Compressive strength of concrete at 28 days.

strength higher. Concrete mixtures containing residual N showed very high air contents and low densities. In general, the density of the concrete decreased as the amount of residuals increased. Density values of residual N mixtures were considerably lower than that of the control mixture. Density values of residuals F and K mixtures were somewhat lower than that of the control mixture.

### 3.2. Compressive and splitting-tensile strengths

Compressive and splitting-tensile strengths of concrete were determined at 28 days by testing three  $100 \times 200$ -mm cylinders for each test type. Test results are presented in Figs. 1 and 2. Most of the F and K mixtures were equivalent to the control concrete in compressive strength and splitting-tensile strength. Within each group of residuals N and K concrete mixtures, compressive and splitting-tensile strengths generally decreased as the amount of residuals increased, probably due to the reduction in the density of the concrete. In addition, a considerable retardation of concrete mixture N7 was noted for about 3 days, due to the use of excessive amount of HRWRA (4.1 vs. 1.3 l [the recommended maximum]/100 kg of cement). Relatively large doses of HRWRA in mixtures N4, N5, and N6 might also have caused some delays in the setting of the concrete. On the other hand, the quantities of HRWRA used in mixtures C, N1, N2, F1, F2, F3, and F4 were lower

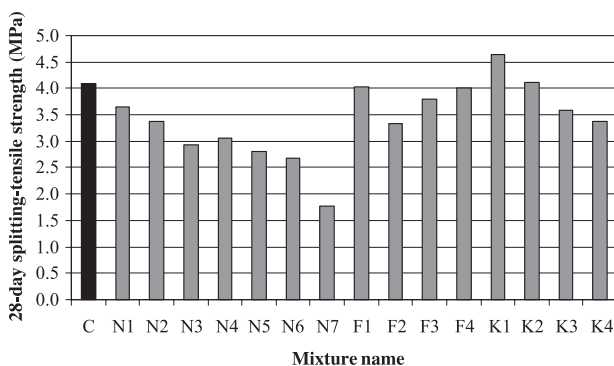


Fig. 2. Splitting-tensile strength of concrete at 28 days.

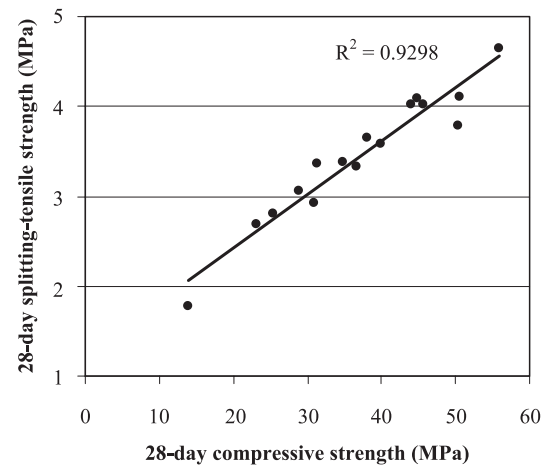


Fig. 3. Relation between the 28-day compressive and splitting-tensile strengths of concrete.

than the maximum recommended by the manufacturer, and no considerably delayed settings of these mixtures are suspected.

Mixtures F3 and F4 showed higher compressive strength than mixtures F1 and F2, respectively. This may be attributed to lower w/c of F3 and F4 mixtures compared with F1 and F2 mixtures, respectively. Splitting-tensile strength values of mixtures F3 and F4 were higher than that of mixture F2 and were nearly equivalent to that of mixture F1. If less water had been used in mixtures F1 and F2, higher strengths of these concrete mixtures would have been obtained; also, the slump would have been comparable to that of mixtures F3 and F4.

Overall, the compressive strength and splitting-tensile strength of concrete showed a very good correlation (Fig. 3). In general, compressive and splitting-tensile strengths of concrete containing the residuals were proportional to the density of concrete (Figs. 4 and 5). To make the strength of

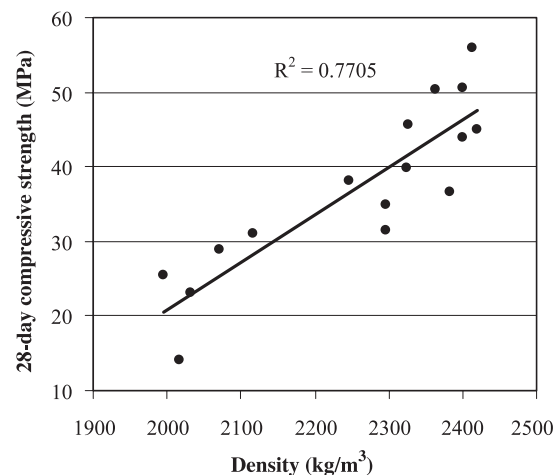


Fig. 4. Relation between density and the 28-day compressive strength of concrete.

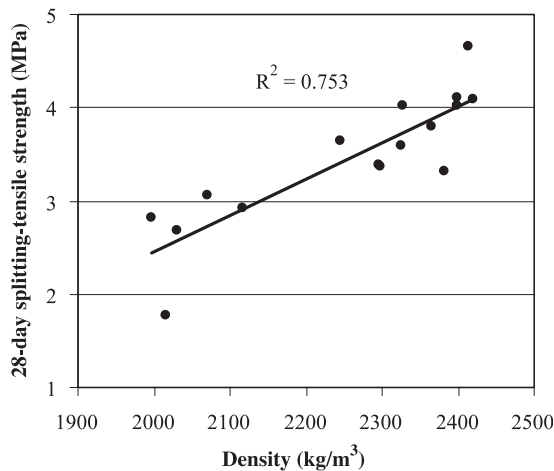


Fig. 5. Relation between density and the 28-day splitting-tensile strength of concrete.

residuals-containing concrete about the same as that of the concrete without the residuals, the density of concrete would have to be consistent regardless of the residuals content in concrete. In the research conducted later [14,15], the equivalent density and strength of concrete were achieved by keeping the sum of the volume of mixing water, fibrous residuals, and HRWRA constant regardless of the amount of residuals.

Varying levels of relationships between w/c and the strength were observed within each group of concrete mixtures containing the residuals (N, F, and K). However, on the whole, no close relationships between w/c and the strength were observed.

#### 4. Conclusions

Based on the initial results presented, the following general conclusions may be drawn:

1. In general, within each group of concrete mixtures containing the fibrous residuals from pulp and paper mills, density, compressive strength, and splitting-tensile strength of concrete decreased with the increase in the amount of the residuals in concrete.
2. Several concrete mixtures containing the residuals showed higher strength than the concrete without the residuals.
3. A very strong correlation was observed between the compressive strength and the splitting-tensile strength of concrete containing pulp and paper mill residuals.
4. A strong correlation was observed between density and strength of concrete containing the residuals.
5. Overall, a low correlation was observed between w/c and strength of concrete containing the residuals.
6. By achieving equivalent density of concrete, the strength of concrete containing the residuals may be

made equivalent to that of concrete without the residuals.

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