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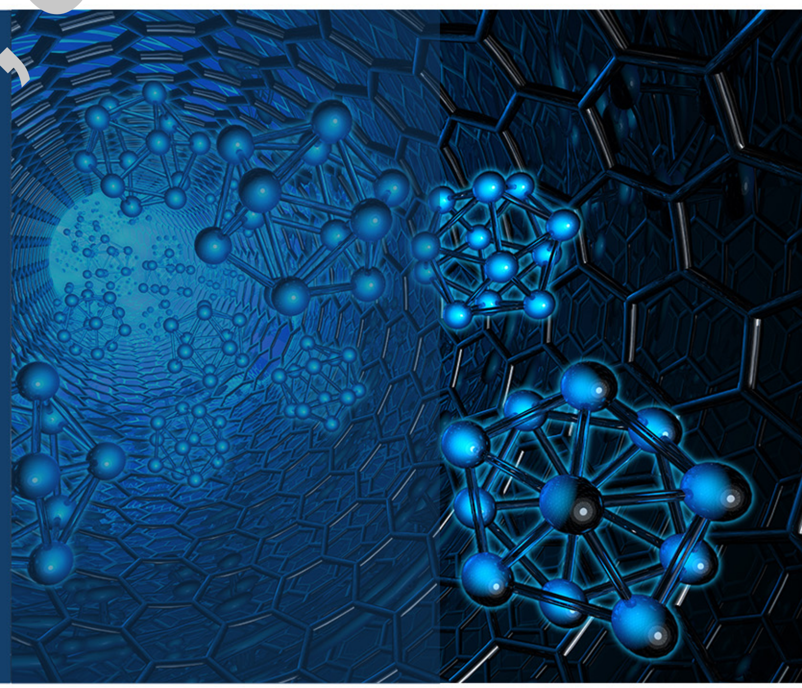
**CSA Z5300:19**
National Standard of Canada

# Cellulose filaments (CF) — Preparing handsheets for physical tests



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*National Standard of Canada*

# *CSA Z5300:19*
# ***Cellulose filaments (CF) — Preparing handsheets for physical tests***

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# Contents


Technical Committee on Cellulose Nanomaterials 3

Preface 5

**0 Introduction** 6
0.1 Cellulose filaments, microfibrils, and nanofibrils 6
0.2 Films and handsheets of CF, CMF, and CNF 8
0.3 CF, CMF, and CNF as reinforcing agents for paper products 8
0.4 Measuring and predicting reinforcement ability of CF 8

**1 Scope** 9

**2 Reference publications** 9

**3 Definitions** 11

**4 General** 14
4.1 User responsibility 14
4.2 Sample handling and storage 14
4.3 Minimum information to report 14
4.4 Optional information to report 15

**5 Disintegration of CF** 15
5.1 General 15
5.2 Apparatus and reagents 15
5.3 Sample preparation for CF suspension at initial consistency < 20 wt% 17
5.4 Sample preparation for CF suspension at initial consistency ≥ 20 wt% and dry film form CF 18
5.5 Hot disintegration (CF suspensions and dry film form CF) 18

**6 Low basis-weight CF handsheets for testing** 19
6.1 General 19
6.2 Apparatus and reagents 20
6.3 Handsheet formation 23
6.4 Couching 24
6.4.1 General 24
6.4.2 Couching with couch roll 24
6.4.3 Couching with automatic couching system 26
6.5 Basis weight adjustment 26
6.6 Pressing 27
6.7 Drying 28
6.8 Physical properties testing 29

**7 CF-reinforced handsheets of low-strength pulp** 30
7.1 General 30
7.2 Apparatus and reagents 31
7.3 Preparation of low-strength pulp 31

7.4 Disintegration of low-strength pulp *31*
7.5 Low-strength pulp handsheets *32*
7.6 CF-reinforced pulp handsheets *32*
7.7 Physical properties testing *32*

Annex A (informative) — Bibliography *33*

# Preface

This is the first edition of CSA Z5300, *Cellulose filaments (CF) – Preparing handsheets for physical tests*.

This Standard describes methods for the preparation of handsheets of cellulose filaments (CF) and CF-reinforced pulp handsheets for physical properties testing. The properties of the handsheets can be studied to give an indication of the performance of CF as a reinforcement additive in the production of paper or tissue.

This Standard is the third in a series on cellulose nanomaterials intended to serve as a foundation for the development of both Canadian and international standards to facilitate the introduction of cellulose nanomaterials into global markets. Other standards in the series on cellulose nanomaterials include CSA Z5100, *Cellulose nanomaterials — Test methods for characterization* and CSA Z5200, *Cellulose nanomaterials — Blank detail specification*.

This Standard was prepared by the Technical Committee on Cellulose Nanomaterials, under the jurisdiction of the Strategic Steering Committee on Health Care and Well-being, and has been formally approved by the Technical Committee.

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   b) *relevant clause, table, and/or figure number;*
   c) *wording of the proposed change; and*
   d) *rationale for the change.*

# CSA Z5300:19
# Cellulose filaments (CF) — Preparing handsheets for physical tests

## 0 Introduction

### 0.1 Cellulose filaments, microfibrils, and nanofibrils

The need to develop high-value applications of wood pulp fibres has prompted significant efforts by the Canadian pulp and paper industry to develop the production of cellulose nanomaterials (CNM) derived from pulp fibres and improve their unique properties.

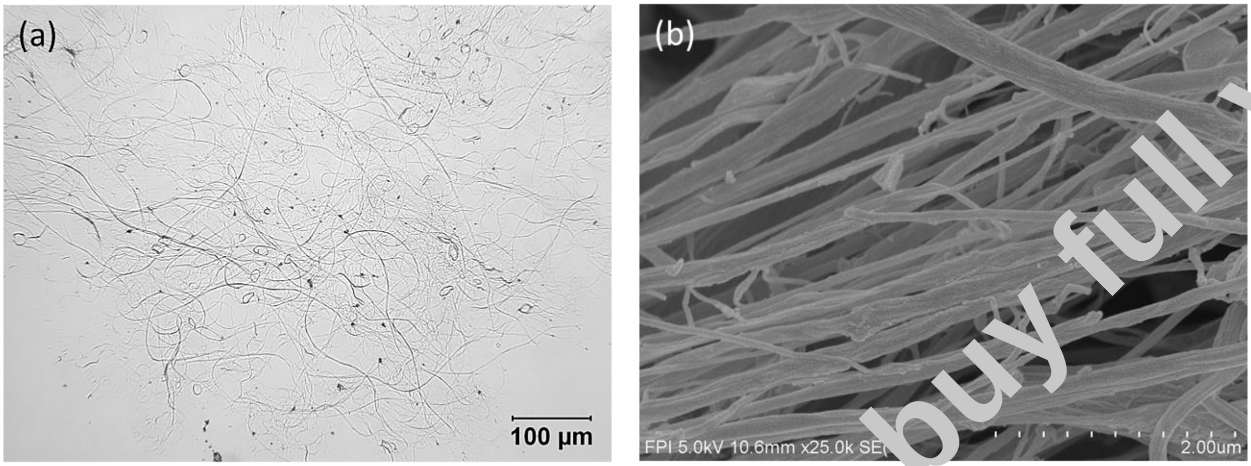
To this end, microfibrillated cellulose (MFC), now called cellulose microfibrils (CMF), was first produced in the 1980s by multi-pass, high-pressure mechanical disintegration of pulp fibres at 2 to 4 wt% consistency in a homogenizer [1, 2]. A very high energy input is required to delaminate the pulp fibres, prohibiting CMF production at an industrial scale. Currently, many alternative multi-pass processes are used to produce CMF and cellulose nanofibrils (CNF), including homogenization, microfluidization, grinding, and low- or medium-consistency refining, often coupled with chemical or enzymatic pre-treatments to reduce the amount of mechanical energy required to generate the fibrils and prevent clogging of the equipment. The typical processing consistency to produce CMF and CNF is about 3 wt% or less.

For the purposes of this Standard, CMF are fibrils (fibre-like strands) composed of cellulose and having cross-sectional dimensions typically larger than 100 nm, while CNF are fibrils of cellulose having cross-sectional dimensions in the nano-scale; that is, between 1 and 100 nm. Both CMF and CNF have lengths of typically less than 100 µm [3–5] and aspect ratio (length-to-width ratio) generally in the range of 100 to 150 [6]. Owing to large variations in the manufacturing processes used to produce them, material designated as CMF can contain a certain (small) fraction of CNF, and vice versa.

Recently, commercially feasible industrial-scale processes to produce cellulose filaments (CF) have been developed. CF are produced from wood pulp by applying mechanical forces, for example, using multi-pass disk refining at a high consistency of > 20 wt%, without chemical or enzymatic pre-treatment.

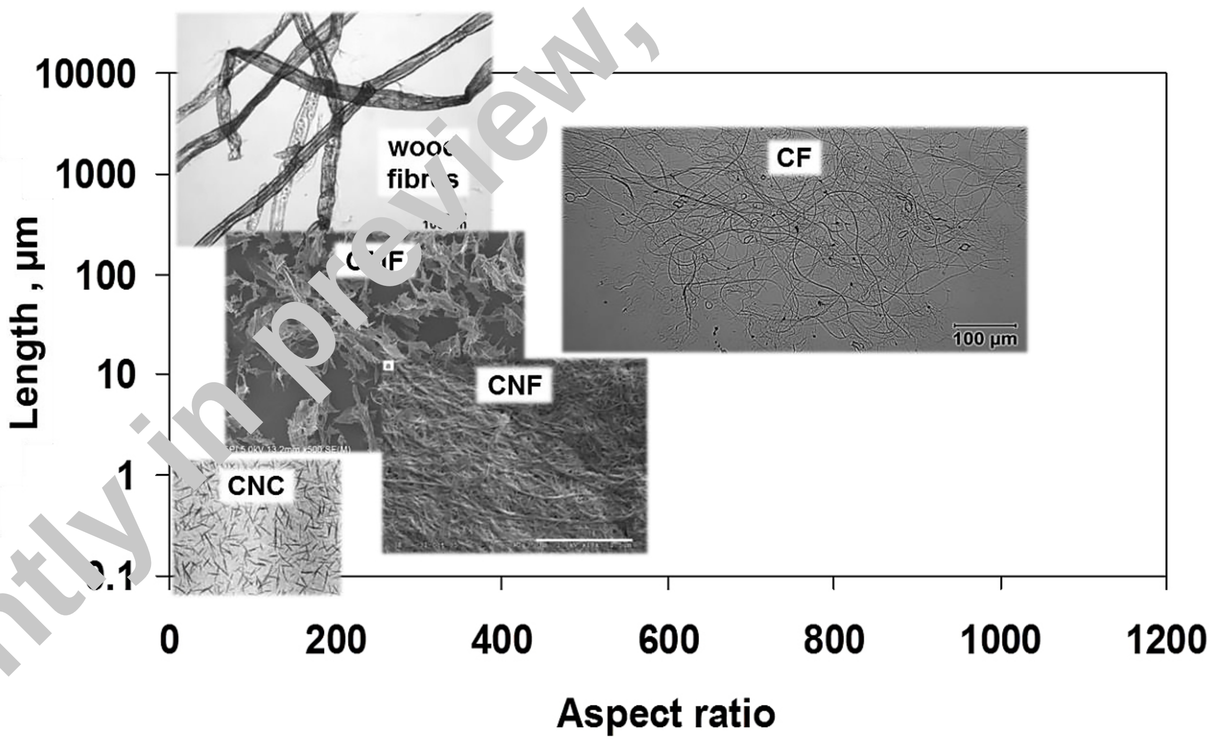
CF is a CNM consisting of individual cellulose filaments or fibrils having a flexible ribbon-like structure (Figure 1), with a length typically > 100 µm on average. Its cross-sectional dimensions generally range from 30 to 500 nm, giving CF a very high aspect ratio of at least 200 and typically > 500 [7, 8]. CF is thus much longer than CMF or CNF (see Figure 2).

**Figure 1**
**Images of cellulose filaments showing their fine ribbon-like structure**
**a) Light micrograph (160 × magnification);**
**b)scanning electron micrograph (25 000 × magnification)**
(See Clause 0.1.)




**Source:** *Image courtesy of FPInnovations.*

**Figure 2**
**Approximate length and aspect ratio ranges for various cellulose nanomaterials and chemical (softwood kraft) pulp fibres**
(See Clause 0.1.)




**Source:** *Modified from Hua et al., 2016.*

## 0.2 Films and handsheets of CF, CMF, and CNF

The methods described in this Standard use a handsheet machine to make CF handsheets in which the cellulose filaments are randomly oriented. A dry film form of CF can also be produced on paper machines using typical fabrics.

Films, handsheets, or "nanopapers" of various types of CMF and CNF have been prepared by various methods, including solvent casting or air-drying [9–13]. The most common method is filtration [14, 15], which leads to a film or sheet in which the fibrils are randomly oriented, similarly to handsheets. To speed the process, filtration is typically vacuum- or pressure-assisted [16], and usually followed by oven-drying [11, 14, 17] or hot pressing [11, 18–21]. Filter pore sizes are typically < 1 µm, as precautions must be taken to avoid loss of shorter, narrower CNF, particularly those produced via TEMPO-oxidation pretreatment [17]. CMF and CNF produced by mainly mechanical means without chemical pretreatments can be retained by meshes of larger pore size [15, 22–24]. Oriented sheets such as those made on the moving fabric of paper machines can be made from larger CNF with the dynamic sheet former [15]. A handsheet maker has also been used to prepare (non-oriented) handsheets of mechanical CNF [23, 24], although a filter paper with a 25 µm pore size was placed over the filter mesh.

## 0.3 CF, CMF, and CNF as reinforcing agents for paper products

CF has been added to papermaking furnish as an additive. Paper and pulp mill trials have demonstrated the superiority of CF as a reinforcement agent for various paper and tissue grades [9, 25]. When it is added to papermaking pulp, CF does not require the use of additional retention agents to prevent its loss through the paper machine fabric, owing to its very long filaments.

CMF and CNF have also been investigated as additives in papermaking by mixing them with pulp fibres, typically weaker hardwood kraft or thermomechanical pulps [26]. The most common application of CMF/CNF is as a dry strength agent [4, 27–29]. The CMF/CNF are mechanically mixed into the pulp slurries [30,30], sometimes during the disintegration of dried pulps [28, 29]. Pulp/CNF blends have also been used in paper machine trials [31, 32]. Additives such as cationic starch and colloidal silica [28,30,33] or cationic polyacrylamide (CPAM) [29, 31, 32] are often included as retention agents for CMF and CNF, as precautions must be taken to avoid losing the shorter, narrower fibrils through the wire mesh during production.

## 0.4 Measuring and predicting reinforcement ability of CF

Long wood fibres such as softwood kraft pulp and narrow CF and CMF/CNF fibrils have an inherent tendency to form tangled networks, and when mixed with other pulps may form a load-bearing network embedded in the larger pulp fibres [27], and also promote adhesion of adjacent pulp fibres by effectively bridging them and increasing the fibre-fibre bonding area.

Handsheets of certain pulps such as Northern Bleached Softwood Kraft (NBSK) are commonly prepared to evaluate their performance as reinforcement additives in paper or tissue made from weaker thermomechanical pulp (TMP) or hardwood kraft pulps. When standard handsheets (60 $g/m^2$) are made from the NBSK pulps alone, their measured strength properties correlate well with their reinforcement ability: Higher tensile index and/or tensile energy absorption (TEA) of the NBSK handsheets in particular indicate higher reinforcement ability of the NBSK pulps. It has also been found that the tensile index and/or TEA of low basis-weight handsheets made from CF alone can be used to assess the reinforcement abilities of, for example, different grades of CF produced with different specific refining energies.

# 1 Scope

## 1.1

This Standard describes methods for the preparation of low-basis weight handsheets of cellulose filaments (CF) and CF-reinforced pulp handsheets to be used for physical tests.

## 1.2

This Standard also covers the preparation of low-basis weight handsheets of cellulose microfibrils (CMF) and cellulose nanofibrils (CNF), and CMF- or CNF-reinforced pulp handsheets, provided that handsheets of acceptable quality can be obtained using the methods described.

## 1.3

This Standard does not cover the preparation of handsheets of CF, CMF, or CNF for optical tests.

## 1.4

In this Standard, “shall” is used to express a requirement, i.e., a provision that the user is obliged to satisfy in order to comply with the Standard; “should” is used to express a recommendation or that which is advised but not required; and “may” is used to express an option or that which is permissible within the limits of the Standard.

Notes accompanying clauses do not include requirements or alternative requirements; the purpose of a note accompanying a clause is to separate from the text explanatory or informative material.

Notes to tables and figures are considered part of the table or figure and may be written as requirements.

Annexes are designated normative (mandatory) or informative (non-mandatory) to define their application.

# 2 Reference publications

This Standard refers to the following publications, and where such reference is made, it shall be to the edition listed below, including all amendments published thereto.

**Note:** *See also Annex A.*

**CSA Group**

Z5100-17
*Cellulose nanomaterials – Test methods for characterization*

Z5200-17
*Cellulose nanomaterials — Blank detail specification*

**ISO (International Organization for Standardization)**

187:1990
*Paper, board and pulps — Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples*

536:2012
*Paper and board — Determination of grammage*