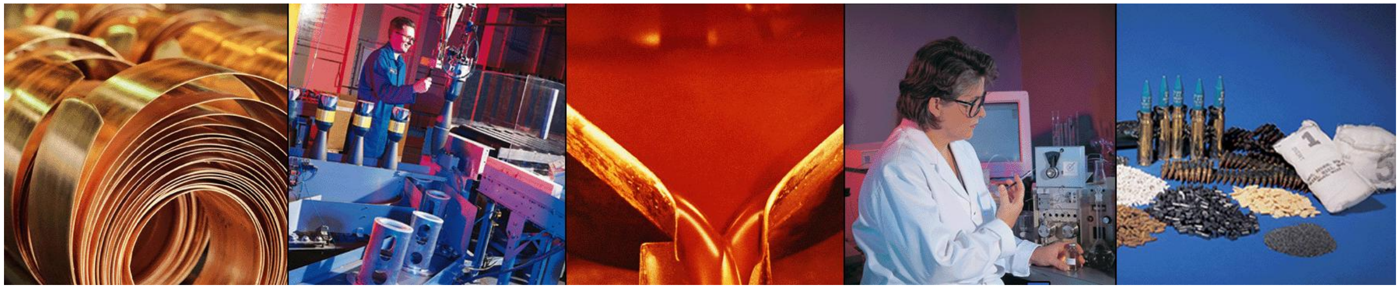
GENERAL DYNAMICS
Ordnance and Tactical Systems–Canada Valleyfield



NC fiber length distribution: Empirical modelling of the mass weighed length distribution

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What prompted this work?

The physical properties of NC are often related to the mass weighted fiber length distribution.

A weighted average length by true mass is difficult/impossible to obtain.

One must settle for a functional estimate based on mathematical/statistical models relying only on fiber length.

-Which set of calculation should be applied on NC data to get useful information?

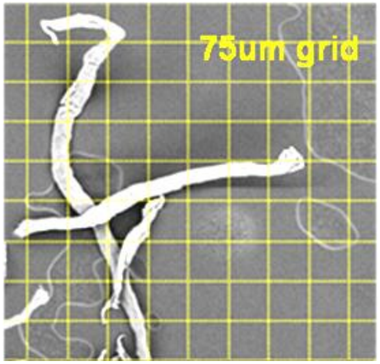
-How should the data be treated and results interpreted?

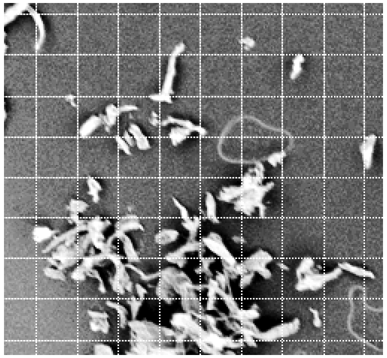
Worked done in GD-Valleyfield in 1980's, confirmed that the **mass content** of fines is somewhere between 5 to 10% range. That work was never completed/published.

How can realistic and useful information on NC length distribution be obtained from modern fiber analyzers?

This presentation only set the table for more discussion/work…

Nitrocellulose Particle Sizing
Classification of NC particles





NC is composed of numerous particles which can be divided in groups/classes.

Fibers vs Fines → Apparent volume density

Fibers are hollow cylinders, volume density << NC density (1.66g/cc)
Fines are not hollow → volume density = NC density (1.66g/cc)

Typically for wood and cotton NC (by mass):

90-97% are fibers and 3-10% are fines

Classification

Major effect on mass distribution :	Fibers vs Fines
Lesser effect on mass distribution:	Fines → Fragments vs Dust
????	Fibers → Short vs long ???

Nitrocellulose Particle Sizing
Primary classification: Fibers vs Fines

Classification
Primary: Fibers vs Fines
Secondary: Fines → Fragments vs Dust
Fibers → ????

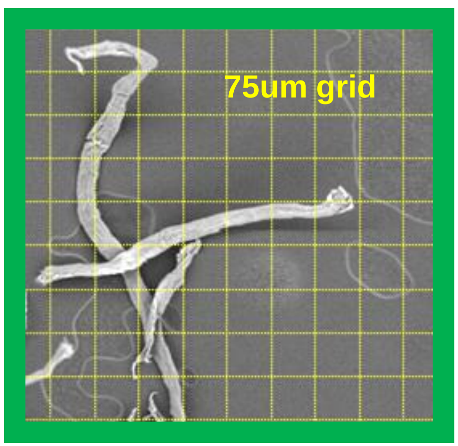
Fibers are hollow cylinders, volume density << NC density (1.66g/cc)
Fines are not hollow → volume density = NC density (1.66g/cc)

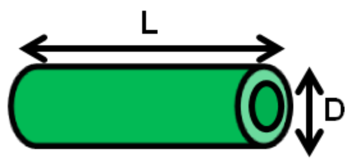
When does particles cease to be hollow cylinders?

Somewhere between 60-100um

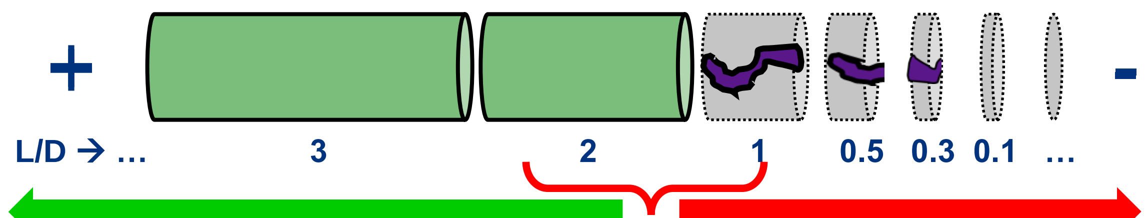
For NC fibers, D is typically between 30-50 um due to nitration swelling (average about 40 um)

Type of wood	Typical External Diameter µm
Douglas Fir	40
Hemlock	40
Spruce/Pine	30
Western Red Cedar	30
White Spruce	30
Lodgepole Pine	30
Alpine Fir	35
Southern Pine	45
Scandinavian Pine	35
Average	**35**
Total Average	35





NC particles cease to be hollow cylinders when L/D is < 2



Can be considered fibers

Must not be considered fibers
will be greatly over estimated

Thus: the distribution must either be trimmed or modeled below 60-100um (taken to be 75um) to account for change in volume density…

Classic approach to estimation of the weight distribution

Very basic approach: Valid only for hollow particles, **For NC with L > about 75um**

$$L_M = \sum_{i=1}^{n} \frac{w_i}{w_{total}} L_i = \frac{\sum_{i=1}^{n} w_i L_i}{\sum_{i=1}^{n} w_i}$$

$$w_i = N_i \, L_i \, c_i$$

$$w_{total} = \sum_{i=1}^{n} w_i = \sum_{i=1}^{n} N_i \, L_i \, c_i$$

Coarseness c is the linear density usually express as mg/m

C = Weight/Length
W= Coarseness x Length

An assumption must be made about coarseness:

L_M for a constant coarseness:

$$= \frac{\sum_{i=1}^{n} N_i \, L_i^2 \, c_i}{\sum_{i=1}^{n} N_i \, L_i \, c_i} = \boxed{\frac{\sum_{i=1}^{n} N_i \, L_i^2}{\sum_{i=1}^{n} N_i \, L_i}}$$

Usually labeled:
L_L , the length weighted average length

L_M for coarseness proportional to length:

So c α L or c = aL

$$= \frac{\sum_{i=1}^{n} N_i \, L_i^2 \, c_i}{\sum_{i=1}^{n} N_i \, L_i \, c_i} = \frac{\sum_{i=1}^{n} N_i \, L_i^2 \, a \, L_i}{\sum_{i=1}^{n} N_i \, L_i \, a \, L_i} = \boxed{\frac{\sum_{i=1}^{n} N_i \, L_i^3}{\sum_{i=1}^{n} N_i \, L_i^2}}$$

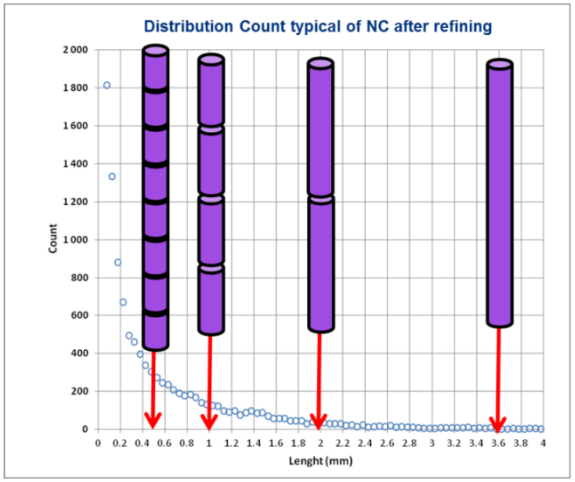
Usually labeled:
L_W , the weight weighted average length

Valid for only 1 common shape and the average coarseness of length fractions either constant or proportional to length

For NC with L > about 75um

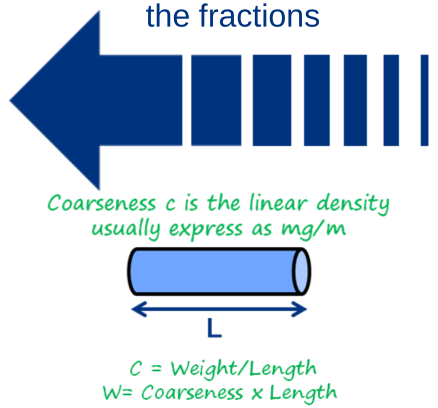
Classic approach to estimation of the weight distribution

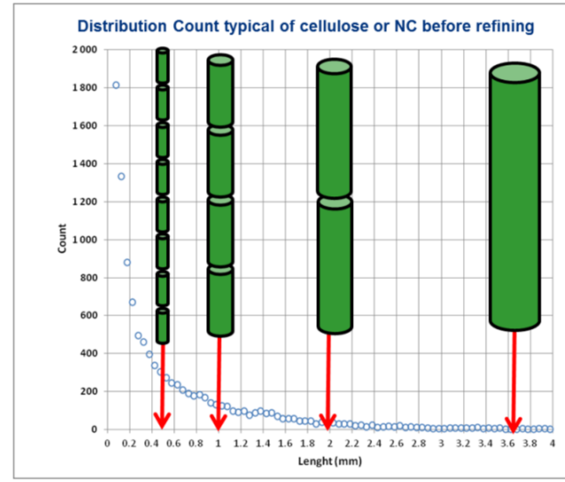
<u>Very basic approach:</u> Valid only for hollow particles, **For NC with L > about 75um**



Average Coarseness of fraction is constant

Leveling out of the average coarseness of the fractions





Average Coarseness of fractions are proportional to length

-Unprocessed fibers (from one specie) → coarseness relatively proportional to length of fibers

Short fibers = lesser coarseness, long fiber = greater coarseness

Increasing wall thickness and/or diameter

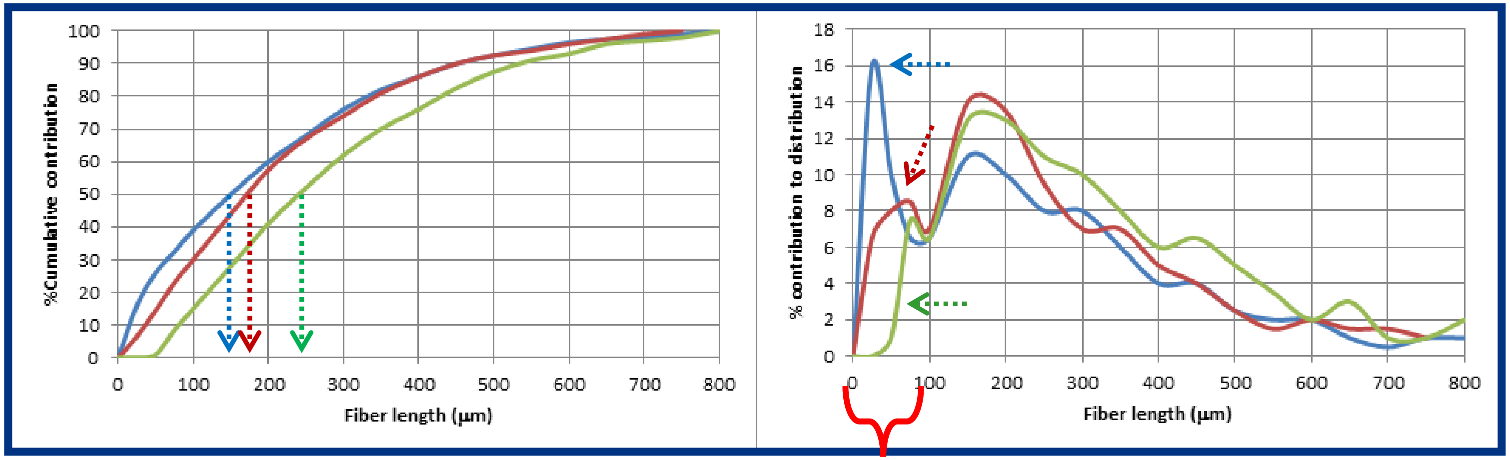
-Processing will cut fibers into smaller fibers increasing the count number in smaller length fractions

→ rapid leveling out of average coarseness between length fractions

At the expense of larger Standard Deviation of the fractions

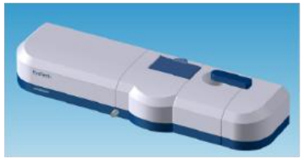
-NC after using refiners: Average coarseness of fractions is nearly constant with considerable increases in standard deviation of fractions Therefore for NC, the constant coarseness assumption is preferred

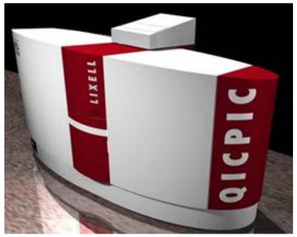
Estimation of the weight distribution relative to length



The resolution in the lower range can be significantly different between instruments

Various fiber analysers produce significantly different mass weighted length averages





Nitrocellulose Particle Sizing

Primary classification: Fibers vs Fines

Classification

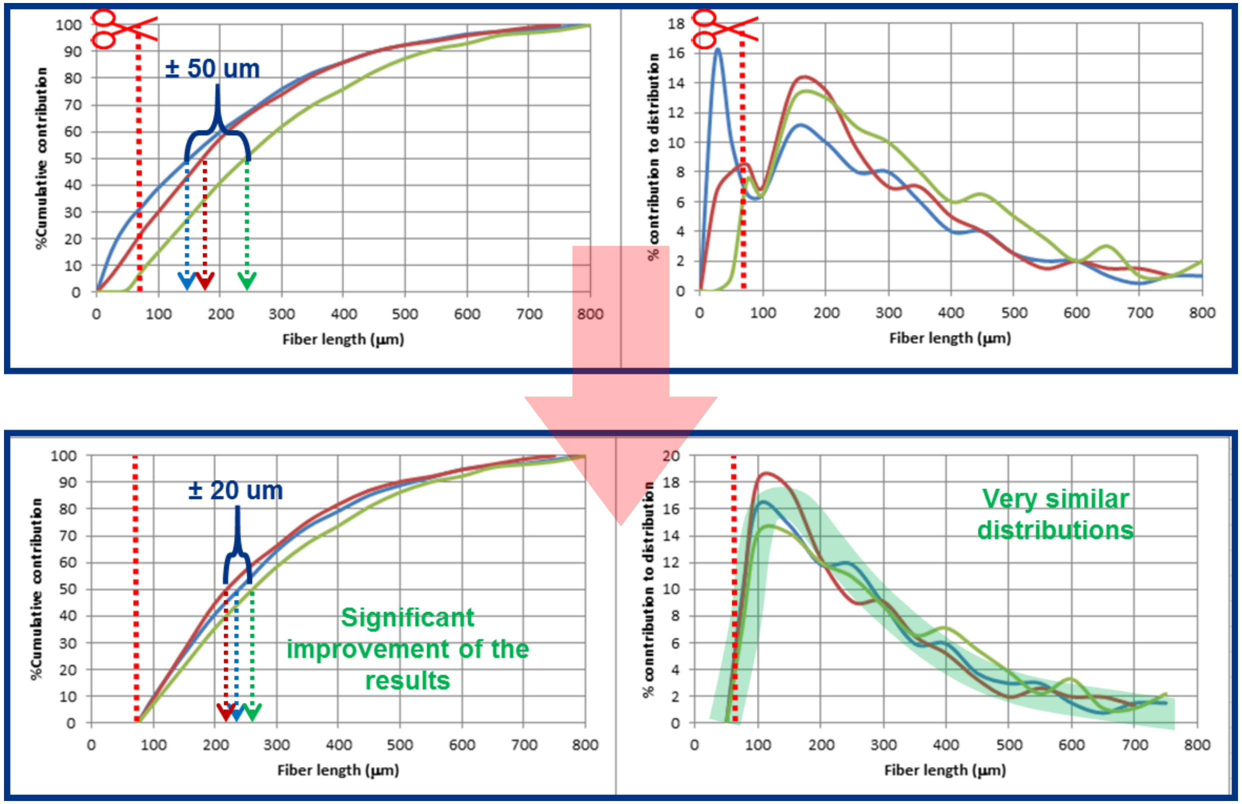
Primary: Fibers vs Fines

Secondary: Fines → Fragments vs Dust

Fibers → ????

Various fiber analysers produce significantly different results:

- **Trimming the low range portion of the distribution** significantly improves results obtained on different analysers



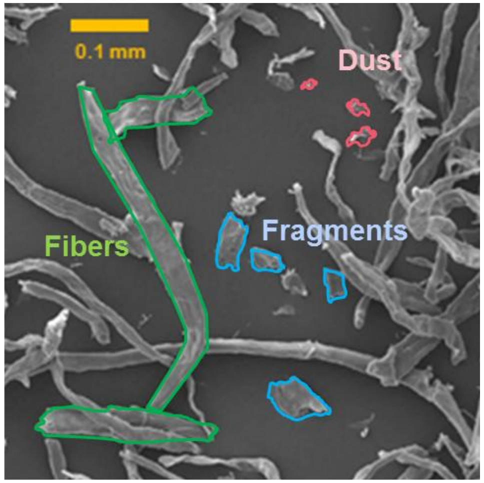
Nitrocellulose Particle Sizing
Primary classification: Fibers vs Fines

Classification
Primary: Fibers vs Fines
Secondary: Fines → Fragments vs Dust
Fibers → ????

-Fines: Small particles of NC/cellulose mainly composed of fiber fragments and dust which cannot be considered of the same coarseness and shape as the bulk of the material

-In pulp: There is no consensual definition of fines.
It is sometime defined as:
- -the fraction of pulp material passing through a 100 mesh (150 um) screen
- -the fraction of pulp material passing through a 200 mesh (75 um) screen
- -material having an upper length limit of about 100 um
- -material having an upper length limit of about 200 um (0.2 mm)

-Threshold between fibers and fines is very important for NC
- -near bulk of the fiber distribution
- -NC results are <u>significantly</u> more affected then cellulose



Wood base NC Grade C1

-A method to quantify Fines in NC by gravimetric determination is essential to determine the validity of a model

In the pulp industry:
There is a strong opinion that the existing analyzers cannot measure or rank correctly the fines content. Until more reliable and faster methods are available, the existing gravimetric methods is relied upon.

Nitrocellulose Particle Sizing

Primary classification: Fibers vs Fines   Gravimetric determination

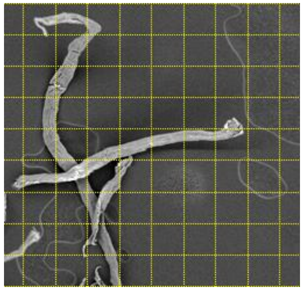
NC settling rate in water

Classification

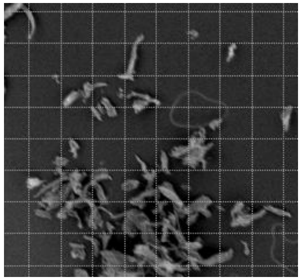
Primary: Fibers vs Fines

Secondary: Fines → Fragments vs Dust

Fibers → ????



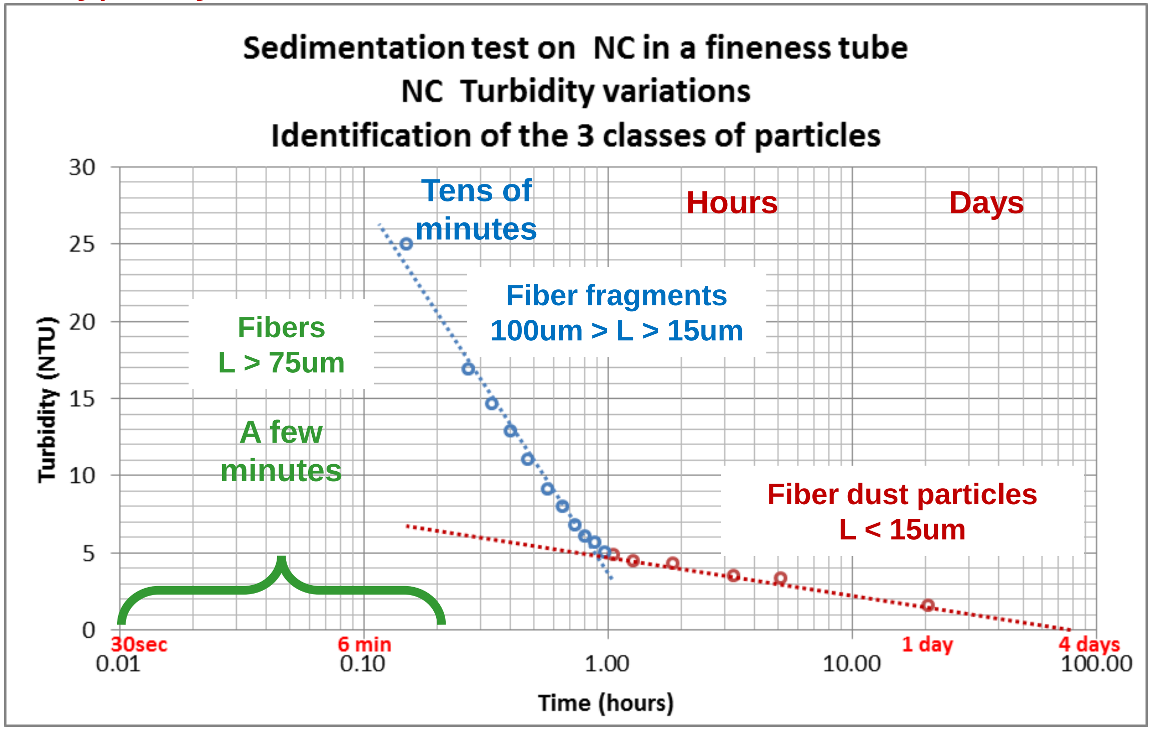
Fibers



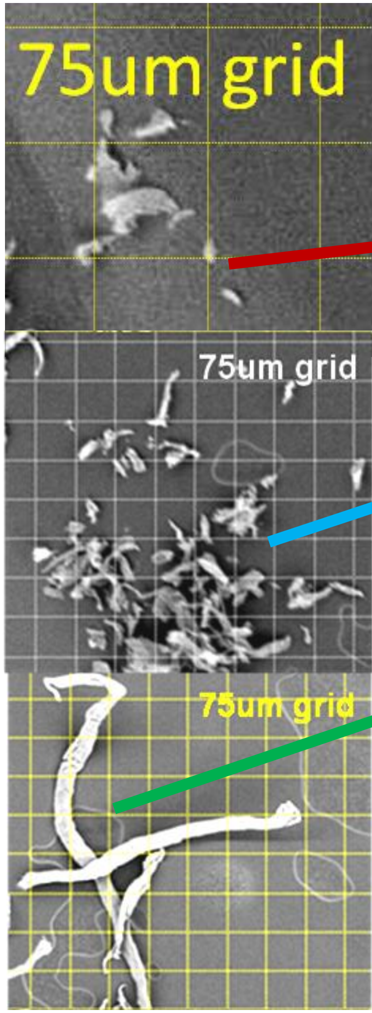
Fragments

Dust

Typically dust account of about 0.2 to 0.4% of the mass

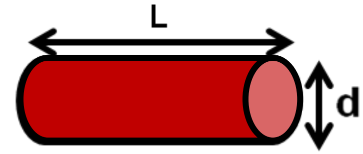


Nitrocellulose Particle Sizing:
Model based on shape of particles

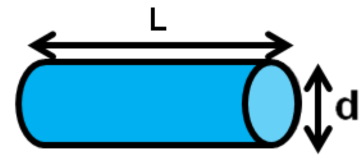


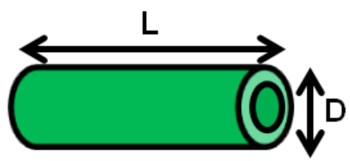
Assuming 3 different types of particles having 3 different ranges of length, 3 different average diameters and 2 different densities

L about < 15 um
d about 4 um (or D/10)
Cylindrical
Density near NC density

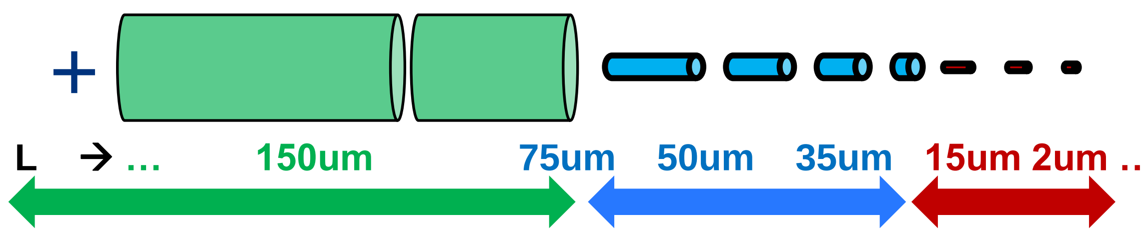


L about < 75 um
d about 8 um (or D/5)
Cylindrical
Density near NC density

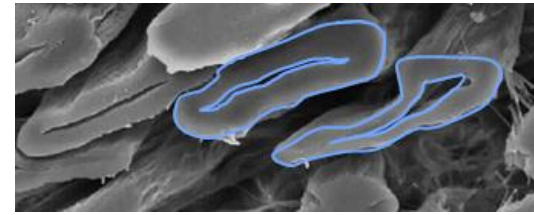


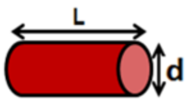
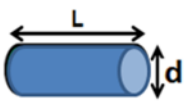
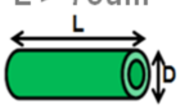


L about > 75 um
D about 40um
Tubular or Hollow Cylinder
Density << NC density



Nitrocellulose Particle Sizing:
Model based on shapes of particles



Type of particles	Approximation Weight$_{particle}$ = V x δ_{NC}
Dust L < 15um 	$12\,L\,\delta_{NC}$
Fragments 15um < L < 75um 	$50\,L\,\delta_{NC}$
Fibers L > 75um  **Constant Coarseness**	$393\,L\,\delta_{NC}$

For a cylindrical model where:

$$V_{Cylinder} = \pi r^2 L = \pi \left(\frac{d}{2}\right)^2 L = \frac{\pi d^2}{4} L$$

Where:
- L = Length of long side of piece (measured) and
- d = Average diameter cylinder → **For NC average about 40 um**

Fragments are filled cylinders with the average density of NC and diameter (d) about the thickness of fiber wall or about fiber diameter (D) divided by 10:

$$W_{Dust} \cong \frac{\pi\,(D/10)^2}{4} L\,\delta_{NC} = \frac{\pi\,(40/10)^2}{4} L\,\delta_{NC} \cong 12\,L\,\cancel{\delta_{NC}} = \mathbf{L/33}$$

Fragments are filled cylinders with the average density of NC and diameter (d) about the fiber diameter (D) divided by 5 :

$$W_{Fragment} \cong \frac{\pi\,(D/5)^2}{4} L\,\delta_{NC} = \frac{\pi\,(40/5)^2}{4} L\,\delta_{NC} \cong 50\,L\,\cancel{\delta_{NC}} = \mathbf{L/8}$$

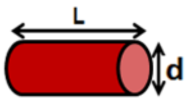
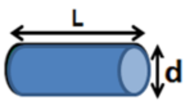
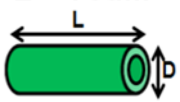
Since fibers are Tubular, their average density is much lower than the average density of NC (by the ratio of cross sectional areas):

$$\delta_{Hollow\ cylinder} = \delta_{NC}/3.2$$

$$W_{Fiber} \cong \frac{\pi D^2}{4} L\,\frac{\delta_{NC}}{3.2} \cong \frac{\pi\,40^2}{4\ 3.2} L\,\delta_{NC} \cong 393\,L\,\cancel{\delta_{NC}} = \mathbf{L}$$

Distribution composed of 2 or 3 types of particles

Process the data as if the distribution is composed of 2 or3 types of particles each with different ranges of length and 2 different densities and 2 or 3 different average diameters

Type of particles	Approximation for a group or class interval W_i	Approximation W_{total}
Dust L < 15um 	$\frac{N_i L_i}{33}$	$\sum_{i=1}^{n} \frac{N_i L_i}{33}$
Fragments 15um < L < 75um 	$\frac{N_i L_i}{8}$	$\sum_{i=1}^{n} \frac{N_i L_i}{8}$
Fibers L > 75um  Constant Coarseness	$N_i L_i$	$\sum_{i=1}^{n} N_i L_i$

Using Fragments and Fibers Constant Coarseness

$$L_M = \frac{\sum_{0-75\mu m} \frac{N_i L_i^2}{8} + \sum_{75\mu m - max} N_i L_i^2}{\sum_{0-75\mu m} \frac{N_i L_i}{8} + \sum_{75\mu m - max} N_i L_i}$$

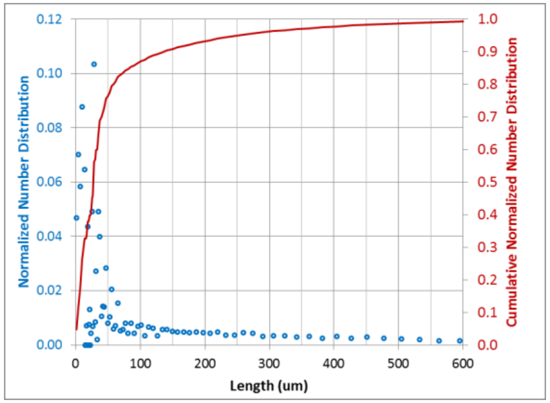
$$W_{\%\,Fines} = \frac{\left(\sum_{0-75\mu m} \frac{N_i L_i}{8}\right) 100}{\sum_{0-75\mu m} \frac{N_i L_i}{8} + \sum_{75\mu m - max} N_i L_i}$$

Using Dust, Fragments and Fibers Constant Coarseness

$$L_M = \frac{\sum_{0-15\mu m} \frac{N_i L_i^2}{33} + \sum_{15-75\mu m} \frac{N_i L_i^2}{8} + \sum_{75\mu m - max} N_i L_i^2}{\sum_{0-15\mu m} \frac{N_i L_i}{33} + \sum_{15-75\mu m} \frac{N_i L_i}{8} + \sum_{75\mu m - max} N_i L_i}$$

$$W_{\%\,Fines} = \frac{\left(\sum_{0-15\mu m} \frac{N_i L_i}{33} + \sum_{15-75\mu m} \frac{N_i L_i}{8}\right) 100}{\sum_{0-15\mu m} \frac{N_i L_i}{33} + \sum_{15-75\mu m} \frac{N_i L_i}{8} + \sum_{75\mu m - max} N_i L_i}$$

Nitrocellulose Particle Sizing:
Theoretical modeling of mass weighted fiber length distribution



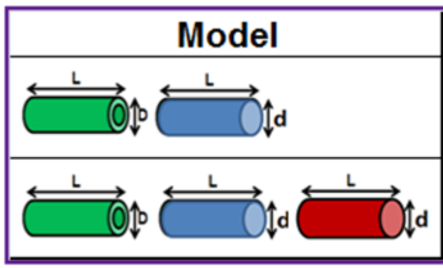
Classic approach: 2 possibilities

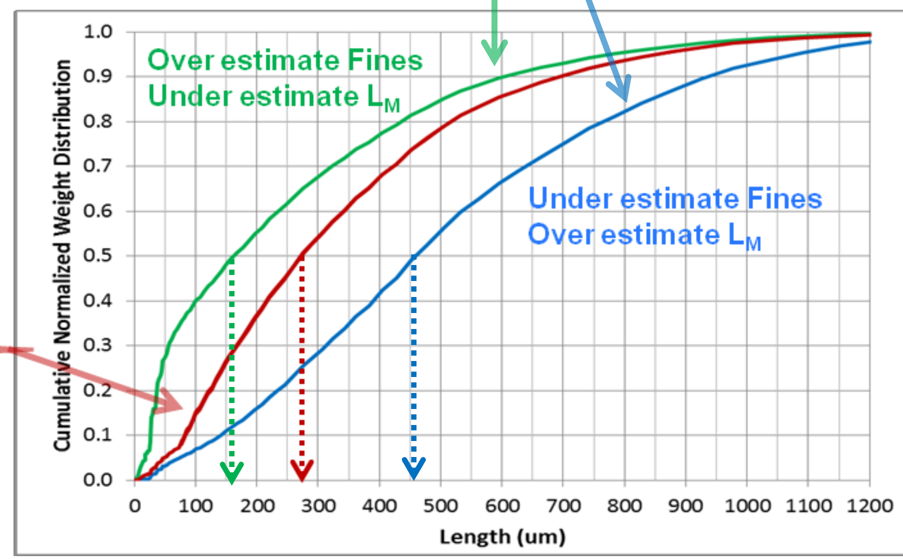
Coarseness proportional to length	L_L $\frac{\sum_{i=1}^{n} N_i L_i^3}{\sum_{i=1}^{n} N_i L_i^2}$	Graph vs length $\frac{N_i L_i^2}{\sum_{i=1}^{n} N_i L_i^2}$
Coarseness constant	L_W $\frac{\sum_{i=1}^{n} N_i L_i^2}{\sum_{i=1}^{n} N_i L_i}$	Graph vs length $\frac{N_i L_i}{\sum_{i=1}^{n} N_i L_i}$

Type of particles	Approximation for a group or class interval W_i
Fibers L > 75um Constant Coarseness	$N_i L_i$

Type of particles	Approximation for a group or class interval W_i
Dust L < 15um	$\frac{N_i L_i}{33}$
Fragments 15um < L < 75um	$\frac{N_i L_i}{8}$
Fibers L > 75um Constant Coarseness	$N_i L_i$

Multi Models
2 possibilities





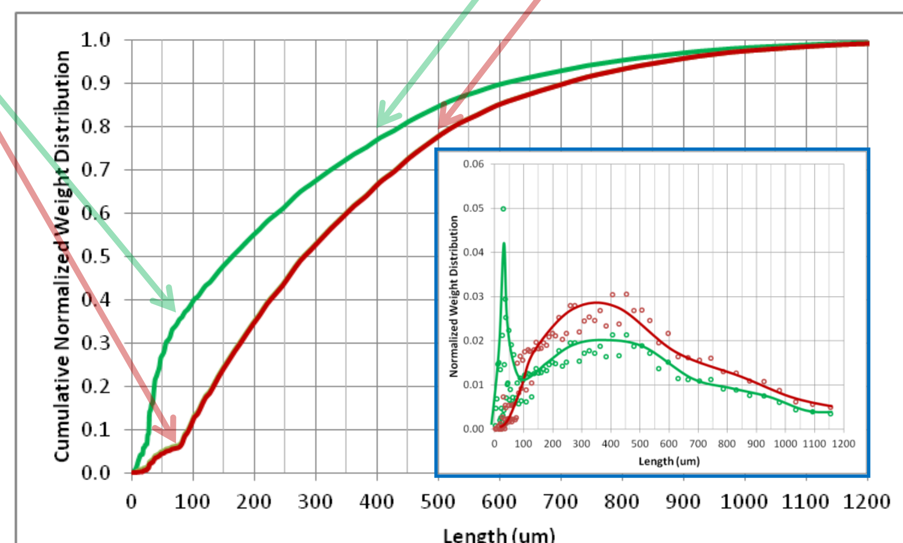
Nitrocellulose Particle Sizing:
Theoretical modeling of mass weighted fiber length distribution

Model	%Fiber	%Fines	%Fragments	%Dust
	94.1	5.9	5.7	0.2
	93.6	6.4	6.4	
	64.5	35.5		

←Dust is treated as Fragments

←Fragments and Dust are treated as Fibers

Method		Average Length um
Classic Ln	NL/N	59
Classic Ll	NL2/NL	255
Lw	NL3/NL2	518
2 shapes model L_M	NL2/NL	353
3 shapes model L_M	NL2/NL	355



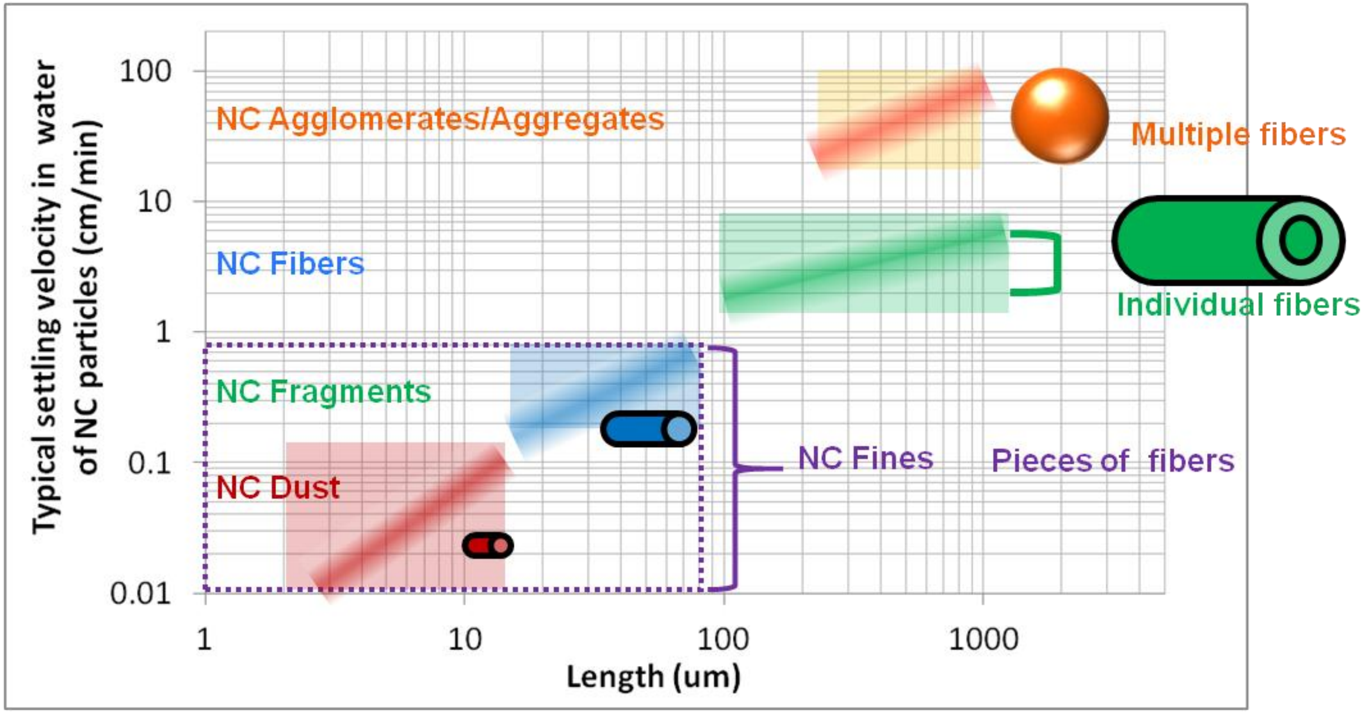
For NC: A method to quantify Fines by gravimetric determination is essential to select or calibrate a model

Connecting to reality

Nitrocellulose Particle Sizing:
Selecting a classification method Gravimetric determination NC fines

Types of NC particles: length and settling velocity

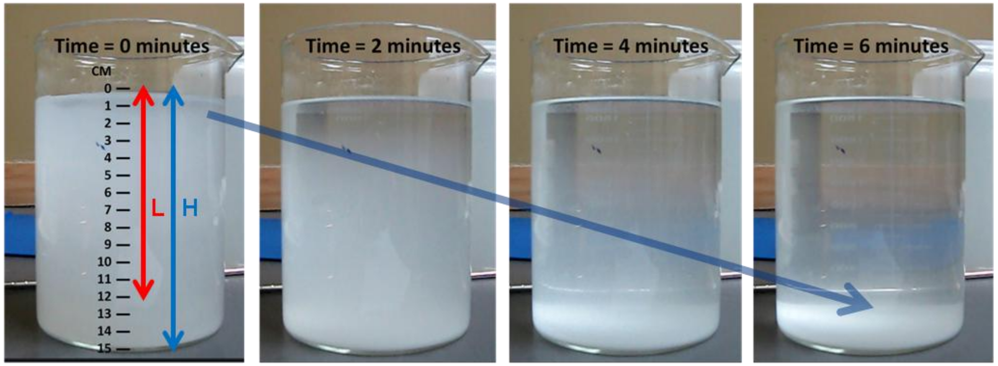
Elutriation and sedimentation: Attempt classification by terminal velocity



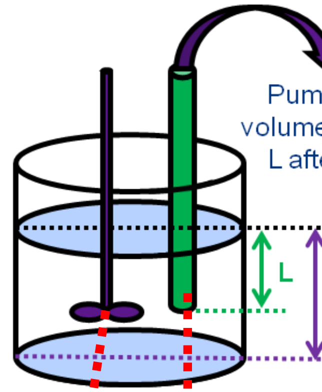
Refer to poster presentation on sedimentation testing for gravimetric determination of fines in NC samples

Screening :
Attempt classification by length

Nitrocellulose Particle Sizing:
Classification by terminal velocity: Gravitational sedimentation



Most NC fibers settle at a rate of 2 - 5 cm/min



For separation of fines from fibers: Settling time about L / 0.75
For separation of dust from Fragments: Settling time about L x 10

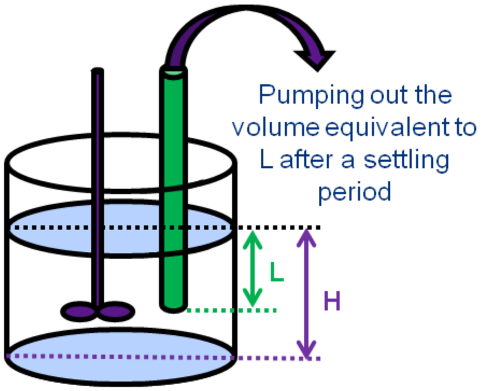
- -# of washes are required to get % Recovery > 95%
  For a L/H ratio of 0.75 = # washes is 2
- -Mixing must provide 3D homogeneous dispersion
- -Initial NC concentration about 0.1%
  (typically 2 g dry NC in 2000 ml of water)
- -Initially, ultra sonication in 50 ml of water is required to insure good dispersion of particles
- -NC should rest in water about 1 hrs before starting test, moisture penetration improves fractionation

Nitrocellulose Particle Sizing:
Classification by terminal velocity: Gravitational sedimentation

Typical fines content for various NC samples from different manufacturers

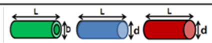
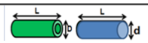
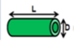
Gravimetric sedimentation method for content in fines in NC		
Supplier	**Grade**	**% Fines**
AA	B	6.5
AA	A	5.5
AA	A	9.7
BB	A	7.5
BB	C	5.7
CC	B	9.9
CC	A	5.4
CC	C	6.5
DD	C	7.8

On 5 replicates: 6.5% ±0.7%
Typical fines content for
most NC cotton or wood : 7% ±3%



Nitrocellulose Particle Sizing:

Theoretical modeling of mass weighted fiber length distribution

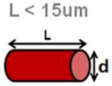
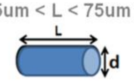
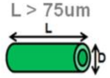
Model	%Fiber	%Fines	%Fragments	%Dust
	94.1	5.9	5.7	0.2
	93.6	6.4	6.4	
	64.5	35.5		

←Dust is treated as Fragments

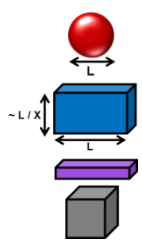
←Fragments and Dust are treated as Fibers

Method		Average Length um
Classic Ln	NL/N	59
Classic Ll	NL2/NL	255
Lw	NL3/NL2	518
2 shapes model L_M	NL2/NL	353
3 shapes model L_M	NL2/NL	355

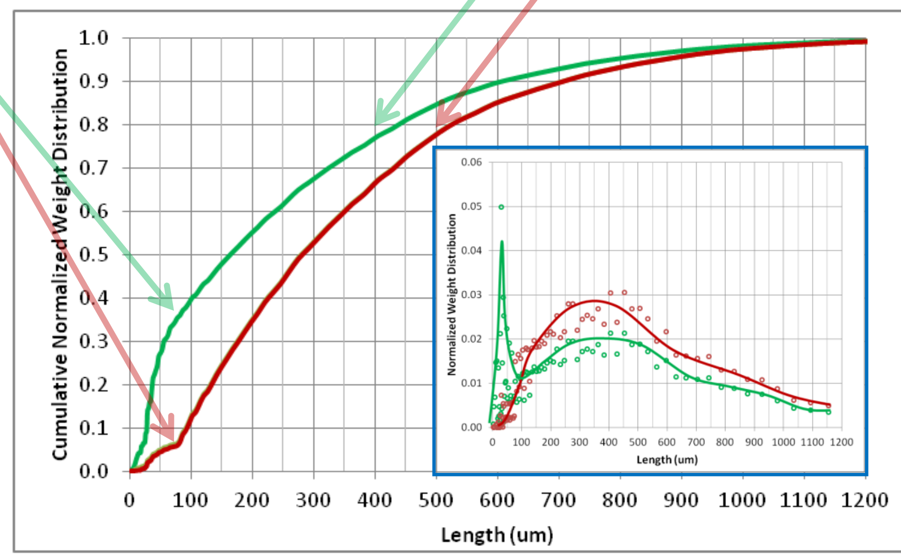
The gravimetric assessment of the fines obtained by sedimentation testing was 8.5%

Type of particles	Approximation for a group or class interval W_i
Dust L < 15um 	$\frac{N_i L_i}{33}$
Fragments 15um < L < 75um 	$\frac{N_i L_i}{8}$
Fibers L > 75um 	$N_i L_i$
Constant Coarseness	

The shape factors can be adjusted to yield different results



Other shapes tested: always come down to nL/Shape Factor

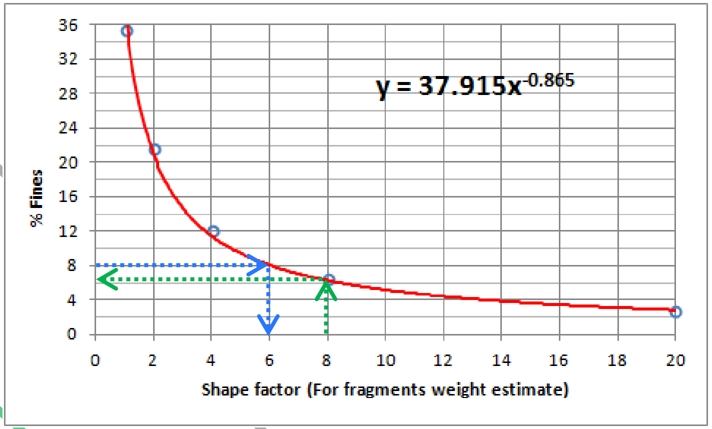


Nitrocellulose Particle Sizing:

Theoretical modeling of mass weighted fiber length distribution

The gravimetric assessment of the fines obtained by sedimentation testing was 8.5%

Model	Shape factor	%Fines
L, D	NA	36
L, D; L, d	1	36
	2	22
	4	12
	8	6
	20	2

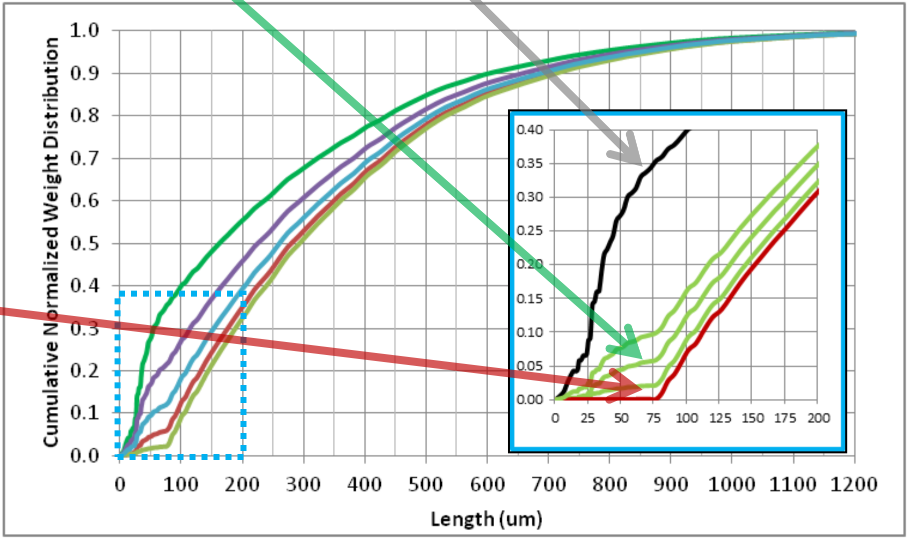


Type of particles	Approximation for a group or class interval W_i
Dust L < 15um	$\frac{N_i L_i}{33}$
Fragments 15um < L < 75um	$\frac{N_i L_i}{8}$
Fibers L > 75um Constant Coarseness	$N_i L_i$

-The fines model is applied between 0 and 75um, it can be applied on a different range (like 0 to 100um)

-If the shape factor is very large (>>20), it produces a distribution similar to a trimmed distribution

-The shape factors can be adjusted to correct results obtained on different equipment



Nitrocellulose Particle Sizing:

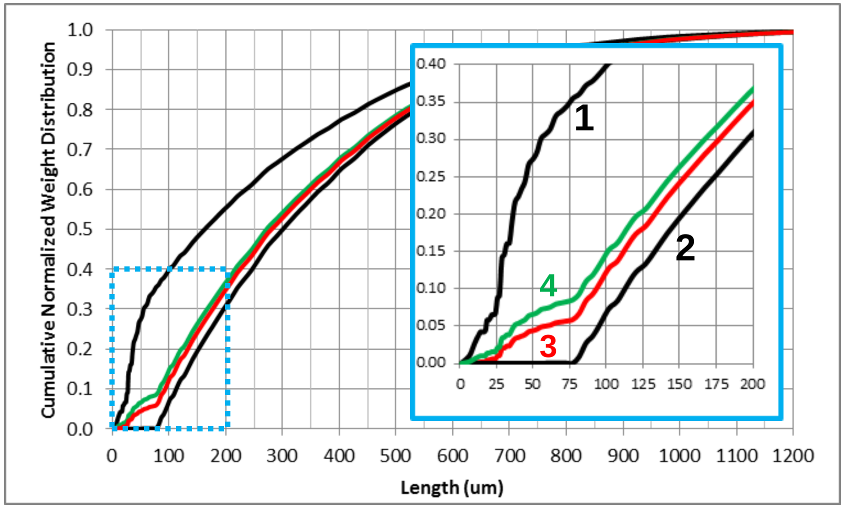
Empirical modeling of mass weighted fiber length distribution

	Model	%Fiber	%Fines	L_M	
1	Classic $L_{Length\ weighted}$	64.5	35.5	255	*Statistical model*
2	Classic $L_{Length\ weighted}$ Trimmed to 75um	100.0	0.0	374	*Statistical model*
3	2 shape model with shape factor = 8	93.6	6.4	353	*Theoretical model assuming 2 shapes*
	Actual %Fines Content Gravimetric method		8.5		*Empirical assessment of fines content in NC*
4	2 shape model with shape factor = 5.9	91.5	8.5	346	*Calibrated model base on empirical assessment*

The shape factor for this equipment is 5.9.
It is expected that once the model is calibrated using one sample of NC, it is valid for all similar samples of NC

For most analysers: a 2 shape model is sufficient
→ however no information provided about dust

For high resolution analysers: a 3 shape model could be used to provide information about dust
→ it should be calibrated accordingly



Nitrocellulose Particle Sizing:

Empirical modeling of mass weighted fiber length distribution

Using the gravimetric content in fines and the trimmed (between 75um and max) particle size analyser data : the L_M can be calculated using this relation:

$$\sum_{i=0}^{75um} N_i L_i \cong \left(\frac{\left(\sum_{75\mu m-max} N_i L_i\right)}{\left(\sum_{75\mu m-max} N_i L_i\right) - \%Fines/100} - 1 \right)$$

$$\sum_{i=0}^{75um} N_i L_i \cong \left(\sum_{75\mu m-max} N_i L_i \right) \left(\frac{100}{100 - \%Fines} - 1 \right)$$

$$L_M = \frac{\left(\sum_{0-75\mu m} N_i L_i\right) L_{M\ 0\ to\ 75um} + \sum_{75\mu m-max} N_i L_i^2}{\left(\sum_{0-75\mu m} N_i L_i\right) + \sum_{75\mu m-max} N_i L_i}$$

$L_{M\ 0\ to\ 75um}$ is typically about 45-50 um, therefore:

$$L_M \cong \frac{50\,(\%Fines) + L_{L\ 75um\ to\ max}\,(100 - \%Fines)}{100} = \frac{50um\ (8.5\%) + 374um\ (100-8.5\%)}{100}$$

Example:

-% Fines by gravimetric method = 8.5%

-$L_{L\ 75um\ to\ max}$ by fiber length analyser = 374um

Then:

-L_M = about 346um

-Compared to 346um by 2 shapes model

Example:

-% Fines is assumed to be 7% then L_M = about 351um (Error of +5um)

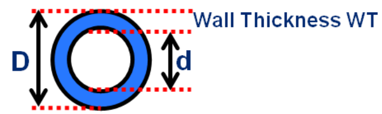
Nitrocellulose Particle Sizing:
Modeling of mass weighted distribution

Classification
Primary: Fibers vs Fines
Secondary: Fines → Fragments vs Dust
Fibers → ????

Again, the physical properties of NC tend to depend more on the mass then on the number of particles of the length fractions.

The coarseness is the linear density and is given by: **C = W/Length = Density x π/4 Diameter²**

Density of fiber (hollow cylinder with wall thickness WT) is given by:



$$Density\ of\ Fibers\ \delta_{Fibers} = \frac{Wall_{Cross\ Sec\ Area}}{Fiber_{Cross\ Sec\ Area}}\delta_{NC} = \frac{Fiber_{Cross\ Sec\ Area} - Lumen_{Cross\ Sec\ Area}}{Fiber_{Cross\ Sec\ Area}}\delta_{NC}$$

$$= \frac{(\pi/4\,D^2) - (\pi/4d^2)}{\pi/4\,D^2}\delta_{NC} = \frac{(D^2 - d^2)}{D^2}\delta_{NC}\quad where\ d = D - 2WT$$

$$C = Density_{Fiber} \times \pi/4\ D^2 = ((D^2\text{-}d^2)/D^2\ \delta_{NC})\ (\pi/4\ D^2) = k\ (D^2\text{-}d^2)$$

$$\textbf{Weight} = \textbf{Length x Coarseness}\ \alpha\ L\ (D^2\text{-}d^2)$$

L and D are external characteristics: L can be measured. What about D?

d (or WT) are internal characteristics: can only be estimated by relying on assumptions

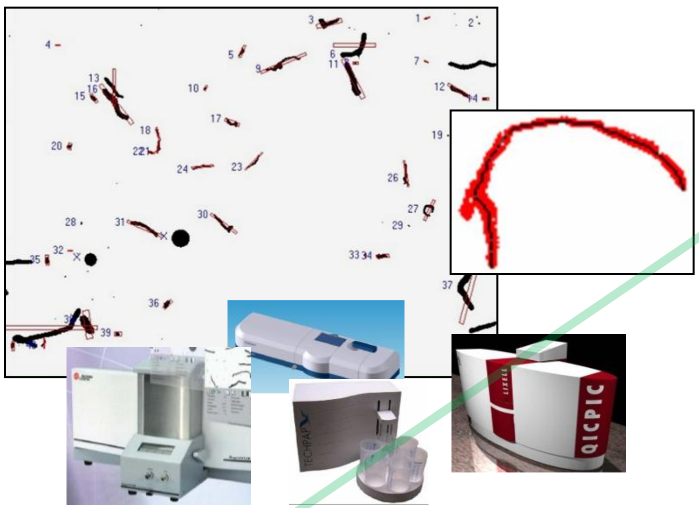
Nitrocellulose Particle Sizing:
Modeling of mass weighted distribution

Classification
Primary: Fibers vs Fines
Secondary: Fines → Fragments vs Dust
Fibers → ????

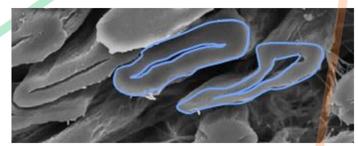
Fiber analyzer can only measure external characteristics.
D and L are external characteristics (Can be measured?)

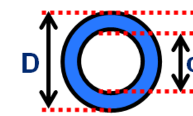
d or WT internal characteristics: estimated by relying on assumptions

Weight = Length x Coarseness α $L\,(D^2 - d^2)$

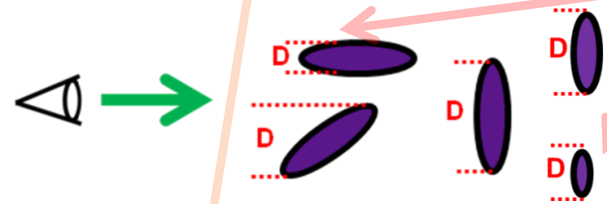


For fiberboard base NC, fibers are compressed hollow cylinders





Cannot differentiate

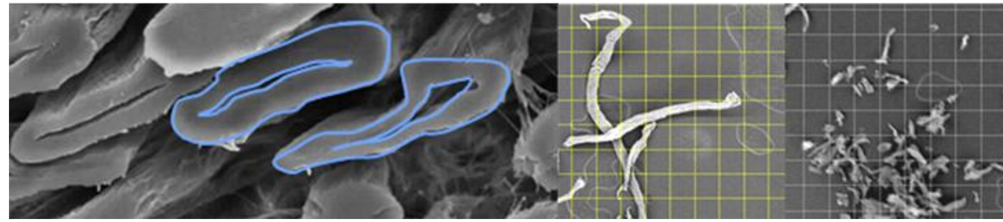


L measured = L of Hollow Cylinder

D measured ≠ D of Hollow Cylinder
Some interpretation will be required
(mathematical relation/statistical model may be?)

Additional efforts will be required to resolve the true distribution of the fiber portion of NC samples using higher resolution fiber analyzers

In summary:



-Equipment for particle sizing has evolved significantly in the last 20 years, however classic data analysis is still very simplistic because in the end,

data analysis is extremely application and/or product specific

-Using more specific forms of modeling, it is possible to obtain more realistic estimates of the NC mass weighted length distribution and also evaluate the content in fibers and fines

-Empirically calibrated NC models can go a long way to compensate for equipment variation

-Primary classification (fibers from fines) has the most effect on estimation of the mass distribution and can reliably be estimated

-Secondary classifications effect on mass distribution are more difficult to estimate, more work will be required using higher resolution fiber analyzers

-NC fines content can be estimated by a gravimetric sedimentation method

-For NC fibers: The trimmed to 75um Classic $L_{Length\ Weighed}$ is the closest substitute to L_M obtained by an empirically calibrated model and the actual weight distribution

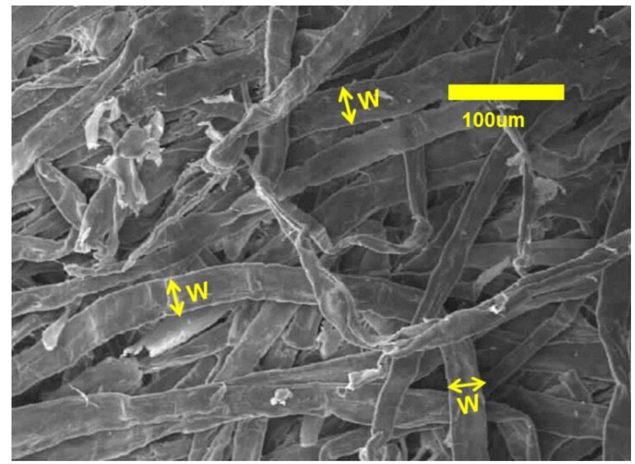
-For NC fibers and fines: For most applications, a 2 shape modeling with empirical calibration is sufficient

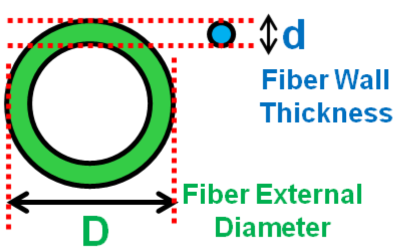
Nitrocellulose Particle Sizing:
Shape model: Fibers as Tubular Cylinder (Hollow Cylinder)

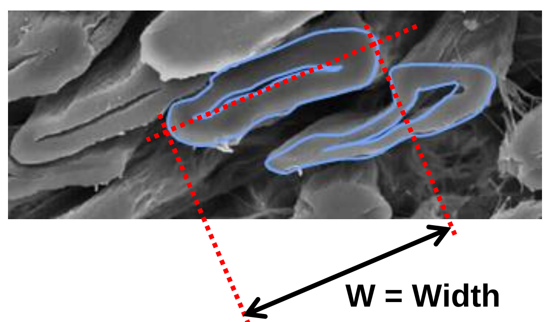
Rule of thumb:

Fiber Diameter ≅ W / 1.6
Fiber Wall Thickness ≅ W / 20

Type of wood	Typical External Diameter µm	Typical Wall Thickness µm	Typical Ratio Ext Dia / Wall Thick
Douglas Fir	40	4.0	10
Hemlock	40	2.5	16
Spruce/Pine	30	2.0	15
Western Red Cedar	30	1.5	20
White Spruce	30	2.0	15
Lodgepole Pine	30	2.0	15
Alpine Fir	35	2.0	18
Southern Pine	45	5.0	9
Scandinavian Pine	35	3.0	12
Average	**35**	**3.0**	**12.7**
Total Average	35	2.7	14.4







$$Circonference = \pi D \cong 2W$$

So $D = \frac{2L}{\pi} \cong W / 1.6$

Also

Wall Thickness ≅ D / 12.5 ± 3
Wall Thickness ≅ W / 20 ± 4