Measuring the content, size and density of fiber agglomerates of nitrocellulose by water elutriation

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Abstract

Fiber agglomerates are to be expected in various amounts in all nitrocellulose (NC) including NC made of baled cotton linters. In investigating the problem of fiber agglomerates, one of the first steps was to produce an analytical method that allows for the quantification of NC agglomerates. We previously presented a method based on the Bauer-McNett classification method using sieves and water wet NC slurries to generate a fractionation of the sample relative to the size of agglomerates. In addition of being time consuming; the sieving method cannot reliably separate fine and extra fine agglomerates because of fiber entanglements at agglomerate contents below 2-3%. We have developed another method which uses water elutriation for separating the free fibers from agglomerates in NC samples. It is more precise and easy to execute than the sieving method and in addition to content and size can provide a semi-quantitative assessment of agglomerates density. The method can also quantitatively differentiate between NC agglomerates and NC. Based on the results obtained with this method; a new nomenclature of NC's various physical shapes is presented.

1 Introduction

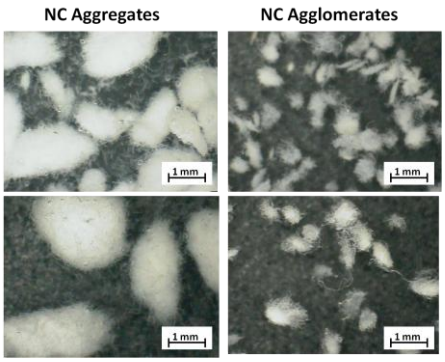
Cellulose is currently available in two forms to NC production facilities: baled cellulose (cotton base) and fiberboard cellulose (cotton or wood base). Baled cellulose is usually a fluff material composed of free fibers with various amount of fiber agglomerates composed of neps (fiber entanglements). Fiberboard cellulose is made of compressed fibers in sheeted or rolled form. Fiberization is the process of converting a fiberboard of cellulose to free fiber cellulose. For fiberboard cellulose prior to nitration: some NC facilities require fiberization of the fiberboard which means disintegration of the fiberboard back to free fibers. Instead other NC facilities require that the fiberboard be disintegrated into chips; small pieces of fiberboard that are obtained by processing the fiberboard through a cutter or shredder. In term of NC manufacturing, there are advantages and disadvantages to both approaches. In term of final applications: the use of fiberboard chips may be an advantage in some final applications which are essentially depending on molecular properties like lacquers and varnishes where they will be dissolve in large quantity of solvent and the use of NC chips may reduce dustiness and facilitate dosing in process equipment. However for other applications which uses some of the fiber properties like extruded propellants (microfibrillar properties) or combustible case (fiber properties); the presence of fiber agglomerates may be quite detrimental.
For NC facilities using fluff cellulose, the fiberization quality of the final NC can be enhanced in 2 ways; by improving the quality of fiberization prior to nitration and also by the deflakers/refiners at the stabilization stage. While for NC facilities using chips cellulose; the fiberization quality of the final NC can be enhanced only by the deflakers/refiners at the stabilization stage. In all cases, the measurement of the quality of fiberization of the NC may be an important quality criterion.

Measurement of the quality of fiberization is achieved by separating free fibers from anything else that is not free fibers in a sample. When measuring the quality of fiberization on the final NC samples, significant amount of fiber aggregates may be present in addition to agglomerates.

-*Agglomerates* are fibers bound together in a solid cluster. Agglomerates are produced from the original cellulose either from the fiberization or shredding of the fiberboard or for baled cotton; from neps which are fiber entanglements. Agglomerates are created before the nitration process and nitration renders fiberization much more difficult; it cannot be fiberized without the help of very energetic process equipment like refiners or deflakers available only in NC facilities.

-*Aggregates* conversely are fibers bound together in less solid cluster and are created after the nitration process usually in the dewatering section where nitrated free fibers in slurry can be compress into hard lumps. Since the dewatering process is the last stage of the NC manufacturing process; refining equipment cannot be used for their elimination. Those aggregates or lumps are typically much larger (10X) than agglomerates and be as high as 5% in some samples. These lumps have much lower mechanical resilience then agglomerates and can usually be eliminated by most processing equipment in the propellant or combustible case process when taken into account.

Figure 1



The aggregates can easily be pulverized by ultra-sonication which is not the case with agglomerates. It therefore can easily be quantified.

We have previously developed a method based on the Bauer-McNett classification method using sieves and water wet NC slurries to produce a fractionation of NC sample relative to size of agglomerates. The method was modified to permit classification of large agglomerates of fibers instead of free fibers. The typical Bauer-McNett classification method uses Tyler standard sieves number 14, 28, 48, 100 and 200 (which are equivalent in size to 1.18mm, 0.60mm, 0.30mm, 0.15mm and 0.075mm) to fractionate cellulose fibers. Because NC fibers are much shorter than pulp and paper fibers (by a factor more than 10X) most of the fibers are easily eliminated leaving only fiber agglomerates on the sieves. This sieving method for agglomerates is based on the Tyler sieves number (or equivalent) 20 (0.85mm), 32 (0.50mm) and 60 (0.25mm). This method is simple, low cost, fast and can easily be modified for semi-quantitative at line measurement. A portion of NC slurry is transferred to the stack of sieves. The sample is washed down with water in order to fractionate by size of agglomerates. The amount can be quantified (by weight) for more precise evaluation or semi-quantified (by volume) for fast analysis.

Classes of agglomerates by size

The fiber agglomerates are divided in 3 classes:

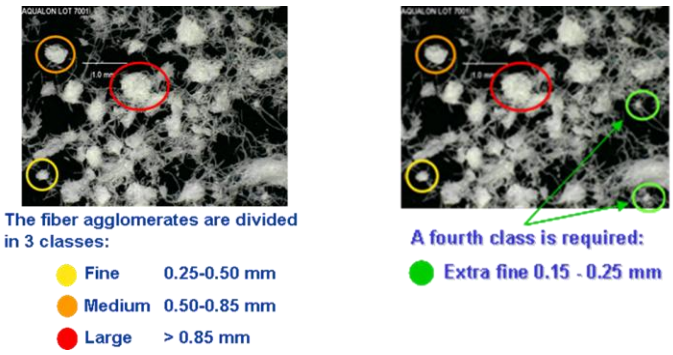
Fine	0.25-0.50 mm
Medium	0.50-0.85 mm
Large	> 0.85 mm

This sieving method was added to the NC specification MIL-DTL-244C [1] and to the STANAG 4178 Ed.2 [2].

However, this size classification may not be sufficient for all applications and more specifically for combustible case applications. Smaller agglomerates are visible in NC samples and a more stringent definition of agglomerates may be necessary. A fourth class may be added:

Extra fine 0.15 - 0.25 mm

Figure 2



It is not possible to reliably separate fine and extra fine agglomerates using the sieving method because very small agglomerates are strongly entangled in the free fibers. The sieving method is able to perform reliable assessment of large and medium size agglomerates; however the assessment of the fine agglomerates is unreliable below 2-3% in agglomerate content. In addition, the sieve method is labor intensive and this becomes a real annoyance with long fiber NC and/or for small agglomerates.

We developed a second method in 1985 which uses elutriation for separating the free fibers from agglomerates and was used to investigate the quality of refining using the Jordan refiners in Valleyfield for conditions where the level of agglomerates was below 2%. The method is identified as the *elutriation method*.

A basic elutriator follows these basic principles:

-various size agglomerates and fibers have in general different masses and shape which imply that they have different terminal velocity when falling in a fluid following the reasoning of Stoke's law

-therefore; they can be separated by elutriation using water as a fluid

-creating an ascending velocity gradient with high velocity in the bottom and low velocity on the top can produce a simple mean of achieving physical separation and quantification

-exception: some agglomerates with significantly lower density (like very fragile pieces of sheet delamination) may be ejected with the fibers)

The basic design of the elutriator is simple using a decantation cone called an Imhoff sedimentation cone. A stable gradient flow is created by feeding tap water from the bottom of the cone. Since the volume of the cone increases with height; a stable gradient of water flow upward is produced. Fiber and agglomerates falling under the influence of gravity in water quickly reach the height in the cone equivalent to their terminal velocity in the fluid causing separation roughly following Stoke law. There is about 3 order of magnitude difference in ascending velocity between the bottom and the top of the cone.

In order to work well; the flow must be as laminar as possible. However turbulences are required to help break-up the NC flocculate which trap agglomerates. High velocity in the bottom of the cone creates a zone of turbulence which is required for deflocculation of the fibers. It is important to limit the zone of turbulence to the bottom of cone to avoid disturbing the laminar flow in the top portion of the cone to prevent ejecting smaller agglomerates.

The testing equipment and conditions are standardized to achieve both these effects consistently in the cone. The ascending velocity of the fluid in the cone is proportional to the flowrate of the fluid. Imhoff sedimentation cones are usually 45.0 cm in height with an opening of 11.6 cm; the velocity at various height in that type of cone is given in the following graphs for various flowrates of water at about 15°C. Imhoff cones are usually graduated in ml; therefore the height can be measured using the graduation marks or by linear measurement using a ruler.

Figure 3

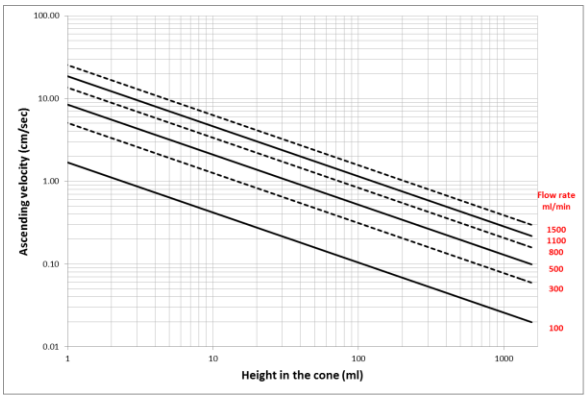
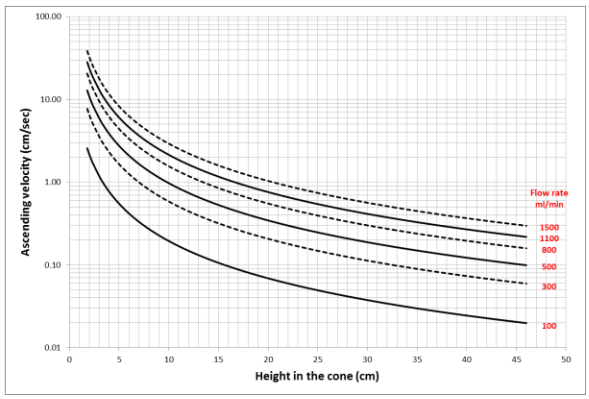


Figure 4

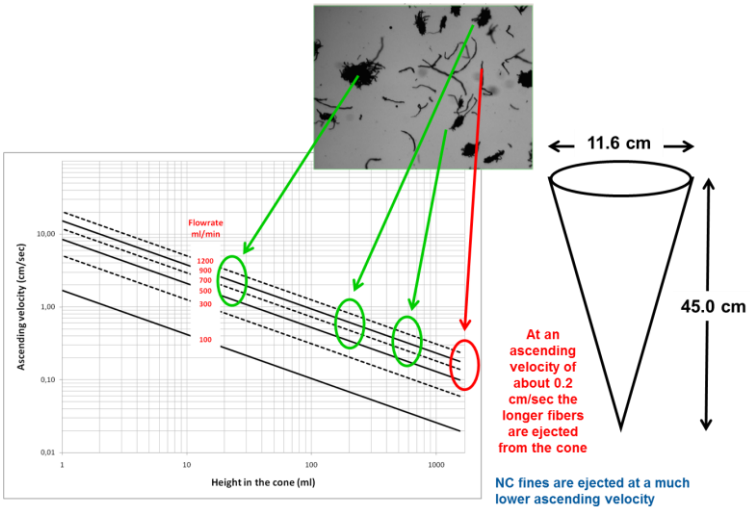


The Imhoff sedimentation cone of 1000 ml is usually graduated to 1000 ml but may contain up about 1500 ml depending on the cone model. The important velocity is the ejection velocity which is the velocity at which the material will be ejected from the cone. That velocity is at the upper rim of the cone which is about the 1500 ml or 45 cm from the bottom.

Thus at a fix flow rate of about 900-950 ml/min:

-Large agglomerates are at the bottom of the cone
-Medium agglomerates are about 1/3 up the cone
-Smaller agglomerates are about mid-cone
-Free fiber will be ejected from the cone eventually

Figure 5



All fibers are rapidly extracted from the cone and separated from the agglomerates by the over flowing water at the top of the cone. Although water could be fed from the bottom; it is easier to add a steel tube centered in the cone at a height of about 4cm above the bottom of the cone. The tube is centered using 2 holders. The bottom holder is important because it creates the right amount of turbulence in the bottom part of the cone to deflocculate NC and speed-up separation of agglomerates from free fibers. Those turbulence must not disturb the laminar flow in the upper part of the cone were separation of large fibers from

small agglomerates it taking place. Typically, the extraction is done on 5g (equivalent dry weight) of NC and the extraction takes about 20-30 minutes.

Once the separation is completed, the water flow is stopped. The agglomerates are allowed to settle in the cone and a volumetric assessment can be done. The gravimetric assessment is more precise and accurate. For a gravimetric assessment; the agglomerates are filter out using cindered glass crucible and dried to constant weight in an appropriate oven. The weight of total agglomerates can be measured on an analytical balance. The size distribution of the agglomerates may be determined using a set of standard sieves or an image analysis system.

For samples containing high contents in agglomerates (>2-3%), the quantification can be done in one extraction. Whenever the content in agglomerates is lower than 1% the presence of residual fibers in the cone with the agglomerates significantly affects the results. When starting the analysis; the concentration in free fiber is high and extraction is rapid. However, as the extraction is continued; the concentration in free fiber rapidly decreases causing a leveling off. Fibers clinging to the side of the cone account for a large proportion of the leveling off. The exponential decrease in concentration of free fibers over time is similar to the following:

Figure 6

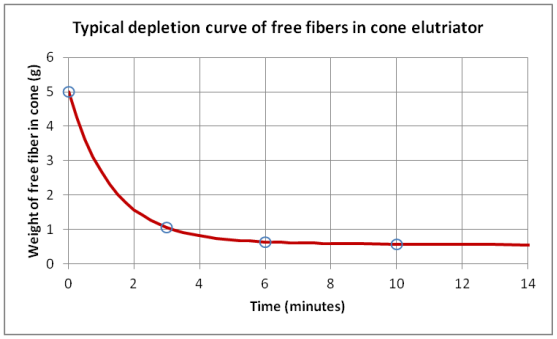
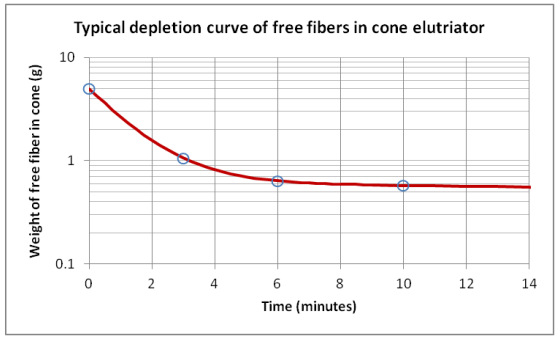


Figure 7



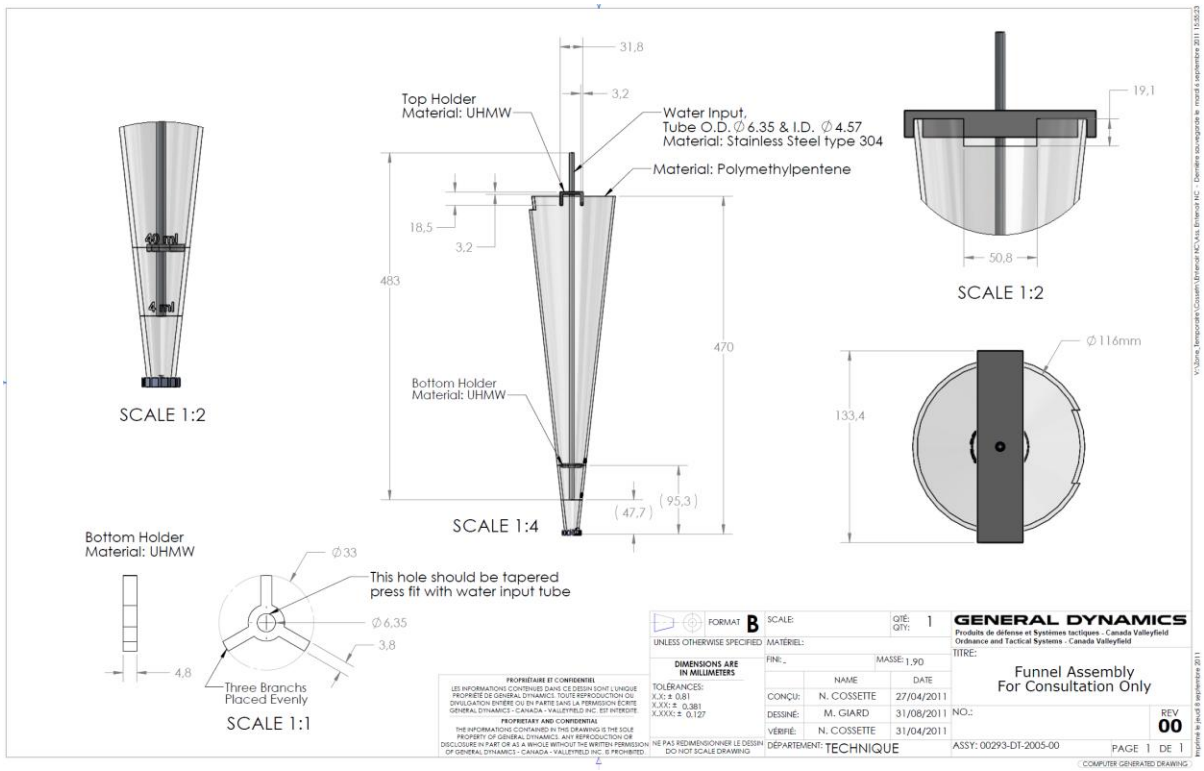
In order to circumvent that problem, the cone method was modified by adding extraction steps to the procedure with clean-up steps in between. Instead of performing the analysis in the cone once and moving on to the quantification using a fritted glass crucible; the material is transferred in a beaker with about 500 ml of water. The cone is then washed and prepared for a repeat of the extraction. A series of washing is done on the 500 ml portion by whirling the content to put the fibers and agglomerates in suspension in the beaker, letting the mixture rest for about 30-90 depending on size of agglomerates and size distribution of free fibers (this can be assess visually) and pouring the water and floating fibers (free of agglomerates and aggregates) down to about 100 ml. The process is repeated up to 3 times and the left-over is transferred in the cone elutriator for a final extraction. The extraction time in the cone can be reduced from 30 minutes to about 10 minutes per extraction, thus the total time is still about 30 minutes for the extraction procedure.

2 Analytical procedure for cone elutriation method

Equipment

The main equipment is an Imhoff sedimentation cone of 1000 ml (similar to Brandtech Scientific Cat.Number 388000) and modified to produce the elutriator as illustrated in diagram 1.

Diagram 1: the cone elutriator



Additional equipment:

-Erlenmeyer about 250 ml fitted with a rubber stopper

-Beaker about 500 ml

-Stainless steel agitating rod about 30 cm in length and about 2 mm in diameter

-Elutriation cone according to provided diagram 1

-Lab stand, clamps and ring holders to support the cone level and vertical

-Flexible Tygon tubing about 6 mm inside diameter (about 2 meters) or equivalent

-1 plastic ball valve with barbs about 6mm outside diameter (similar to Cole-Palmer Catalog Number R-01377-30)

-1 plastic Y connector with bards about 6mm outside diameter (similar to Cole-Palmer Cat Numb ED-06296-20)

-1 connection to tap water with adapter with barb about 6 mm outside diameter to connect a Tygon tubing or equivalent

-Crucible (fritted or Gooch) about 2 cm in diameter and fitted with filter paper. Crucible are dried in oven a about 100°C for about 1 hrs prior to use.

-Crucible filtration set up

-Oven set a temperature preferably >65°C to dry NC to constant weight.

-Dessicator

-Analytical balance with a precision of ±0.1 mg

-Laboratory ultrasonic bath or probe

- Flowmeter with metric-reading scale about 100-1500 mL/min (similar to Direct Reading Flowmeter from Cole-Palmer Cat Numb R-32461-40)

Chemicals

-Tap water

Procedure

Setting up the cone elutriator

-Set up the elutriator according to diagram 2 using the lab stand, clamps and ring holders to support the cone level and vertical above a collection system, filter system or drain which can receive/dispose of water contaminated with NC fibers.

-Connect the stainless steel flow tube (from diagram 1) to the tap water using the tap water adapter, the flexible Tygon tubing, the Y connector, the plastic ball valve and the flowmeter according to diagram 3.

Diagram 2: Set up for the cone elutriator

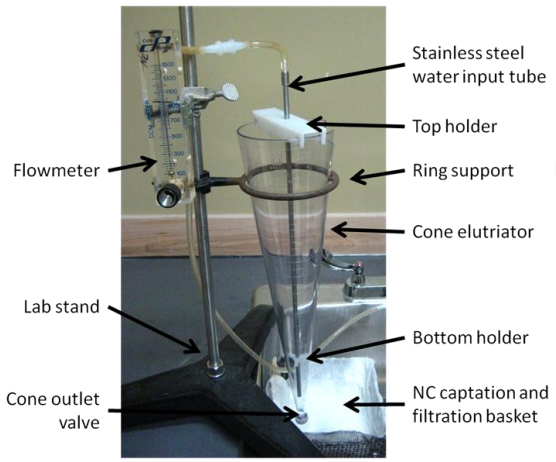
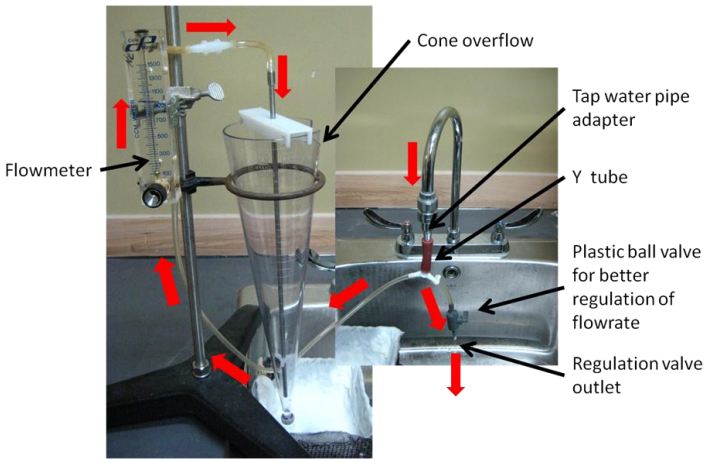


Diagram 3: Water flow diagram for the cone elutriator



First elutriator extraction:

-Transfer about 5 g of dry nitrocellulose or the equivalent (if the content in water is known; the estimated dry weight can be determined) into a clean 250 ml Erlenmeyer. Weigh and record the weight to a minimum precision to ±0.01 g ($W_{Dry\ NC}$).

-Wet the NC with 200 ml of water and seal the Erlenmeyer with the rubber stopper.

-Shake vigorously for about 30 seconds to wet all the NC.

-Install the water feed stainless steel tube into the cone. Ensure that the tube is centered and that the plastic bottom holder is at the proper height: the bottom of the tube should be near the 4 ml line and the plastic bottom holder near the 40 ml line.

-Transfer the NC from the Erlenmeyer to the elutriation cone and rinse both the Erlenmeyer and stopper into the cone.

-Start the water flow and set the water flow rate at about 950 ml/min.

-Elutriate the NC for about 10 minutes. During the elutriation, the stainless steel rod can be used to agitate the slurry at regular interval of about 3 minutes to help break-up the flocculation, speed up the separation and clean the side of the cone. This agitation must be very slow and for short periods of time (about 3-6 seconds). The central tube with both holders can also be agitated to remove clinging NC.

-After 10 minutes, stop the flow of water and remove the water feed stainless steel tube from the cone. Using the stainless steel rod wipe the side of the cone to ensure that all agglomerates are in suspension. Wait 2 minutes to decant the agglomerates and transfer about 500 ml of the water and NC agglomerates/aggregates into a 500 ml beaker. Clean and prepare the cone for another elutriation.

Beaker extraction

-Using the stainless steel rod, agitate the slurry in the 500 ml beaker to suspend all the solids. Wait about 30-90 seconds to decant all the heavier material than remove and discard about 400 ml of the water containing free fibers. Add about 400 ml of fresh tap water.
-Repeat the last operation two more time after which the remaining solid in the beaker should consist essentially of agglomerates or aggregates with a minimal quantity of free fibers.

For analysis of agglomerates and aggregates:
-The solution does not require any addition treatment and is ready for quantification. Move to Quantification.

For analysis of agglomerates only:
-Install the 500 ml beaker containing the agglomerates into a sonication bath or install the sonication probe to sonicate the content of the beaker. The sonication is done for about 15 minutes. This can be adjusted depending on the quality of the NC.
-The solution is now ready for the final elutriator extraction. Move to the final elutriator extraction.

Final elutriator extraction
-Install the water feed stainless steel tube into the cone. Ensure that the tube is centered and that the plastic bottom holder is at the proper height: the bottom of the tube should be near the 4 ml line and the plastic bottom holder near the 40 ml line.
-Transfer the all NC from the 500 ml beaker to the elutriation cone and rinse the beaker into the cone.
-Start the water flow and set the water flow rate at about 950 ml/min.
-Elutriate the NC for about 10 minutes. During the elutriation, the stainless steel rod can be used to agitate the slurry at regular interval of about 3 minutes to help break-up the flocculation, speed up the separation and clean the side of the cone. This agitation must be very slow and for short periods of time (about 3-6 seconds). The central tube with both holders can also be agitated to remove clinging NC.
-After 10 minutes, stop the flow of water and remove the water feed stainless steel tube from the cone. Using the stainless steel rod wipe the side of the cone to ensure that all agglomerates are in suspension. Wait 2 minutes to decant the agglomerates and transfer about 500 ml of the water and NC agglomerates/aggregates into a 500 ml beaker. Clean and prepared the cone for another elutriation.

Quantification:
The content in agglomerates or agglomerates/aggregates can be done gravimetrically or volumetrically. The gravimetric assessment is more precise. The volumetric assessment is more rapid.

Volumetric assessment:
-Transfer the content of the 500 ml back to the cone fitted with a volumetric stopper. The cone is capable to perform volumetric assessment to a minimum volume of 0.1 ml. A decantation bulb capable of measuring down to 0.1 ml can also be used; the proper amount of water can be discarded from the 500 ml beaker in order to reduce the volume of the solution to the volume of the decantation bulb. After about 5 minutes, the approximate volume can be estimated. Since the method may produce results that cover more than 3 orders of magnitude; it is recommended to express the results to the following precision:

For final volume below 0.05 ml:	<0.05 ml
For final volume between 0.05 and 1 ml:	with a precision of 0.05 ml
For final volume between 1 and 2 ml:	with a precision of 0.1 ml
For final volume between 2 and 10 ml:	with a precision of 0.5 ml
For final volume between 10 and 40 ml:	with a precision of 1 ml

Gravimetric assessment:

-Obtain a crucible fitted with a filter paper that was dried to constant weight and maintained in a dessicator to room temperature. Weigh to a precision of 0.1mg and record the weight of the crucible ($W_{crucible}$).

-Filter the content of the 500 ml in the prepared crucible.

-Transfer the crucible with the agglomerates or agglomerates/aggregates into a drying oven and dry to constant weight.

-Transfer the crucible with the agglomerates or agglomerates/aggregates into a dessicator for cooling.

-Once at room temperature weigh and record (to a precision of 0.1mg) the weight of the crucible with the agglomerates ($W_{crucible+agglo}$) or agglomerates/aggregates ($W_{crucible+agglo+aggre}$).

Calculation:

Eq 1 % agglomerates/aggregates = $(W_{crucible+agglo+aggre} - W_{crucible}) / W_{Dry\ NC} \times 100$

Eq 2 % agglomerates = $(W_{crucible+agglo} - W_{crucible}) / W_{Dry\ NC} \times 100$

Eq 3 % aggregates = % agglomerates/aggregates - % agglomerates

3 Precision of the method

The method can produce results that cover about 3 orders of magnitude. Several samples of various contents in agglomerates spanning the range of possible results were tested using this method (from 0.05% to 25%). The test was done by two operators: Operator 1 is newly trained to the method while operator 2 is very familiar with the method and has analyzed hundreds of sample. Four repeats on each sample. The following results were obtained:

Table 1

	Operator 1		*Operator 2*		*Average of sample*	*Repeatability*	Reproducibility			
Sample	Order of testing	Results	Order of testing	Results		Average Standard Deviation of operators	Standard Deviation of measurement averages (between operator variation)	R and R	Expected precison of a duplicate analysis	Expected precison of a triplicate analysis
		%		%	%	Sigma	Sigma	Sigma	Sigma	Sigma
High concentration sample										
	8	25.6	4	24.5						
	16	20.6	12	25.4						
	23	23.3	18	23.5	**24.0**	1.92	1.70	2.56	1.81	1.48
	32	21.7	28	27.4						
Mean		22.8		25.2						
Standard Deviation		2.17		1.66						
Medium concentration sample										
	5	1.51	2	0.82						
	13	0.79	10	0.74						
	22	0.86	19	1.07	**0.98**	0.26	0.07	0.26	0.19	0.15
	31	0.95	26	1.11						
Mean		1.03		0.94						
Standard Deviation		0.33		0.18						
Low concentration sample										
	6	0.15	1	0.16						
	14	0.28	11	0.17						
	24	0.08	20	0.20	**0.17**	0.062	0.028	0.068	0.048	0.039
	29	0.09	27	0.23						
Mean		0.15		0.19						
Standard Deviation		0.092		0.032						
Very low concentration sample										
	7	0.02	3	0.06						
	15	0.06	9	0.03						
	21	0.04	17	0.04	**0.04**	0.018	0.009	0.020	0.014	0.011
	30	0.03	25	0.07						
Mean		0.038		0.050						
Standard Deviation		0.017		0.018						

Operator 1 with less experience and tend to produces lower average results than operator 2. This is attributed to the fact that operator 1 stirs the NC slurry frequently and with more energy causing substantial disruption in the laminar flow in the cone and expelling more agglomerates. The effect is less important with sample containing large quantity of agglomerates but can affect results of samples containing low level of agglomerates. Ideally, the results obtained from a duplicate analysis made by 2 operators (one test each) produce more reliable results.

The test is designed to establish the content in agglomerates in rough order of magnitude type of evaluation. With low content samples: retaining or losing a few agglomerates will cause significant variation in the precision of the method. The relative precision of duplicate analysis will increase with content typically following this relation:

Figure 8

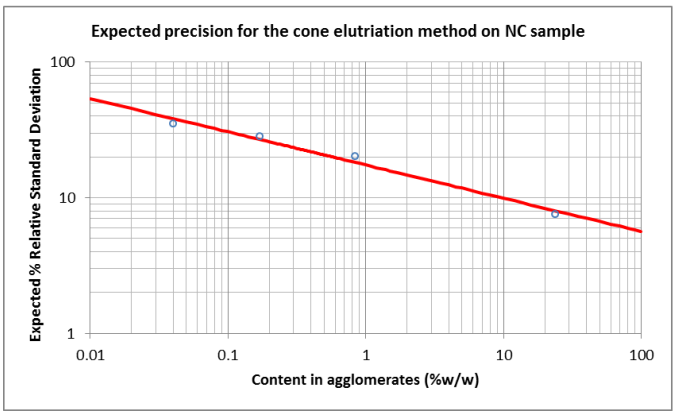
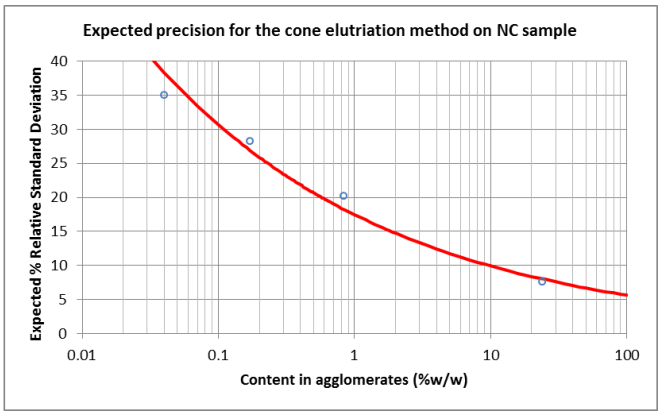


Figure 9



4 Examples of results obtained using the method

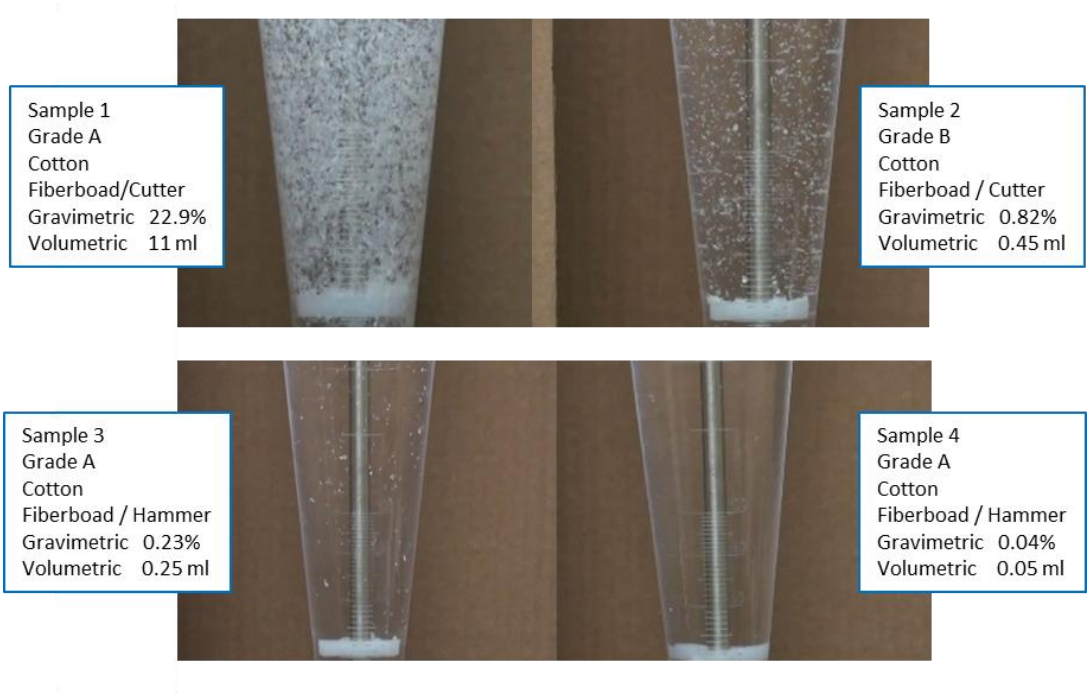
The following are examples of cone elutriation extraction on samples from various suppliers. All samples are NC prepared from fiberboard cellulose using either cutter or hammermill cellulose preparation equipment:

Table 2

Identification	Cellulose	Cellulose preparation	Cellulose preparation	Agglomerates		Aggregates
				Gravimetric	Volumetric	Gravimetric
				% w/w	ml	% w/w
Sample 1	Cotton	Fiberboard	Cutter	21.9	11	0.90
Sample 2	Cotton	Fiberboard	Cutter	0.82	0.45	0.02
Sample 3	Cotton	Fiberboard	Hammermill	0.23	0.25	0.28
Sample 4	Cotton	Fiberboard	Hammermill	0.04	0.05	0.67

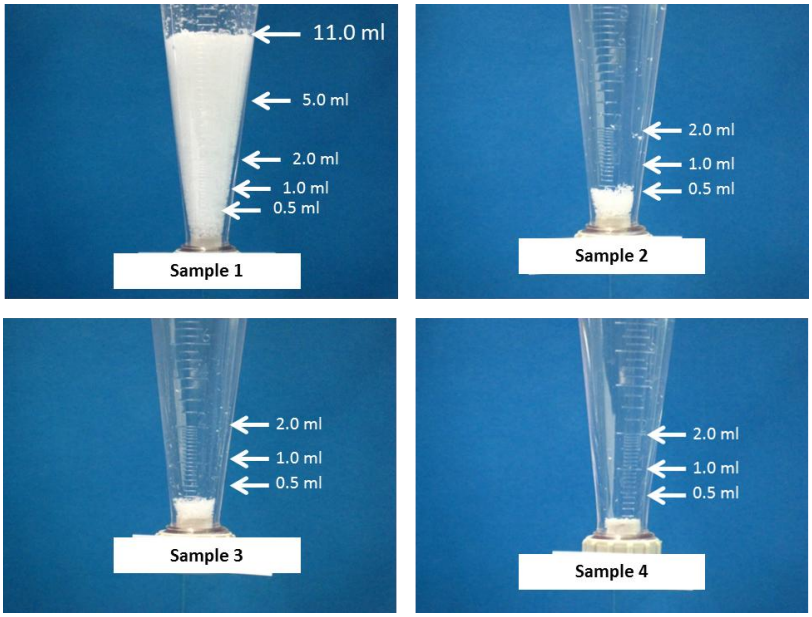
For these samples, the following pictures of the NC agglomerates in the cone elutriator are taken just before quantification (results for sample 1 to 4):

Figure 10



Once the agglomerates are decanted, volumetric quantitative assessment can be made directly into the cone (results for sample 1 to 4):

Figure 11



Additional quantification

The method can also be modified to quantify the size of agglomerates and to some extent the density of the agglomerates.

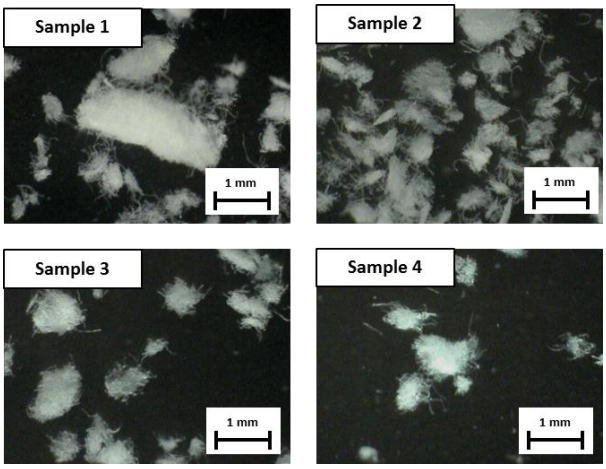
Size quantification

The agglomerates, now free from the fibers can easily be separated by size using sieves of various sizes. Typically the sieves of interest are: 0.85mm 0.50mm 0.25mm 0.15mm.

Size quantification can also be done using a fiber length analyzer with the proper rejection filter for the free fibers.

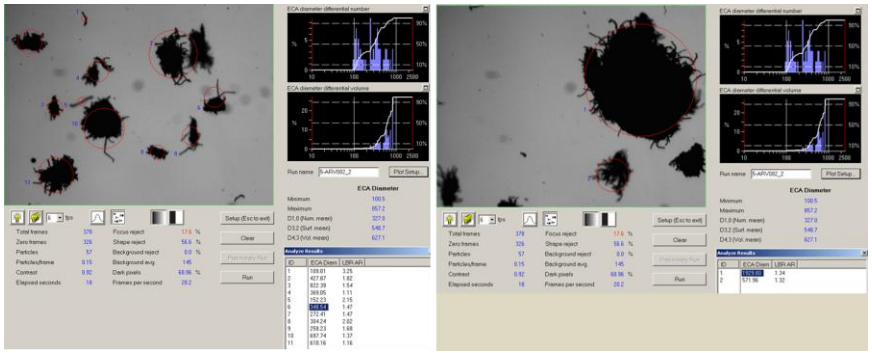
In addition to a qualitative assessment of agglomerates size, the microscope pictures of the isolated agglomerates allow for quantitative assessment of their size and distribution using an image analysis software. The following are pictures are eloquent examples of agglomerates isolated from the previously listed NC samples:

Figure 12



A similar assessment can be done by using a fiber analyzer which is programmed to calculate the average diameter of a sphere equivalent to the agglomerates with the adequate filters to reject fibers. In the following example, agglomerates with an average diameter from about 0.25mm to 2.0mm are displayed:

Figure 13



Density quantification

Density can be roughly be estimated by the elutriator if the size of agglomerates is known. Low density agglomerates (0.3-0.5 g/ml) will be floating in the upper portion of the elutriator during testing while high density agglomerates (0.6 to 0.8 g/ml) of equivalent size will be floating in the lower portion of the cone. At this time no other method is available however, looking at the volume occupied by the agglomerates versus the weight of agglomerates enable a relative ranking of density:

Table 3

Identification	Cellulose	Cellulose preparation	Cellulose preparation	Agglomerates		Ranking the density of agglomerates
				Gravimetric	Volumetric	
				% w/w	ml	
Sample 1	Cotton	Fiberboard	Cutter	21.9	11	High
Sample 2	Cotton	Fiberboard	Cutter	0.82	0.45	High
Sample 3	Cotton	Fiberboard	Hammermill	0.23	0.25	Low
Sample 4	Cotton	Fiberboard	Hammermill	0.04	0.05	Low

Sample 1 contains about 22% of agglomerates in 11 ml of volume (a ratio of 2) which is about the same ratio for sample 2 (about 0.8% in 0.45ml). However sample 3 is 0.23% in 0.25ml and is similar in density to sample 4 which is about 0.04% in 0.05ml both with a ratio near 1. This is confirmed by the elutriation testing: agglomerates from sample 1 and 2 are heavier and hand lower in the cone relative to sample 3 and 4 which are much liter and hang higher in the cone during elutriation. The agglomerates relative size distributions are all similar except for sample 1 which has the largest agglomerates.

5 Conclusions

Agglomerates quantification is an integral part of acceptance testing of nitrocellulose as a measure of the quality of fiberization. The actual method base of wet sieving approach was developed for samples containing large content in agglomerates and agglomerates of large sizes. The method using elutriation is developed mainly to improve quantification of agglomerates and aggregates in samples of low content and/or containing small agglomerates. However it can just as well analyse samples with large content in agglomerates. In addition it improves the quantification of agglomerates in several ways: It is not so labour intensive, the precision of the method is not so much dependent on the patience of the technician, it can isolate smaller agglomerates in addition to larger sizes agglomerates and some additional information is obtained about the density of agglomerates.

Among the great challenges that NC facilities have met in the past with various success and must meet in the future is the introduction of fiberboard cellulose (cotton or wood). This can be done only with a clear quantitative understanding of the performance required from the NC in various applications in order to adapt the facilities to produce the required material. This inexpensive method can easily be introduced at line to monitor the NC refining process in addition to fineness and/or freeness. This early in the process, there is still hope and means of improving the NC and since at that stage NC is still unstable, this method does not require drying of the NC and using volumetric assessment can easily be adapted to fit the purpose.

References

[1]
Nitrocellulose Specification MIL-DTL-244 Revision C
Detail specification for nitrocellulose approved for use by all departments and agencies of the US Department of Defense.
Project Manager for Combat Ammunition System, Picatinny Arsenal, N.J. 07806-5000.

[2]
NATO Standardisation Agreement (NATO STANAG) 4178, edition 2.
Test procedures for assessing the quality of deliveries of nitrocellulose from one NATO nation to another.
Military Agency for Standardisation, NATO Headquarters, Blvd Leopold III, 1110 Brussels, Belgium.