

Characterization of nitrocellulose agglomerates within as-collected samples

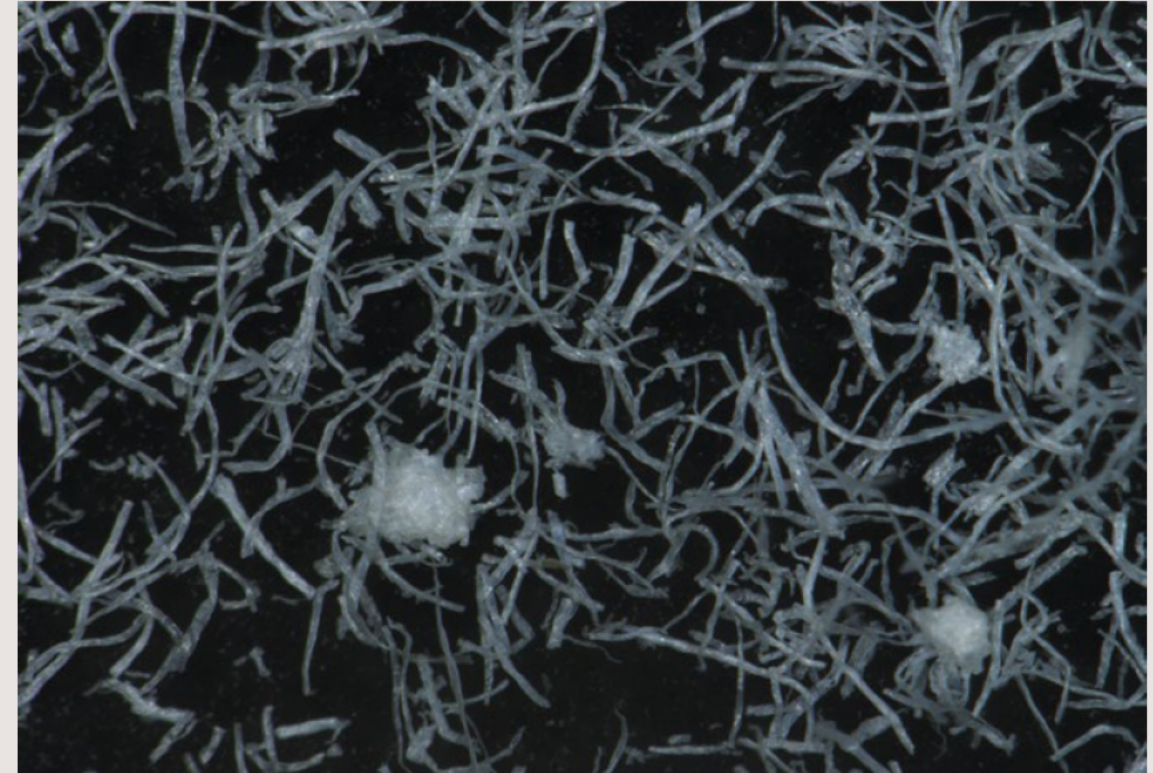
Dr. Lee A Goetz, Amy Morris, Roger Hollins, Kelly Moran

Ordnance Systems, Inc.
BAE Systems
Radford, Virginia USA

Project Overview

Overview

- Agglomerates Characterization
- Methodology
- Results
- Lessons Learned
- Summary
 - Path Forward
 - Conclusions



Agglomerates Characterization

Nitrocellulose Characterization

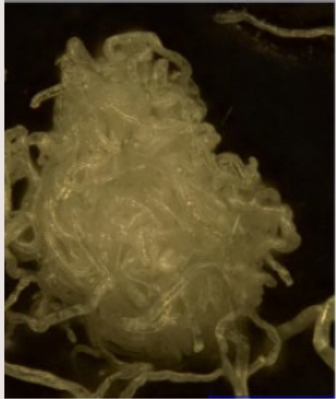
- Improved Quantification of Standardized Testing (MIL-DTL 244(C))
- Nitrocellulose properties' effect on downstream products and manufacturing
- Nitrocellulose manufacturing process evaluations
 - Cellulose raw material source evaluations
 - New and existing
 - Overall NC manufacturing process comparisons
 - Cellulose Cutting Methods
 - Nitrocellulose Refining Process Comparisons

Final Product
AND
In-Process
Nitrocellulose
Sampling



Agglomerate Characterization

- Agglomerates definition:
 - Bundles of nitrocellulose fibers that have not been separated into individual fibers during the overall nitration manufacturing process



Characterization Methods

- Sieve Analysis,
- Elutriation,
- Microscopy

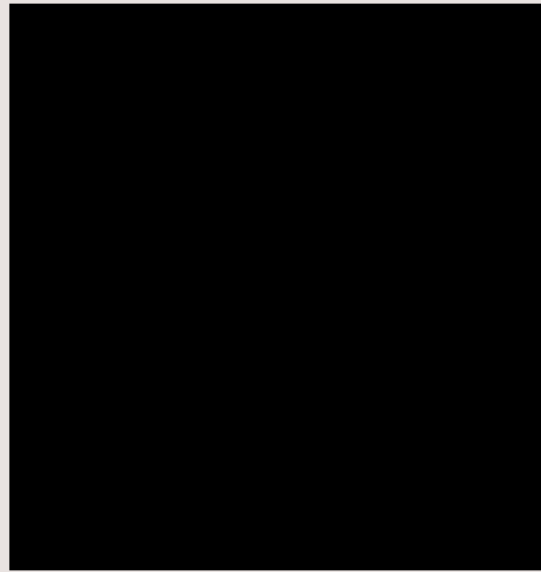
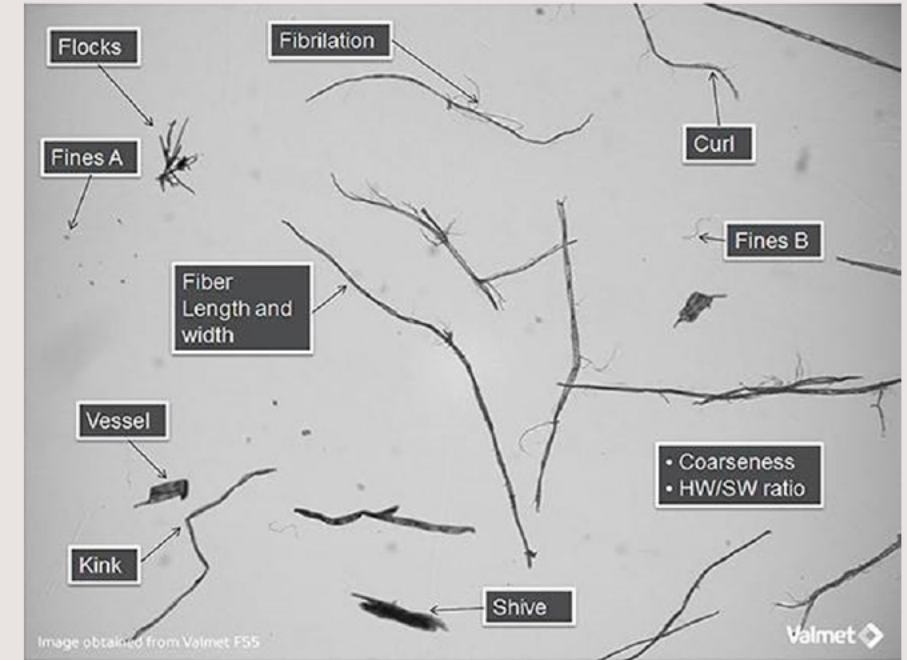


Limitations

- Provide limited information on agglomerate dimensions, area, and shape
- Requires test samples to be stable, 'final' process
- In-process NC samples are unstable
 - Special handling requirements, or
 - In-lab stabilization
 - Test time requirements

Fiber Analyzers

- Image analysis of pulp fibers dilutely suspended in water
- Developed for pulp and paper industry
- Fiber length and width distributions
- What opportunity is there to fine-tune image analysis to identify and quantify 'non-fibers'?



Can we use image analysis to better quantitatively analyze agglomerates in nitrocellulose?

Methodology

Methodology

NC Grade Selection

Training sets of NC
(historical and current
lots)

"As Received"
samples
analyzed

Samples wet
sieved to
separate by
sieve size for
training

Image
Analysis
films

Identification and Classification

Image
Analysis
films

Major Classes
aka "Object"

Able to set sub-
classifications

Agglomerate

Not an
Agglomerate

Object
Identification
Files for each
film

Compilation of Object Identification Files

Select and combine
to create targeted
neural network
identification
method

Program Validation
using training sets
(half of identified
sample set is tested
against other 50%)

Validation Results
Data/squares
(Good = 75%, ours were
typically >97%)

Testing

Run same sample as used for
identification/validation with
trained data set; Compare
results (area, length, width)

Repeat same films with trained
data set and compare how
repeatable the identification
set is

Analyze NC lot not used in
identification/classification/cre
ation

Down select to "best" neural
network identification model

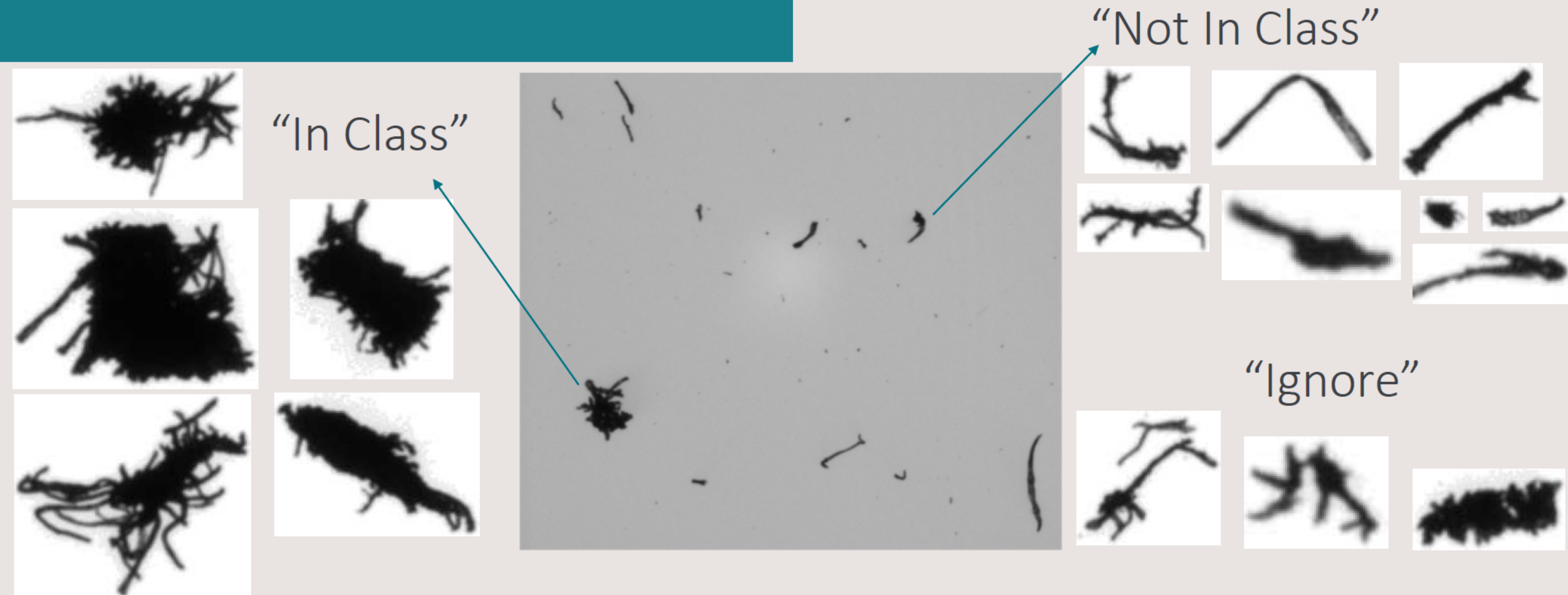
Implementation

Analyze additional NC lots for targeted
analysis

Future Work

Classification

Classification Process



Every sample of interest is run through the analyzer, an image file generated, and undergoes classification process

Classification Summary Info

Sample ID	Number of Particles Identified as "In Class"	Number of Particles Classified as "Not in Class"	Number of Particles Marked as "Ignored"
694	5	6	2
695	3	3	1
677	10	14	0
678	9	13	1
679	10	18	0
670	258	6	5
667	334	9	4
648	231	1	4
650	283	2	2
658	1939	236	216
647	1315	110	106
649	1064	159	76
643	331	1499	185
622	254	1013	76
625	318	1096	1587

- Examples of class selection for selected sample files
- Generate the neural network agglomerate "Identification Output" files

Testing and Validation

- Created multiple neural network “Identification files” for pathfinder, other single lots, and
 - Selected classification files specific to pathfinder lot and compiled together to create neural network
 - Pathfinder lot
 - Representative lots
 - Multi-lot combinations
- Evaluated to down-select to one ‘combined’ identification network file

Testing/Validation Results

Object
Identification Files
for each film

Select and combine to create
targeted neural network
identification method

Program Validation using training
sets (half of identified sample set
is tested against other 50%)

Validation Results Data/squares
(Good = 75%,
ours were typically >97%)

Testing		
	Predicted class Negative	Predicted class Positive
Actual Class Negative	True Negative	False Positive
Actual class Positive	False Negative	True Positive
% Classified Correctly		
Number of Epochs		

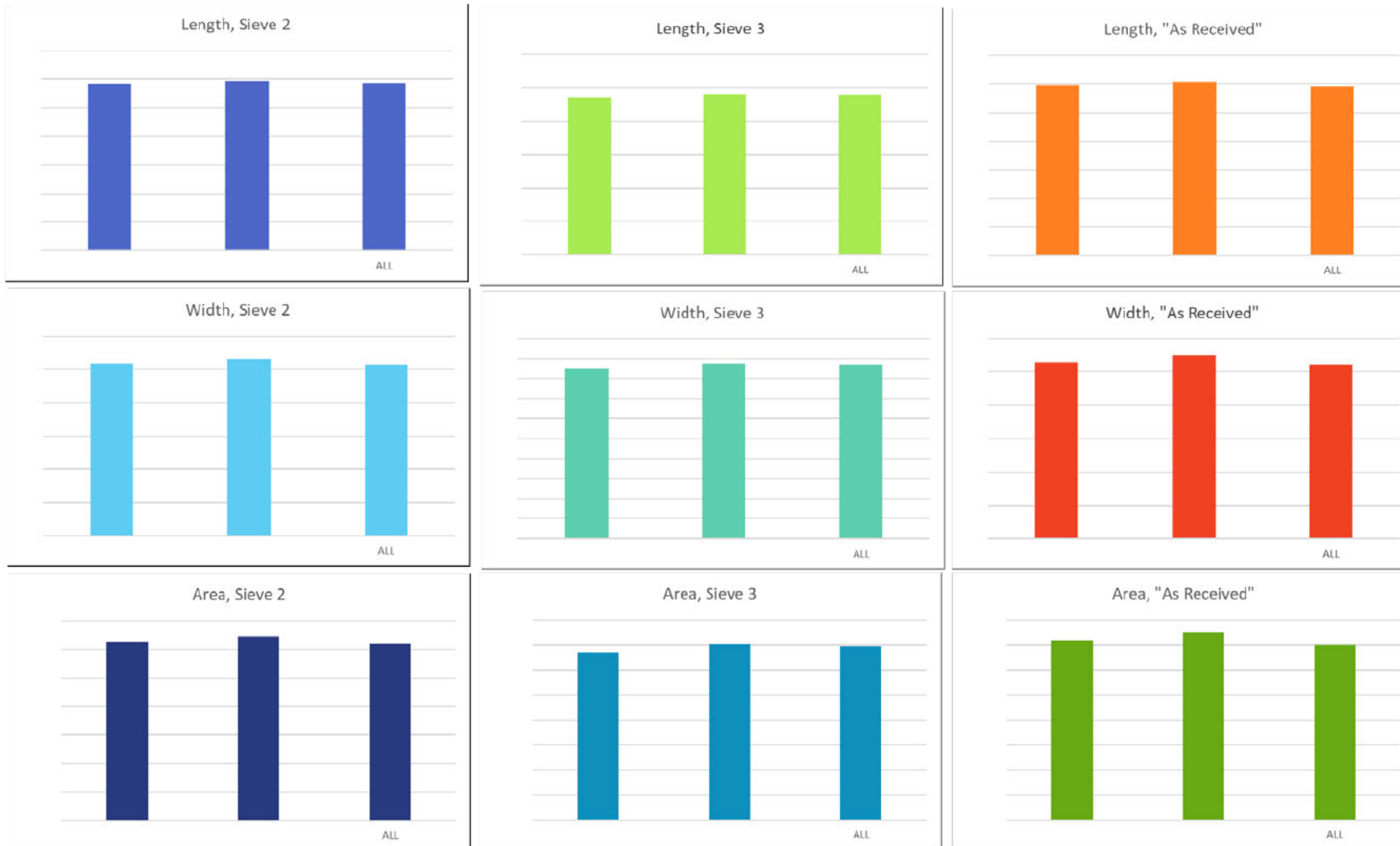
Validation		
	Predicted class Negative	Predicted class Positive
Actual Class Negative	True Negative	False Positive
Actual class Positive	False Negative	True Positive
% Classified Correctly		
Number of Epochs		

Testing		
	Predicted class Negative	Predicted class Positive
Actual Class Negative	1166	28
Actual class Positive	28	1544
% Classified Correctly	98.5	
Number of Epochs	398/400	

Validation		
	Predicted class Negative	Predicted class Positive
Actual Class Negative	1168	42
Actual class Positive	35	1524
% Classified Correctly	97.2	
Number of Epochs	398/400	

Model Analysis

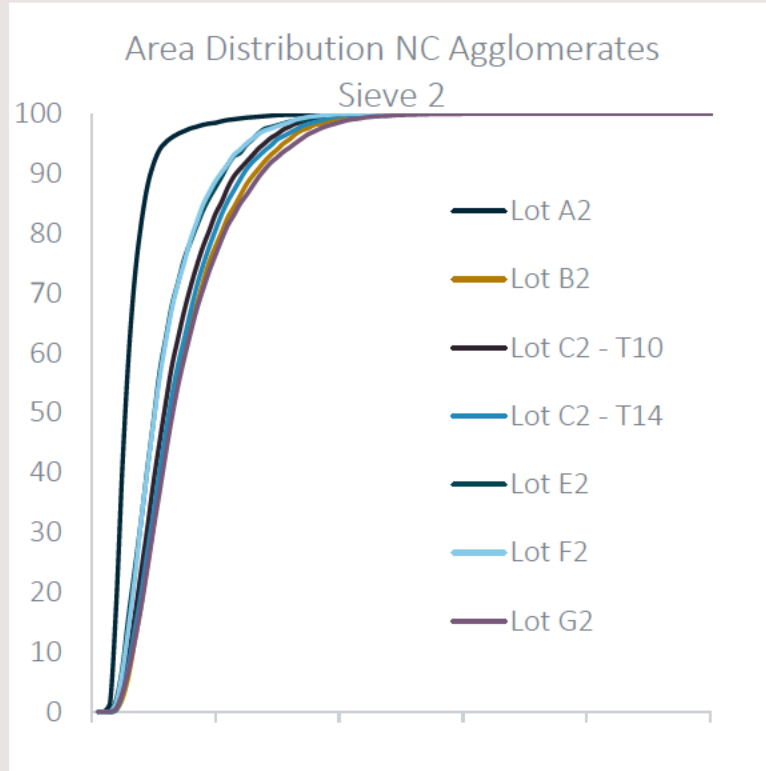
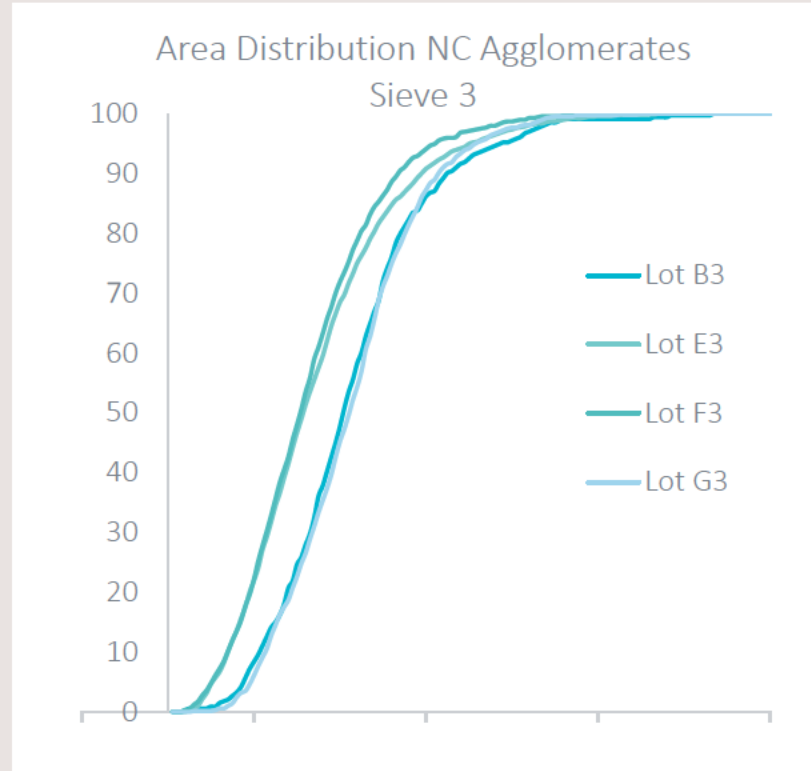
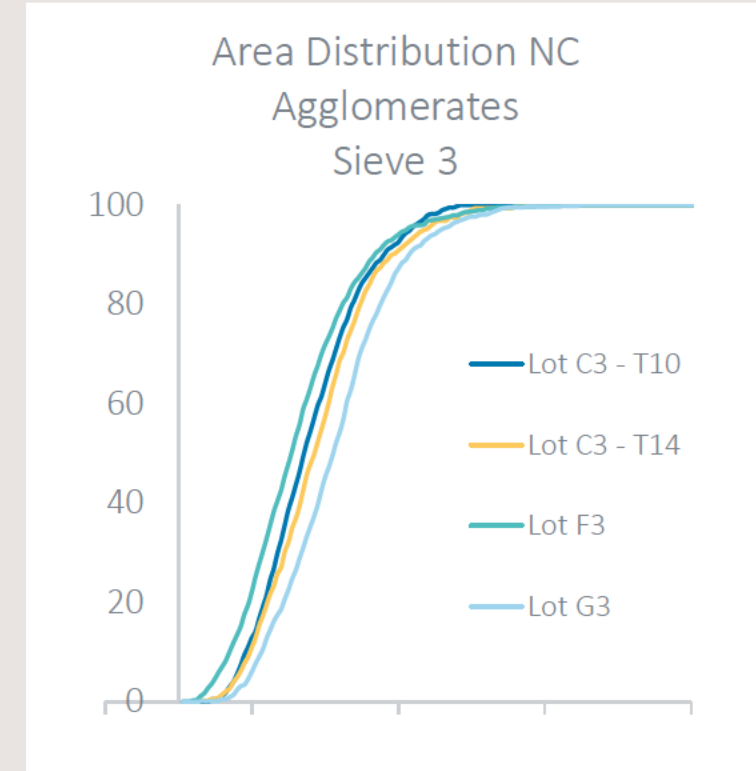
Dimensional Results of Independent Lot vs Single/Multi-Lot Trained



- RSD % vs All/Multi:
0.1 – 0.4%
- RSD% vs Single Lots:
0.1-2.4%

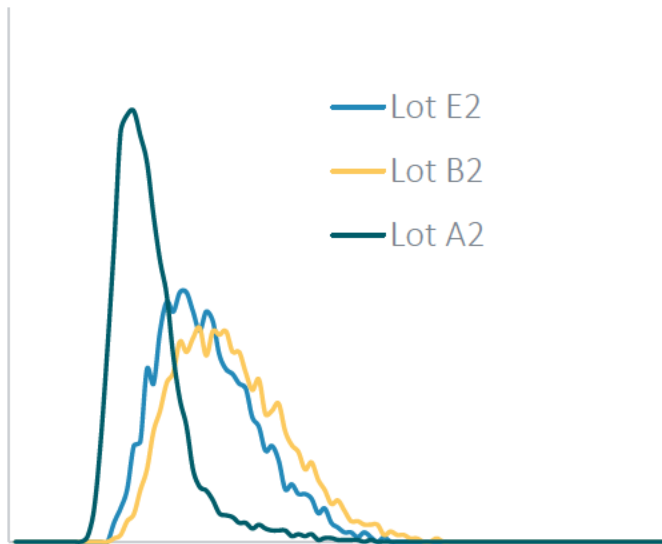
Results

Down-selected Neural Network Model Results Agglomerate Area Distributions

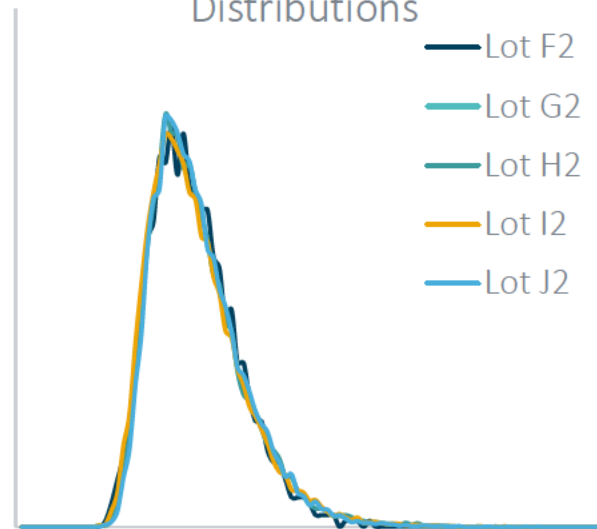


Down-selected Neural Network Model Results Agglomerate Length Distributions

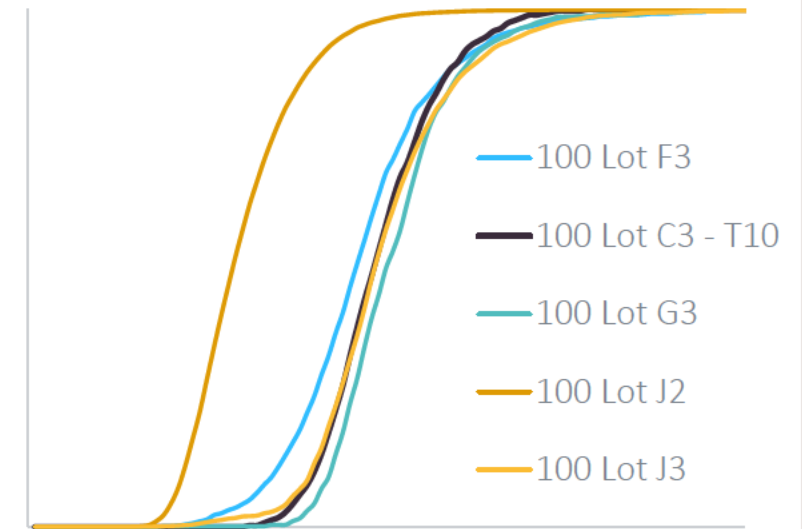
Agglomerate Length Distributions



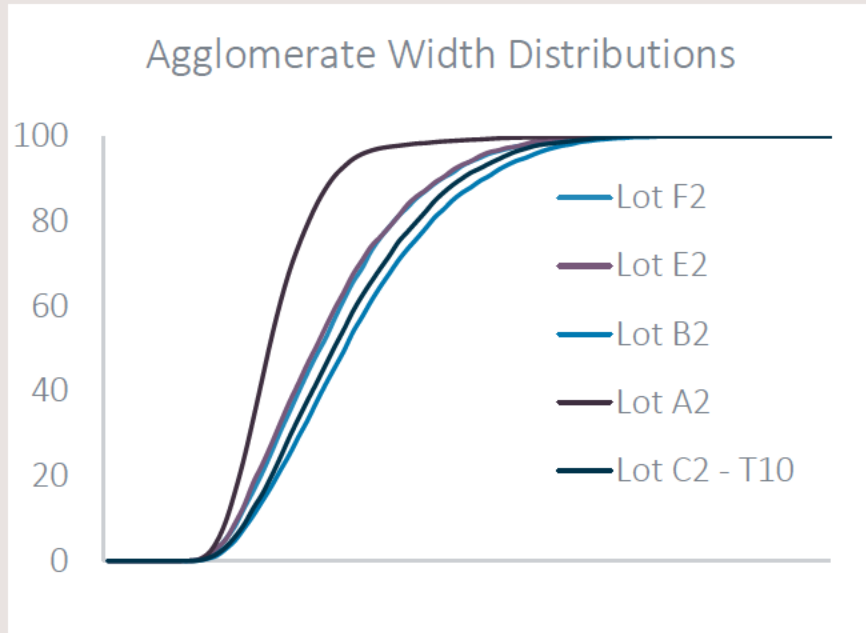
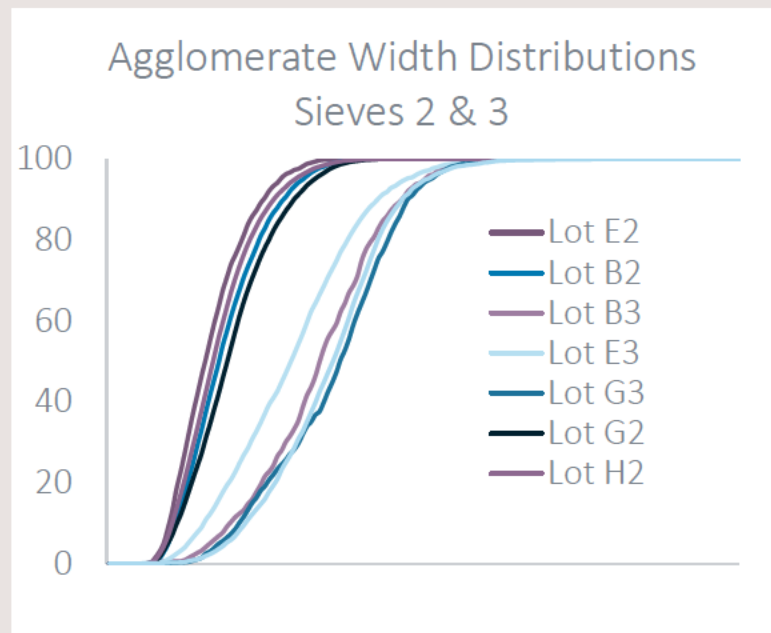
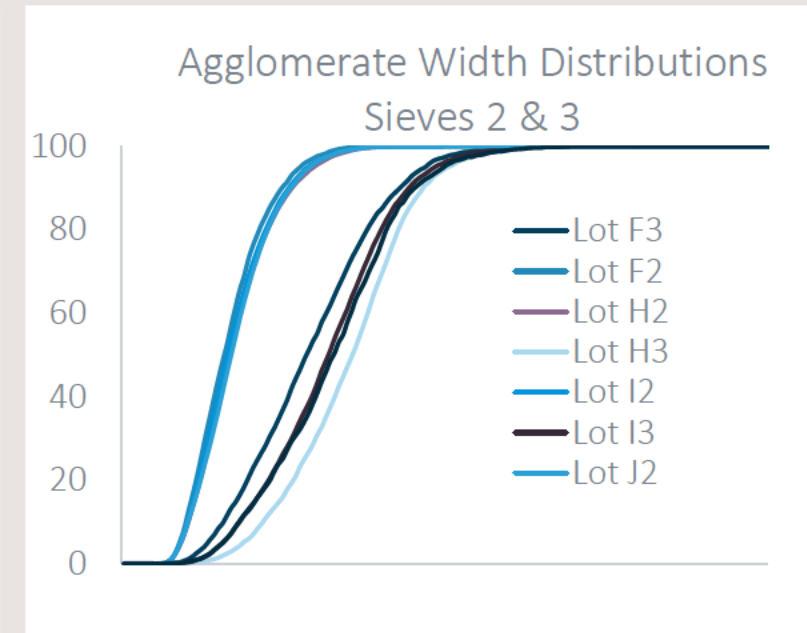
Agglomerate Length
Distributions



Agglomerate Length Distributions



Down-selected Neural Network Model Results Agglomerate Width Distributions



Conclusions

Conclusions and Path Forward

Conclusions

- Able to create a method for identifying, classifying, training, and validating for agglomerates in nitrocellulose
- Data generated from identification allows to better characterize nitrocellulose agglomerates of in-process and final product nitrocellulose
 - Length, width, area – averages and distributions
- Any sample of interest – ‘as-is’, sieved, grab samples throughout the process
- Ability to track changes within processes, R&D programs, process stability over time (retained samples and ‘films’), process optimization and continuous improvement

Path Forward:

- Additional NC types/grades to generate new networks via this classification, training, validation and testing methodology
- How do NC type/grade/cellulose source/processes affect/effect agglomerate dimensional properties?
 - Utilize data for continuous improvement and research and development programs (propellant and NC)

Acknowledgements

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BAE Systems Operations Team

BAE Systems Quality Lab Team

Thank you for your attention!