Defence Ordnance Safety Group – Science & Technology

Qualification of Cotton Linters UK Joint Industry & MoD Approach

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Presentation Overview

- Sources of Cellulose for propellant manufacture
- Linter production
- Why is understanding NC so important?
- Joint UK MoD and Industry approach to characterise and benchmark alternative suppliers
- Changes in cellulose supplier source requiring re-qualification of existing propellant formulations
- Highlighting the successful re-qualification of various propellant formulations in UK service

- Questions and short discussion period open to delegates

Source of Cellulose

- Multiple sources of cellulose are available however Propellant manufacture tends towards two primary sources:

- Cotton Linters:
- Cotton Linters a commodity material produced across Globe and traded on World Markets
  - European source of choice
  - High grade material ~94% cellulose content
  - Physical form can be as a sheet or flock

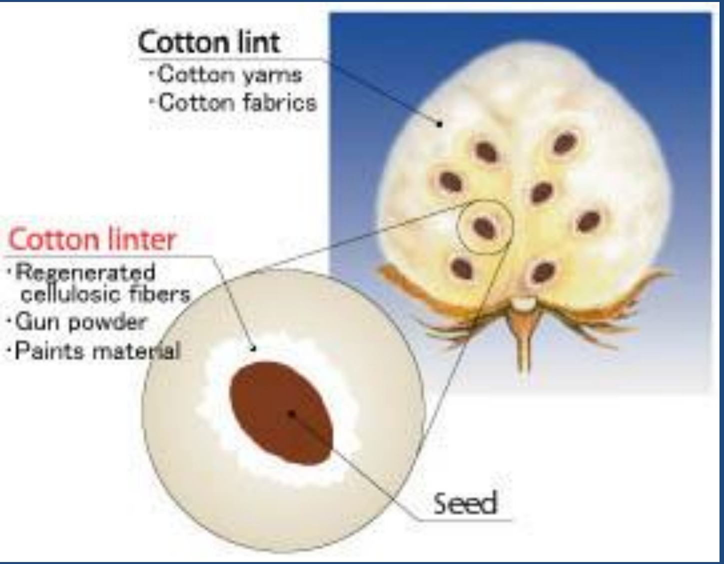
- Wood Pulp:
  - Predominantly used in North America
  - Low grade source ~50% cellulose content

Source of Cellulose

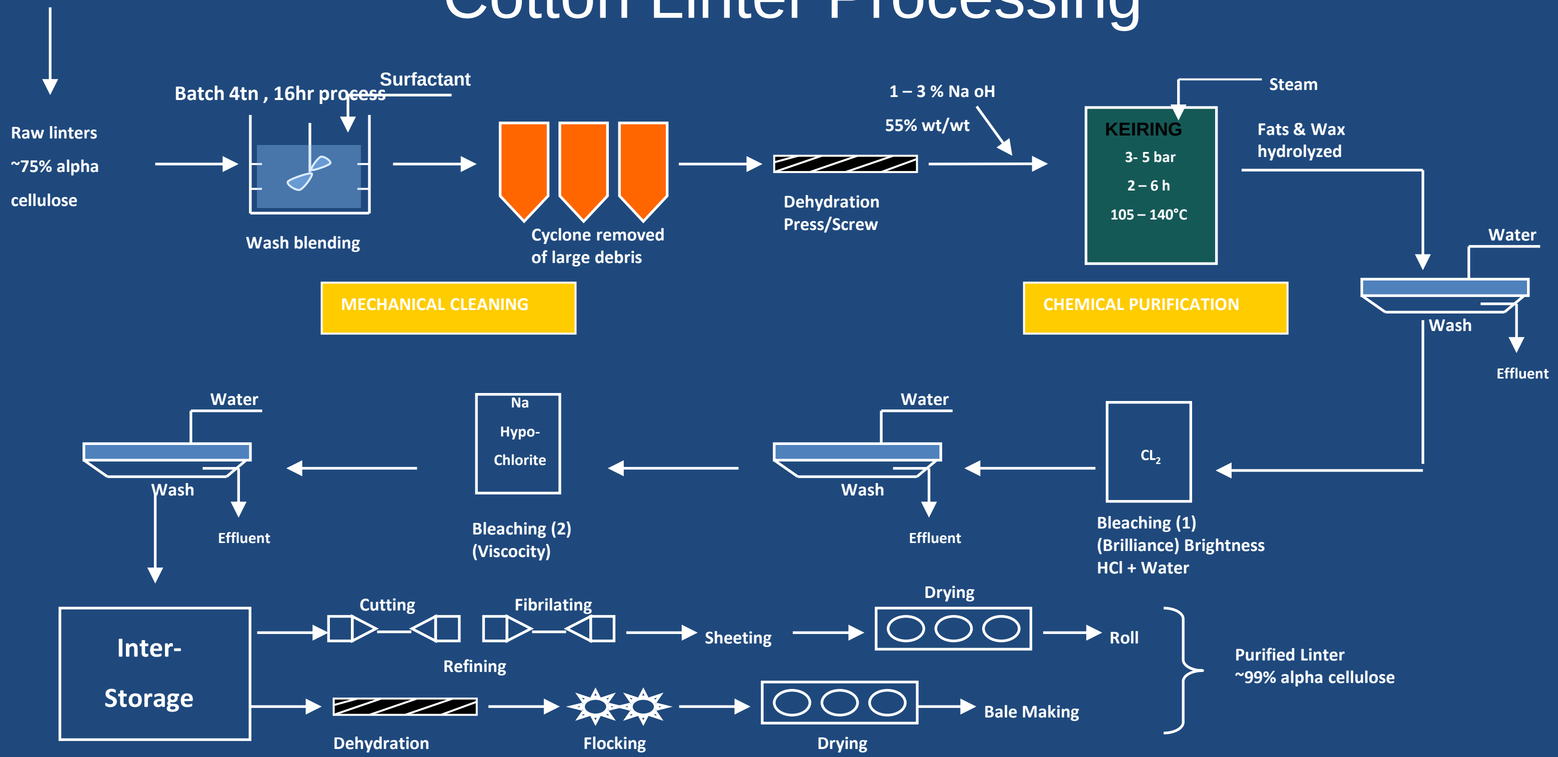
- Diagram shows the cotton plant and the fluffy white exterior known as the lint is what is used in the textile business

- The material surrounding the see is the Cotton Linter

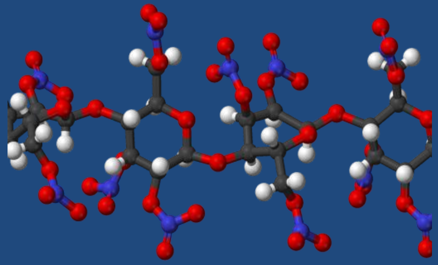
- Natural product with variation in composition



Cotton Linter Processing



Why is Understanding Nitrocellulose so Important?



- Ballistic properties (burning rate, temperature coefficient)
- Chemical stability and service life
- Design choice – chemical energy available can be 'tuned'
- Provides mechanical strength and necessary resilience (Handling & firing over temperature range)
- Bonding – can bond DB RM propellants to the motor casing.

- **Provides confidence in final Propellant properties**

Joint UK MoD & Industry Approach to Qualification

UK MoD Prime Requirements:

- Continuity of Supply of Nitrocellulose Propellants
- Maintain Consistency of Propellant Quality
- Consistent Performance Levels
- Safety Characteristics
- Long Service Life

- Propellant Supply Chain therefore needs to be as ‘stable’ as possible.

Background to Changes in Cellulose Source

- Due to requirement for ammunition UK MOD had no option but to undertake a number of changes in cellulose / nitrocellulose sources

- Process of manufacturing NC then conversion to Paste and then into Propellant

- Followed by the Material Qualification and Type Qualification (High cost and time)

- Long Re-Qualification programmes increase the risk of further material changes during development

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- **UK Cellulose Supply Background since 1990**
  - Holden Vale Linters, Dumfries NC
  - Holden Vale Linters, UK Manufactured NC
  - NATO Source 1 Linters, UK Manufactured NC
  - NATO Source 1 Linters, Manufacturer A NC
  - NATO Source 2 Linters, Manufacturer A NC
  - Non-NATO Source 1 Linter, Manufacturer A NC
  - Non-NATO Source 2 Linter, Manufacturer A NC

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- **2001: Initial Process Changes:**

- NATO Source 1 (UK Manufacture) to NATO Source 1 (Manufacturer A)
- Comparable manufacturing results using NATO Source 1 linter feedstock reported.

Property	Specification		NATO Source 1 UK Manufacture	NATO Source 1 Manufacturer A
	Min	Max		
Settling test (cm)		95	90	87
Nitrogen (%)	12.35	12.75	12.68	12.68
Mineral matter (%)		0.8	0.43	0.47
Alkalinity (%)	0.2	0.5	0.54	0.40
E/A Solubility (%)	95		99.4	99.4
Acetone Insol (%)		0.50	0.15	<0.10
Sulphate (%)		0.10	<0.05	<0.01
B/J test (mgN/g)		1.25	0.84	1.06
Abel HT (mins)	10		11	15
Viscosity (p)	5	10	5.2	7.7

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- NC/NG Paste was converted in to propellant by Manufacturer C

Propellant underwent the following analysis.

- Mechanical Properties
- Chemical Stability Testing
- Small Scale Hazard Testing

- Successful change programme for UK MoD and Industry.
- Propellant formally UK National Authority Qualified.

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- **2009: First Material Changes:**

- NATO Source 1 (Manufacturer A) to NATO Source 2 (Manufacturer A) following the withdrawal of NATO Source 1 from the market
- NATO Source 2: Several batches of Manufacturer A NC converted to NC/NG paste by Manufacturer B

NC Type	Molecular weight	Molecular No	Viscosity (Poise)
NATO Source 2	454946	3.4	14.0
NATO Source 1	473796	4.5	17.0

Paste Type	BAM Impact LIE (Kg.cm)	BAM Friction LL (N)
A	>320	157
B	>320	157
C	>320	158

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- NC/NG Paste converted in to three types of propellant by Manufacturer C

Propellant underwent the following analysis:

- Mechanical Properties Analysis
- Chemical Stability Testing
- Small Scale Hazard Testing

- Propellants produced from NC/NG pastes incorporating NC produced from NATO Source 2 linters exhibited acceptable chemical, ballistic and rheological properties.

- A further successful change programme reported for UK MoD and Industry.

- Propellant formally UK National Authority Qualified.

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- **2009: Further Material Changes**

- NATO Source 2 (Manufacturer A) to **Non-NATO** Source 1 linters (Manufacturer A)
- Non-NATO Source 1: Batch of Manufacturer A NC converted to NC/NG paste by Manufacturer B
- An increased level of NC analysis was conducted:

Requirement	Min	Max	Result
Nitrogen (%)	12.1	12.3	12.24
Mineral matter (%)		0.8	0.3
Settling test (cm)		75	63
Alkalinity as $CaCO_3$ % m/m	0.2	0.4	0.2
Insoluble in acetone % m/m		0.5	0.02
Ether / alcohol solubility % m/m		95	99.7
Sulfate as H2SO4 % m/m		0.1	0.03
Stability at 132°C mg/g		1.25	1.13
Heat test 77°C minutes	10		14-16

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- Mw Comparison reported for the two sources of cellulose:

NC Type	Molecular weight	Polydispersity Mw/Mn
NATO Source 2	441214	4.01
Non-NATO Source 1	431339	4.01

- NC/NG Paste converted in to two types of propellant by Manufacturer C

Propellant underwent the following analysis:

- Mechanical Properties Analysis
- Chemical Stability Testing
- Small Scale Hazard Testing

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- **Propellant test results fully met specification.**
- Propellants produced from NC/NG pastes incorporating NC produced from Non-NATO Source 1 linters exhibited acceptable chemical, ballistic, rheological and explosive hazard properties.
- **Successful change programme for UK MoD and Industry.**
- Propellant formally UK National Authority Qualified.

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- **2011: Further Material Changes:**
- Qualification work covering a range of cotton linters from NATO Source 1 to Non-NATO Source 2 processed by Manufacturer A
- Manufacturer A NC converted to NC/NG Paste by Manufacturer B and then further processed to various types of propellant by two different European manufacturers.
- **Comprehensive series of reports issues by each manufacture covering:**
  - **Cotton Linter Analysis**
  - **NC Analysis**
  - **Propellant Analysis**

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Analytical test		NATO Source 1a	NATO Source 1b	NATO Source 2	Non-NATO Source	Non-NATO Source
Alpha-Cellulose	min %	99	99	98.5	98.4- 99.7	98.3 – 99.5
Viscosity	centipoise	40-60	70-110	43-53	-	-
Viscosity ISO 5351	ml/g	-	-	737	753	765
DCM Extract	max %	0.3	0.1	0.025	0.06 – 0.14	0.10 – 0.13
Ash	max %	0.25	0.25	0.1	0.06 - 0.13	0.06 – 0.13
Chloride	max ppm	60	60	60	10 - 40	10 - 20
Solubility L 7.14 NaOH Sol^{n}	max %	3.5	3.5	3.5	1.4 – 2.4	1.4 – 2.9
Moisture	%	4.5 – 8.5	4.5 – 7.5	5 – 8	4.7 – 7.1	5.5 – 6.3
Degree of Polymerisation		-	-	1090	1121 - 1179	1032 - 1156

Bale Cotton linter cellulose analysis results

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- NC Analysis data across range of Cotton Linter Stock

Components	Typical NC Spec	NATO Source 1	NATO Source 2	Non-NATO Source	Non-NATO Source
% Nitrogen	12.45-12.75	12.54 – 12.66	12.64	12.66	12.66
Viscosity	55-85	66-79	78.5	78	78
Stability B& J Test mg NO/g	1.25 max	0.99 -1.2	1.23	1.18	1.17
Abel Heat Test @ 77 °C (mins)	>10	12 -21	18	19-20	13-14
Ether Alcohol Solubility %	95 min	98.8 -99.8	99.3	99.4	98
Molecular Weight (Mw) X1000	-	835*	875* 632**	677**	640**
Polydispersity Index (Mw/Mn)	-	4.08*	3.96* 3.34**	3.19**	3.38**

Note: Mw and Polydispersity Index results:* from 2007 are comparable while the results** from 2010 are comparable.
Differences are due to significant changes in analytical techniques following the the STANAG 4178 (Edition 2) round robin test organized by UK.

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- **Analysis of Cotton Linters**: No significant difference in the chemical properties.
- **Analysis of the Nitrocellulose:** Complied with the requirements of the specification
- **Chemical Analysis of the Propellants:** Manufactured propellants fully met specification
- **Physical Properties:**
- Dimensional analyses:
- Density and Tensile strength:
- Closed Vessel testing:

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- **Stability Testing:** Met criteria for a 5 year shelf life under normal magazine storage conditions.

- **Heat Flow Calorimetry:** 10 year life for all propellants tested at 80ºC for 10.6 days

- **EMTAP Sensitiveness Testing:** All propellants tested demonstrated that the new supply of Non NATO cotton linters did not significantly change the sensitivity of the propellants.

- **Gun Firings:** Trial results indicated acceptable ballistic levels relative to control samples

- All propellants made with Nitrocellulose from Non-NATO sourced linters were not significantly different from propellants made with NATO Source Linters, and were recommended as being safe and suitable for UK service use.

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- **Conclusions**

- Various Propellant types have proven tolerant to linter source changes.

- Lower Performance, Cartridge load propellants have also shown tolerance to source changes.

- MoD overview of the reported data from the Linter Stock and Process Changes since 2001 demonstrate that throughout:

  - Nitrocellulose produced has complied with specification.

  - Propellants manufactured have fully complied with Chemical and Physical Properties and met Manufacturers Specifications.

  - Propellants have fully met the criteria for UK National Authority Qualification.

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- **Further Conclusions**

- No real predictive capability to understand how Cellulose source / type will effect Propellant properties

- While these programmes were entirely successful, based largely on a trial and error approach

- Better understanding of cellulose and NC properties will allow for a more targeted and scientific approach to material supplier change in the future

- All work now successfully completed leading to the UK Qualification of a number of propellant formulations

- Strategy worked with few problems due to the expert knowledge of the manufacturers

- But, can we take this approach again? Given it may take 5 years work to take to completion

What's the Alternative to Understanding the Science?

Full Re-Qualification

OR?

We propose a much more resilient science based approach, to de-risk the range of options such that at any point we can switch proven supplier route within 18 months. Propellant level qualification. De risk scientifically

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Thank you for your attention

Any questions?