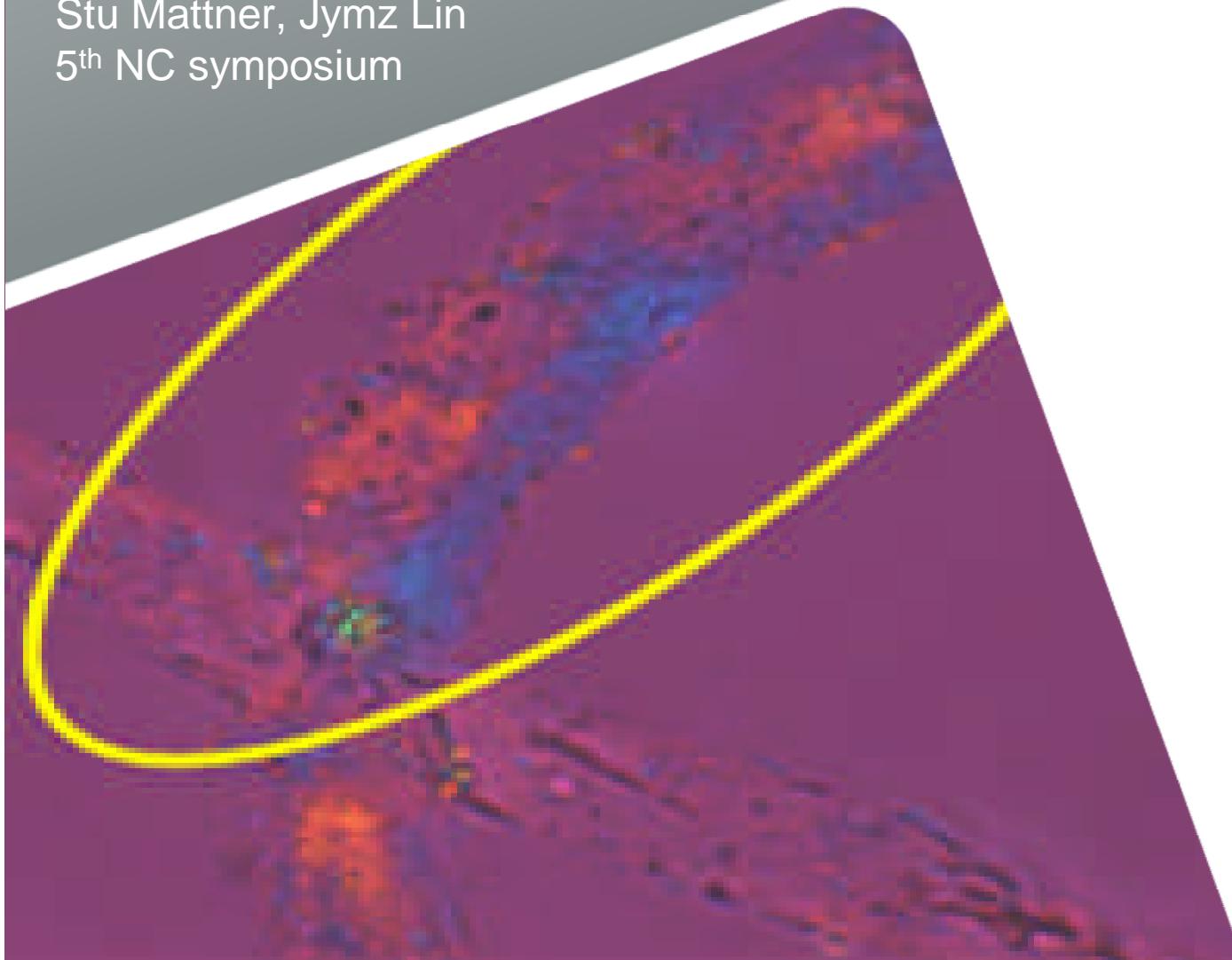


Nitrocellulose: EOE MSc Projects

PP Gill, N Mai, J Bellerby, M Monir

Stu Mattner, Jymz Lin

5th NC symposium



Overview

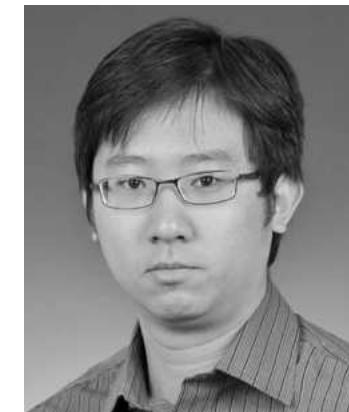
- Cranfield Uni: Explosive Ordnance Engineering MSc
<http://www.cranfield.ac.uk/students/courses/page1409.html>
- NC Mechanical properties (*FL Lt Stuart Mattner*)
 - Thin Film technology
 - DMA, Effect of plasticiser/Mw/Stabiliser
- NC Nitrogen Distribution (*Jymz Lin Zhi Yuan*)
 - Polarised Light Microscopy
 - C¹³ NMR

2011 EOE MSc Project Students

- **NC Mechanical properties**
- FL Lt Stuart Mattner
- Royal Australian Air Force



- **NC Nitrogen Distribution**
- Jymz Lin Zhi Yuan
- Defence Science & Technology Agency, SINGAPORE



Previously.....

NC Thin Films

2007 NC symposium

Experimental

Preparation of NC films (12 % N)

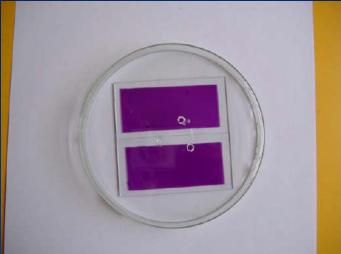
- Preparation of solution
 - Dye 2% *w/w* of NC
 - DOP 5% *w/w* of NC
- Casting films on glass slides

Drying

- Ambient temperature
- Under vacuum

Aging

- Aged at 40 °C, 51 °C, 60 °C, 70 °C



Determination of the Activation Energy of Nitrocellulose Decomposition from UV-visible Spectroscopy of Thin Films

By
M Moniruzzaman
&
J M Bellerby*

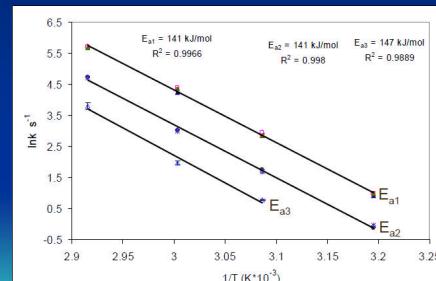
Department of Materials and Applied Science,
Cranfield University, Defence Academy of the
UK, DCMT, Shrivenham, Swindon, SN6 8LA, UK

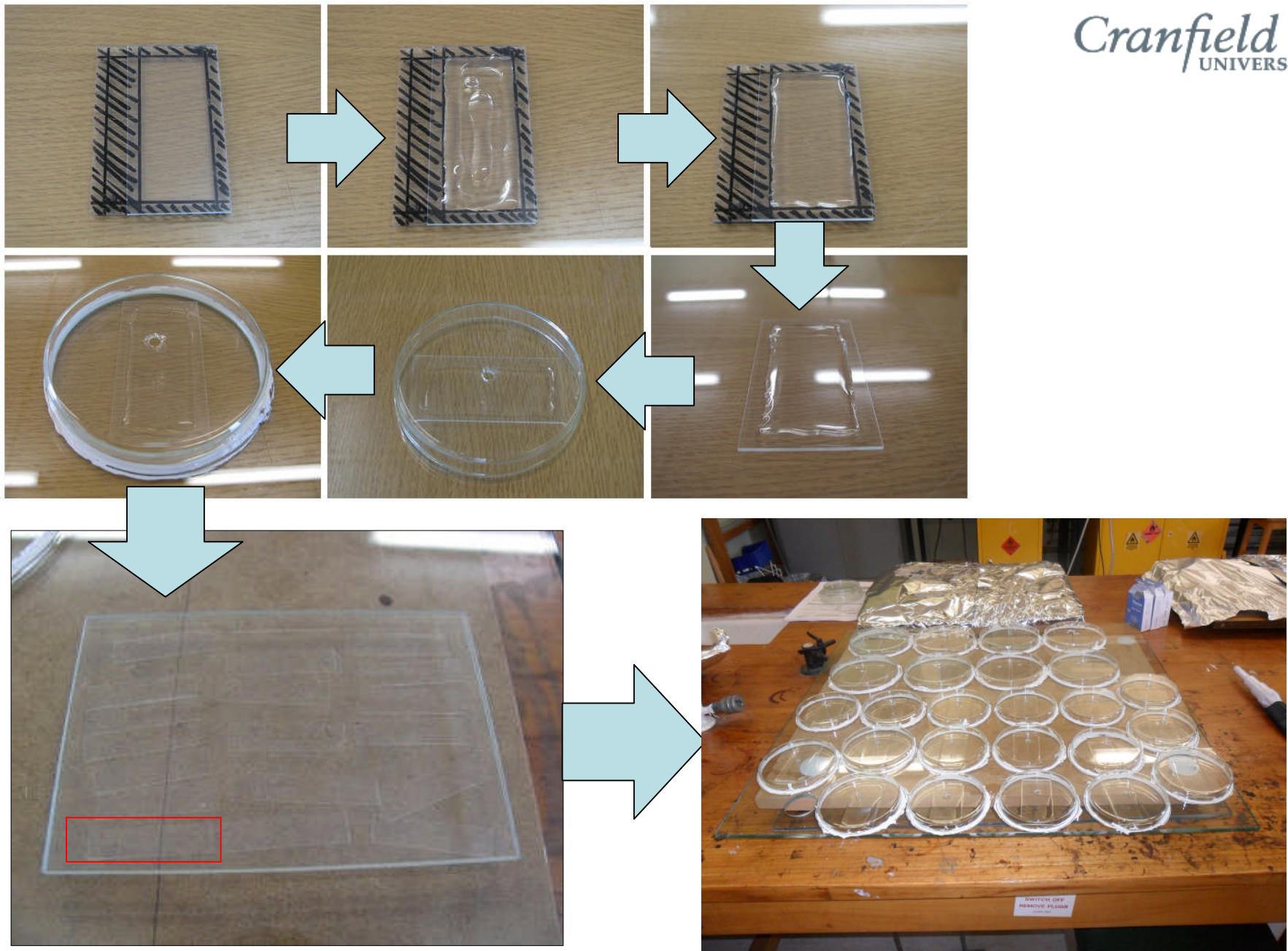


E_a for NC decomposition

- Activation energy for the -ONO₂ decomposition
 - 141 kJmol⁻¹ - position 3
 - 141 kJmol⁻¹ - position 2
 - 147 kJmol⁻¹ - position 6

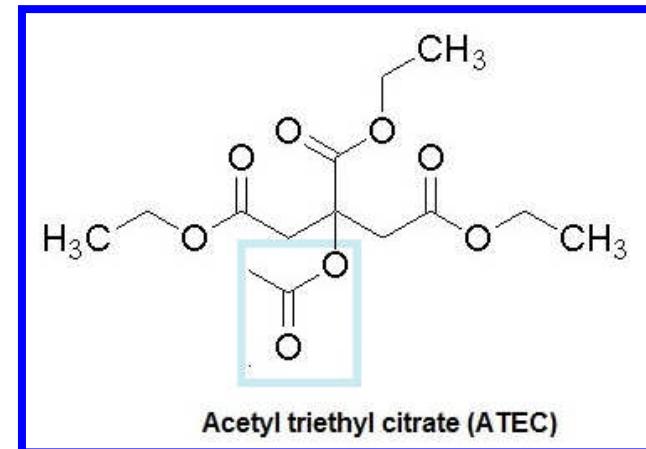
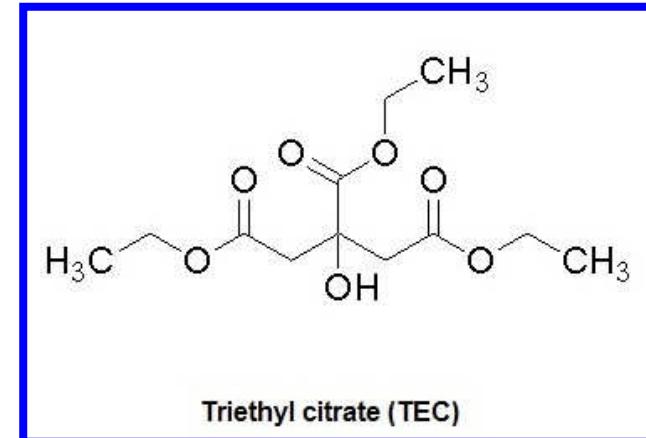
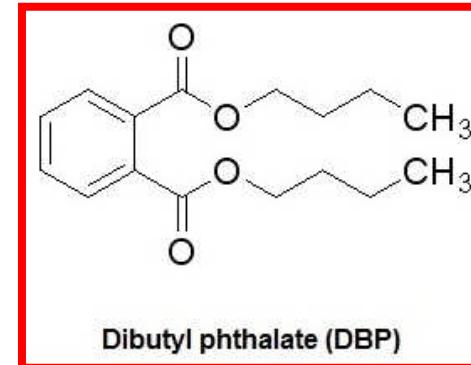
Arrhenius plots





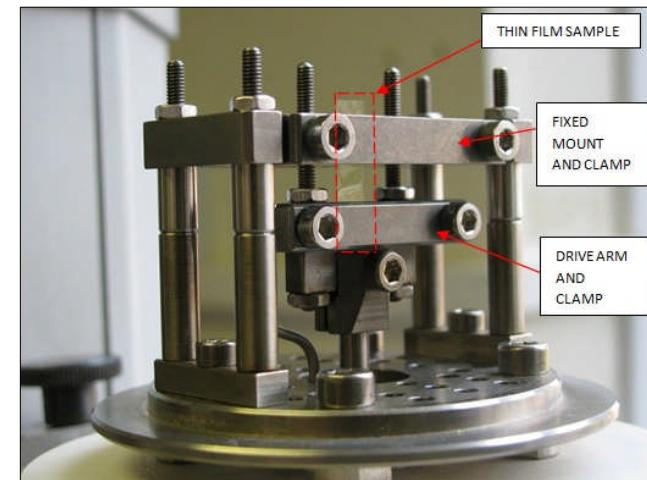
Plasticisers & Ageing regime

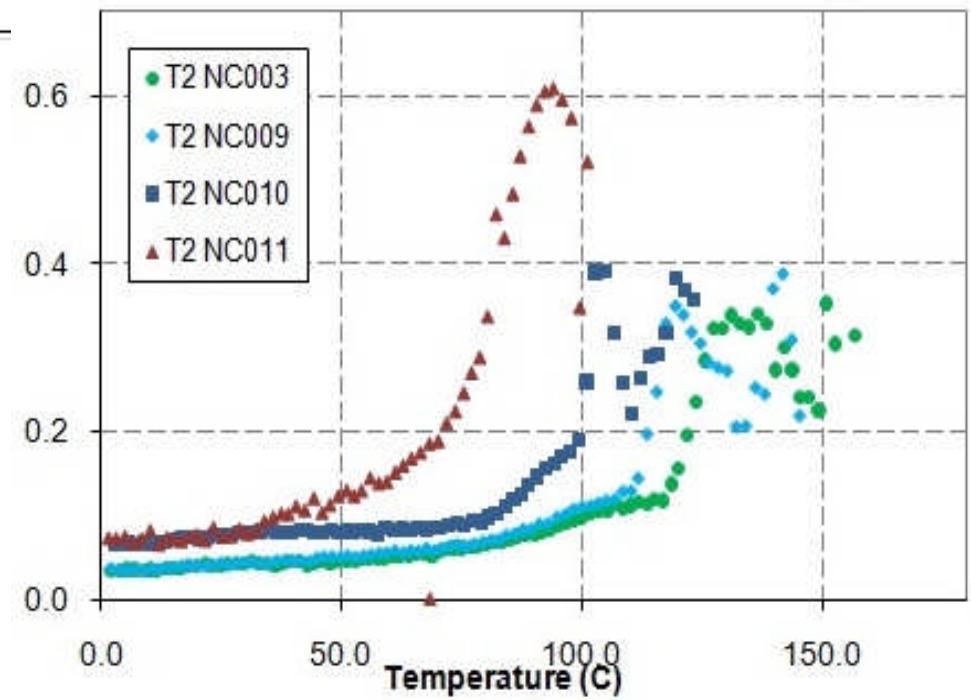
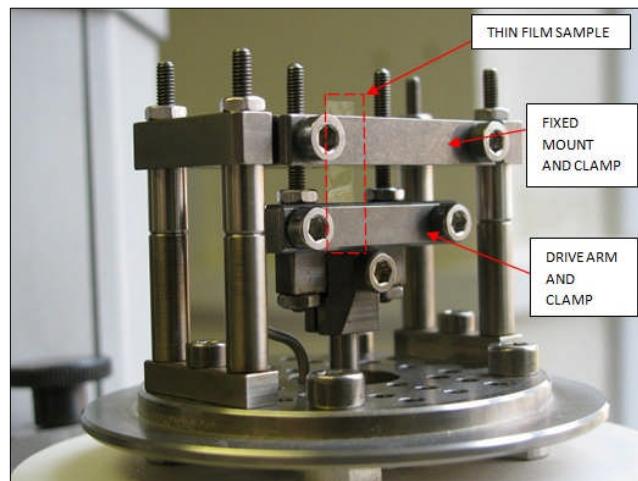
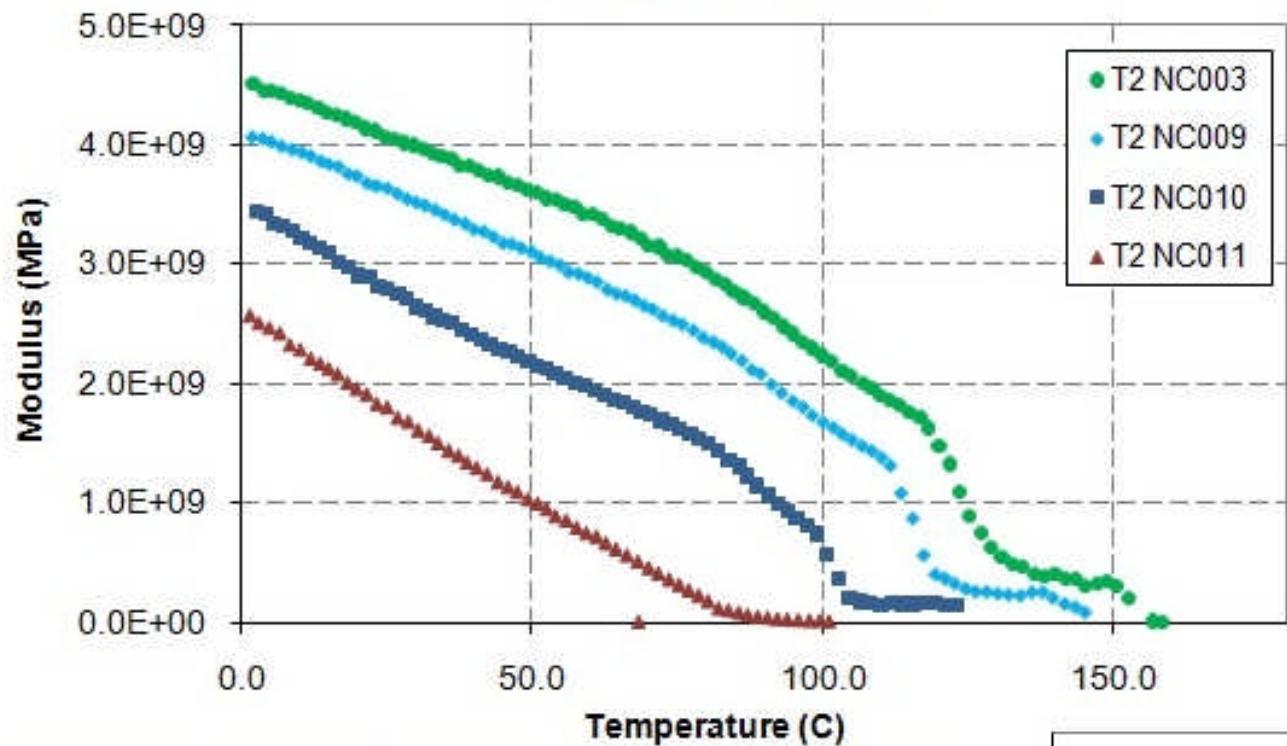
- Samples sealed in polymer-coated aluminium foil bags (AOP-48 Ed 2)
- Ageing timeframes:
 - 60°C, 70°C, 80°C
 - Up to 18 days



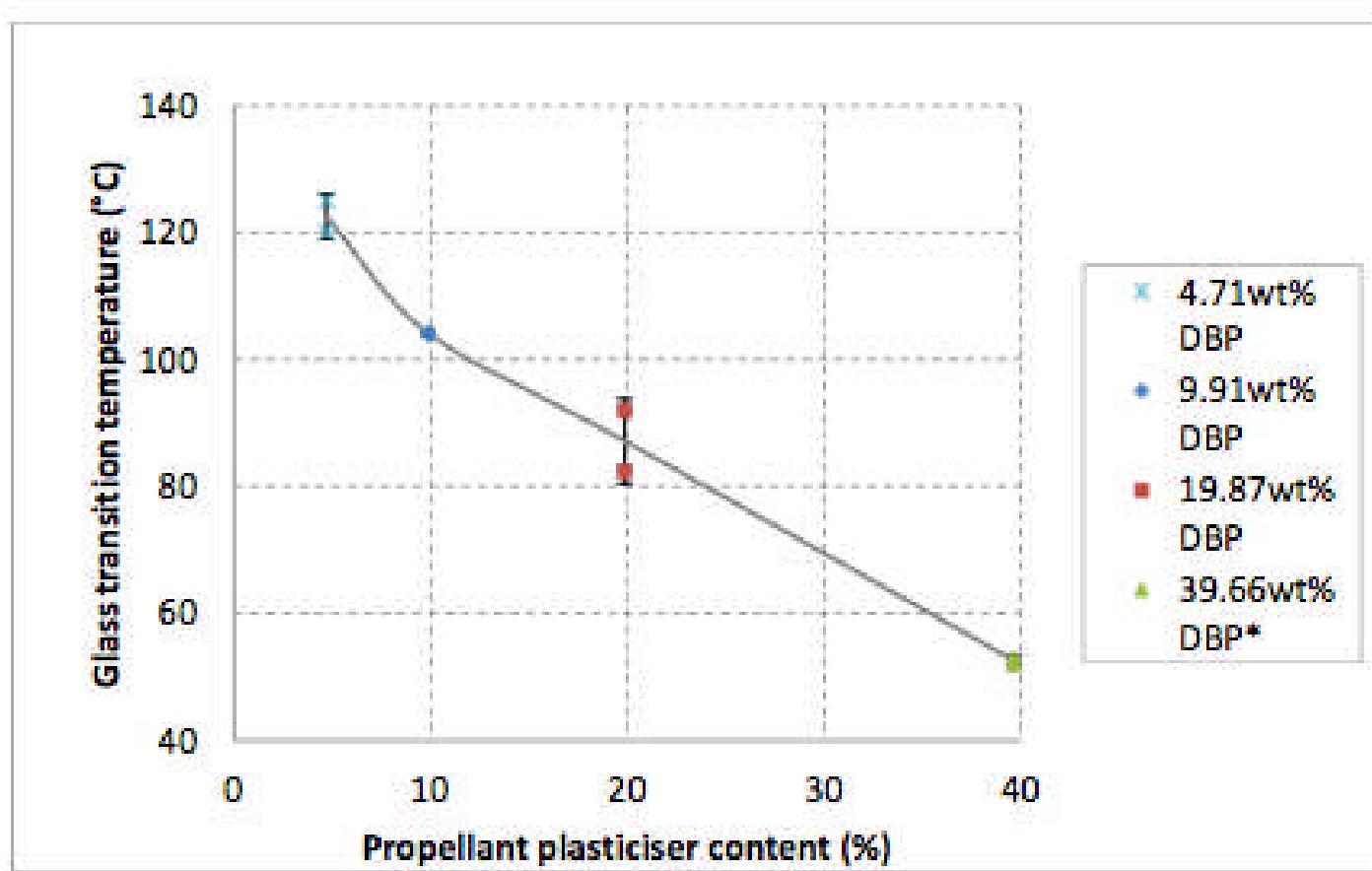
Dynamic Mechanical Analysis

- Tension mode – thin films
- Measures
 - storage (elastic) modulus (E')
 - loss modulus (E'')
 - $\tan \delta$ (the ratio of E' to E'')
- Glass transition region / temperature (T_g)





Tg Vs Plasticiser content



Tg Vs Mw

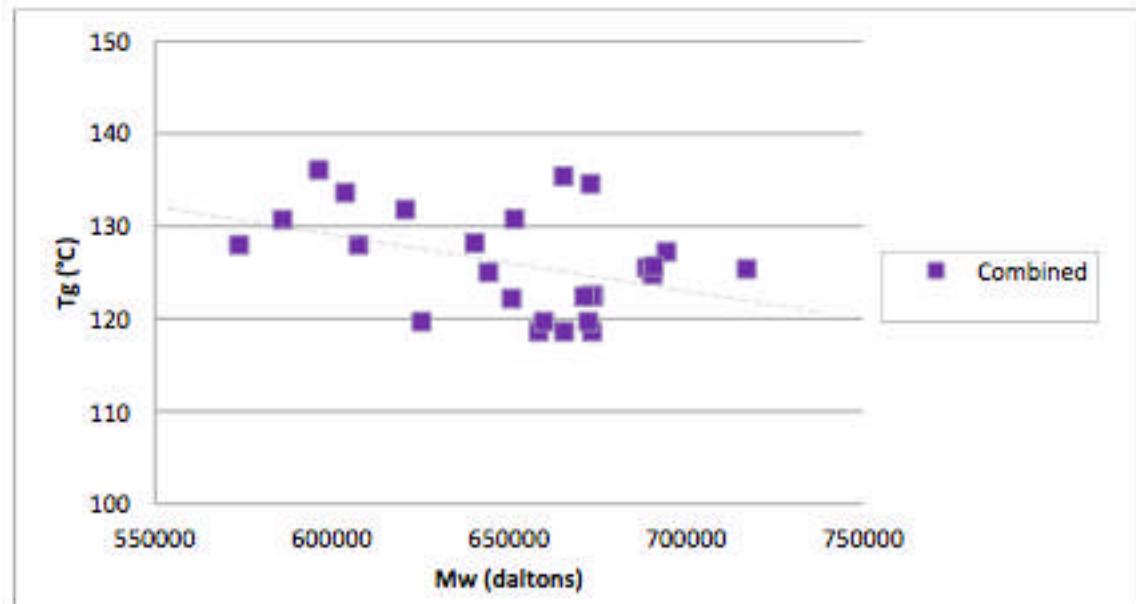


Figure 5.37: Combined T_g against M_w for plasticised samples
(4.71wt% DBP, 4.72wt% TEC, 4.70wt% ATEC)

Tg Vs Stabiliser Content

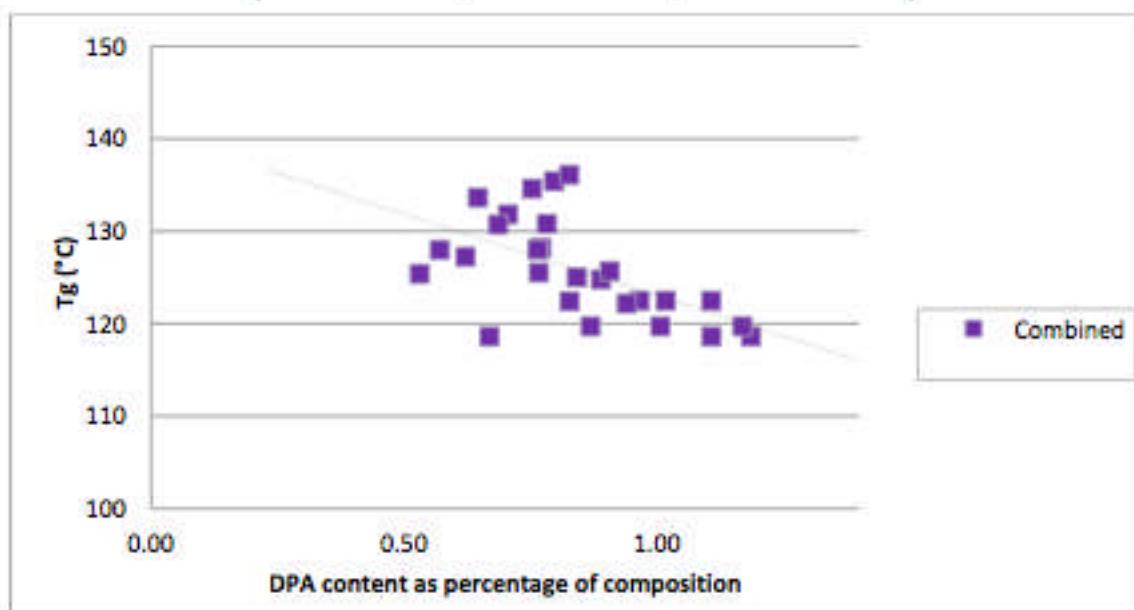


Figure 5.33: Combined T_g against DPA concentration for plasticised samples
(4.71wt% DBP, 4.72wt% TEC, 4.70wt% ATEC)

Conclusions

NC Mechanical properties

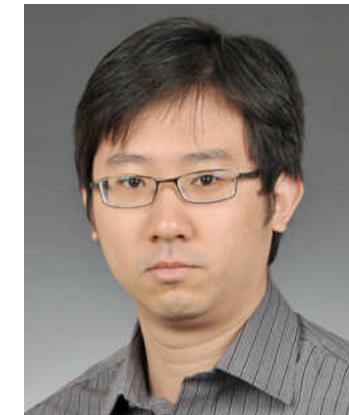
- DMA:
 - Tg identified
 - Shift in Tg observed: increases with plasticiser content & ageing
- Data Scatter prevents comment upon effectiveness of novel plasticisers
- Future work: larger films, more replicates

2011 EOE MSc Project Students

- NC Mechanical properties
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- **NC Nitrogen Distribution**
- Jymz Lin Zhi Yuan
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Previously.....

NC Nitrogen Distribution

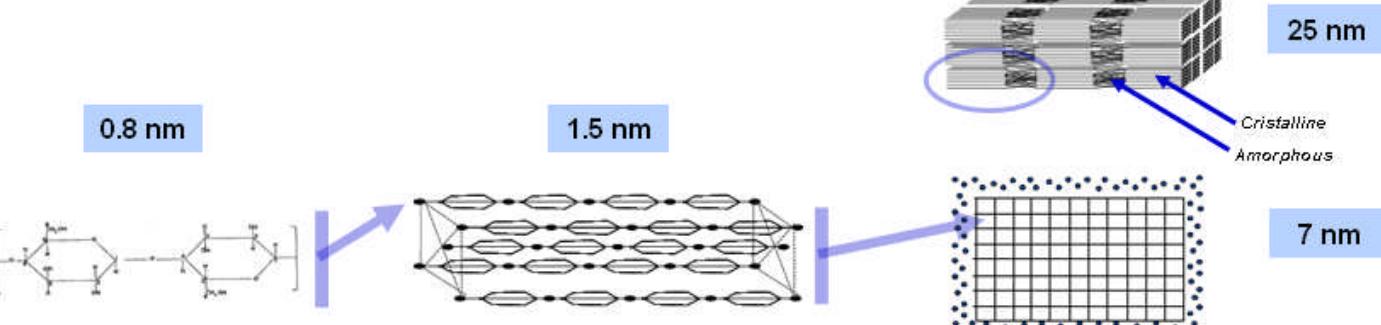
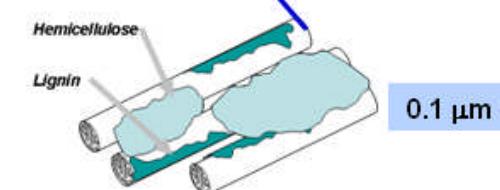
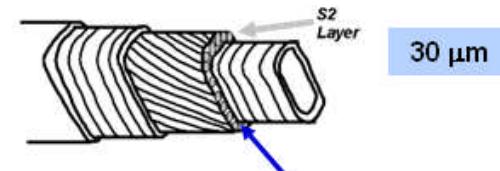
ref: 2010 – Mario Paquet

Quality of nitration: Nitrogen variations



Nitrocellulose nitration challenge:

- Nitrogen average
- Nitrogen distribution within an agglomerate
- Nitrogen distribution between agglomerates
- Nitrogen distribution within a fiber
- Nitrogen distribution between fibers
- Nitrogen distribution on the molecule



Methods

- Distribution between fibres
 - Distribution within fibres
 - Distribution on molecular chains
-
- The diagram illustrates the classification of methods. Three main categories are listed on the left: 'Distribution between fibres', 'Distribution within fibres', and 'Distribution on molecular chains'. To the right of these, two specific methods are listed: 'Polarised Light Microscopy' and 'Nuclear Magnetic Resonance Spectroscopy'. A large brace on the right side groups the 'Distribution within fibres' and 'Distribution on molecular chains' items together, indicating they belong to the same broader category of microscopic and spectroscopic techniques.
- Polarised
Light
Microscopy
- Nuclear
Magnetic
Resonance
Spectroscopy

Polarised Light Microscopy

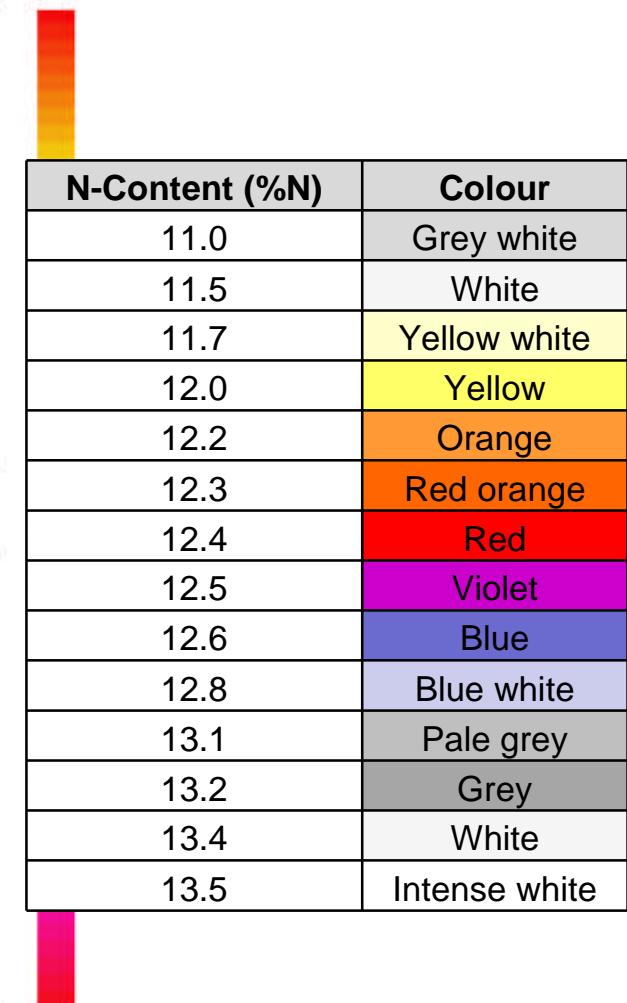
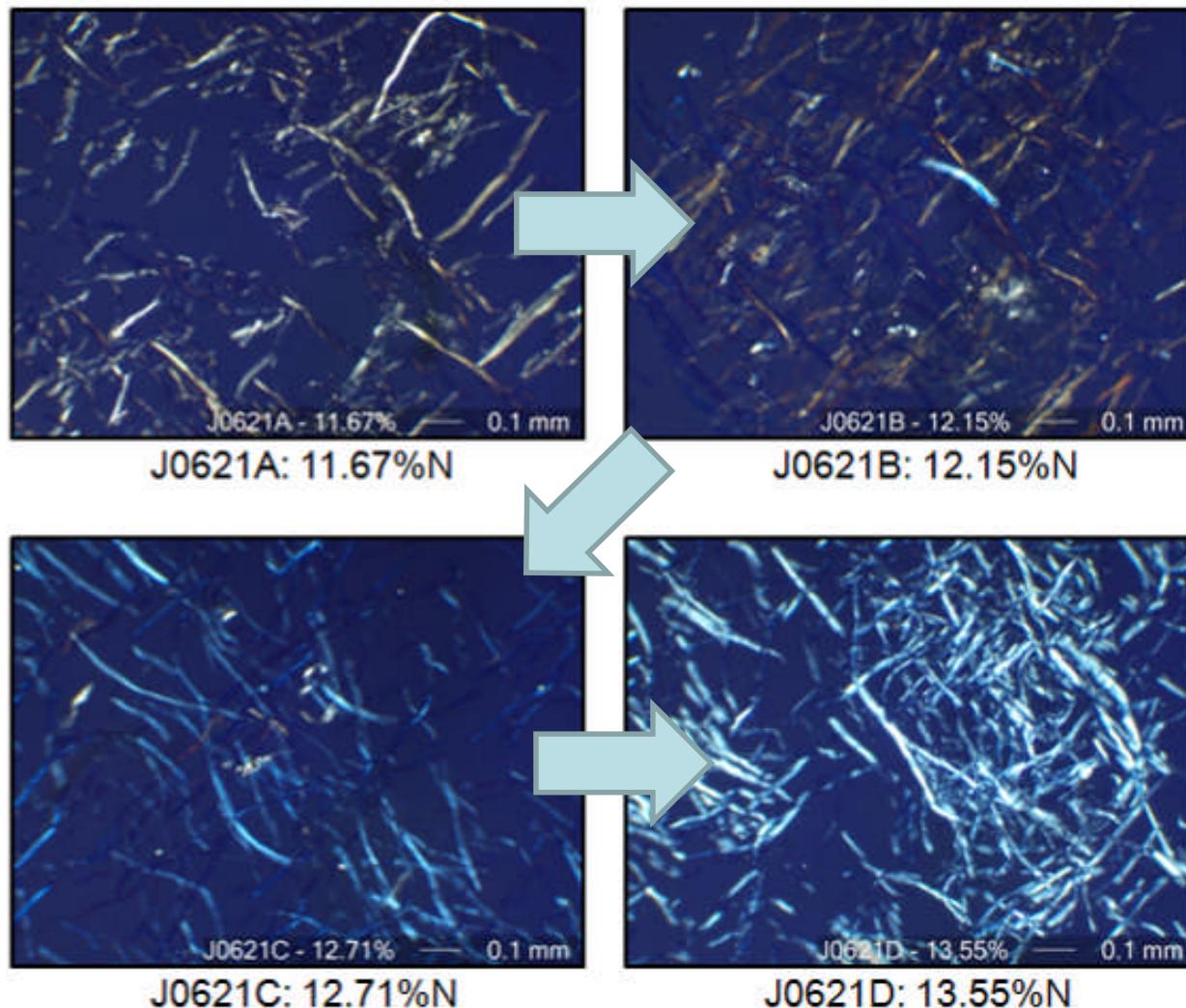
N-Content (%N)	Colour
11.0	Grey white
11.5	White
11.7	Yellow white
12.0	Yellow
12.2	Orange
12.3	Red orange
12.4	Red
12.5	Violet
12.6	Blue
12.8	Blue white
13.1	Pale grey
13.2	Grey
13.4	White
13.5	Intense white

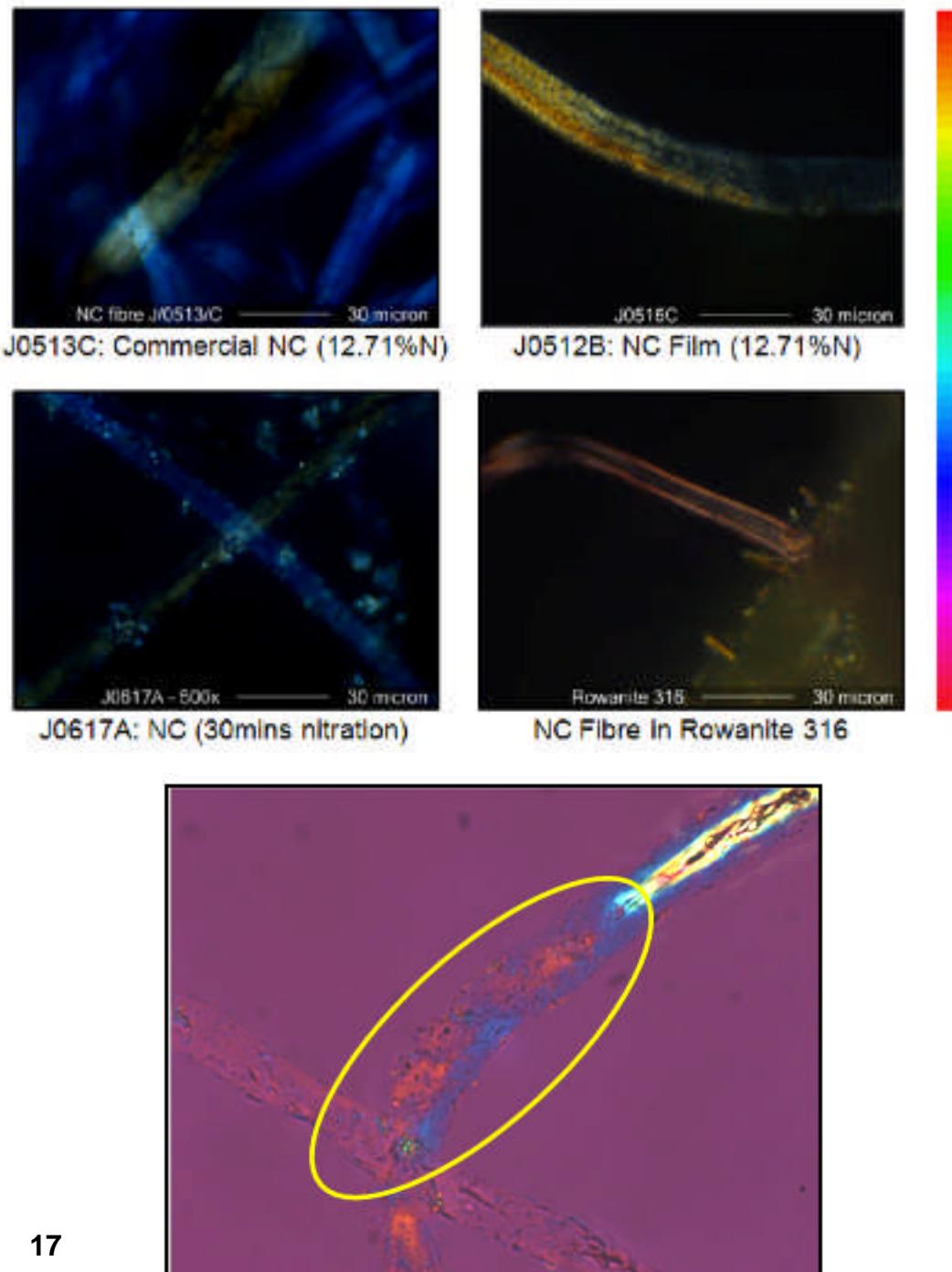
- Different N-content have slightly different crystalline structure, hence different polarisation colours



Polarization colours of nitrocellulose,

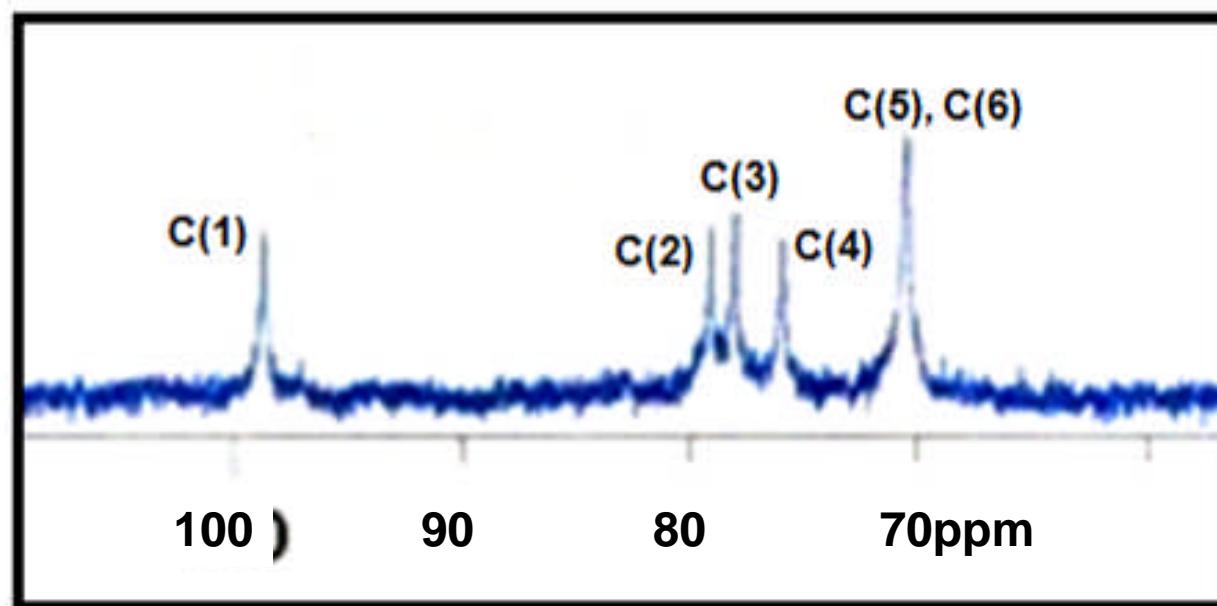
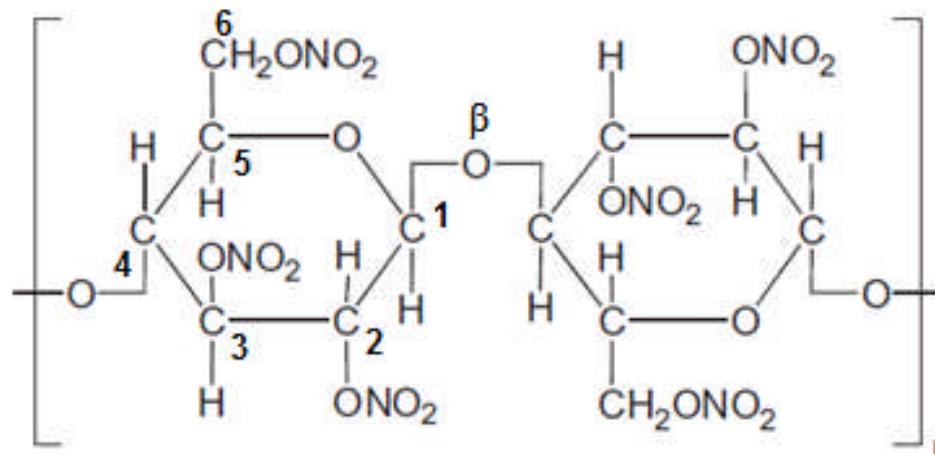
J.A. Kohlbeck and W.T. Bolleter, Journal of Applied Polymer Science, 20, 1976, 153-156



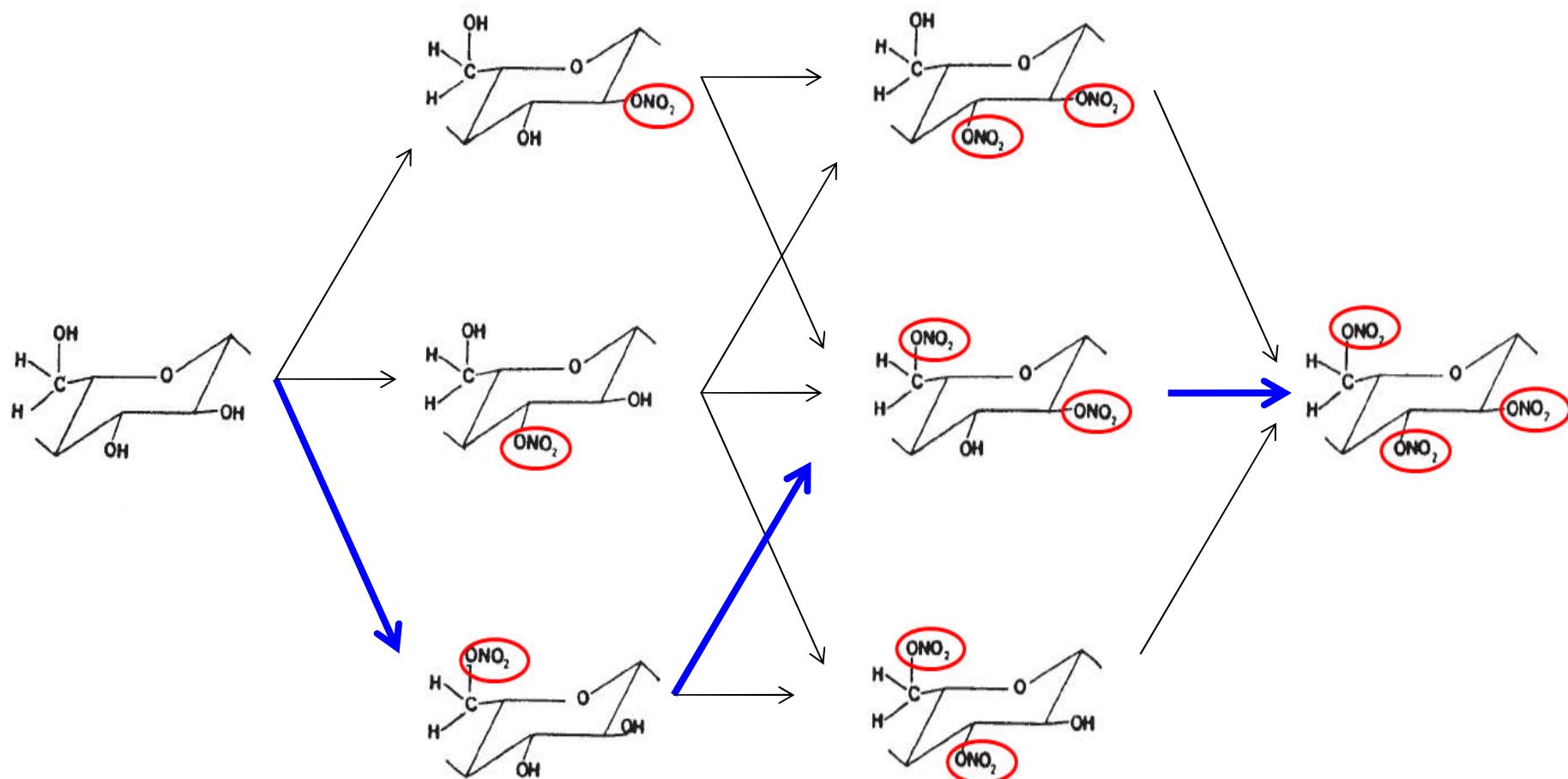
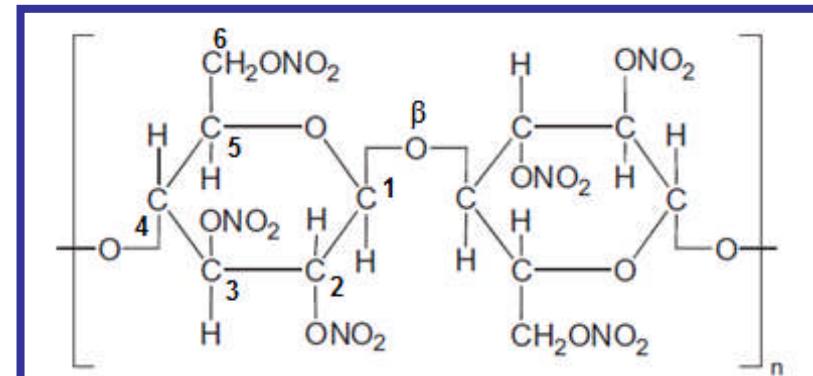


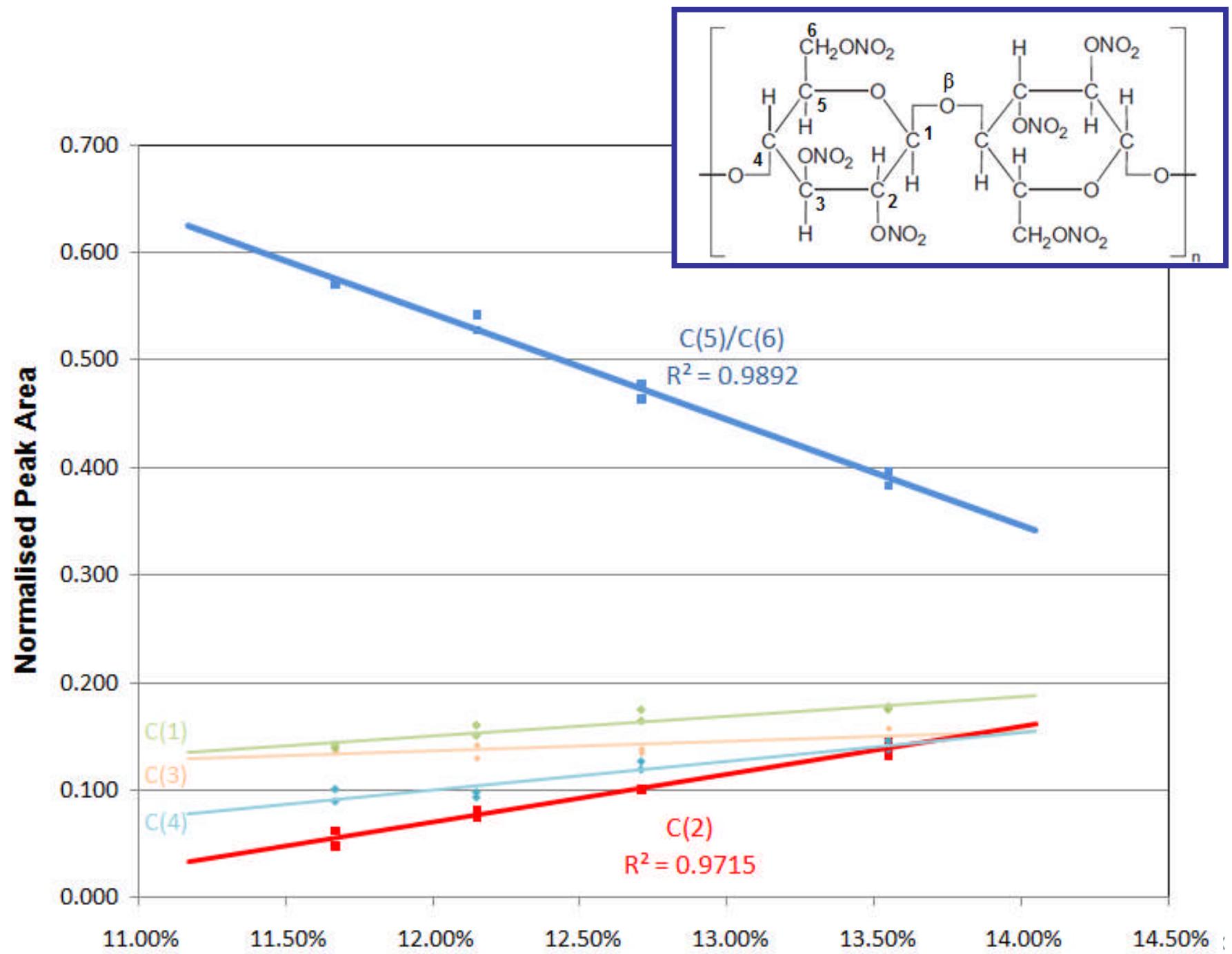
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13.1	Pale grey
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13.5	Intense white

C^{13} NMR of Nitrocellulose

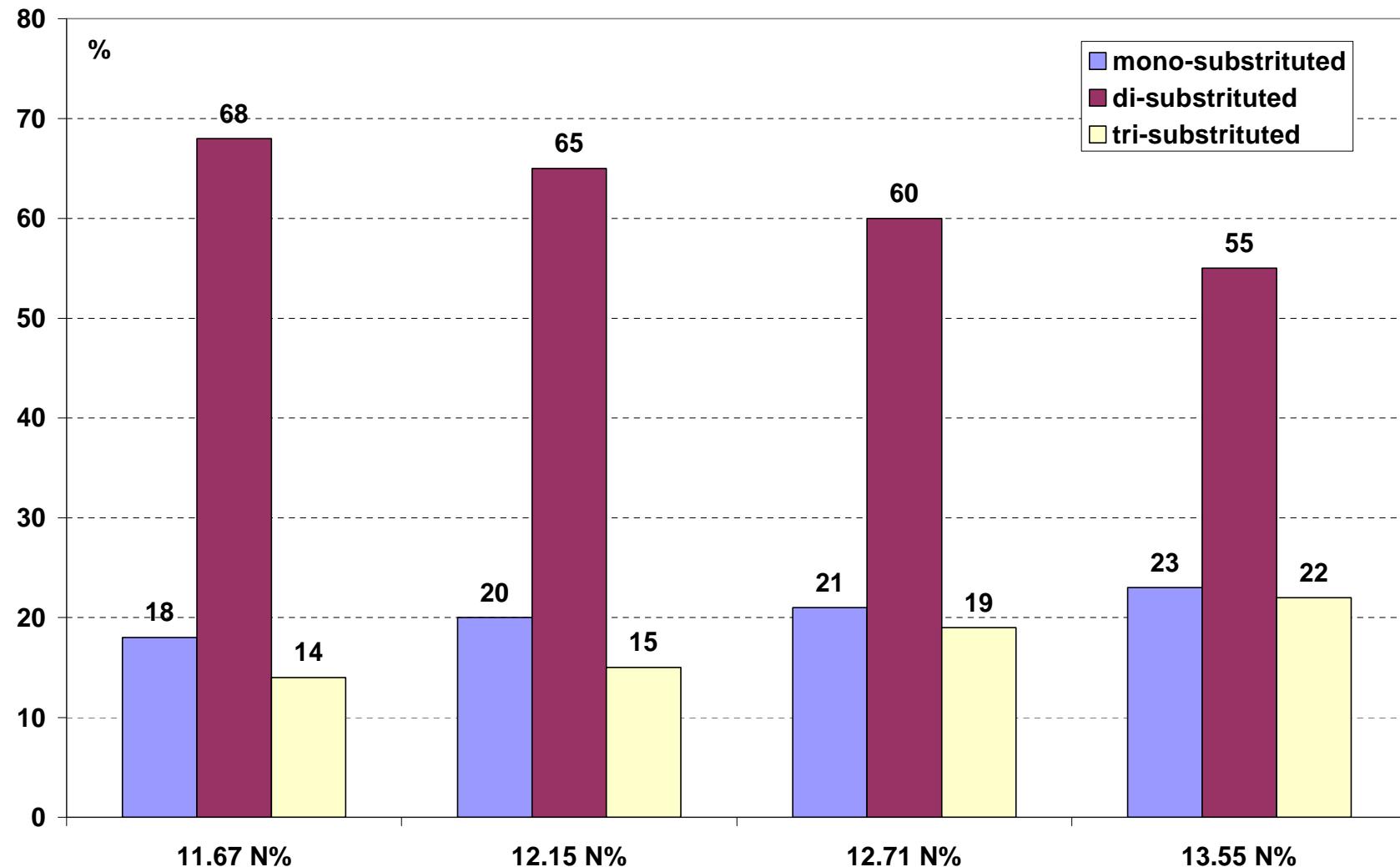


Nitrogen Distribution





Nitrogen Distribution



Conclusions

NC Nitrogen Distribution

Polarised Light Microscopy

- Qualitative, simple, quick
- Distribution between and within fibres

NMR Spectroscopy

- Quantitative, not as simple, not as quick
- Distribution on molecular chain
- *As N-content increases, more of the di-substituted units are being nitrated to tri-substituted units*

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Dr Philip P Gill

Centre for Defence Chemistry

Cranfield University

Shrivenham, SN6 8LA, UK

E: p.p.gill@cranfield.ac.uk



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