



# EFFECT OF CRYSTALLINITY ON AGEING BEHAVIOUR OF NITROCELLULOSE

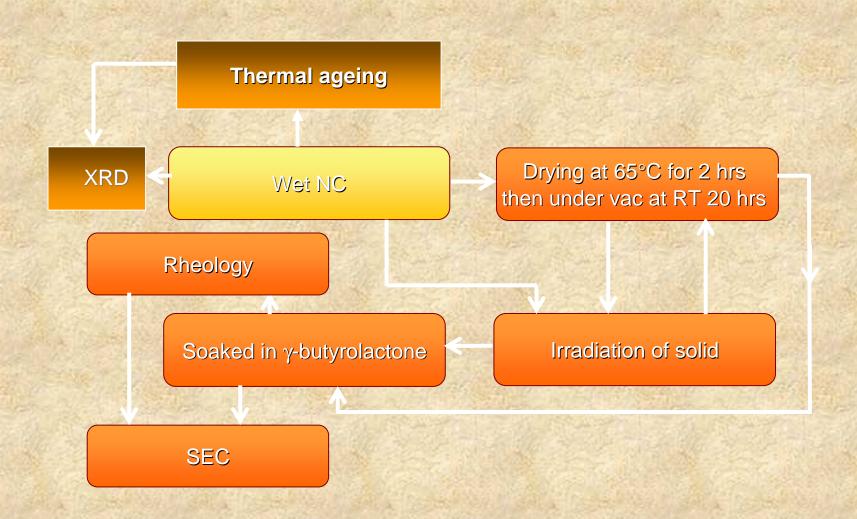
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#### **Programme**

- **→** Crystallinity measurement of NC by XRD
- ◆ Age NC by heat and light
- **→** Measure in-situ continuous viscosity
- **→** Determine molecular weight by SEC
- Try to understand the effect of crystallinity on NC ageing

### Experimental

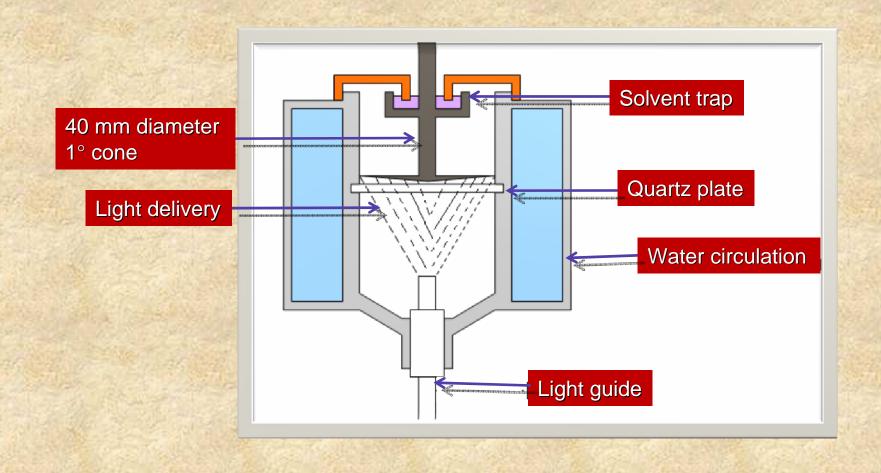


#### Photo-viscosity measurement

- Modified Bohlin CVOR 150
- → 1°/40 mm cone and plate
- Continuous real-time measurements
- **→** In-situ irradiation
- Controlled temperature
- Free from solvent evaporation



## Modified sample cell

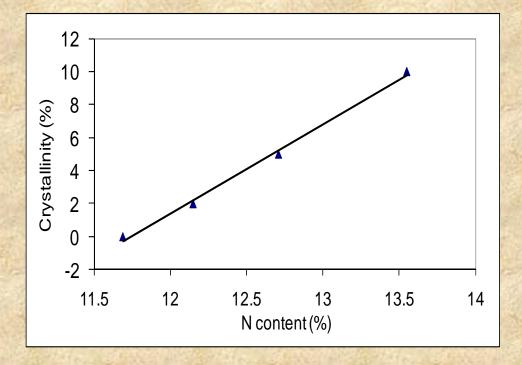




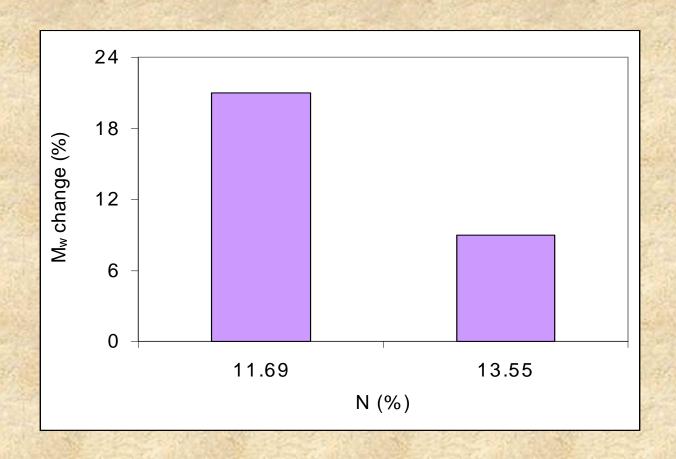
## Crystallinity vs N Content

Degree of nitration dominates

the degree of crystallinity

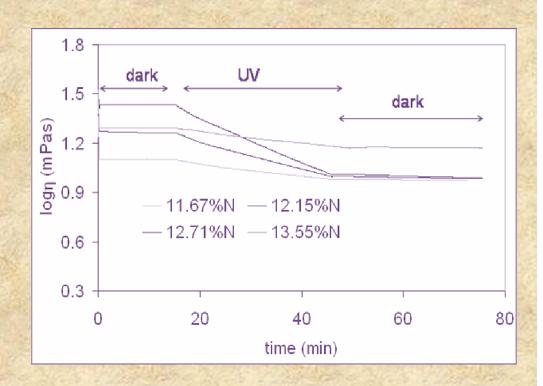


## Mw Changes of thermally aged NC



#### Viscosities for different samples

- UV wave-length was320-390 nm
- Concentration of 10 mg/ml
- → Solution viscosity increases with increasing N content
- Exceptional viscosity behaviour for 13.55%N

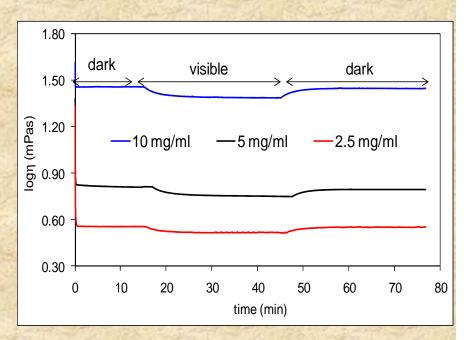


## η<sub>sp</sub> and M<sub>w</sub> changes in samples

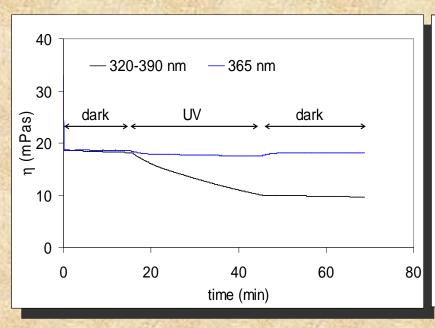
N content in NC (%)	C (mg ml <sup>-1</sup> )	M <sub>w</sub> pre-UV (g mol <sup>-1</sup> )	M <sub>w</sub> post-UV (g mol <sup>-1</sup> )	M <sub>w</sub> decrease	η <sub>sr</sub> /c change (%)
11.69	10	4.157 x 10 <sup>5</sup>	1.726 x 10 <sup>5</sup>	<b>58</b>	<b>28</b>
12.15	10	5.629 x 10 <sup>5</sup>	2.359 x 10 <sup>5</sup>	<b>58</b>	60
12.71	10	7.689 x 10 <sup>5</sup>	3.402 × 10 <sup>5</sup>	<b>56</b>	66
13.55	10	4.045 x 10 <sup>5</sup>	$3.370 \times 10^{5}$	17	26

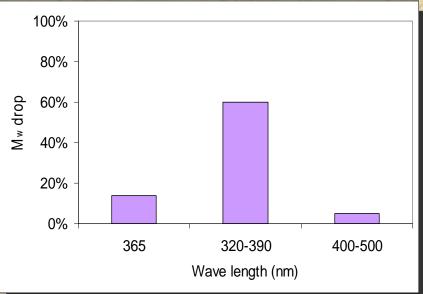
## Effect of visible light (400–500 nm) on η and Μ<sub>w</sub>

- → 12.71% N content studied
- No major viscosity changes
- **→** No significant M<sub>w</sub> change
- η drop is due to heating
  effect
- Viscosity recovered after irradiation ceased



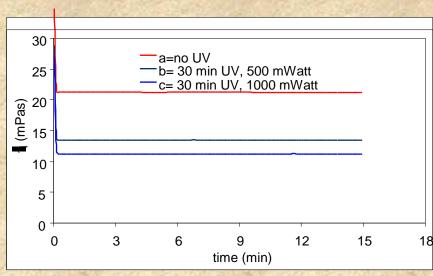
### Effect of UV light on $\eta$ and $M_w$ (12.15%N)

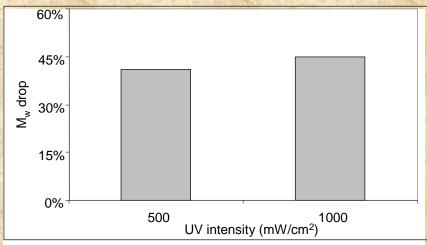




### Effect of UV intensity on $\eta$ and $M_w(12.15\%N)$

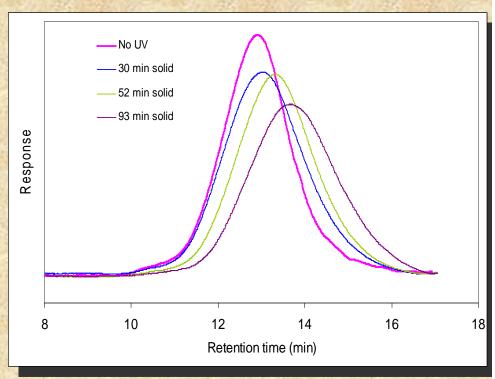
- η decreases are UV
  intensity dependent
- M<sub>w</sub> changes are not greatly influenced by UV intensity
- Effects are non-linear





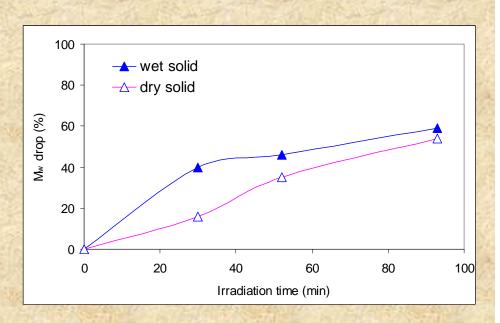
## SEC of solid NC containing 12.15%N, dried (1000 mW/cm<sup>2</sup>)

- Retention time increases
  with UV irradiation
  time
- No higher M<sub>w</sub> shoulder
  is observed



## Changes in M<sub>w</sub> of solid (12.15%N)

- ◆ Light intensity was 1000
  mW/cm²
- Water accelerates photoaging and M<sub>w</sub> drop
- More acid is formed in wet NC than dry NC
- M<sub>w</sub> drop is non-linearly dependent on irradiation time



#### Conclusions

- **◆** Crystallinity is linearly dependent on N content in the NC
- **→** M<sub>w</sub> changes of thermally aged NC is significantly influenced by crystallinity
- In-situ photo-viscosity measurement with modified rheometer gave better results with good precision than a conventional method
- UV irradiation causes NC chain scission and hence drop in η but not at 365 nm
- → Visible light shows no effect apart from small amount of heating
- η and M<sub>w</sub> decrease faster in wet NC than in dried NC

### Acknowledgement

- **→** Thanks to
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  - **→ John Bellerby**
  - **→ Nathalie Mai**
  - **→ Phil Gill**