

Oscillatory rheology of plasticised nitrocellulose binder formulations

Adam Halford

www.awe.co.uk

© British Crown Owned Copyright 2018/AWE



Contents

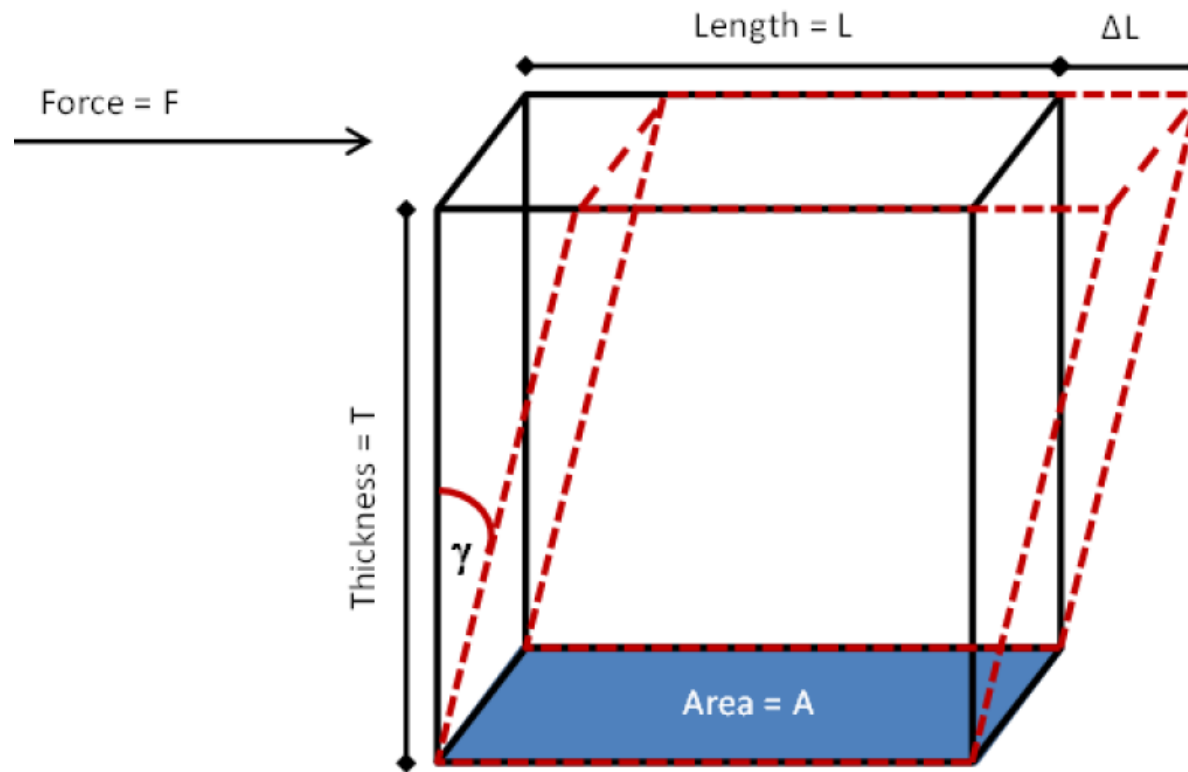


- Introduction to technique and what we are looking for.
- Justification for testing binder materials.
- A case study of work being carried out.
- Some results obtained.
- A few conclusions that can be drawn and some recommendations for future work.

Introduction

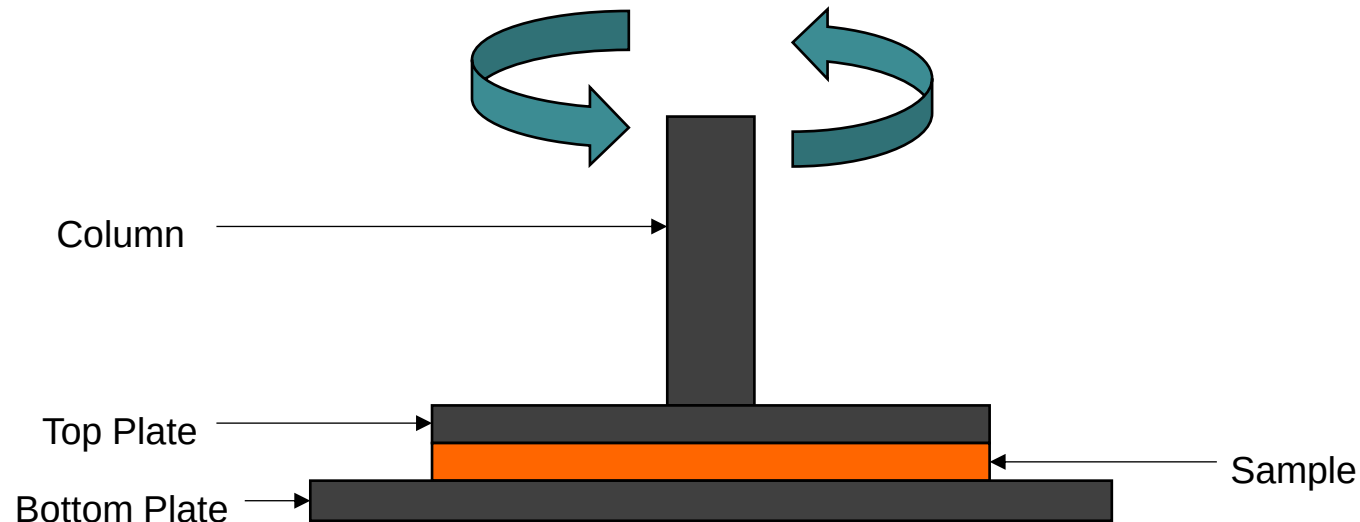


- Oscillatory rheology is a mechanical assessment technique that characterises a materials properties by applying dynamic stimuli – much like DMA.
- A rotational shearing force is applied to the material in a controlled manner (stress or strain) to obtain various parameters dependant on the test.



Oscillatory rheology

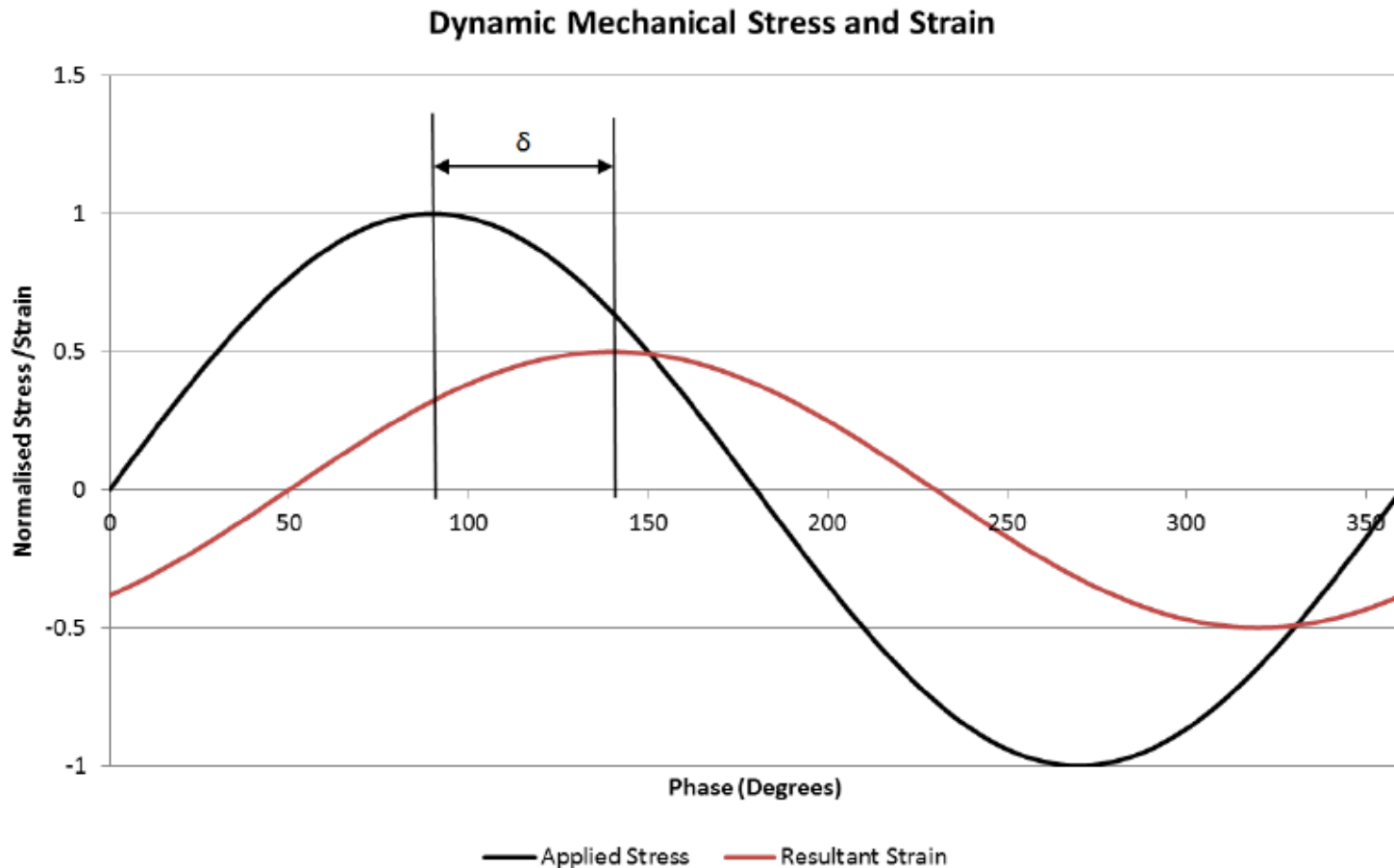
- A sample is placed between a temperature controlled base plate and a plate attached to a moveable column.
 - Many geometries of top plate available to best fit the sample.
- A normal force is applied to ensure good contact but also small enough to not induce significant additional energy into the material.
- Suitable values were obtained through experimentation and monitoring of normal force relaxation times and resultant properties.



Oscillatory rheology



- The stress is applied to the material sinusoidally and the resultant strain is measured by the instrument.
 - Other types of rheometers can operate with a controlled strain with the stress being the measured value.

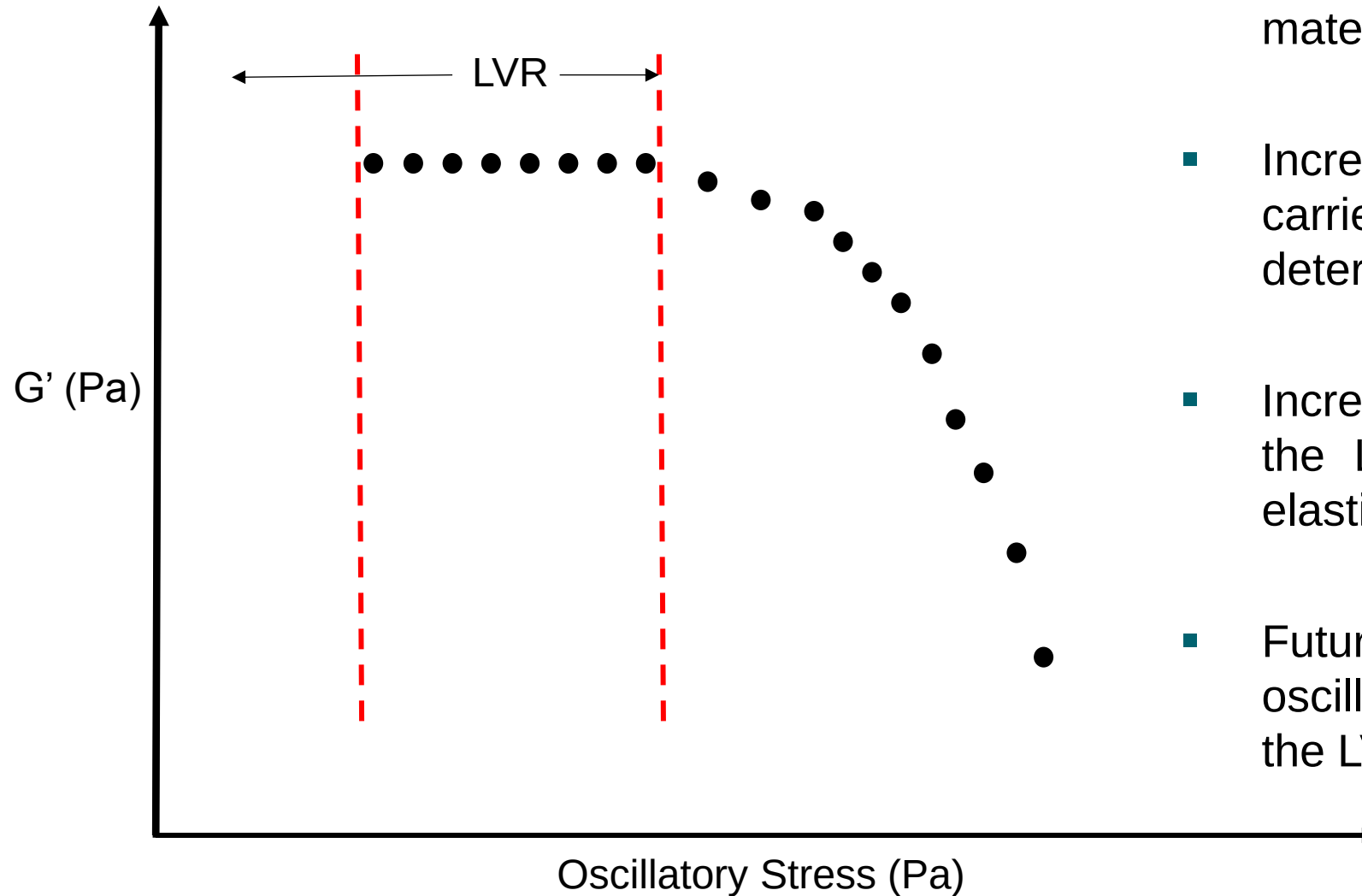


$$G'(t) = \cos \delta \left(\frac{\sigma(t)}{\varepsilon(t)} \right)$$

$$G''(t) = \sin \delta \left(\frac{\sigma(t)}{\varepsilon(t)} \right)$$

$$\tan(\delta) = \frac{G''}{G'}$$

Linear viscoelastic region



- A representation of the determination of the LVR of a material.
- Increase of oscillatory stress is carried out logarithmically to determine LVR.
- Increasing the applied stress in the LVR does not affect the elastic modulus (G').
- Future testing will utilise an oscillatory stress value from the LVR.

What we are looking for

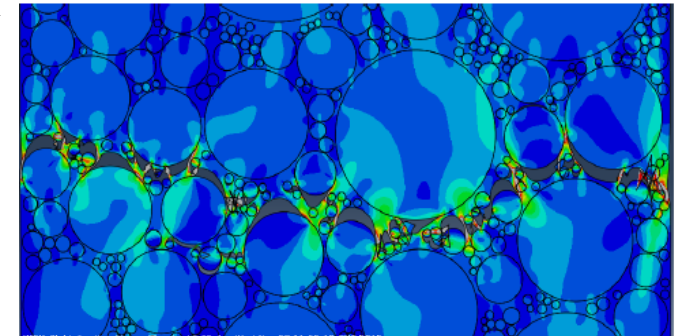
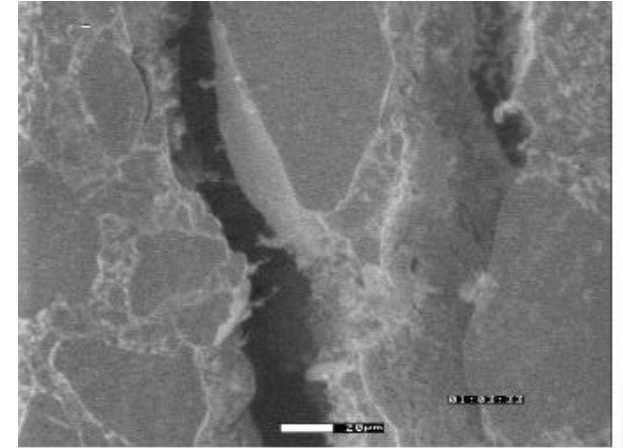


- To test the material within the linear viscoelastic region (LVR).
 - Where the material moduli are independent of the applied deformation.
- Region is identified using a stress sweep with a broad range of applied stresses.
- When the region is identified, subsequent testing utilises a determined value of stress from within the LVR.
- The results from the other tests, e.g. frequency sweep, temperature sweep can therefore be attributed to a specific material response.

Why test binder materials?

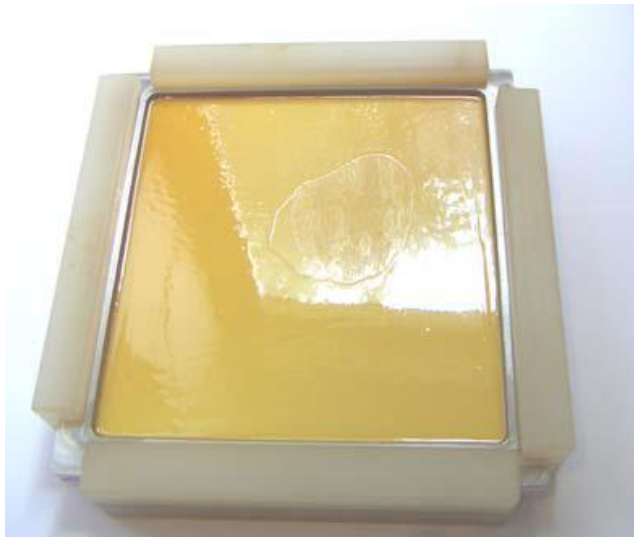


- They are a safer alternative to testing PBX formulations – minimising the amount of testing on PBXs is always beneficial.
- The particulate fill of the PBX of consideration has a stiffness of many orders of magnitude greater than that of the binder.
- As a result, the mechanical properties of the PBX can be derived from the properties of binder and the interface with the hard inclusions in the formulation.
- The adhesion between the binder and the hard inclusions is known to be a function of the binder material properties and the thermodynamic work of adhesion.
- As a result, the mechanical properties of the PBX under consideration can be approximated to the binder material.
- Therefore by physical testing of the binder material, differences due to formulation changes or ageing can be investigated.

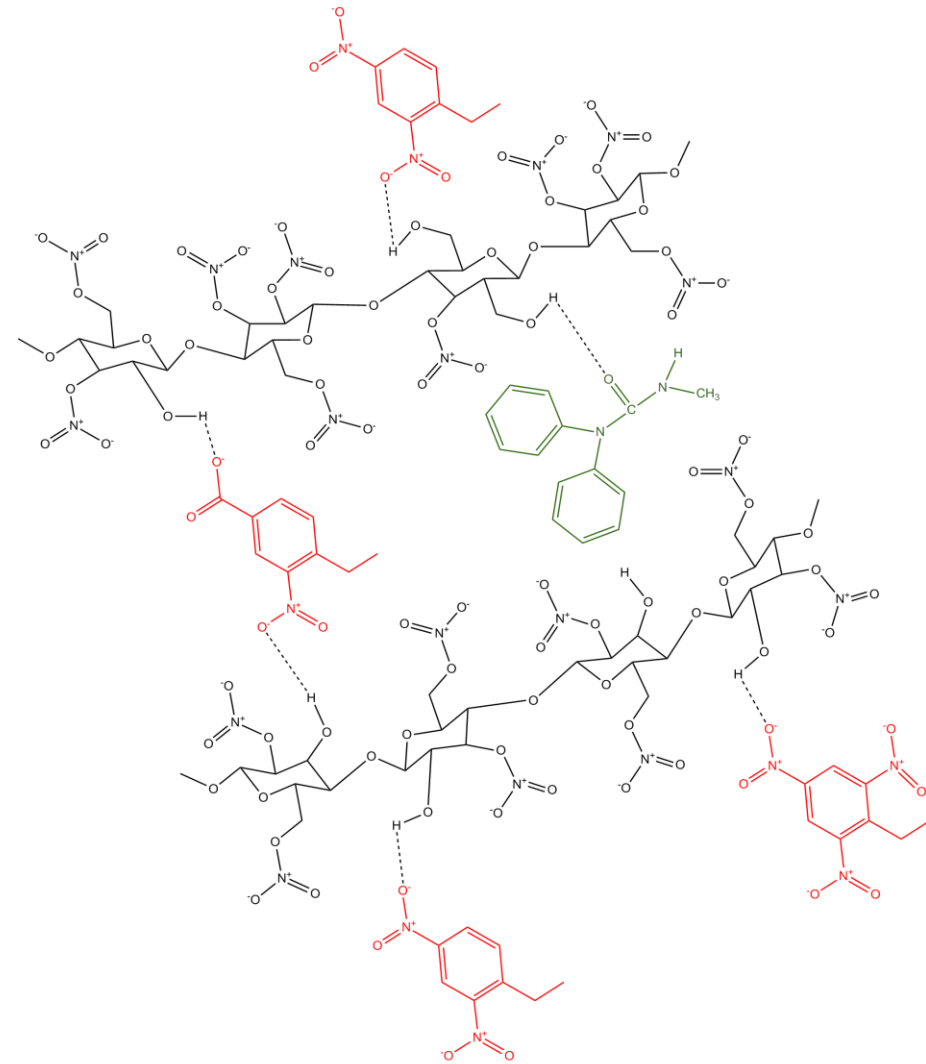
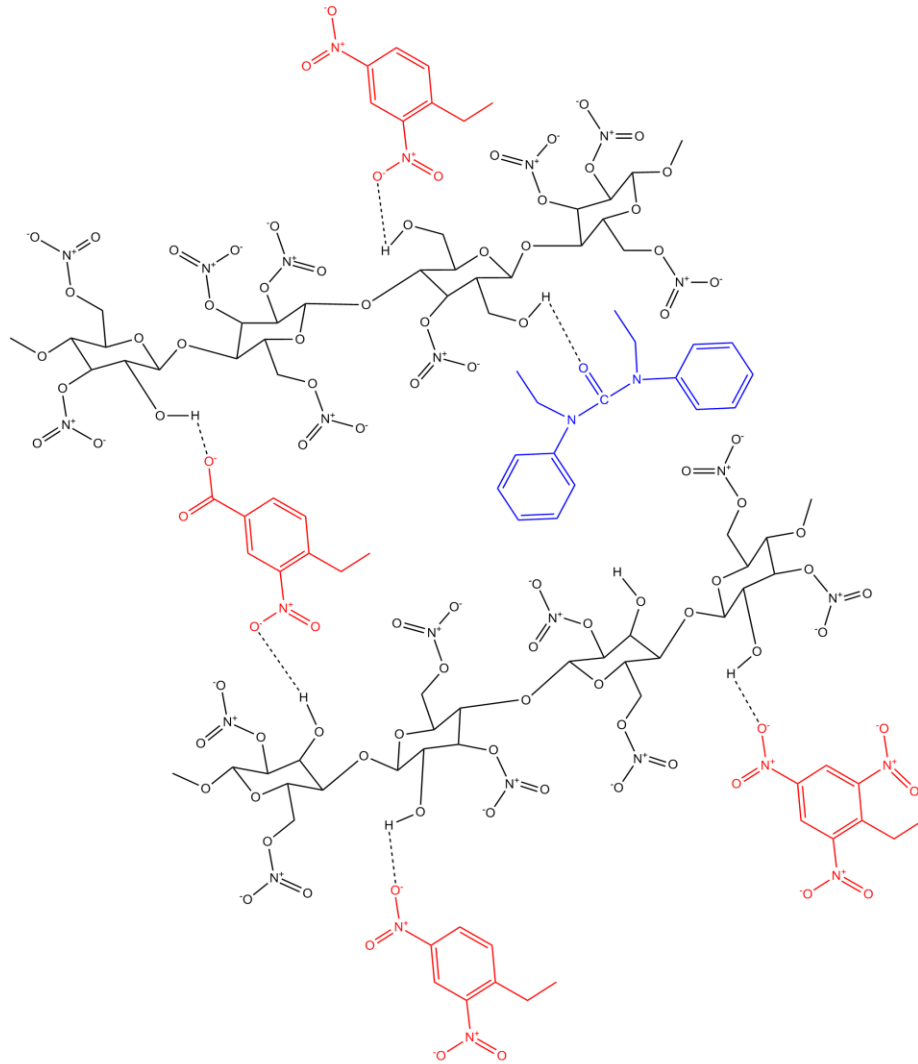


Case study

- An investigation into the differing rheological properties of plasticised nitrocellulose as a result of thermally accelerated ageing.
- Variety of binders manufactured and subsequently placed in thermal chambers for a period of 12 months with withdrawals at 1, 3 and 6 months also carried out.



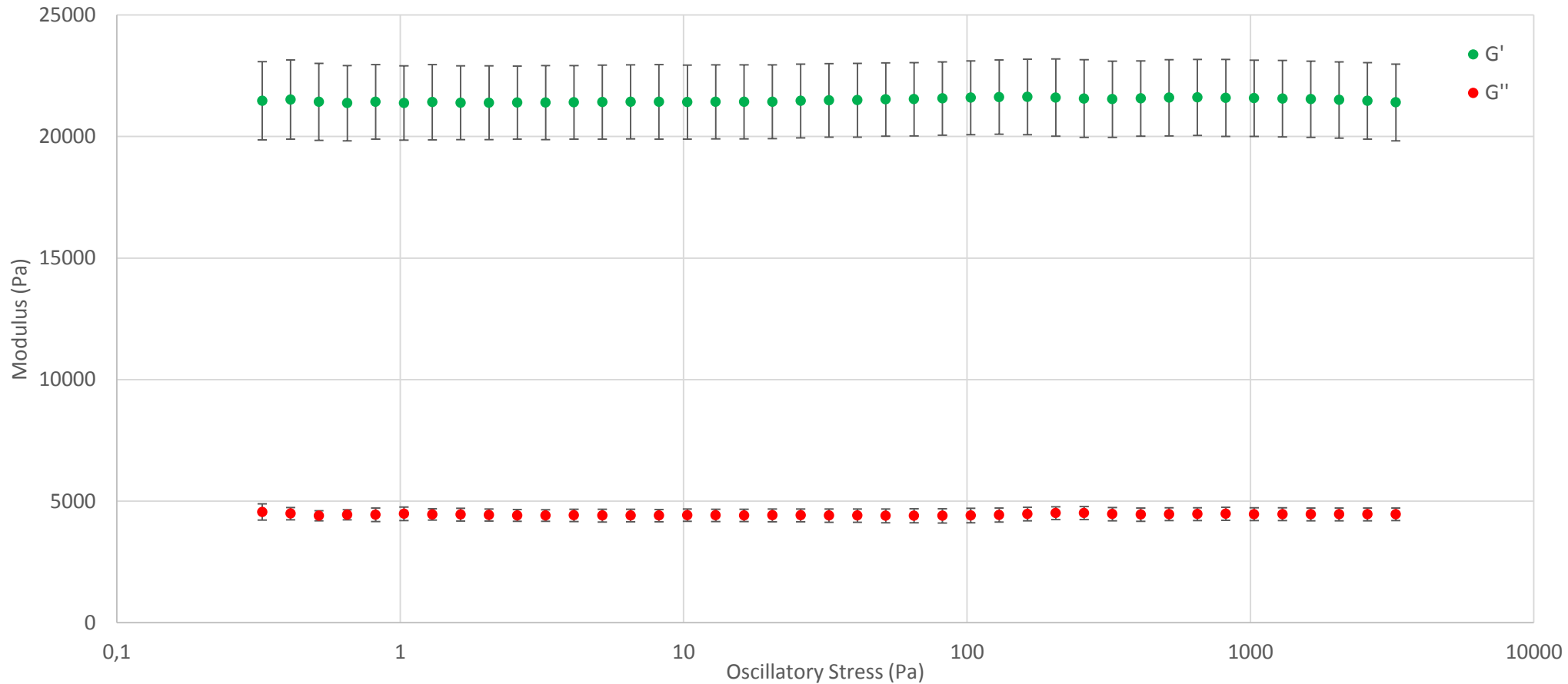
Gel structure of formulation



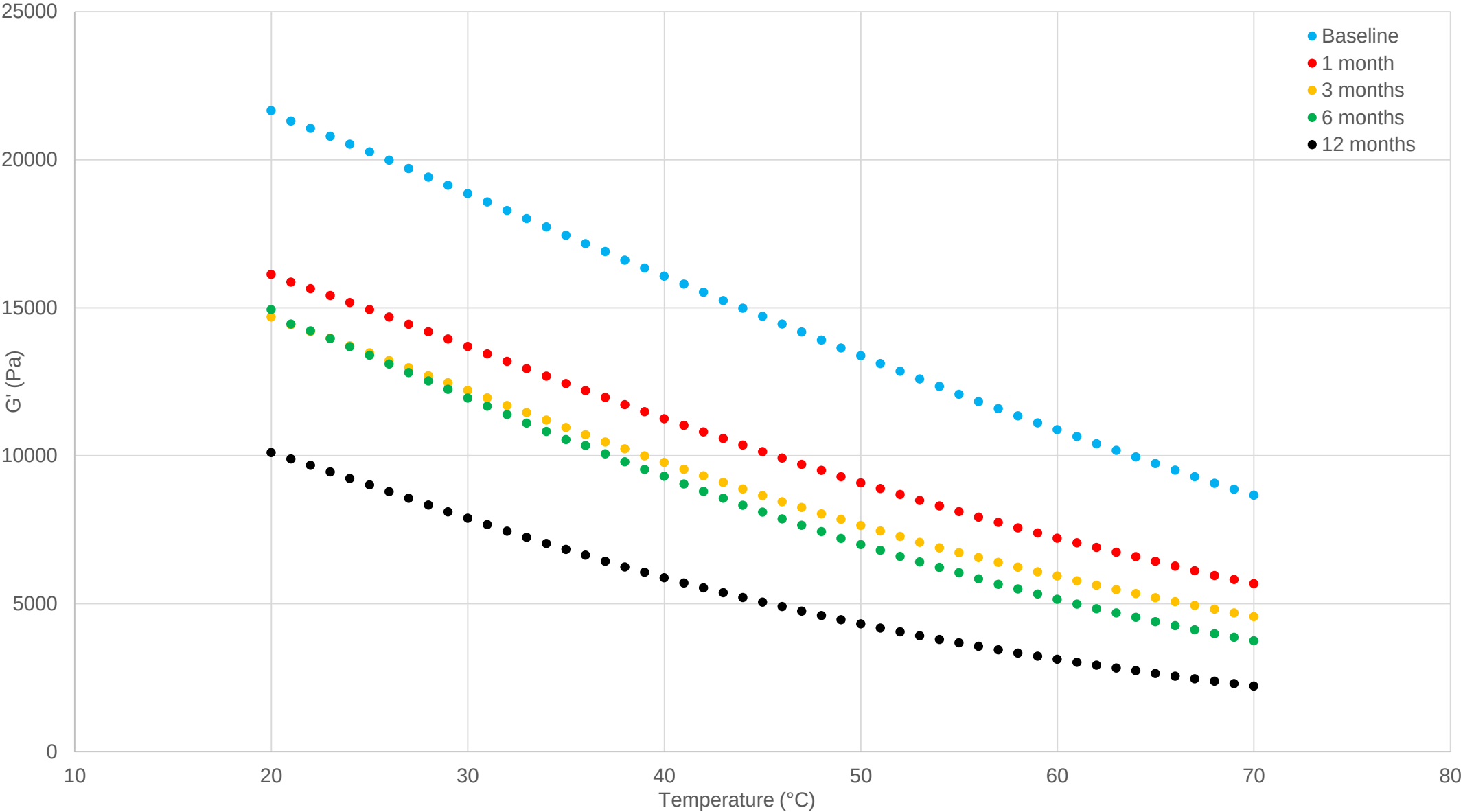
Some results



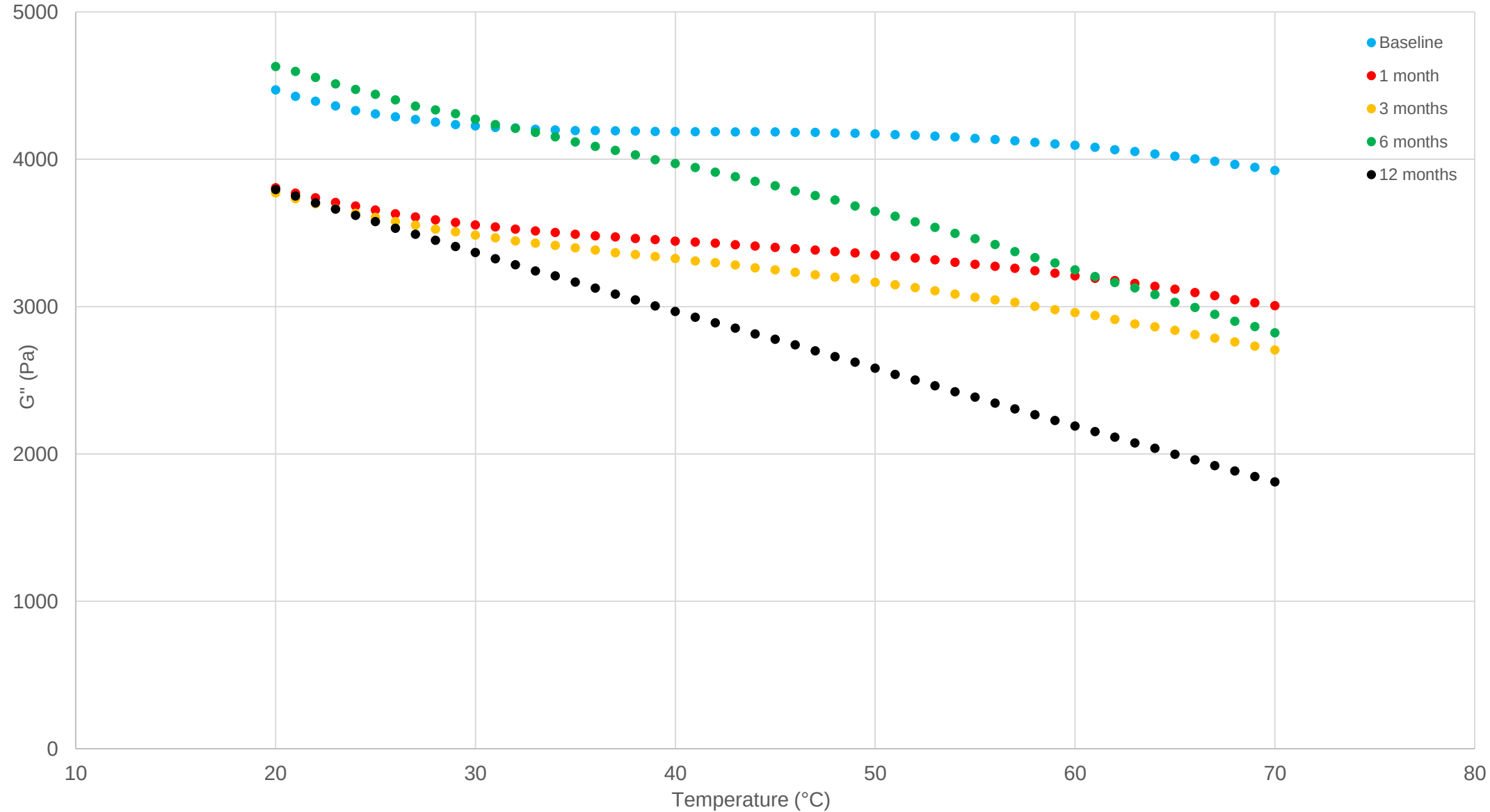
- Shown is a plot of the average values obtained from stress sweeps – with increasing the stress, the value for G' was unaffected hence by picking a stress value from within this region for subsequent test, we can assume we are probing the material within the LVR.



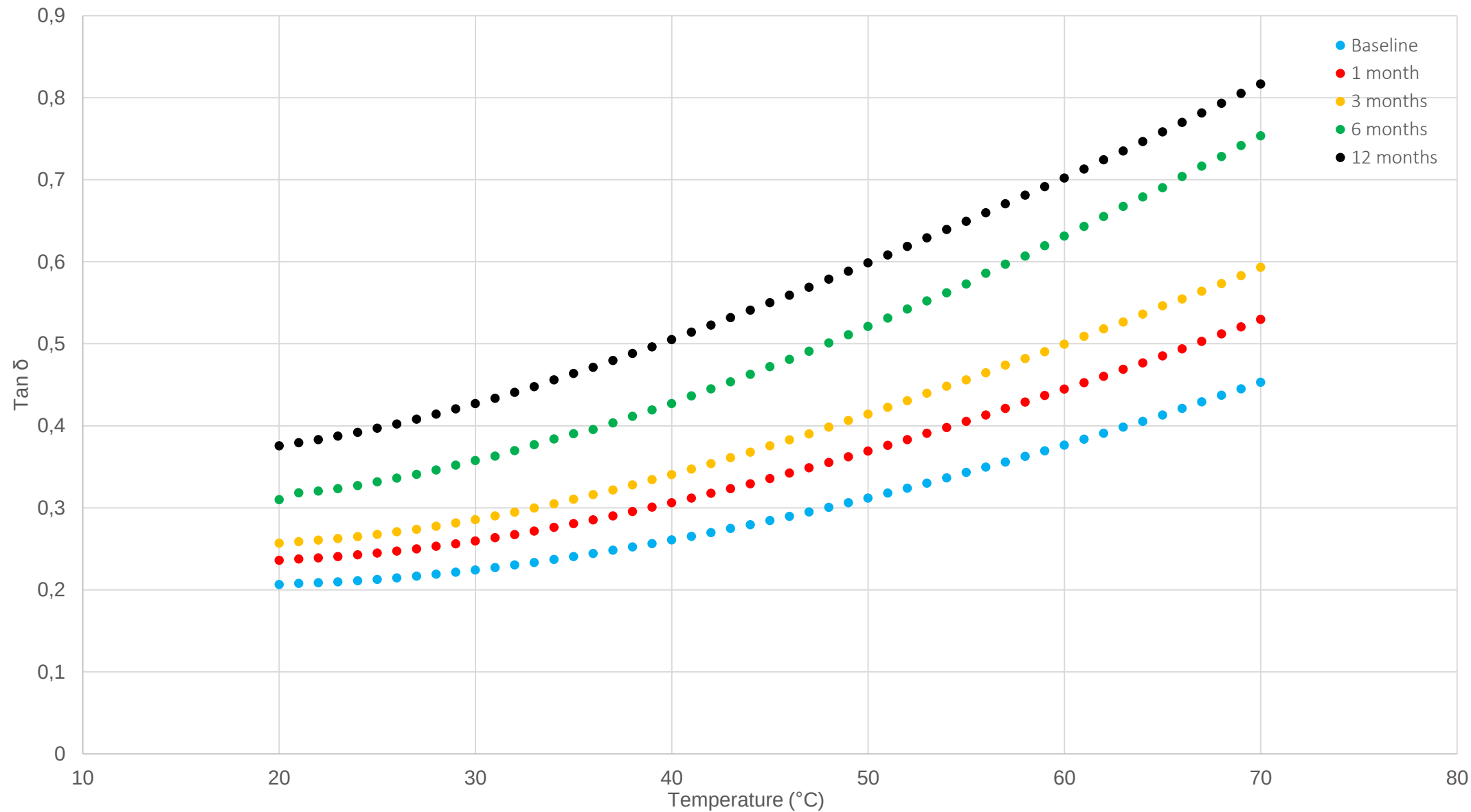
Some results



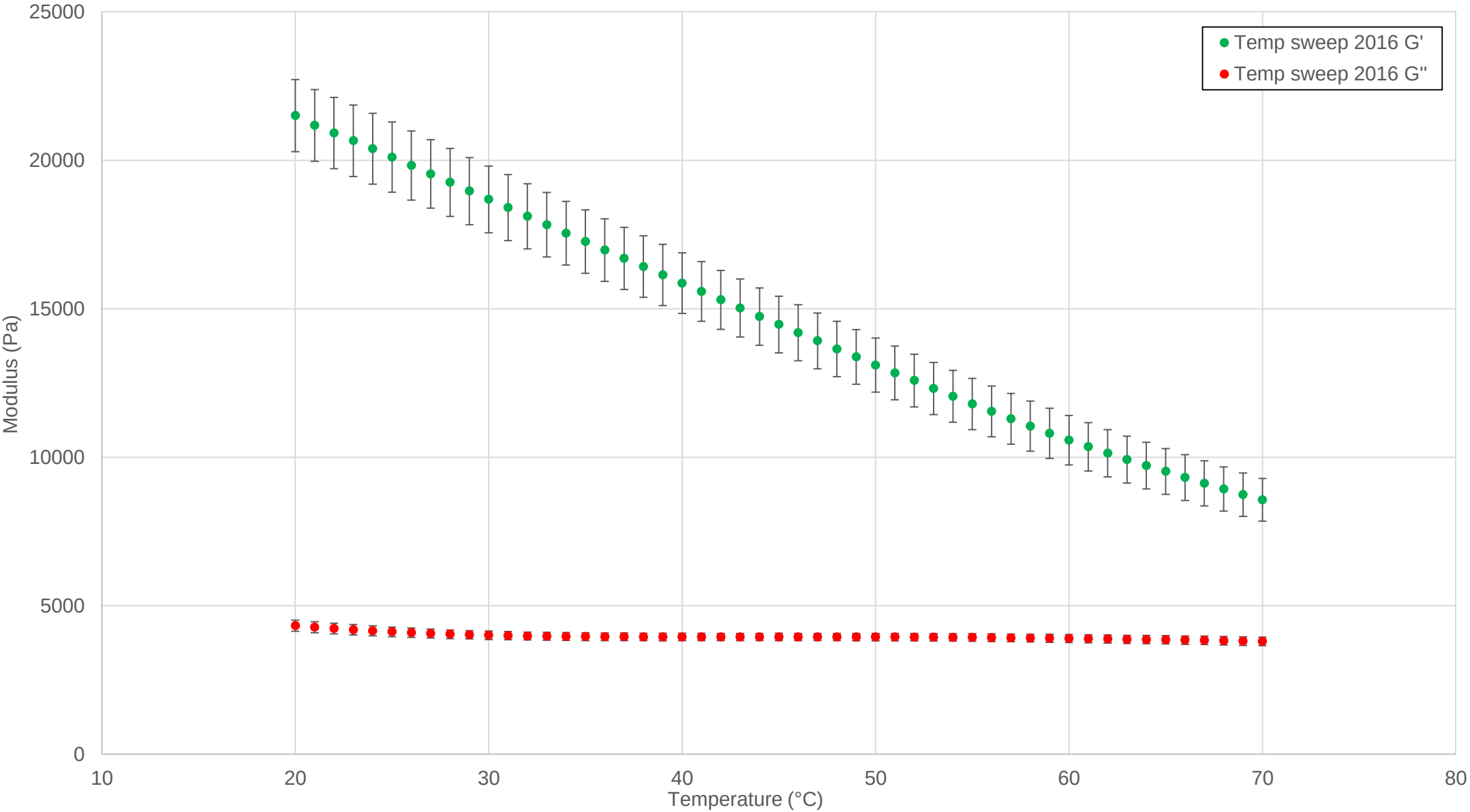
Some results



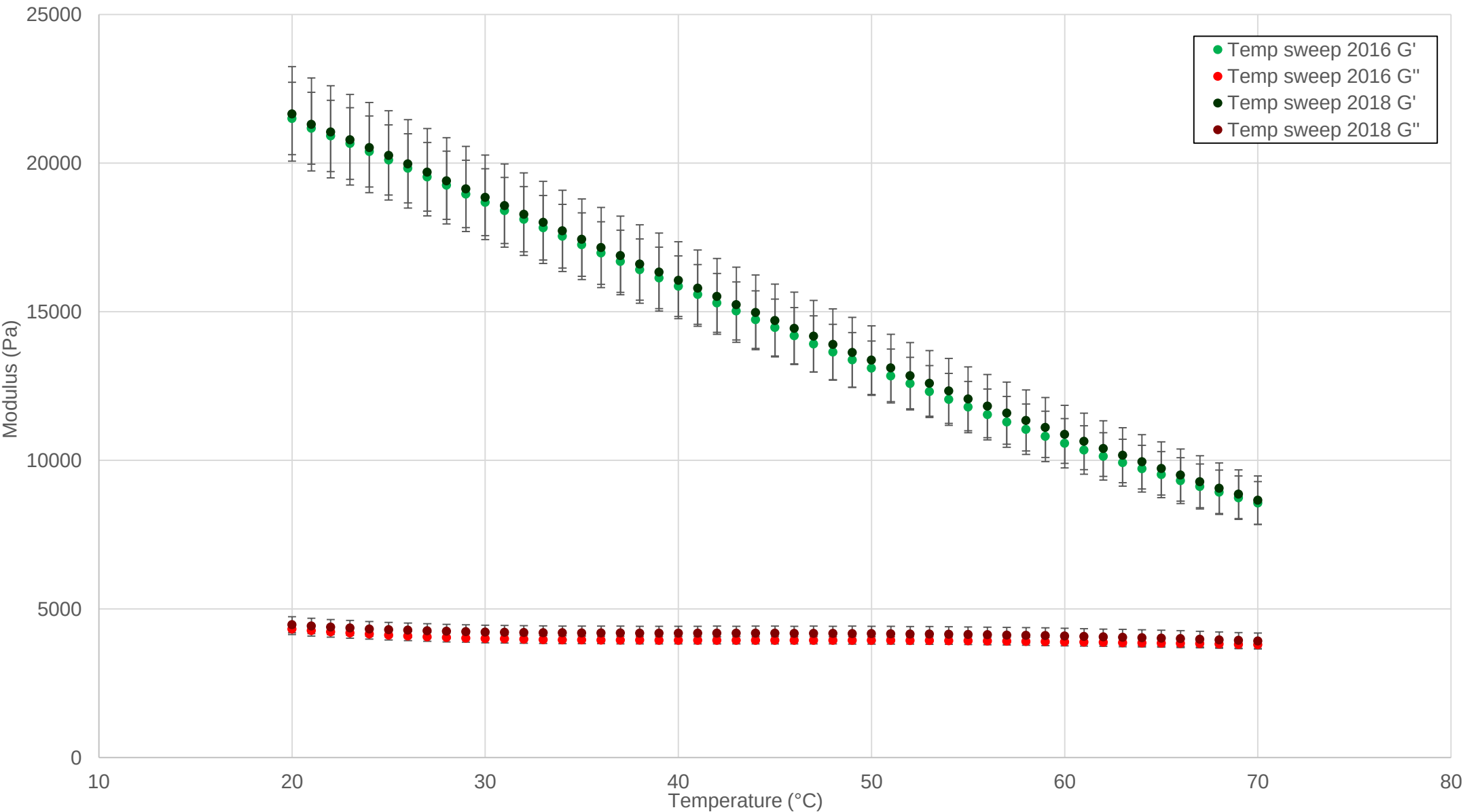
Some results



Some results



Some results



Conclusions

- Thermally accelerating the ageing process of plasticised nitrocellulose has an adverse effect on the mechanical properties.
- Storing an aged binder at sub-ambient temperature for long periods of time appears to prevent any further degradation to the mechanical properties.

Future work

- Continuation of thermal ageing to increase the volume of available data in order to improve the confidence in the fitted model.
- Complete rheological analysis on available binders to further investigate the degradation of mechanical properties as a result of thermally accelerated ageing.

Thanks for listening

Any questions?