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MEASUREMENT OF STORAGE MODULUS IN UNCURED ELASTOMERS USING PLATE-PLATE AND MOVING DIE TECHNIQUES

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INTRODUCTION

Development of new polymers and their recycling technology depend on dynamic-mechanical properties [1]. High viscosity and elasticity of synthetic cis-1,4-polyisoprene rubber (p-isoprene) could be challenging properties for the rubber industry. Therefore, the reduction of viscosity by mastication is an important process for p-isoprene based compounds, which are used in the tires industry and technical rubber products. At low temperatures, mechanical mastication happens when shear forces are applied to the polymer chains during mixing [2]. During this process lamellae, crystals and similar macromolecular agglomerates homogeneously distribute within the elastomer without much chemical degradation. However, viscosity, elasticity and many dynamic-mechanical properties of the elastomer can be affected by ageing. In order to observe the ageing effect on the synthetic cis-1,4-polyisoprene rubber (p-isoprene), mastication was performed and storage modulus G' was recorded with the moving-die rheometer D-MDR 3000 (Montech, Germany).

EXPERIMENTAL

The discs were placed in between disposable polymer sheets of 23 µm thickness and tested in a moving-die rheometer D-MDR 3000 (Montech, Germany). Frequency sweep was used to record G' in 0.01 – 50 Hz frequency range, 3 cycles for each frequency under 0.5% strain concurrently on the same specimen at 25°C and 50°C with 5 min preheating.
Built-in software calculated storage modulus G' whose values from multiple runs (not fewer than 5) were processed using Origin software to derive the representative curves with error bars.

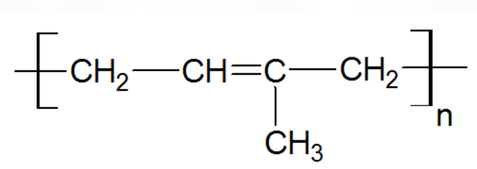
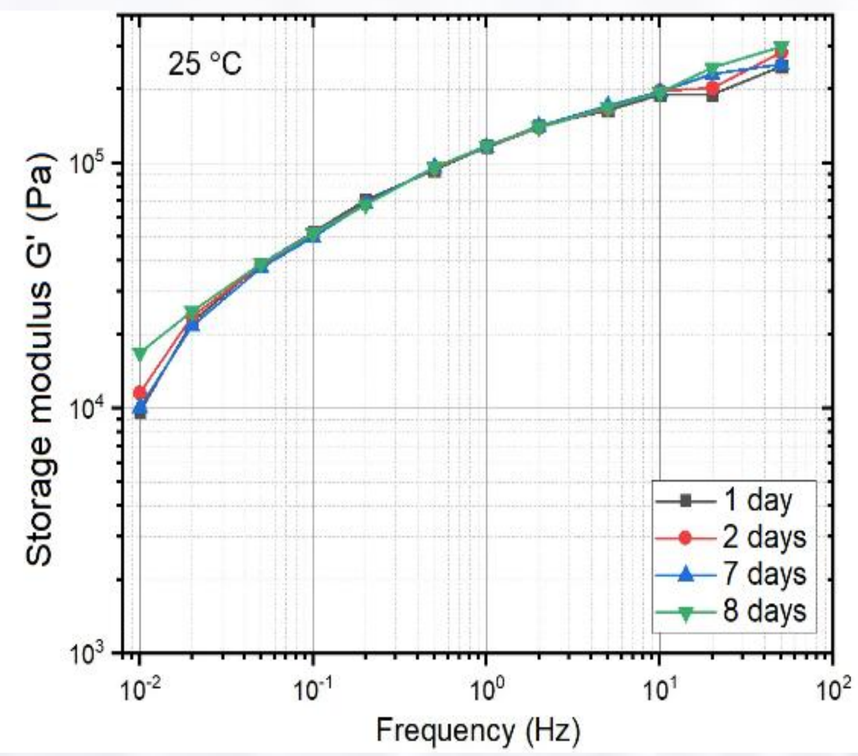


Fig. 1 Polyisoprene [3] structural formula

MATERIALS

In this study p-isoprene without added components was selected to observe ageing effects on its viscoelasticity. Storage modulus G' was monitored after masticating the elastomer and storing it up to 8 days at room temperature. Bulk p-isoprene was sliced into small slabs of ~30 g and masticated on the two-roll mill HTR-300 (Hartek, China) for 25 passes. Because the efficiency of mechanical mastication drops with rising temperature due to the softening of the polymer [2], for the mastication procedure the lower temperature (22°C) was chosen. Rolls of Ø 360 mm were compressed to near-minimal nip size of 0.8 mm ± 30% and their velocity was set at 8 rpm with 1:1.25 sliding ratio.

Afterwards, the sheets were further shaped into specimens. Smooth and flat surfaces were obtained after compressing them between PTFE or glass plates for 16 hrs or longer at room temperature. An effort was made to avoid bubbles, wrinkles or other structural imperfections. Specimens of polyisoprene were shaped into discs Ø ~35 mm and 4-6 mm thick, weighing ~5 g, and were stored in dark cabinet.
For dynamic-mechanical testing the D-MDR 3000 rheometer (Montech, Germany) with biconical dies was employed per ASTM D6204.



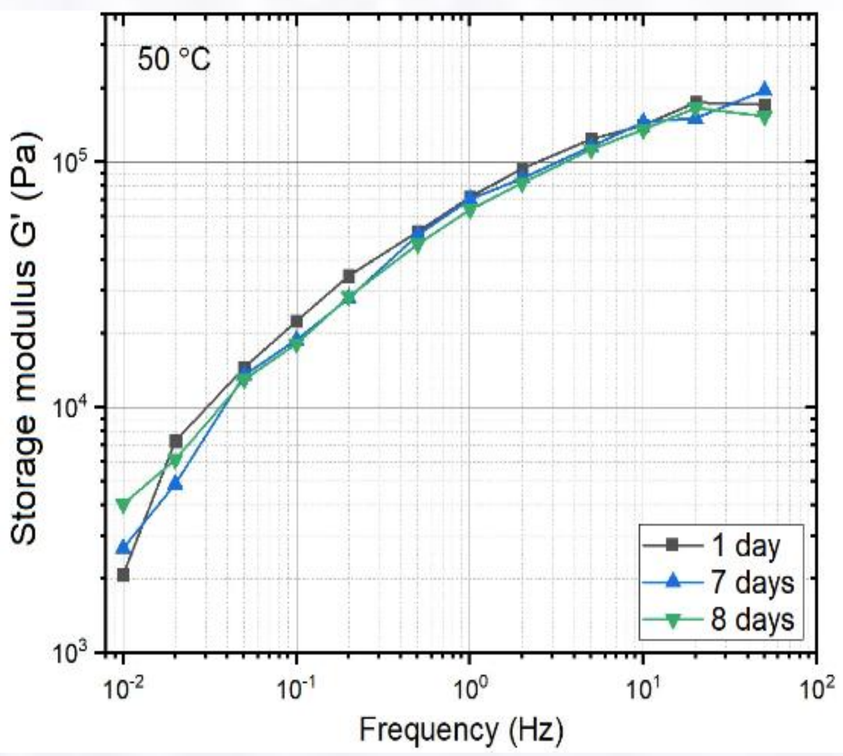


Fig. 3 Effects of mild ageing at room temperature on viscoelasticity of p-isoprene

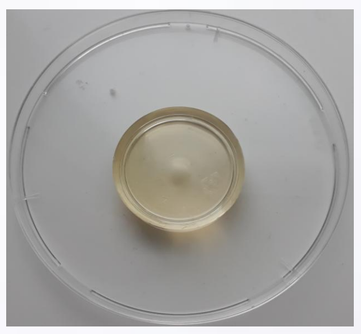


Fig. 2 D-MDR 3000 (Montech) and applied sample before and after test

RESULTS

The results of frequency sweep tests of polyisoprene specimens showed a tendency of rising storage modulus G' under increasing frequencies. The ageing effect on storage modulus after mastication was not detectable at 25°C. Repeatability of G' at this temperature is good.
However, the values of storage modulus at 50°C revealed the possible ageing effect on p-isoprene specimens. Although the differences of curves were not large and might be considered negligible in some cases, Noticeably the graphs of 7 and 8 days mostly had lower values than of 1 day after mastication. Thus, significant alterations in viscoelasticity of p-isoprene specimens could be observed 1 week after mastication.

CONCLUSIONS

- The frequency sweep tests showed a tendency of rising storage modulus G' of p-isoprene under increasing frequencies.
- The ageing effect on G' was not significant at 25°C and its repeatability is good.
- Possible ageing effect on p-isoprene specimens could be detected at 50°C as the curves of 7 and 8 days most often had lower values than of 1 day after mastication.
- 1 week after mastication revealed detectable changes in the dynamic-mechanical properties of p-isoprene.
- Elastomer formulations with compounding ingredients possibly could experience more rapid and noticeable transformations.

REFERENCES

1. X. Sun, A. I. Isayev, Ultrasound devulcanization: comparison of synthetic isoprene and natural rubbers, J. Materials Sci. 42 (17), 7520-7529 (2007).
2. C. Wortmann, P. Lindner, F. Dettmer, F. Steiner, T. Scheper, Mastication Behavior of cis-1,4-Polyisoprene as a Model for Natural Rubber, Journal of Applied Polymer Science 131 (7), 39989 (2014).
3. https://polymerdatabase.com/Elastomers/Isoprene.html

ACKNOWLEDGMENTS

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