DIELECTRIC PROPERTIES OF POLYMERS KRASOL AFTER TEMPERATURE AGING

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ABSTRACT

The objective of this research is to measure and to analyze dielectric properties of KRASOL oligomer materials. The other objective is to make the methodical process of this measuring on the liquid materials. The analysis and subsequent interpretation is based on a detailed observation of temperature dependencies of the relaxation processes prior to and after the aging process in a broad frequency range. Measurements in the frequency domain were carried out on the Hewlett Packard HP 4284A precision LCR meter using the dielectric test fixture HP 16451B. The sample was analyzed in the temperature chamber, which was specifically developed for this experiment.

1 DIELECTRIC RELAXATION SPECTROSCOPY

The research of material characteristics necessitates the selection of an appropriate analytical method. One of the modern experimental methods is dielectric relaxation spectroscopy (DRS). In general, DRS studies molecular dynamics of current carriers and dipoles. DRS is based on a set of theories and methods dealing with this dynamics. KRASOL samples are analyzed and evaluated in the frequency domain at different temperatures.

2 SAMPLE

Two butadiene types were studied in the experiment. The first one is KRASOL LB and the other one is KRASOL LBH. KRASOL LB is a liquid, low-molecular-weight polymer of butadiene, without functional groups, produced by a special anionic polymerization technology. The polymer is fully linear and exhibits very narrow molecular weight distribution. Due to its low molecular weight, KRASOL LB is liquid at ambient temperature. It is manufactured in three grades KRASOL LB 2000, 3000 and 5000; numbers indicate the molecular weight. Typical chemical structure is as follows: **$H – (-CH_2-CH=CH-CH_2-)_n – H$.**

Applications of KRASOL LB are based either on its physical properties (viscous behavior and tackiness) or on chemical reactivity of double bonds [6]. The properties of KRASOL LBH are much the same as those of KRASOL LB; the main difference is that KRASOL LBH has terminal secondary hydroxyl groups. The most significant applications of KRASOL LBH are those in polyurethane production [7].

3 MEASURING EQUIPMENT

Measurements in the frequency domain were carried out on the Hewlett Packard HP 4284A precision LCR meter [1] using the dielectric test fixture HP 16451B [2]. Measuring with the precision LCR meter is based on bridge techniques with auto-calibration; results are available over the frequency range 20 Hz – 1 MHz. The aging of samples was made in the STERICELL 55 temperature chamber. At lower temperatures two cooling devices were used. One is a commercial CALEX fridge and the other is designed, developed and manufactured for this experiment and is based on the Peltier cell. The experimental temperature range is from – 25 °C to ambient temperature of about 20 °C.

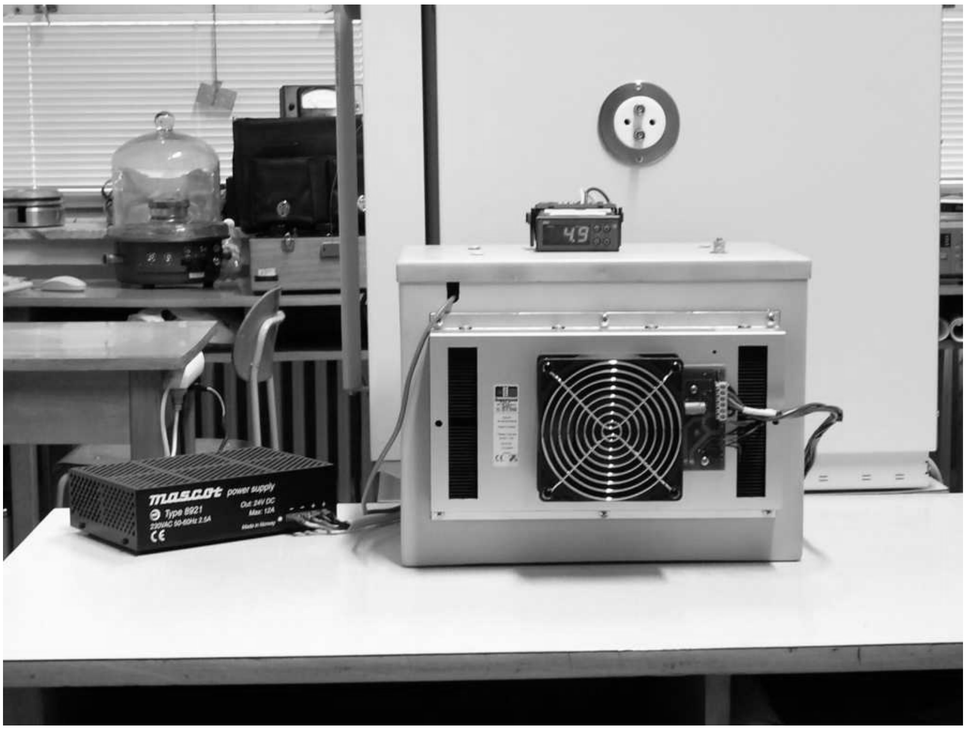

Fig. 1: *Made temperature chamber for experiment*

4 EXPERIMENT EVALUATION

Because the oligobutadiene is at room temperature very viscous we used three 100 μm glass fibers to keep constant distance between the electrodes. These fibers were lying on the bottom electrode and a droplet of oligobutadiene was expelled from a syringe. Then the top electrode was screwed down, so that the droplet flowed away and formed a dielectric layer between both electrodes. After preparing the sample the measuring process in the temperature chamber was started. Experiments were made first on samples without aging and then on samples after aging for 100 and 200 hours. The aging temperature was 130°C. The characteristics ε"=F(f) were measured and evaluated experimentally for the temperatures in the range from –25 °C to 20 °C. The analysis of the experimental data indicates the presence of relaxation processes in the oligobutadiene sample. The relaxation maximums move towards lower frequencies with the decreasing temperature and that is why the relaxation maximums were perceptible. The position of relaxation maximums matches with the straight-line characteristic and therefore Arrhenius activation law was used for its description.

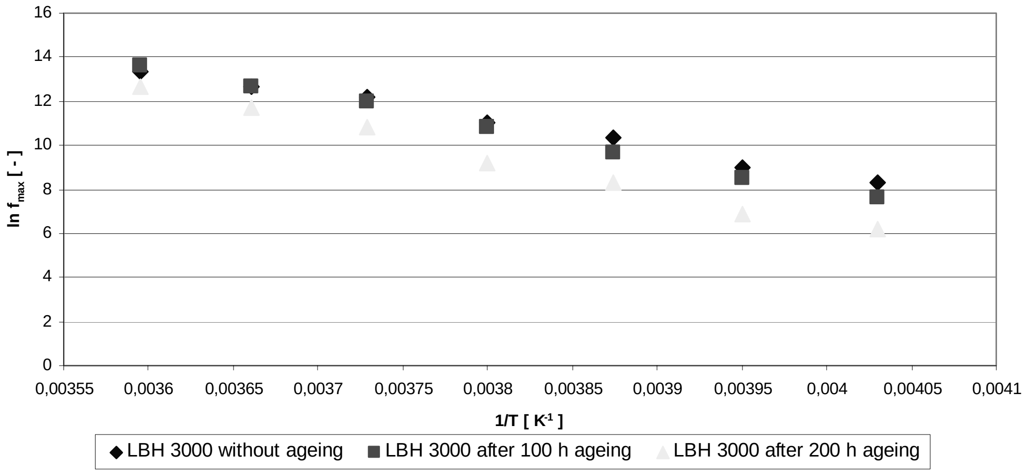

Fig. 2: *Relaxation map of LBH 3000 sample*

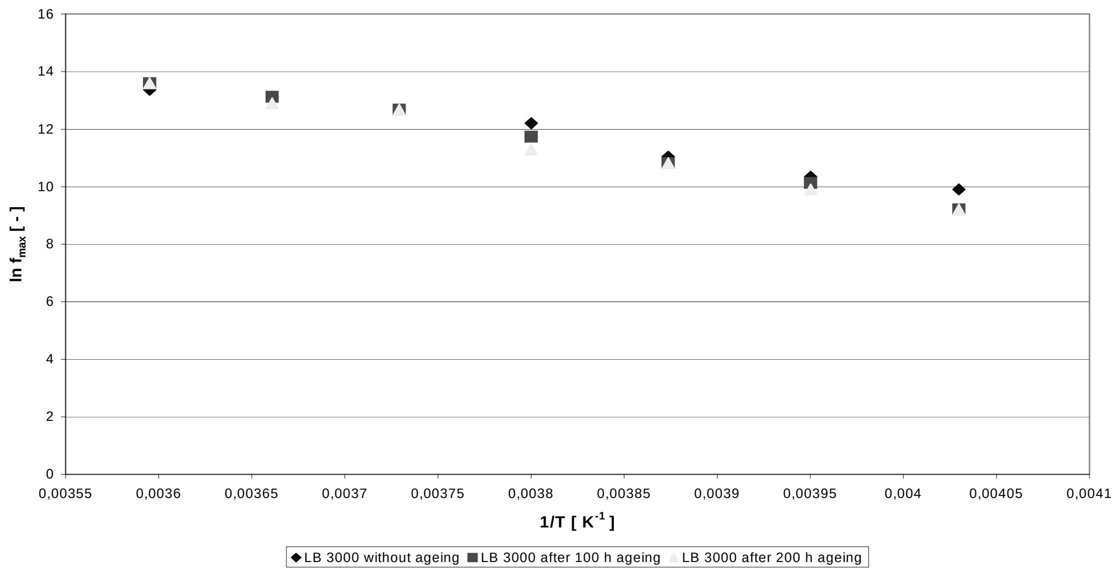

Fig. 3: *Relaxation map of LB 3000 sample*

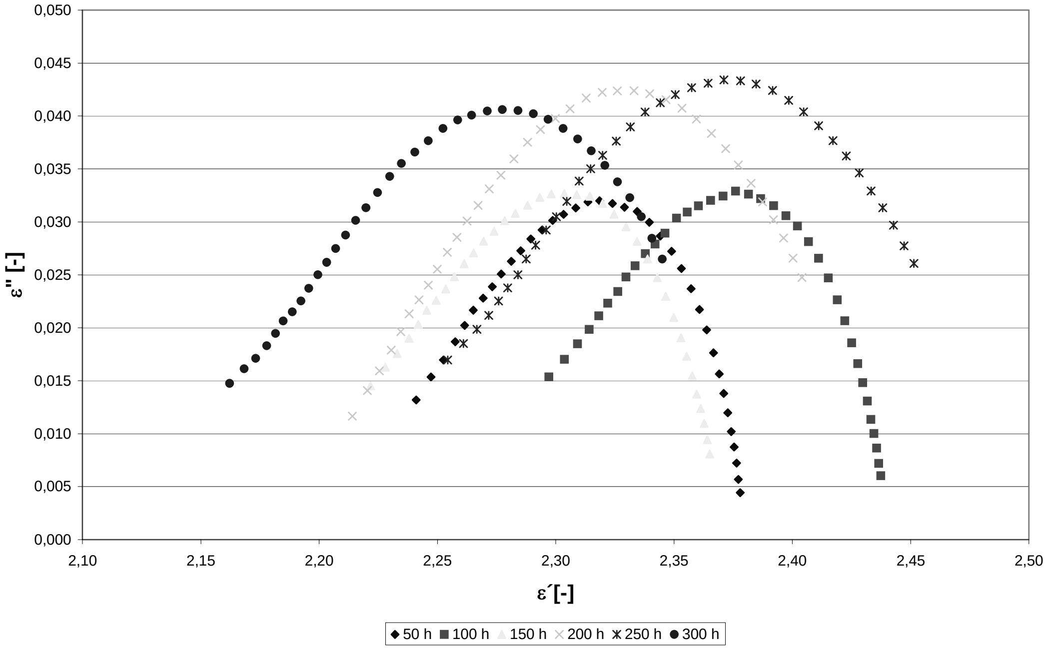

Fig. 4: *Cole-Cole diagrams of LBH 3000 sample on temperature -15°C*

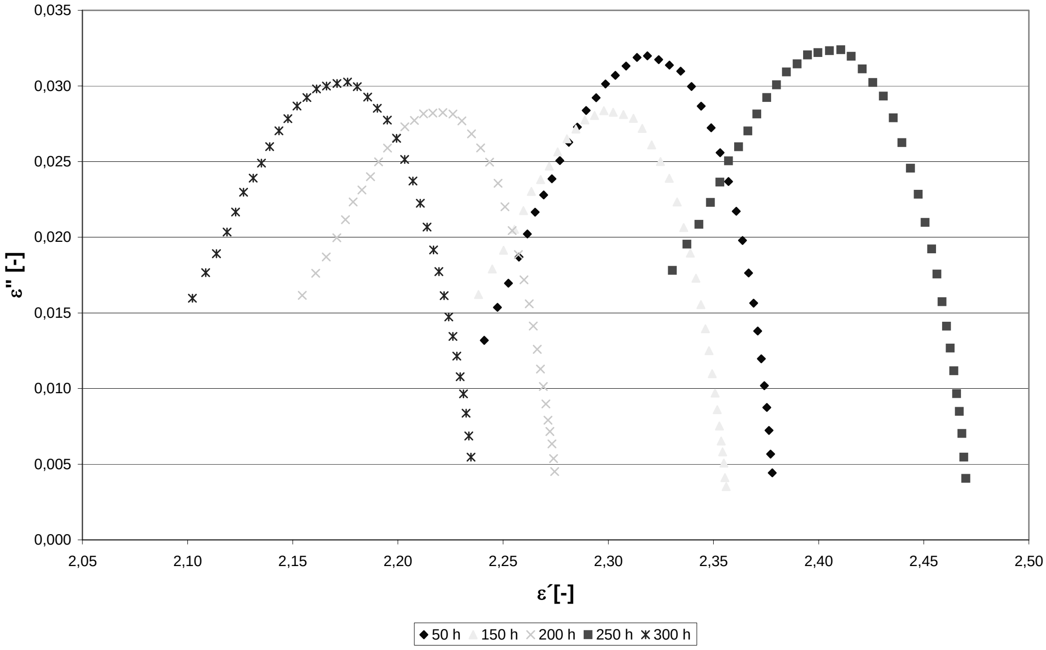

Fig. 5: *Cole-Cole diagrams of LBH 3000 sample on temperature -15°C*

5 CONCLUSION

Dielectric relaxation spectra of oligobutadiene LBH and LB were measured in the frequency range from 100 Hz to 1 MHz and in the temperature range from 20 °C to –25 °C.

The measurements were made on three kinds of samples. The first set of them included samples without any aging. The others were after temperature aging on 130 °C in a temperature chamber. The aging time was 100 a 200 hours. The changes in dielectric properties are shown in fig. 4 and 5. The loss number increases and the maximum relaxation frequency is going down with the decreasing temperature. Experimental work with the sample of oligobutadiene has brought the next results:

- It can be observed α type relaxation mechanism throughout defined temperature and frequency range.
- α type of relaxation mechanism corresponds with turning whole chain of hydroxyled oligobutadiene.
- The comparison of $\varepsilon'' = F(\varepsilon')$ for samples LBH 3000 and LB 3000 has discovered, that the hydroxyled oligobutadiene has higher level of dielectric loss and has wider range of frequency dispersion.
- Hydroxyled oligobutadiene has higher activation energy.

Experimental work with the oligobutadiene has been still going on and its results will be published continuously.

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