

RELAXATION EFFECTS IN DIELECTRIC SPECTRA OF OLIGOMER BUTADIENE MATERIALS AT CONSTANT TEMPERATURE AND TIME DEPENDENCE

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ABSTRACT

The object of this research is to measure and to analyze dielectric spectra of oligomer of butadiene. The analysis and subsequent interpretation is based first on detailed observation of frequency dependencies of relaxation processes and second on the comparison of dielectric measurements made at constant temperatures (-25 °C) and at various times.

1 DIELECTRIC RELAXATION SPECTROSCOPY

The purpose of our research is to investigate dielectric relaxation spectra, which necessitated the selection of the appropriate analytical method for the research of material characteristics. One of the available experimental methods is dielectric relaxation spectroscopy (DRS). In general, DRS studies molecular dynamics of charge carriers and dipoles and it consists of the set of theories and methods using this dynamics for experimental research. DRS can be used to observe many different material systems, from gases to different sorts of solid substance. This research is engaged in DRS of oligomers of butadiene. Surveyed sample is analyzed and evaluated in the frequency domain, at constant temperatures (-25 °C) and various times.

2 SAMPLES

The subject of experimental research and consequential analysis of dielectric spectra has been commercially available hydroxyl-oligobutadiene. Oligobutadiene belongs to synthetic rubbers, which are matters containing dual bonds determining their characteristics.

3 PHYSICAL AND CHEMICAL CHARACTERISTICS

In the ordinary way and at room temperature, oligomers of butadiene are a clear, colorless till yellowish viscous liquid, which is non-miscible with water and alcohols. However it is well miscible with non-polar organic liquids, oils and pitches. Oligobutadiene is

soluble easily in some different solvents and appertains to unsaturated alkaline hydrocarbons, which contain the (OH) functional group.

4 APPLICATION

Liquid oligobutadiene is used primarily in the production of polyurethane. Oligobutadiene containing polyurethane exhibits an excellent hydrolysis resistance. These polyurethanes are also highly elastic, provide excellent insulating qualities for the electric current and leak very little moisture. Utilization of liquid oligobutadiene in practice is presented in [2].

5 MEASURING INSTRUMENTS

During the experiment use has been made of the equipment for the measurement in the frequency domain. Measurements in the frequency domain were carried out by the Hewlett Packard HP 4284A precision LCR meter and dielectric test fixture capacitor HP 16451B. Measuring with the HP precision LCR meter is based on bridge techniques with auto-calibration; measured results are available over the frequency range 100 Hz to 1 MHz.



Fig. 1: *Equipment for the measurements in the frequency domain*

6 EXPERIMENTAL DETAILS

In our laboratory we examined dielectric properties of oligobutadiene. Samples of oligobutadiene supplied were at the room temperate very viscous so that a small amount of

oligobutadiene was expelled from a syringe in the form of a droplet onto the bottom electrode and then the top electrode was screwed down, so that the droplet flowed away and formed a dielectric layer between both electrodes. The distance between electrodes (sample thickness) was kept constant at 10 μm by glass fibers. An important issue was the selection of the sufficiently small amount of oligobutadiene.

7 EVALUATIONS

Loss (imaginary part of the complex permittivity) versus frequency characteristics $\epsilon'' = f(\omega)$ were measured and evaluated experimentally for the constant temperatures and various times. With the help of dielectric relaxation spectroscopy method we obtained the results, which were subsequently analyzed. The data analysis has substantiated the appearance of relaxation processes in the sample of oligobutadiene. Figure 2 shows the relation between the loss number and $\log f$. [Tb0m] is the start time, i.e. immediately after the sample has been inserted in to the refrigerator, we can see that the onset of the decrease of imaginary part of the complex permittivity is decreasing with increasing freezing time. As much as the freezing time increases the relaxation frequency is decreasing. Figure 3 shows the relation between the real number and $\log f$ and confirms the finding obtained from the imaginary part: As much as the freezing time increases the relaxation frequency is decreasing and in other way as much as the sample is getting close to solid state the orientation of dipoles is getting more easier.

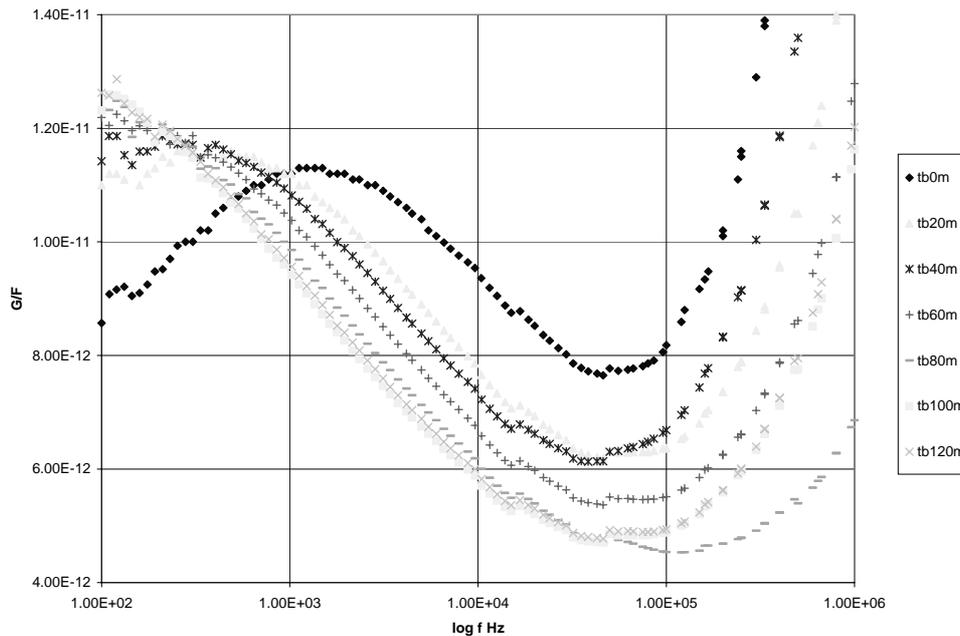


Fig. 2: *The relation between the G/F and the log f*

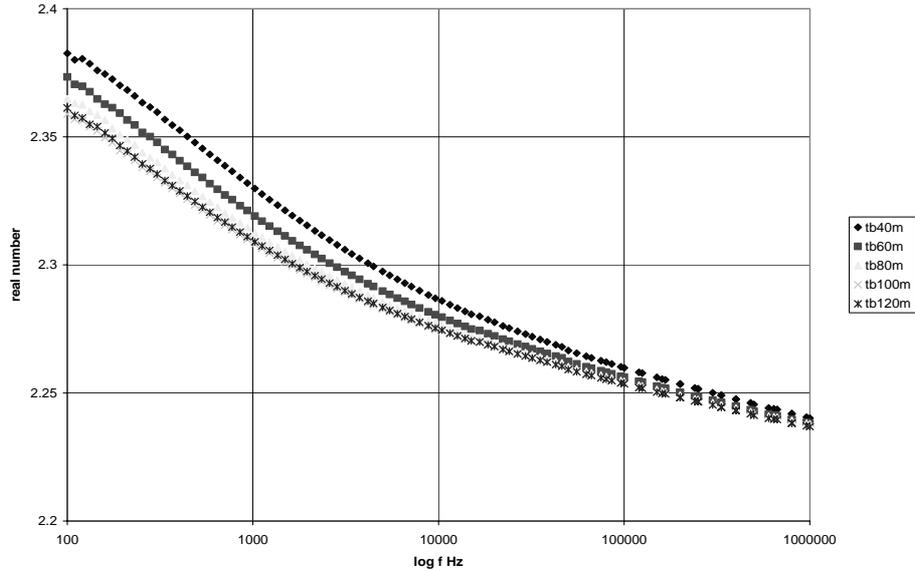


Fig. 3: *The relation between the real number and the log f*

8 CONCLUSION

Dielectric relaxation spectra of hydroxylated oligobutadiene were measured in the frequency range from 100 Hz to 1 MHz in the constant temperature and various times. The measurements might be interpreted as the evidence of the presence of at least one relaxation process right side. As much as the freezing time increases the relaxation frequency is decreasing and in other way as much as the sample is getting close to solid state the orientation of dipoles is getting more easier. Experimental work with the oligobutadiene will go on and its results will be published continuously.

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REFERENCES

- [1] Hedvig, P.: Applied Polymer Analysis and Characterization. Volume II, 1991, Hanser, New York
- [2] Oligobutadiene production manual. <http://www.kaucuk.cz/>
- [3] Bottcher, C. J. F., Bordewijk, P.: Theory of Electric Polarization. Volume I: Dielectric in static fields, 1973, Elsevier, Now York
- [4] Havriliak, S., Havriliak, S. J.: Dielectric and Mechanical Relaxation in Materials. 1997, New York