RELAXATION EFFECTS IN DIELECTRIC SPECTRA OF OLIGOMER MATERIALS

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ABSTRACT

The object of this thesis is to measure and to analyse dielectric spectra of oligomer materials. The analysis and subsequent interpretation is based on detailed observation of temperature dependencies of the relaxation processes in different frequency range.

1 DIELECTRIC RELAXATION SPECTROSCOPY

It's necessary select appropriate analytical method for a research characteristics of materials. Ones of the modern experimental methods is dielectric relaxation spectroscopy (DRS). In general, DRS studies molecular dynamism of current carrier, respectively dipoles and is created by set of theories and methods using to experimental research those dynamism. This thesis is engaged in DRS of oligohydroxibutadiene. Surveyed sample is analyzed and evaluated in the frequency domain by the different temperatures.

2 SAMPLE

The subject of experimental research and consequential analysis of dielectric spectra has been hydroxyled oligobutadiene LBH, made in Kaucuk Kralupy Inc., which producer sells with trademark KRASOL LBH. Oligobutadiene belongs to synthetic rubbers, which are matters containing dual bindings determining their characteristics.

2.1 PHYSICAL AND CHEMICAL CHARACTERISTICS

In the ordinary way, hydroxyled oligobutadiene is clear, colourless till yellowish viscous liquid, that is non-miscible with water and alcohols. However it's well miscible with non-polar organic liquid, oils and pitches. Oligobutadiene is soluble easily in some different solvents and appertain to unsaturated alkaline hydrocarbons, which contain functional group

OH. It is produced by using polymeric reaction.

2.2 APPLICATION

Liquid oligobutadiene KRASOL LBH is used by production of polyurethane. Oligobutadiene's polyurethanes are characteristic its 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 KRASOL LBH in practice is presented in [2].

3 EQUIPMENT FOR THE MEASURING IN THE FREQUENCY DOMAIN

Measurements in the frequency domain were carried out by measuring on the Hewlett Packard HP 4284A precision LCR meter and dielectric test fixture capacitor HP 16451B. Measuring with the precision LCR meter is based on bridge techniques with auto-calibration and its measured results are capable over the frequency range 20 Hz - 1 MHz. It is necessary to carry out corrections before each measuring to avoid errors during the measuring. In this case were corrected errors caused by cable lenght, thus self-impedance and admittance between the wires. The sample was analysed in the temperature chamber, which was for this experiment made.

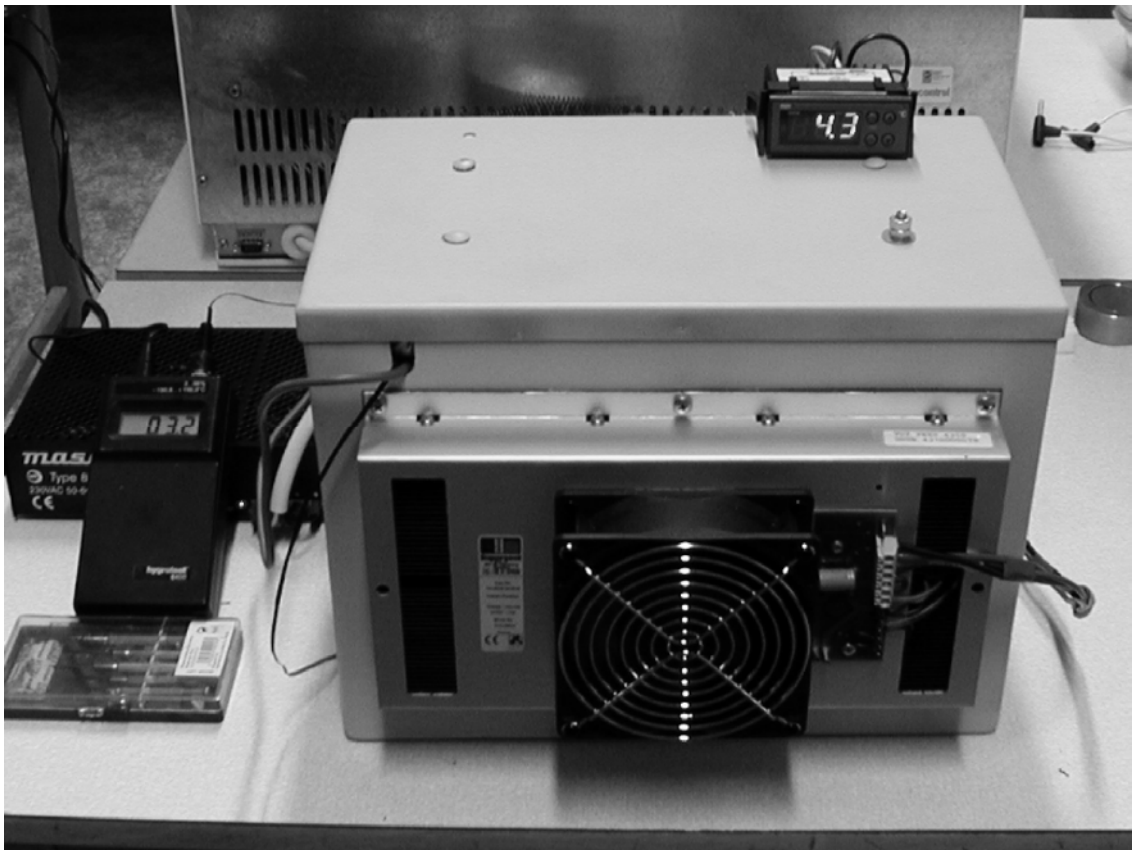

Fig. 1: *Made temperature chamber for experiment*

4 EXPERIMENT EVALUATION

The characteristics ε"=F(f) were measured and evaluated experimentally for the temperatures in the range from –25 °C to 60 °C. With the help of dielectric relaxation spectroscopy method were obtained the results, which were analysed. Data analysis has substantiated appearance relaxation processes in the sample of oligobutadiene. The relaxation map of the tested sample was putted together with usage measured functions ε"=F(f) for the temperatures lower than 20 °C, and decreasing to –25 °C. The relaxation maximums were moving towards lower frequencies with the decreasing temperature and that is why the relaxation maximums were perceptible. The rate of correlativity means cooperation parameter α, that slightly decreases with the increasing temperature. It means, that the orientation each of the molecules is less affected by others. The position of relaxation maximums matches with straight characteristic and therefore it was used to its description Arrhenius activation law with the calculated activation energy $W_0 = 33$ MJ/kmol.

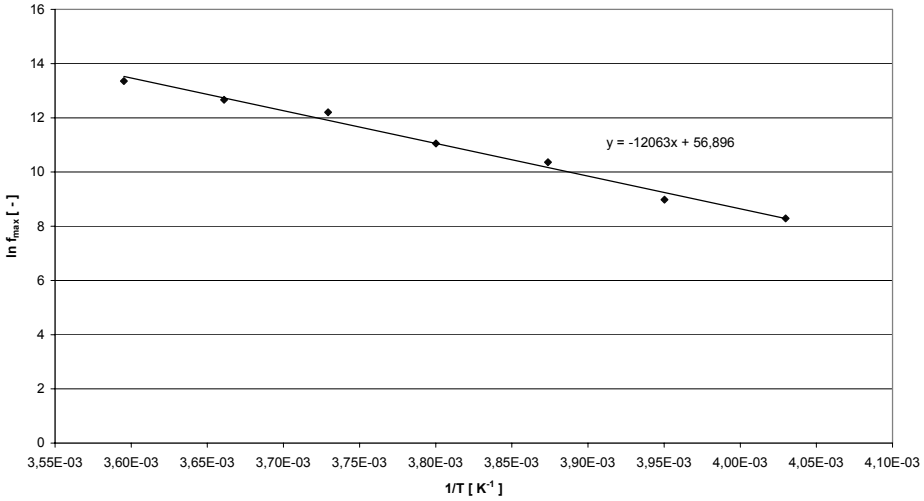

Fig. 2: *Reciprocal absolute temperature dependence of the ln f_{max} (relaxation map)*

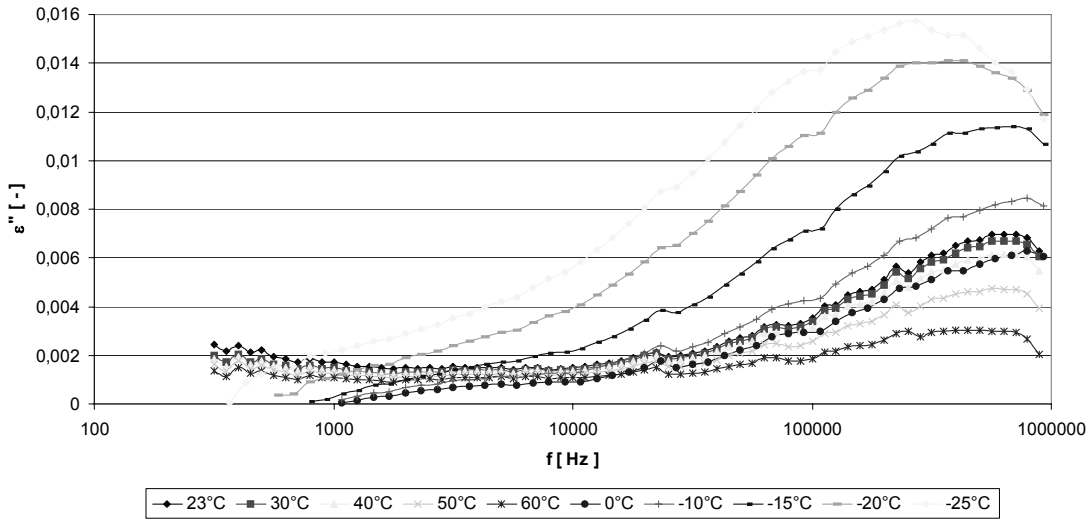

Fig. 3: *Frequency dependence of the loss number ε" for the butadiene LBH 3000 sample without the time exposition*

During the run of experiment was observed, that the relaxation is depended on the temperature and the temperature's operating time. By the two hours operating time of temperature, the relaxation process came out up to –15 °C and by the thermal incidence on the sample for the 24 hours, relaxation process was perceptible only to 0 °C. This phenomenon can be explained by transition amorphous phase of oligobutadiene sample to crystalline phase of oligobutadiene, in which rise crystals and disappears relaxation mechanism. By the lower temperatures under the same conditions only electric conductivity of oligobutadiene sample is appearing.

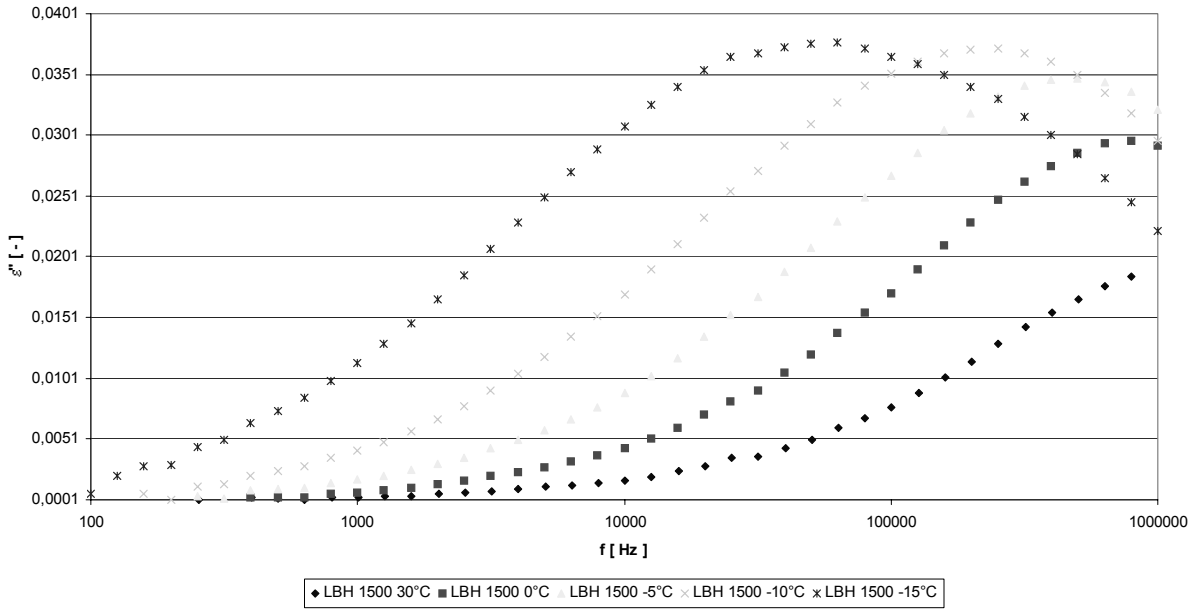

Fig. 4: *Frequency dependence of the loss number ε" for the butadiene LBH 1500 sample without the time exposition*

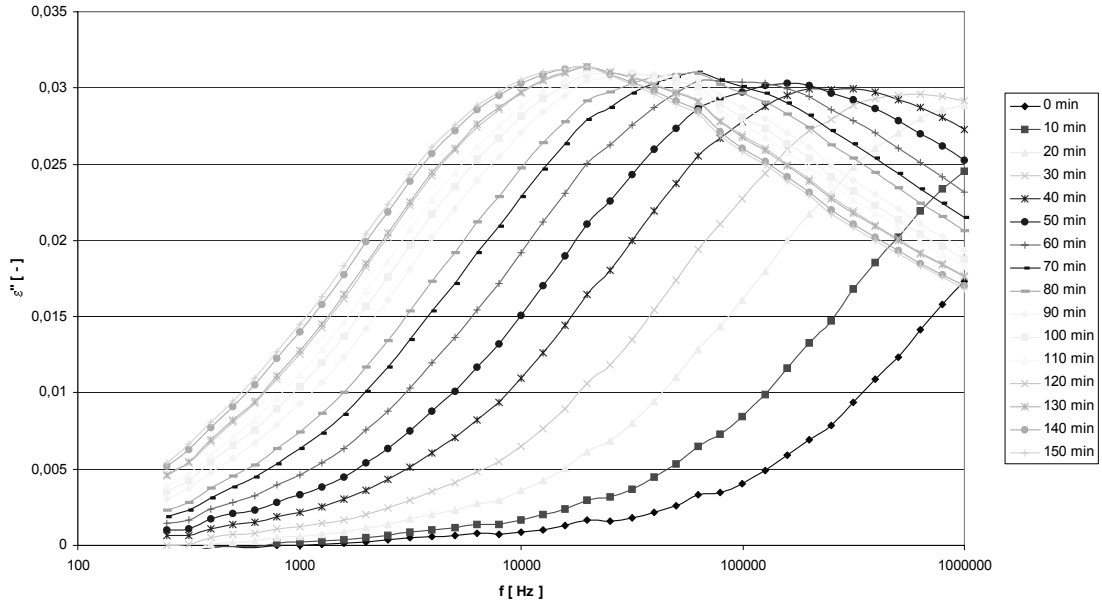

Fig. 5: *Frequency dependence of the loss number ε" for the butadiene LBH 3000 sample by –15 °C with the exposition time*

5 CONCLUSION

Dielectric relaxation spectra of hydroxyled oligobutadiene LBH were measured in the frequency range from 20 Hz to 1 MHz. In the frequency domain were measured dependencies of imaginary part of complex permittivity in the frequency range from 20 Hz to 1 MHz by temperature limit from –25 °C to 60 °C. Experimental work with the sample of oligobutadiene has brought the next results:

The rate of cooperation – cooperation parameter α HN function slightly decreases with the increasing temperature.

It can be observed β type relaxation mechanism throughout defined temperature and frequency range. β type of relaxation mechanism corresponds with turning whole chain of hydroxyled oligobutadiene.

The value of the activation energy is 33 MJ/kmol.

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

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