

DIELECTRIC PROPERTIES OF OLIGOMERS MATERIAL

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ABSTRACT

The object of this research is to measure and to analyze dielectric spectra of oligomer of butadiene. The measurement of dielectric constant and dielectric loss was carried out over the frequency range 100 Hz to 1 MHz at temperatures of 10 °C to -25°C. The analysis and subsequent interpretation is based on detailed observation of temperature dependencies of the relaxation processes.

1 DIELECTRIC RELAXATION SPECTROSCOPY

The purpose of my research is to measure the dielectric relaxation spectra of oligobutadiene. usually in the form $\epsilon'' = \epsilon''(f)$. In general, dielectric relaxation spectroscopy studies molecular dynamics of current carriers, respectively dipoles. Dielectric relaxation spectroscopy can be used to observe several material systems, from gas to different sorts of solid substance. Dielectric spectra exhibit generally local maxima, which are associated with individual relaxation processes and at low frequencies – hyperbolic section, which are due to conduction processes. Relaxation maxima may be analyzed in terms of the maximum height $[\Delta\epsilon_i = (\epsilon_s - \epsilon_\infty)_i]$, timescale of the relaxation (position on the frequency axis, τ_i) and the shape of the maxima (symmetry, width). Most frequently relaxation maxima are mathematically described by (Havriliak – Negami equation which in the complex number notation reads as follows

$$\hat{\epsilon}(\omega) = \epsilon_\infty + \frac{\epsilon_s - \epsilon_\infty}{[1 + (j\omega\tau)^\alpha]^\beta}$$

However if the above expression is separated into the real and imaginary part the formula for ϵ'' (imaginary part) is much more complex:

$$\epsilon''(\omega) = (\epsilon_s - \epsilon_\infty) \frac{\sin \beta\varphi}{\{1 + 2(\omega\tau_0)^\alpha \sin \frac{1}{2}(1 - \alpha)\pi + (\omega\tau_0)^{2\alpha}\}^{\beta/2}}$$

In analogous manner the conductivity component is usually best described by hyperbolic formula: $\epsilon'' = A \cdot \omega^{-n}$

In the case $n = 1$ corresponds to the standard DC conductivity of the material (free charge carriers). [3]

2 SAMPLES

The subject of experimental research and subsequent analysis of dielectric spectra has been hydroxyl-oligobutadiene LBH, made in Kaucuk Kralupy Inc., sold under the trademark KRASOL LBH. Oligobutadiene belongs to synthetic rubbers, which are matters containing dual bonds determining their characteristics.

2.1 PHYSICAL AND CHEMICAL CHARACTERISTICS

Oligomer of butadiene is a clear, colorless till yellowish viscous liquid, which is non-miscible with water and alcohols. However oligobutadiene is well miscible with non-polar organic liquids, oils and pitches. Oligobutadiene is easily soluble in some different solvents and appertain to unsaturated hydrocarbons, which contain functional group OH.

2.2 APPLICATION

Liquid oligobutadiene KRASOL LBH 3000 is used in the production of polyurethane. Oligobutadiene polyurethanes exhibit excellent hydrolysis resistance. These polyurethanes are also highly elastic, provide excellent insulating qualities for the electric current and leak very little moisture.

3 MEASUREMENTS

Measurements 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. Results are available over the frequency range 100 Hz to 1 MHz. The experimental setup is shown in figure (1).



Fig. 1: Equipments for the measurements in the frequency domain.

It is necessary to carry out corrections before each measuring to avoid errors due to cable length, self-impedance and admittance between the wires. Samples of oligobutadiene supplied were at the room temperature very viscous therefore in order to prepare a sample a droplet of oligobutadiene was expelled from a syringe in the form of a droplet to the bottom of electrode, then the top of electrode was screwed down, so that the droplet flowed away and formed a dielectric layer between both electrodes. The distance between samples was kept constant 30 μm by glass fibers. An important issue was the selection of the sufficiently small amount of oligobutadiene.

4 RESULTS AND EVALUATION

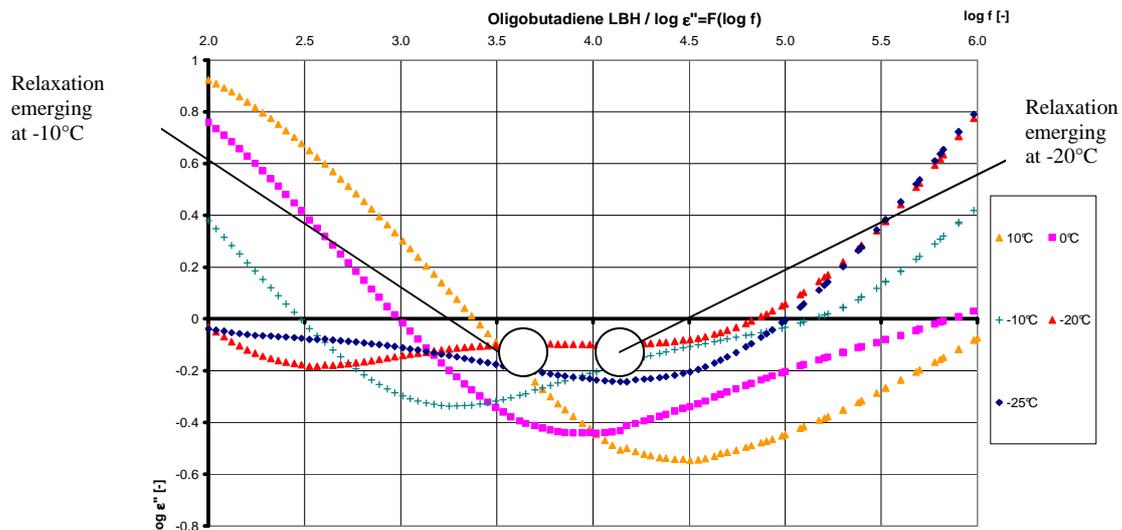


Fig. 2: Relation between the log loss number and the log frequency.

Figure (2) shows (loss number ϵ'' part of the complex permittivity). The figure includes the measured functions $\epsilon'' = F(f)$ for temperatures 10°C, 0°C, -10°C, -20°C and decreasing to -25°C which measured after 24hr. At 10°C the plot exhibits the V-type ship which is interpreted as the presence of two relaxation maxima one at the left side and the other one on the right-hand side, (physically the increase of the loss number is always outright by its decrease so that the increase can only be due to the presence of a relaxation maximum). At lower temperature both maxima move to the lower frequencies. The right-hand side relaxation process shifts from the high-frequencies side to the low frequencies side and becomes unsymmetrical at the temperatures below -10°C. The lack of symmetry might be due to the presence of another relaxation process which is can be seen at temperatures -10°C and -20°C (see inset circles on the figure2). The relaxation process at the left hand side also shifts with the decreasing temperature to the left so that at the -20°C it is almost completely disappears from the experimental frequency window there is not much change of the relaxation spectrum at the temperatures -25°C when the sample was kept at this temperature for 24 hours.

5 CONCLUSIONS

Dielectric relaxation spectra of hydroxylated oligobutadiene were measured in the frequency range from 100 Hz to 1 MHz in the temperature range from 10 °C to -25 °C. The measurements might be interpreted as the evidence of the presence of at least one relaxation process right side, perhaps two at lower temperatures below -10°C. The slope of the relaxation maxima at the room temperature (not shown here) has the value of (0.48) which indicates a partially cooperative character of dipole motions. At decreasing temperature the relaxation process shifts from the high frequency side to the low frequency side and becomes unsymmetrical as the experimental temperature decreases. The lack of symmetry might be due to the presence of either another relaxation process or to the onset of DC conductivity, which is seen at room temperature

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