MEASURING OF RELAXATION TIMES OF NATRIUM IONS BY MAGNETIC RESONANCE METHODS

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Introduction

Nuclear Magnetic Resonance (NMR) experiments enable describing of the structure and the internal mobility of a wide spectrum of materials and systems from solutions, very soft and movable gels over synthetic polymers to organic and inorganic crystals and very hard rigid glass.

The paper deals with a study of the relaxation times of the nearly liquid and gel electrolytes with content of natrium ions by the NMR technique. Newly designed method of solid electrolytes samples compounding and a suitable method for measurement of the relaxation times during the samples solidification were established.

Experimental

The relaxation times T_1 and T_2 of ^{23}Na nuclei in the gel electrolytes and their changes during the gel solidification were experimentally measured. Relaxation time T_1 was measured by the Inversion Recovery and the Saturation Recovery techniques application. On the other hand, the relaxation time T_2 was measured by use of the Spin Echo (SE) method and established from half-width of spectral line according to

$$T_2 = \frac{1}{\pi \cdot \Delta \nu},$$

where Δu is the half-width of the spectral line measured.

The gel is based on polymethylmethacrylate (PMMA). Its preparation consists of mixing three convenient components

- liquid monomer methylmethacrylate (MMA 99%),
- solid oligomer, including an initiator and a matter for polymer netting,
- optional components ensuring conductivity of the ions 0,5 ÷ 1,5 M solution of waterless salt as sodium perchlorate in waterless propylenecarbonate (PC 99,7%).

The use of $NaClO_4$ salt appeared to be the best. Due to its good solubility in propylenecarbonate the 0,5 M, 1 M and 1,5 M solution could be concocted.

Table 1 *Saturated solution of $NaClO_4$*

Electrolyte	Saturation (mol)	Content of PC (ml)	Content of salt (g)
$NaClO_4$	0,5	25	3,061
	1,0	25	4,591
	1,5	25	6,122

Results and discussion

The designed methods were experimentally tested on a MR tomograph at the premises of ISI ASCR, Brno with 4,7 T induction of the basic magnetic field, nuclei resonance frequency 200 MHz. The nuclei of ^{23}Na resonate on 51 MHz frequency. The specimen was a glass phial of 11 ml cubature filled by either 4 ml of liquid electrolyte, or 10 ml gel electrolyte. Data obtained in both time and frequency domains were processed in the MATLAB program.

First, the sample of liquid $NaClO_4$ with 1M saturation was measured under conditions described above. 24 hours after measurement the gel electrolyte was mixed.

In Fig. 1 the relaxation times T_1 and T_{12} in dependence on the gel ageing are illustrated. Relaxation time T_{12} was established by a more accurate approximation by two exponential functions.

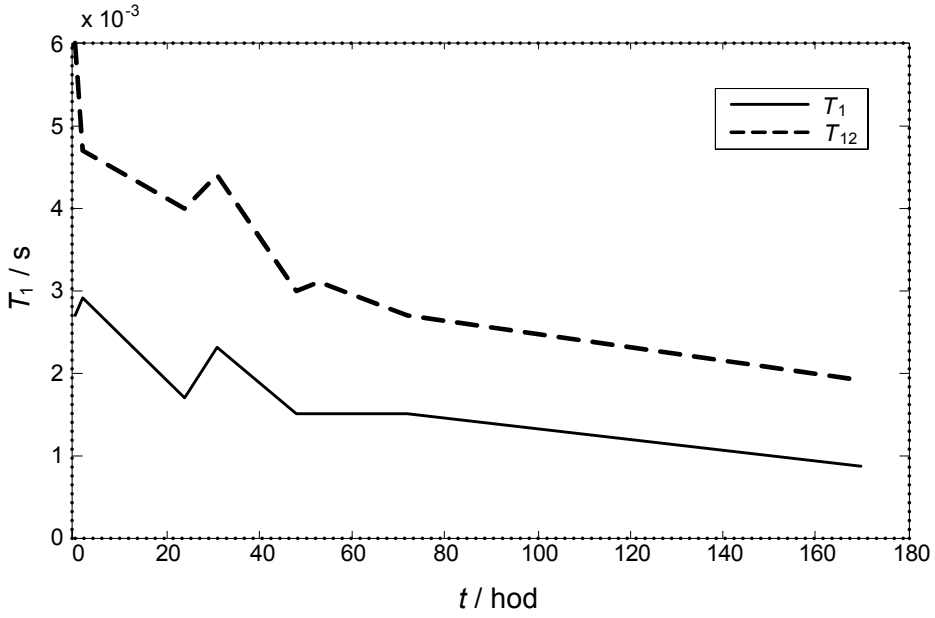

Fig. 1 *T_1 and T_{12} relaxation times as a function of gel aging.*

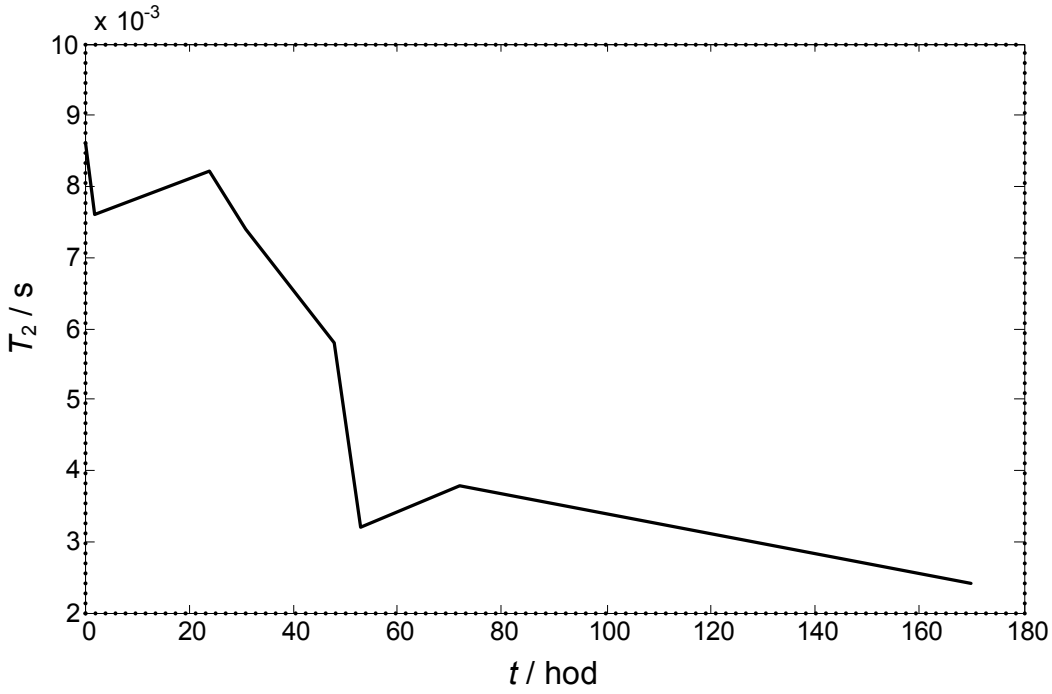

Fig. 2 *Relaxation time T_2 as a function of gel aging.*

From the experimental measurement arise the following conclusions:

- Relaxation times of the liquid electrolyte are in ms. For 1 M concentration they are T_1 = 3 ms and T_2 = 8 ms.
- By better approximation of data measured with two exponential functions it is T_1= 5 ms, physically it corresponds to two groups of nuclei with different relaxations, or to systematic errors.
- During the gel electrolyte solidification lasting 160 hours, the relaxation times decreases to values T_1 = 1 ms, and T_2 = 2 ms. This decrease is not steady still, but due to very low S/N ratio of FID signal it was not possible to measure for a longer time interval.
- The sensitivity of the measurement is not high because of the low S/N ratio neither for 50 signal accumulations. In time domain is S/N ratio 1, in frequency domain 5.
- The relaxation times for liquid and gel electrolyte were the same immediately after the addition of a hardener.

Conclusions

Experimental results indicate the main problem in the relaxation times in gel electrolytes measurement. It consists in very low S/N ratio and their small values, simultaneously; the problem is in the approximate equality of both relaxation constants. The measurements performed are the basis for additional experimental work.

Acknowledgements

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