

^7Li NMR Studies of Li^+ Insertion in V_2O_5 Xerogels

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Vanadium pentoxide has been studied for decades because of its ability to reversibly intercalate Li^+ . The xerogel (XRG) form of V_2O_5 is particularly interesting due to its ability to intercalate up to 3.3 mol lithium/mol V_2O_5 XRG.¹ Recently, there has been considerable effort in the use of sol-gel methods to rationally design new cathode materials. These layered materials have shown superior electrochemical properties such as efficient cycling² and high specific energy (1137 Wh/kg).³ The xerogel form of V_2O_5 is easily synthesized through sol-gel methods⁴⁻⁷ with the resulting product being described as an amorphous, fibrous-like material with short-range order.⁴ ^7Li NMR has been used previously to study Li^+ insertion in vanadium oxides.⁸ It has been shown that the NMR data can be used to characterize the different Li^+ environments in the material, as well as other materials characteristics. We describe here a ^7Li solid state NMR study of Li^+ insertion in such materials, with emphasis on use of the NMR results in combination with other experimental results to understand how the microscopic structure of the material influences its electrochemical behavior.

Lithium can be inserted into the xerogel host material *via* electrochemical reduction at various potentials in a LiClO_4 propylene carbonate electrolyte. Following this, the samples are rinsed, dried at 100 °C on a high vacuum line and transferred into an Ar-filled glove box. NMR rotors are loaded and sealed in this inert atmosphere environment.

Figure 1 shows the MAS spectra of several $\text{Li}_x\text{V}_2\text{O}_5$ xerogel samples at various levels of Li^+ insertion (i.e. various x values) ranging from 0.03 to 0.98. Two resonances are resolved: a narrow resonance near 0 Hz and a broader resonance shifted upfield. The narrow resonance is relatively invariant with x value. The broader resonance increases in intensity and shifts upfield as x increases. We also observe a trend in the d -spacing for these samples (as obtained from X-ray diffraction) with the x -value. This trend will be correlated with the NMR results. The interpretation of these data will be presented.

REFERENCES

1. H.K Park, W.H. Smyrl, and M.D. Ward, *J. Electrochem. Soc.*, **142**, 1068 (1995).
2. K. West, B. Zachau-Christiansen, T. Jacobsen, and S. Skaarup, *Electrochim. Acta*, **38**, 1215 (1993).
3. D.B. Le, S. Passerini, A.L. Tipton, B.B. Owens, and W.H. Smyrl, *J. Electrochem. Soc.*, **142**, L102 (1995).
4. J. Livage, *Chem. Mater.*, **3**, 578 (1991).
5. M. Nabavi, C. Sanchez, and J. Livage, *Philosophical Magazine B*, **63**, 941 (1991).
6. P. Aldebert, N. Baffier, J. Legendre, and J. Livage, *Rev. Chim. Miner.*, **19**, 485 (1982).
7. J. Livage, *Mat. Res. Bull.*, **26**, 1173 (1991).

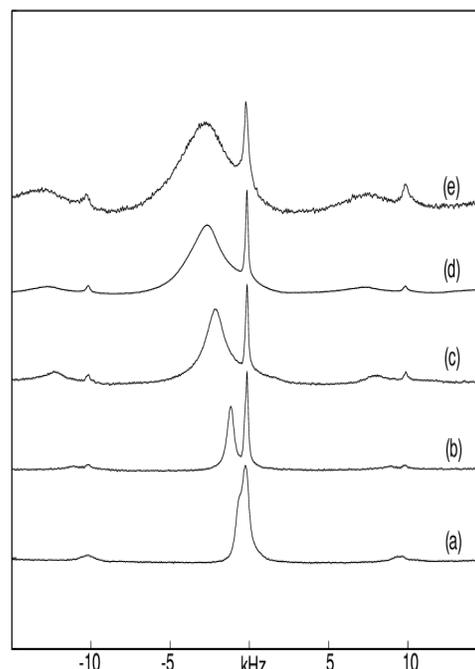


Figure 1. ^7Li NMR static spectra of $\text{Li}_x\text{V}_2\text{O}_5$, $x = 0.03$ (a), 0.17 (b), 0.55 (c), 0.84 (d), 0.98 (e).

8. P. E. Stallworth, S. Kostov, M. L. denBoer, S. G. Greenbaum and C. Lampe-Onnerud, *J. Appl. Phys.*, **83**, 1247 (1998).