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Enhanced Laboratory Methods for Shale Analysis using Low Field NMR Relaxometry

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Summary

Recent improvements in low-field nuclear magnetic resonance (LF-NMR) instrumentation have extended its application beyond measuring pore fluids to include assessment of highly viscous and even solid organic phases within reservoir rocks. Longitudinal (T_1) and transverse (T_2) relaxation responses are different for solids and liquids, therefore, the relationship between these two modes of relaxation can be used to differentiate organic phases in rocks or to characterize isolated organic materials. Using T_1-T_2 correlation data, organic components in shales, such as kerogen and bitumen, can be examined by laboratory relaxometry measurements. In addition, implementation of a solid-echo pulse sequence to refocus homonuclear dipolar couplings that affect T_2 values allows for improved resolution of solid phase protons.

Introduction

LF-NMR relaxometry is a non-invasive technique commonly used to assess hydrogen-bearing fluids in petroleum reservoir rocks. T_1 and T_2 relaxation time distributions are measured on reservoir rocks to obtain information on rock porosity and pore size distributions, as well as fluid types and saturations in some cases. This approach has found wide application in the study of sedimentary rock and unconsolidated porous media due to the robust nature and modest equipment demands of the methods. Improved instrumentation now allows for measurement of shorter relaxation times and assessment of fluids in very small pores and additional non-fluid hydrogen-bearing phases present in organic-rich shales. Application of LF-NMR relaxometry to the characterization of organic matter in shales is in the early stages of development and new techniques are currently being tested to improve the quality of results generated.

Methods

Measurements of T_1 and T_2 relaxation time distributions were carried out on three sets of dry samples from the Eocene Green River Formation: 1) raw oil shales, 2) samples of these shales processed by hydrous pyrolysis and techniques designed to mimic surface and in situ retorting, and 3) samples that have undergone low temperature ashing using oxygen plasma (60 °C) to remove organic matter. The pyrolyzed samples ranged in thermal maturity from bitumen and early oil generation through to depletion of oil generating potential. T_1-T_2 correlation data sets were acquired using methods combining the Carr-Purcell-Meiboom-Gill (CPMG) approach with spin and solid echo techniques for measuring T_1 and T_2 distributions simultaneously.

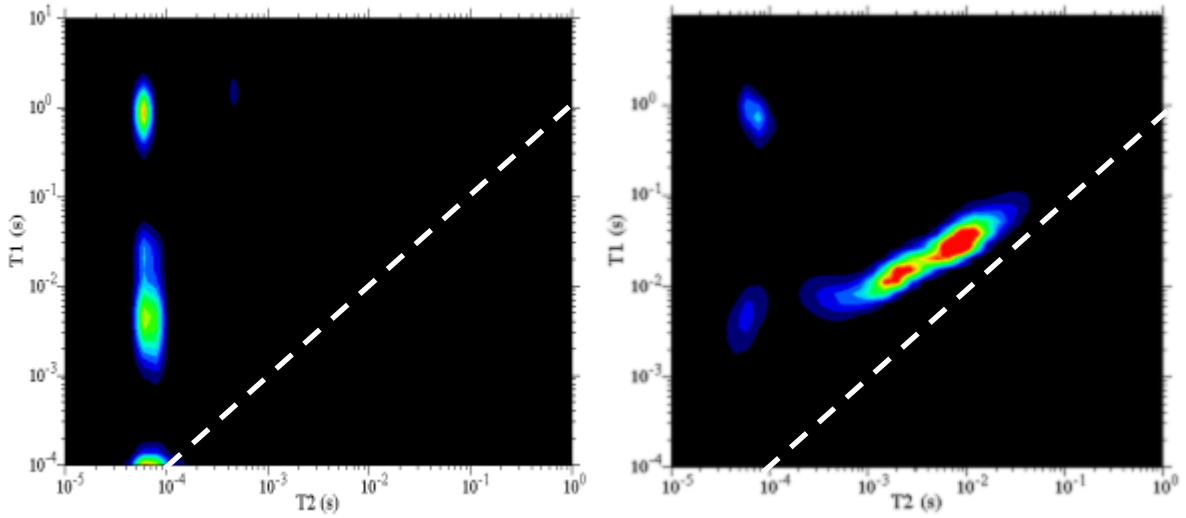

Figure 1: T_1-T_2 correlation plots for raw Mahogany zone oil shale (left) and Mahogany zone oil shale after pyrolysis at 360 °C for 72 hours.

Examples

Standard T_1-T_2 correlation plots (figure 1) revealed distinct peaks representative of solid and liquid organic phases present in the samples. The results on the pyrolyzed shales and ashed samples reflect changes occurring during processing (e.g., conversion of kerogen to bitumen) and peaks attributable to mineral phases, respectively. Solid-echo T_1 and T_2 measurements were used for improved assessment of the solid organic phases, specifically kerogen, thermally degraded kerogen, and char. Integrated peak areas from the LF-NMR results representative of kerogen and bitumen were found to be well correlated with S1 and S2 parameters from Rock Eval programmed pyrolysis.

Conclusions

This study demonstrates that LF-NMR relaxometry can provide a wide range of information on shales and other reservoir rocks that goes well beyond porosity and pore-fluid analysis.

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