Solid-State and Gas-Phase NMR Spectroscopy for Characterizing CO2 Uptake in Solids

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In this work we describe an experimental approach that combines solid-state and gas-phase nuclear magnetic resonance (NMR) measurements to probe gas uptake from two points of view. First, we demonstrate the utility of the method on a simple reaction between CO_2 gas and $Ca(OH)_2$. With vapor-phase NMR measurements, we observe the decrease in the CO_2 signal over time as it is converted into carbonate. This data is useful for determining the total amount of CO_2 uptake that occurs during the measurement. In addition, the vapor-phase measurements are rapid, taking tens of seconds, so details about the rate of the reaction are obtained at different temperatures and initial loading pressures. After these experiments are complete, the solid material is investigated using solid-state NMR. These measurements are useful to confirm the presence and amount of carbonate that is formed during the reaction. After discussing the experiments, we consider potential applications of the work, including monitoring CO_2, or other gas uptake, in industrially relevant materials such as polymers, cements, and concretes. Following this, we describe some of the advantages of this approach. For example, the NMR measurements described can distinguish between chemical and physical adsorption and sample conditions can be cycled to monitor desorption or capacity fade over time. We conclude with a discussion on the current challenges and pitfalls of the method, such as the necessity to work with ^{13}C labeled CO_2 to avoid long experiment times and the need to validate the method against more established analytical techniques.