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Project Summary

Development of Infrared Methods for Characterization of Inorganic Sulfur Species Related to Injection Desulfurization Processes

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The report gives results of using photoacoustic (PA) and diffuse reflectance (DR) detection methods in Fourier transform infrared spectroscopy (FTIRS) to evaluate the reaction of particulate CaO, $CaCO_3$, and $Ca(OH)_2$ samples with SO_2 at temperatures ranging from 25 to 900°C.

This Project Summary was developed by EPA's Air and Energy Engineering Research Laboratory, Research Triangle Park, NC, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Three Types of Studies

Photoacoustic (PA) and diffuse reflectance (DR) detection methods in Fourier transform infrared spectroscopy (FTIRS) were used to evaluate the reactions of particulate CaO, $CaCO_3$, and $Ca(OH)_2$ samples with SO_2 at temperatures ranging from 25 to 900°C. More specifically, this work involved three types of studies: 1) post-exposure FTIR-PAS (25-900°C); 2) *in situ* FTIR-DRS (25-700°C); and 3) *in situ* FTIR-PAS (25-400°C). The emphasis of this work was on determining the utility of FTIR-PA and FTIR-DR techniques for understanding the mechanisms by which SO_2 is captured by particulate samples of CaO, $Ca(OH)_2$, and $CaCO_3$.

Post-exposure FTIR-PAS experiments involved obtaining spectral measurements of particulate CaO, $CaCO_3$, and $Ca(OH)_2$ samples after they had been exposed to 1.5% SO_2 in He as a function of exposure time, temperature, and gas composition. These data indicate that : 1) the initial product formed is $CaSO_3$; 2) at temperatures above 500°C, $CaSO_3$ is converted to $CaSO_4$ predominantly through the reaction of $CaSO_3$ with SO_2; 3) additional $CaSO_4$ production, as well as the production of CaS_2O_3, occurs via $CaSO_3$ disproportionation; 4) the formation of $CaSO_4$ is not as extensive for exposures of 900°C as it is at 750°C; 5) in the presence of O_2, the production of CaS_2O_3 is reduced; 6) the products form as a thin layer on the surface of the 50-100μm particles; and 7) the extent of SO_2 capture by the reactants decreases in the order $Ca(OH)_2$ > $CaCO_3$ > CaO.

In situ FTIR-DRS experiments confirmed post-exposure FTIR-PAS results. In addition, the inherent sensitivity and *in situ* capabilities of FTIR-DRS enabled the detection and identification of: 1) two different $SO_3^=$ species and 2) an intermediate monodentate $SO_4^=$ species. FTIR-DRS also indicated that the formation of $S_2O_3^=$ depends on the concentration of CaO, $CaCO_3$, or $Ca(OH)_2$ in the NaCl diluent.

To be able to perform *in situ* high temperature FTIR-PAS measurements the development of a photoacoustic cell with elevated temperature and atmospheric control was necessary. The design of the cell is described. The

interaction of 1.5% SO_2 in N_2 with $Ca(OH)_2$, $CaCO_3$, and Na_2CO_3 as a function of time and temperature of exposure was monitored. Preliminary data, depicting the presence of physisorbed SO_2 on the $CaCO_3$ surface at 360°C, demonstrated the sensitivity of *in situ* FTIR-PA detection methods for both gas- and solid-phase reaction intermediates.

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The complete report, entitled "Development of Infrared Methods for Characterization of Inorganic Sulfur Species Related to Injection Desulfurization Processes," (Order No. PB 90-231-275AS; Cost: $23.00, subject to change) will be available only from:
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