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Silica Immobilized Thiophene Model Compounds As Calibrants For Reductive Pyrolytic Experiments

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Atmospheric Pressure Temperature Programmed Reductions (AP-TPR) can be used to determine sulfur functionality in solid materials, such as coal. However, AP-TPR needs reference materials to assign the H_2S profile to certain sulfur groups present in the initial sample. This can be achieved in two ways: one by using special synthesized model compounds or by sulfur selective treatments of real coal samples. A drawback of the latter method is that the selective treatment can cause structural changes in the sample, and thus will influence the environment of other sulfur forms present in the sample. Therefore, model compounds containing a thiophene functional group will be investigated by AP-TPR. These compounds will be covalently attached to a silica surface, through a Si-O-C_{AR} linkage, to prevent the premature sublimation of the compound.

In this investigation, AP-TPR coupled with mass spectrometry (MS) was used to study the hydrogenation and degradation behavior of silica-immobilized benzylthiophene and dibenzothiophene was investigated under the same condition that was used for analysis of real coal samples. For benzylthiophene, it was found that not only H_2S was formed under AP-TPR conditions, but also other volatile sulfur compounds such as thiophene and methylthiophene were formed. For dibenzothiophene, on the contrary, H_2S was the only volatile sulfur containing species found. The char and tar fractions

from the AP-TPR-MS experiments were also analyzed by an AP-TPO-MS experiment using an oxidative atmosphere. SO_2 was found in these experiments indicating that residue sulfur containing species were present, which were not released by hydrogenation in the AP-TPR experiment. For dibenzothiophene, the SO_2 profiles were more complex than for the benzylthiophene indicating a variety of sulfur-containing species were present.

Key Words: Thiophene, Model Compound, AP-TPR, sulfur characterization

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