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High sensitivity analysis of nitrogen in carbon materials using temperature-programmed desorption up to 2100 ℃

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Nitrogen-doped (N-doped) carbon materials are attracting attention in various fields, such as catalysis for oxygen reduction reaction. CHN elemental analysis and XPS were conventionally used for analysis of N species, but their measurement accuracy is about 0.1 wt%. Thus, a technique for detailed qualitative and quantitative analysis of N species is desired. Temperature-programmed desorption (TPD) has been popularly used for the analysis of inorganic catalysts and carbon materials. In conventional TPD, a sample is heated to typically around 1000 ℃ at a fixed heating rate and the desorbed gases are detected for chemical identification as a function of temperature. However, it has not been developed as a quantitative method for an effective determination of the N species, mainly due to the higher thermal stability of N in carbon materials than oxygen. Herein, we propose high-temperature vacuum TPD up to 2100 ℃ as a new high-sensitivity analytical method for N-doped carbons. In TPD result of N-containing mesoporous carbons, desorption of N-containing gases, NH3, HCN, and N2, was observed in a wide temperature range of 300-1900 ℃. The nitrogen content calculated from the total amount of desorbed gas was 7.9 wt%, which is in close agreement to the CHN analysis (8.0 wt%) and XPS analysis (8.5 wt%). Combined with the results of XPS analysis and DFT calculations, we found that the gas emission patterns of NH3, HCN, and N2 obtained by TPD can provide quantitative and qualitative information on the different types of N species including pyrrolic N, pyridinic N, and graphitic N. Furthermore, TPD analysis was performed on a sample with a very small amount of N content and showed a high quantitative accuracy of the order of 10 ppm (0.001 wt%).

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