Synthesis planning & reaction mechanism in ionic liquids

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Ionic liquids (ILs) have been used increasingly in the synthesis of inorganic particles for more than ten years. In the development of new syntheses, parameters such as reaction temperature and reaction time are often obtained by trial-and-error-methods. This procedure involves a lot of time and costs. In addition, the mechanisms involved in the course of the reaction are only examined in detail in individual cases.

Using a model system, which consists of the ionic liquid $[C_4C_1im]BF_4$ and the oxide SeO_2, as well as the additional reducing agent $NaBH_4$, a method is to be developed to derive suitable synthesis parameters with minimal effort. A screening with DSC has shown that with the addition of $NaBH_4$, the oxide SeO_2 could be completely reduced. However, it could also be shown that even without the addition of the extra reducing agent a complete reduction takes place. The time-dependent thermal stability – MOT [1–3] – of $[C_4C_1im]BF_4$ was investigated to determine whether the reduction was due to the IL itself or to decomposition products of the IL. The kinetic model of the MOT describes the temperature at which not more than 1% of the ionic liquid was thermally decomposed within a certain application time. The obtained time-dependent temperatures correlate with the onset of the exothermic effect of the DSC curve at 240 °C (β = 10 K·min^{-1}), which corresponds to the reduction of SeO_2. TGA-MS measurements were performed to determine which species is responsible for the reduction. It could be shown that the gaseous species NH_3, and especially B_2H_6, are probably responsible for the reduction. The obtained product could be identified as red amorphous selenium by XRD. A more detailed investigation by SEM has shown that amorphous red selenium consists of both irregular regions and spherical particles (diameter 20 µm).

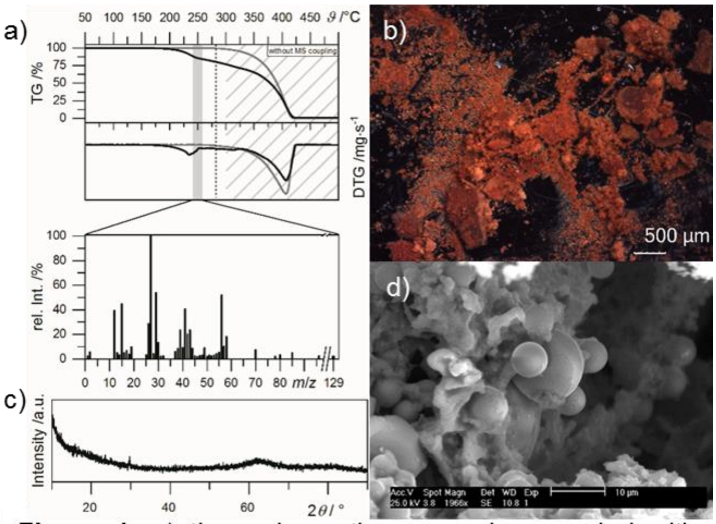

Figure 1. a) thermal reaction screening coupled with MS and b) light microscope image, c) X-ray diffractogram, d) SEM image of red amorphous selenium

The derivation of suitable synthesis parameters as well as the analysis of the reaction mechanism are obtained.

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