Material characterization down to ppm range by elemental analysis

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For the development and production of advanced technical ceramics, extensive quality control along the entire process chain is necessary. To achieve this, the qualification of the raw materials as well as the control of the whole manufacturing process up to the evaluation of the final product must be considered. Minor deviations in composition can result in a major impact on the material performance. Furthermore, there is an increasing interest in analysis of the material quality regarding purity to evaluate its suitability as a recycling product. The systematic use of chemical elemental analysis contributes to process reliability and helps to significantly reduce costs caused by errors.

With ICP-OES (inductively coupled plasma-optical emission spectroscopy), which uses inductively coupled plasma to determine elemental concentrations down to the ppm range by means of optical emission spectroscopy, process monitoring like this becomes possible. It can be used to characterize both raw materials and substances along the value chain and to analyze a large range of elements in the periodic table (Fig. 1). Additionally, elemental analysis can be extended by analysis of the element content of oxygen, carbon, sulphur, nitrogen and hydrogen by using an enhanced procedure.

For analysis using ICP-OES, at first the elements of interest must be determined. Afterwards, digestion of the substrate is conducted using a suitable method for conversion of the solid-state into liquid where it can then be analyzed.

By using ICP-OES, it is then possible to characterize the sample regarding the desired individual elements. Depending on the element, accuracies down to the lower ppm range can be achieved for most elements (Fig. 1).

In addition to applications in research, Fraunhofer IKTS can also support industrial customers in the quality assurance of large-scale manufacturing processes using ICP-OES elemental analysis. For instance, mixed metal oxides such as $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-d}$ (BSCF), $Ca_{0.5}Sr_{0.5}Fe_{0.2}Mn_{0.8}O_{3-d}$ or La-CoO_{3-d} that are frequently used to produce ceramic components for gas permeation and storage processes as well as for catalytic reactions especially depend on maintaining the exact

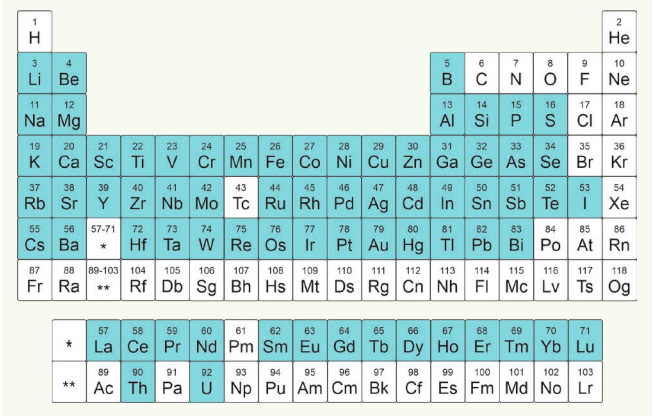

Fig. 1: Elements that could be analyzed by ICP-OES (blue).

stoichiometric ratios of the metal ions. Even small deviations from the desired composition, e.g. the Ba:Sr ratio in BSCF, can result in a significant influence on the functional properties, such as electronic and ionic conductivity. In addition, larger concentrations of foreign ions can also show a significant effect on the physical, mechanical and functional properties. They can be already contained in the raw materials (e.g. sulphur) or be brought into the material during the manufacturing process (e.g. Al, Zr). Therefore, it is highly relevant to monitor the quantities and types of impurities as well as the desired cation ratios of the produced oxides covering the entire process chain starting from the raw materials (Fig. 2). If there are any deviations, the manufacturing or recycling process can be adjusted to make targeted changes.

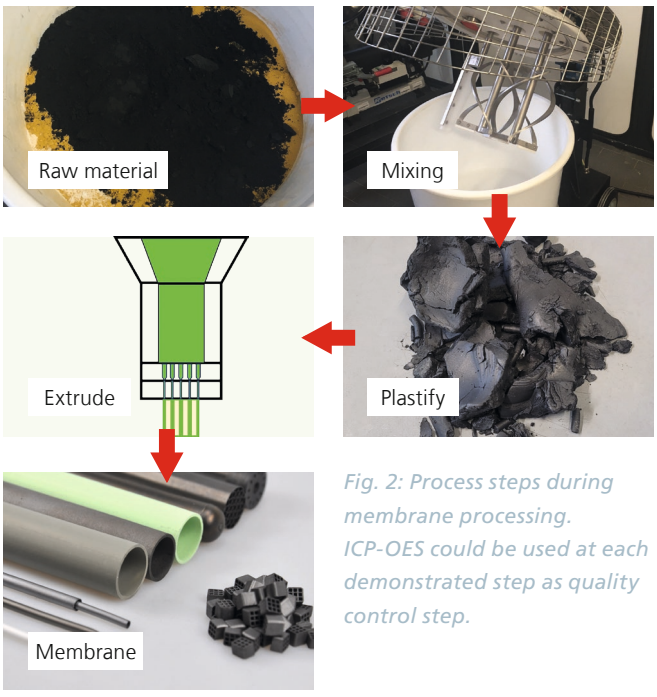

Fig. 2: Process steps during membrane processing. ICP-OES could be used at each demonstrated step as quality control step.

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