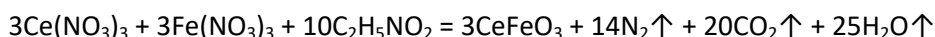


2. Experimental

2.1 Synthesis

Cerium orthoferrite nanopowder was synthesized by the solution combustion synthesis method using glycine as fuel. The following reagents were used as starting materials: cerium nitrate (III) $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ and iron nitrate (III) $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (chemically pure, Neva Reactive), taken in a stoichiometric ratio, and glycine or glycine acetic acid $\text{C}_2\text{H}_5\text{NO}_2$ (chemically pure, Neva Reactive), the amount of which was calculated relative to the amount of nitrate groups in the reaction solution (glycine to nitrate ratio or G/N) according to the glycine-nitrate combustion reaction:



The crystal hydrates of cerium (III) and iron (III) nitrates, as well as glycine, were completely dissolved in the minimum necessary amount of distilled water (40 ml) with constant stirring until a homogeneous reaction solution was formed. The resulting solution was heated in a glassy carbon flask until almost all of the water evaporated, forming a viscous gel-like product, which then self-ignited and burned to form a foam-like solid brown substance. The synthesized product by solution combustion was ground in an agate mortar to form a homogeneous powder and sent for physico-chemical and electrocatalytic testing.

2.2 Physicochemical characterization

The powder obtained as a result of the synthesis was investigated using a complex of physicochemical analysis methods. The elemental composition and morphology of the nanopowder were studied by energy-dispersive X-ray spectroscopy (EDXS) and scanning electron microscopy (SEM), respectively, using a Tescan Vega 3 SBH scanning electron microscope equipped with an Oxford INCA 200 energy-dispersive detector. The morphology of individual nanoparticles and their aggregates was studied by transmission electron microscopy (TEM) using a JEOL JEM2100F transmission electron microscope. The specific surface area and porous structure of the nanopowder were investigated by adsorption-structural analysis (ASA) using a Micromeritics ASAP 2020 surface area and porosity analyzer. Powder X-ray diffraction analysis (PXRD) of the sample was performed using a Rigaku SmartLab multifunctional X-ray diffractometer equipped with a $\text{CuK}\alpha 1$ radiation source, $\lambda = 0.154056$ nm. The average crystallite size was calculated based on the line broadening of X-ray diffraction lines using the fundamental parameters method implemented in the Rigaku SmartLab Studio II software package. Refinement of the elementary cell parameters was carried out using the Rietveld method implemented in the same software package. Vibrational modes of the sample were determined using Raman spectroscopy in the geometry of combined backscattering using a SINTERRA Raman microscope equipped with a laser emitter with an excitation wavelength of $\lambda = 532$ nm. The local environment of iron atoms in the sample was investigated by Mössbauer spectroscopy using a Wissel GmbH Mössbauer spectrometer 1104EM at a temperature of 300 K in the transmission geometry using a radioactive $^{57}\text{Co}(\text{Rh})$ γ -source.

2.3 Electrocatalytic test

The electrocatalytic and electrically conductive characteristics of the sample were investigated using cyclic voltammetry and impedance spectroscopy in a three-electrode electrochemical cell with an Elins P-45X potentiostat equipped with an FRA24M impedance measurement module. The working electrode was made by applying a suspension of the studied substance onto the surface of nickel foam, followed by air-drying. The suspension of the studied substance was prepared by ultrasonic treatment of nanopowder in a 0.5% Nafion solution in isopropyl alcohol for 30 minutes. A platinum foil and a silver chloride electrode were used as the auxiliary electrode and reference electrode, respectively. Measurements were

conducted in an alkaline aqueous electrolyte (1M KOH) at room temperature and atmospheric pressure. Electrochemical impedance spectroscopy (EIS) was conducted for the HER at an overpotential of -176 mV in the frequency range of 0.1 to 50,000 Hz. The alternating current voltage was 5 mV.