Supplementary Information

2.1. Electrochemical characterization

In the study, all electrochemical analyzes were performed with a potentiostat/galvanostat (Gamry Reference 3000) device. For electrochemical measurements, a conventional three-electrode system was used. This electrode system; It consists of working electrode (glassy carbon-GCE), counter electrode (platinum plate) and reference electrode (Ag/AgCl). Before the electrochemical measurements were made, the working electrodes were cleaned with alumina (Al_2O_3), ultrasonically treated, and then washed with methanol. The resistance measurements of the working electrode were optimized by measuring with a multimeter. Optimized electrocatalyst coating was modified by drop casting method. For the modification solution, 10 mg of NP powder, 500 µL of dH_2O, 37.5 µL of Nafion D-521 and 75 µL of DMF were added and sonicated for 30 min [32]. Then, a three-electrode system was established and cyclevoltammetry (CV),chronoamperometry (CA), 500 cycle, Scan Rate (SR), CO stripping and Impedance (EIS) measurements were performed. Measurements were made in a 1 M KOH solution containing 1 M CH_3OH in the potential range of −0.8 V to 0.2 V. SR measurements to see the activity of the catalyst at different scanning speeds; Scanning rates of 50 mV/s, 100 mV/s, 150 mV/s, 200 mV/s, 250 mV/s were performed in a 1 M KOH solution containing 1 M CH_3OH. At the same time, electrochemical impedance spectroscopy (EIS) measurements were made. EIS measurements were made in a 1 M KOH solution containing 1 M CH_3OH in the frequency range of 10 kHz–0.01 Hz.

2.5.Material structure

For the characterization of the obtained nanostructures, the functional groups and elemental characterizations of, Transmission Electron Microscopy (TEM) (Hitachi- HT780), X-Ray

Diffraction (XRD) (Rigaku Miniflex) devices, Scanning Electron Microscope (SEM-EDX mapping) (JEOL JSM-5600LV) were used in the analyses.