Influence of microwave synthesis conditions on the performances of nitrogen-doped carbon-based catalysts for hydrogen peroxide electrosynthesis

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Hydrogen peroxide (H_2O_2) ranks among the top 100 most significant chemicals, finding extensive use across various chemical and medical end-user industries including paper and pulp bleaching, water treatment, food processing, electronics, and disinfection in medical and pharmaceutical sectors. The electrochemical two-electron ($2e^-$) oxygen reduction process (ORR) offers a sustainable pathway for decentralized H_2O_2 production, diverging from the energy-intensive centralized industrial anthraquinone process [1]. Metal-free nitrogen-carbon-based catalysts have emerged as promising alternatives for expensive, toxic, and scarce noble-metal or alloy catalysts like Pt, Pd, Au, Pt–Hg, and Au–Pd for $2e^-$ ORR [2].

In this study, we synthesized nitrogen-doped carbon-based catalysts (NCC) from polyaniline (PANI) using a microwave-assisted method (MW). MW heating offers a compelling alternative to conventional thermal methods, providing quicker heating, enhanced energy efficiency, and the potential for nanostructure formation with distinct morphological or structural properties [3]. The MW synthesis of NCC was performed in a microwave calorimeter equipped with a dual-mode cylindrical cavity. This setup allowed simultaneous heating of the sample and measurement of the material's dielectric properties relative to the carbonization temperature [4]. Control of heating in the PANI sample was achieved by tuning the resonant frequency near 2.45 GHz, regulating the resulting MW power delivered to the cavity. Oscillations in MW power within the 20±10 W range facilitated rapid heating and cooling cycles in the 350 to 500 °C temperature range, yielding uniform NCC structures ($NCC_{heating\text{-}cooling}$). Conversely, continuous MW power increase led to thermal run-away and localized superheating of the material. The ORR performance of the synthesized NCC was assessed using a rotating ring-disk electrode (RRDE) with a Pt ring and glassy carbon (GC) disk in O_2-saturated 0.1 M KOH media. $NCC_{heating\text{-}cooling}$ samples exhibited 93 % selectivity to H_2O_2, diffusion-limited current densities of 2.6 mA cm^{-2}, and H_2O_2 production current of 3.9 mA cm^{-2}, surpassing values reported for nitrogen-doped carbon-based catalysts synthesized by conventional heating documented in the literature.

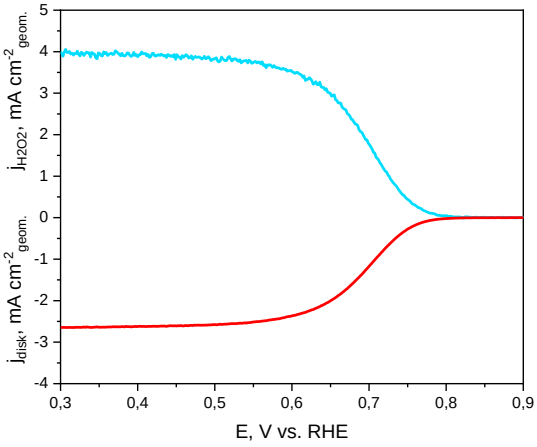

Figure 1. The RRDE polarization curves of $NCC_{heating\text{-}cooling}$ samples in O_2-saturated 0.1 M KOH, produced through rapid heating and cooling stages during MW heating.

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