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Microplastic removal from aqueous solutions using a gas diffusion electrode coated with a very low content of Pd for H₂O₂ production

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The increasing prevalence of microplastics in aquatic environments presents a significant environmental challenge due to their persistence and potential toxicity. Polyethylene (PE), one of the most widely used plastics globally, is a major contributor to this issue, given its extensive applications worldwide [1]. Its durability, while beneficial for its use, makes it particularly resistant to degradation, exacerbating the accumulation of microplastics in water systems. Among the most effective methods for degrading such pollutants is the electro-Fenton process, which relies on the *in situ* production of hydrogen peroxide (H₂O₂) and its subsequent catalytic conversion into hydroxyl radicals (•OH) [2]. However, the efficiency of the electro-Fenton process is directly linked to the availability of H₂O₂, highlighting the need for optimized electrode materials that can enhance its production through oxygen reduction reaction (ORR) (3). In this study, we developed a gas diffusion electrode (GDE) modified with 0.1% Pd-ZrO₂, optimized for *in situ* hydrogen peroxide production under mild conditions. By increasing H₂O₂ availability, we aim to boost the overall efficiency of the electro-Fenton process for the degradation of PE from aqueous solutions. The Pd-ZrO₂ GDE was engineered to optimize H₂O₂ production while maintaining high faradaic efficiency and stability. The modified electrode demonstrated a 200 mV reduction in the overpotential for H₂O₂ production compared to a palladium-free GDE, while maintaining a current efficiency above 95%. H₂O₂ production reached 90 mg L⁻¹ at an optimized potential of -0.4 V vs Ag/AgCl. This potential was applied for the degradation of 10 mg L⁻¹ polyethylene using the following process: H₂O₂, electro-Fenton (EF), and photoelectro-Fenton (PEF). In the PEF process, a UVC Pen-Ray lamp (model 11SC-2.12) was used. Polyethylene concentration was determined using pyrolysis coupled with gas chromatography-mass spectrometry (Py-GC/MS), by employing a calibration curve with key pyrolysis products (1-heptene, 1-octene, 2-nonene, 2-decene, 1-undecene, and 1-dodecene). After 120 minutes of electrolysis, PE degradation rates were 8.8%, 20.6%, and 47.1% for the H₂O₂, EF, and PEF processes, respectively. Non-purgeable organic carbon (NPOC) analysis revealed the formation of soluble organic compounds, with the highest values observed in the H₂O₂ process (5.6 mg L⁻¹), followed by EF (3.9 mg L⁻¹) and PEF (3.2 mg L⁻¹). Although PE degradation values in the process using only H₂O₂ production was poor, the increased NPOC indicated partial breakdown of the microplastic particles by H₂O₂, albeit with low power to mineralization of the soluble compounds. In contrast, EF and PEF showed higher degradation efficiency and lower accumulation of soluble organics, attributed to the hydroxyl radicals (•OH) generated, which effectively attacked both the microplastics and organic intermediates, promoting mineralization. The PEF process proved to be the most efficient, achieving ~50% PE removal after 120 minutes of electrolysis due to the synergistic activation of H₂O₂ by both Fe²⁺ and UVC photocatalytic enhancement, leading to superior •OH production. This study highlights the potential of the Pd-ZrO₂ GDE for enhancing H₂O₂ production and improving the degradation of microplastics through advanced electrochemical oxidation processes.

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