

Eco-Friendly Synthesis, Characterization and Cytotoxicity of Zero-Valent Iron Nanoparticles from Spent Coffee Grounds

Filipe Fernandes ^{1,2}, Maria Freitas ¹, Cláudia Pinho ³, Ana Isabel Oliveira ³, Cristina Delerue-Matos ¹, Clara Grosso ^{1,*}

¹ REQUIMTE/LAQV, Instituto Superior de Engenharia do Porto, Instituto Politécnico do Porto, Rua Dr. António Bernardino de Almeida, 431, 4249-015 Porto, Portugal;

² Faculdade de Ciências da Universidade do Porto, Rua do Campo Alegre, s/n, 4169-007 Porto, Portugal;

³ REQUIMTE/LAQV, Escola Superior de Saúde, Rua Dr. António Bernardino de Almeida, 400, 4200-072 Porto;

* claragrosso@graq.isep.ipp.pt

Background: The agrifood sector generates substantial waste and by-products, creating opportunities for sustainable valorization. The coffee industry, for instance, produces spent coffee grounds (SCG), which can be utilized for eco-friendly nanomaterial production [1,2]. **Objectives:** the valorisation of spent coffee grounds, the synthesis and characterization of zero-valent iron nanoparticles (nZVI), and the assessment of their toxicity to human cells. **Methods:** nZVI, widely studied for soil and water remediation, can be synthesized using SCG hydromethanolic extracts, eliminating toxic solvents. These green nZVIs were further characterized by ultraviolet-visible spectroscopy (UV-Vis), fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS) thermogravimetric analysis (TG), dynamic light scattering (DLS), and magnetic properties. SCG extract and nZVI cytotoxicity was assessed via MTT assay in MRC-5 (human lung fibroblast) and U-87 (human glioblastoma) cell lines. **Results:** HPLC-DAD analysis of SCG extract revealed caffeoylquinic acids, feruloylquinic acids, di-caffeoylquinic acids, caffeine, and trigonelline as key components. SEM and EDS analyses revealed that nZVIs presented spherical shapes with particle sizes of 83.50 ± 14.69 nm and 72.47 ± 12.56 nm for SCG40°C-nZVI and SCG60°C-nZVI, with an elemental composition of carbon (47%), oxygen (34%), and iron (16%). DLS presented relatively lower sizes but in the same order of magnitude (14.64 ± 0.76 nm and 22.68 ± 6.79 nm, with a zeta potential of -5.99 ± 1.71 mV and -6.97 ± 1.15 mV, respectively). TG confirmed EDS results, showing approximately 20% residual mass at 1400 °C. nZVI were shown to be paramagnetic, and FTIR spectra showed the peaks corresponding to the phenolic compounds being incorporated into the nZVI. **Conclusions:** Compared to chemically produced nZVI, green nZVI demonstrated improved stability due to organic matter on the surface. The adoption of SCG to produce nZVI is a low-cost approach without toxic reagents and provides new understanding into waste recycling and nZVI synthesis.

Keywords: Spent coffee grounds, nano zerovalent iron, low-cost approach, green synthesis

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References

- [1] Franca AS, Oliveira LS. Potential Uses of Spent Coffee Grounds in the Food Industry. *Foods* 2022, 11. <https://doi.org/10.3390/foods11142064>.
- [2] Park MH, Lee J, Kim JY. Oxidation resistance of nanoscale zero-valent iron supported on exhausted coffee grounds. *Chemosphere* 2019, 234, 179–86. <https://doi.org/10.1016/J.CHEMOSPHERE.2019.06.035>.