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ACCEPTED MANUSCRIPT

Ultrasonic Spray Pyrolysis Synthesis of Nitrogen-Doped Porous Fe/C Composites from

Glycerol for Hexavalent Chromium Removal

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Abstract

Nitrogen-doped porous carbon spheres impregnated with iron nanoparticles (Fe/C-N) are

prepared from glycerol, a by-product of the biodiesel industry, and ferric ammonium citrate

(FAC) via ultrasonic spray pyrolysis (USP). Carbon microspheres are generated during rapid (<

10 sec) pyrolysis of an aerosolized liquid precursor, resulting from acid induced dehydration,

polymerization, and carbonization of glycerol. Fe/C-N composites are predominantly meso and

macroporous, caused by iron-mediated carbon gasification during heating and salt templating.

Iron impregnation (up to 6.6 wt%) and nitrogen doping (up to 3.3 wt%) are achieved

simultaneously because FAC acts as both iron precursor and reducing agent. Reducing gases

released from thermally decomposed FAC react with carbon surfaces to deposit nitrogen while

facilitating in situ generation of Fe⁰ and Fe₃O₄. Cr(VI) removal capacity up to 33 mg/g is

reported, attributed to combined adsorption and reduction when applying Fe/C-N composites.

The described synthesis, therefore, converts an industrial by-product into value-added materials

with potential for use in environmental remediation.

Keywords: Ultrasonic spray pyrolysis, glycerol, ferric ammonium citrate, nitrogen-doped carbon,

hexavalent chromium removal

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