Synopsis

The increasing scarcity of clean water, coupled with the continuous release of toxic contaminants from industrial and agricultural activities, has emerged as a critical global concern. This calls for the advancement of sustainable water treatment technologies that are efficient, selective, reusable, and environmentally friendly. In this dissertation, four novel carbonaceous nanocomposites, namely, Ni-CNF/ACF, Cu-CNF/ACF, ZnO/g-C_3N_4-HT, and Fe_3O_4/rGO are synthesized, characterized, and evaluated for their applicability in removing a wide range of pollutants from wastewater, including synthetic dyes (Methylene Blue, MB; and Congo Red, CR), heavy metals (Cr(VI) and Pb(II)), and nitroaromatics (para-nitrophenol, 4-NP; and para-nitroaniline, 4-NA).

In one of the studies, Ni-CNF/ACF was synthesized through a sequential process involving calcination, H_2-reduction, and catalytic chemical vapour deposition (CCVD) using C_2H_2 as the carbon source, resulting in the formation of carbon nanoforests anchored on a porous ACF matrix. The nanocomposite demonstrated outstanding adsorption performance toward 4-NP (325.80 mg/g at pH 2) and MB (480.90 mg/g at pH 12). This enhanced uptake was attributed to synergistic contributions from π–π interactions, electrostatic attraction, high specific surface area, and the hydrophilic nature of the adsorbent surface, which collectively facilitated strong binding of pollutant molecules to the multi-scale web of carbon-micro-nano matrices.

In another study, ZnO/g-C_3N_4-HT nanocomposite was synthesized via a hydrothermal treatment at 150 °C, followed by calcination in air at 500 °C. FE-SEM and TEM analyses confirmed a uniform dispersion of rod-shaped ZnO nanoparticles onto the g-C_3N_4 matrix. Under visible-light illumination, the composite exhibited efficient photocatalytic activity, achieving ~90% degradation of 10 ppm MB within 210 min at pH 7 with a catalyst dose of 0.6 g L^{-1}, following pseudo-first-order kinetics (pseudo-first-order rate constant of 0.0113 min^{-1}). The enhanced photocatalytic performance was attributed to the reduced optical band gap and efficient generation of reactive oxygen species ($\cdot OH$ and $\cdot O_2^-$), which actively contributed to MB degradation.

Another objective of the study was to fabricate a multi-scale web of carbon micro/nanofibers (CNF/ACF) matrix through sequential calcination, H_2-reduction, and CCVD using C_2H_2 as the carbon precursor and Cu NPs as the catalyst. TEM observations confirmed a tip-growth mechanism for CNFs anchored onto the highly porous ACF framework. As evaluated using the Liu isotherm model, the exceptional adsorption capacities for CR (288.13 mg/g at pH 4) and Pb(II) (423.78 mg/g at pH 6) were governed by multiple synergistic mechanisms, including π–π and n–π interactions, electrostatic attraction, hydrogen bonding, surface chelation, and pore filling. The adsorption performance of the synthesized CNF/ACF in natural river water medium remained largely unaffected, indicating strong resistance to interference from dissolved ions and organic matter.

The Fe_3O_4/rGO nanocomposite was synthesized via a solvothermal approach with an optimized Fe_3O_4 loading of 27% by weight. FE-SEM analysis revealed a quasi-uniform dispersion of Fe_3O_4 NPs across the porous rGO nanosheets. Adsorption equilibrium for 4-NA and Cr(VI) was achieved within 4 and 6 h, respectively, following pseudo-second-order kinetics. The Liu isotherm model estimated maximum adsorption capacity (Q_{max}) values of 264.21 mg/g for 4-NA and 324.56 mg/g for Cr(VI) at pH 2, 303 K, and an adsorbent dose of 0.25 g/L. The removal of 4-NA was dominated by hydrogen bonding, electrostatic attraction, and π–π stacking, whereas Cr(VI) uptake proceeded via chelation with surface oxygenated functional groups and Fe_3O_4 assisted reduction to Cr(III).

It is worthwhile to mention that all the studied nanocomposites retained above 80% of their initial adsorption performance after at least four regeneration cycles, without noticeable structural deterioration, chemical modification, or metal leaching. Collectively, these findings highlight the broad-spectrum efficiency, durability, and practical applicability of the engineered nanomaterials for advanced wastewater treatment.

Keywords: *Carbon, Chemical Vapour Deposition, Nanomaterial, Dyes, Heavy Metals, Adsorption, Photodegradation, Wastewater remediation*