## **Abstract**

## Carbon-Metal Micro-Nanocomposites for Advancing Environmental Remediation and Sustainable Agriculture

Addressing the critical challenges of environmental remediation and sustainable agriculture requires advanced multifunctional materials with tailored physicochemical properties and robust catalytic performance. Carbon-based micro-nanomaterials including graphene, carbon nanofibers, and reduced graphene oxide-exhibit the high electrical conductivity, large specific surface areas, and tuneable surface functionalities, which if coupled with metal nanoparticles or metal-based semiconductors, enhance charge separation and catalytic efficiency.

In the first study, Ni-Sn-based perovskite (NiSnO<sub>3</sub>, NSO) was anchored onto graphitic carbon nitride (g-C<sub>3</sub>N<sub>4</sub>) sheets to synthesize binary composite for nitrogen reduction reaction. The NSO-gCN composite achieved an ammonia evolution rate of  $\sim$ 566  $\mu$ mol g<sup>-1</sup> h<sup>-1</sup> under visible light, surpassing pure g-C<sub>3</sub>N<sub>4</sub> and NSO by factors of 3.6 and 40, respectively. The relatively improved performance of NSO/gC<sub>3</sub>N<sub>4</sub> composite is attributed to the efficient charge separation via S-scheme mediated charge transfer mechanism between NSO and gC<sub>3</sub>N<sub>4</sub>, supported by the findings of XPS, radicals scavenging tests, photoluminescence, and electrochemical impedance spectroscopy analyses.

In the second study, a NiCo2S4 (NCS)-decorated phosphorus-doped g-C3N4 (PCN) composite was developed for photocatalytic nitrate reduction. The optimized NCS-PCN (30 wt% NCS) system achieved >99% nitrate removal within 4 h, with ammonia selectivity of ~96%. EPR, XPS, KPFM analyses confirmed the presence of an internal electric field between NCS and PCN which is supporting an S-scheme charge transfer mechanism The photocatalyst demonstrated minimal activity loss (<1%) after five reaction-regeneration cycles, underscoring its durability.

In the third study, an UV–Vis spectroelectrochemical sensor utilizing laser-induced graphene (LIG) electrodes modified with copper nanoparticles (Cu-LIG) was developed for the selective detection of vanillin. The high electrochemical surface area (0.907 cm²/cm²) and electrical conductance (0.002 S) contributed to rapid electron transfer. The sensor exhibited a linear detection range from 0.25 to 40  $\mu$ g/mL (R² = 0.993) with a limit of detection of 0.14  $\mu$ g/mL. The selectivity was validated against common interferents including glucose, fructose, and ascorbic acid.

In the fourth study, a novel dolomite-supported carbon nanofibers (CNF) composite loaded with the oxides of Cu, Zn, Mo, and B micronutrients (MO-BO-CNF/dolomite) was synthesized as a controlled-release fertilizer. The synthesized material was characterized using different

physicochemical techniques. SEM images, EDS, XPS and XRD anlyses confirmed the formation of metal oxides and CNFs. The average diameter of CNFs was determined to be ~107 nm. Seed germination assays on *Cicer arietinum* using MO-BO-CNF/dolomite at the dose of 1 mg/mL, showed ~95% germination percentage, while 30-day plant growth experiments demonstrated significant improvements in the root and shoot length, biomass, chlorophyll, and protein content compared to control (without any treatment) group. A 7.8-fold increase in total nitrogen conetent of the soil post MO-BO-CNF/dolomite treatment was observed and an enhanced populations of nitrogen-fixing bacteria (*Bacillus sp., Acetobacter sp.*, and *Rhizobiales*), highlighting the role of MO-BO-CNF/dolomite fertilizer in the nutrient bioavailability and soil fertility.

Carbon–metal composites serve as a robust and adaptable materials platform for advancing solutions across clean energy, environmental monitoring, and sustainable agriculture.