Electronic Supplementary Material (ESI) for Environmental Science: Nano.

Supplementary Information

Fe-carbon nanofiber-modified Mo-MOF for the controlled release and translocation of micronutrients in plants

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Soil-moisture content

In the plant growth experiments, 50 mL of Milli-Q water was supplied after every 4th day to maintain a consistent moisture content throughout the experiments. The loss of soil-moisture content over time was determined by measuring the weight (g) of soil before (dry soil) and after supplying water (moist-soil). 500 g of agriculture soil was filled in a planter pot and weight was determined at day 0. Pot was supplied with 50 mL of Milli-Q water and the weight of moist-soil of measured. The pot was then maintained at photo-period cycle (10 h light: 14 h dark) for 4 days and weight-loss of moist soil was measured. After 4th day, we observed the weight of soil close to the dry soil, indicating loss of moisture content (data not shown). Therefore, 50 mL of water was supplemented every 4th day throughout the plant growth experiment.

Bacterial count in soil

Bacterial count in the soil sample was determined using the colony count method.[1] Approximately, 1 g of soil sample was collected from the Rhizospheric zone of the control- and Fe-CNF/Mo-MOF-treated pots at 0, 5, 15, and 30 days. The samples were dispersed in 10 mL sterilized DI water. The mixture was serially diluted by the factor of 10^5. Approximately 0.1 mL mixture was collected from each sample of different days and spread over the nutrient agar plates using a glass spreader. The plates were incubated for 24 h at 37 ^{O}C. After the incubation period, the number of colonies was counted and the colony forming unit (CFU) was calculated using the following formula:

$$CFU = \frac{Number\ of\ colonies\ x\ Dilution\ factor}{amout\ of\ sample\ (mL)}$$

Characterization of Fe-CNF

ESI Fig. S3a displays the transmission electron microscopy (TEM) image of Fe-CNF revealing a fiber-like morphology of the particles. Further, the Fe nanoparticles were found to be located at the tip of the CNFs, and were visible as spheres with dark contrast. The average hydrodynamic size of Fe-CNF was approximately 50 nm (ESI Fig. S3b). X-ray diffraction (XRD) spectra indicate a dominant presence of the Fe nanoparticles, with traces of Fe_2O_3, as evidenced by the presence of a prominent peak at 44O (3 1 0 planes of Fe) and a small peak at 38O (3 1 1 planes of Fe2O3) (ESI Fig. S3c). The FTIR spectra of Fe-CNF particles reveal the presence of the –OH (broad peaks ~3420 cm-1), COO-

(~1640 cm-1), C=C (~2250 cm-1), N-H (~3200 cm-1), and C-N (981 cm-1) groups at the particle surface (ESI Fig. S3d). The BET analysis of Fe-CNF reveal the pore volumes and surface area of Fe-CNF particles (Fig. S3e). These characteristics of Fe-CNF are in agreement with the previous studies [22,25,26].

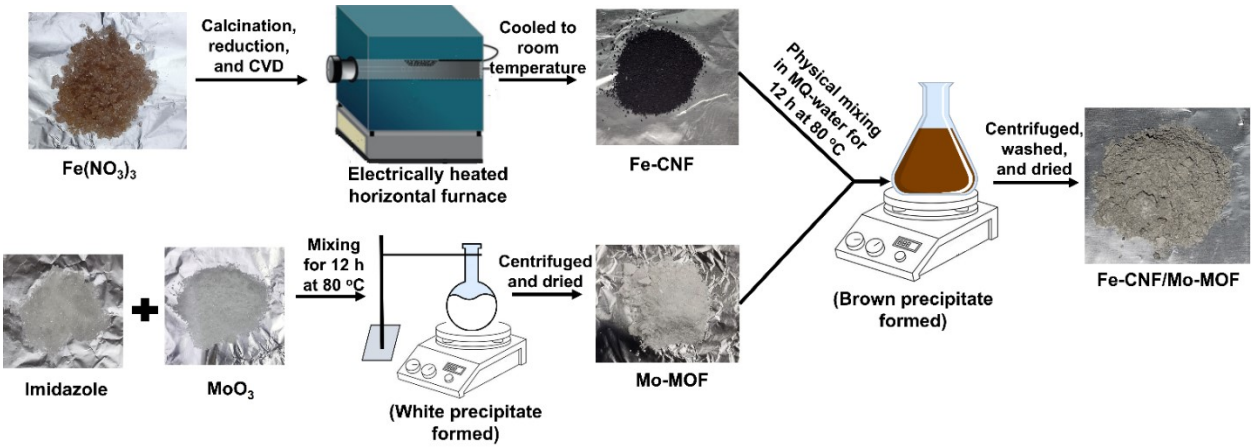

Fig. S1: Schematic representation of the steps involved in the materials synthesis.

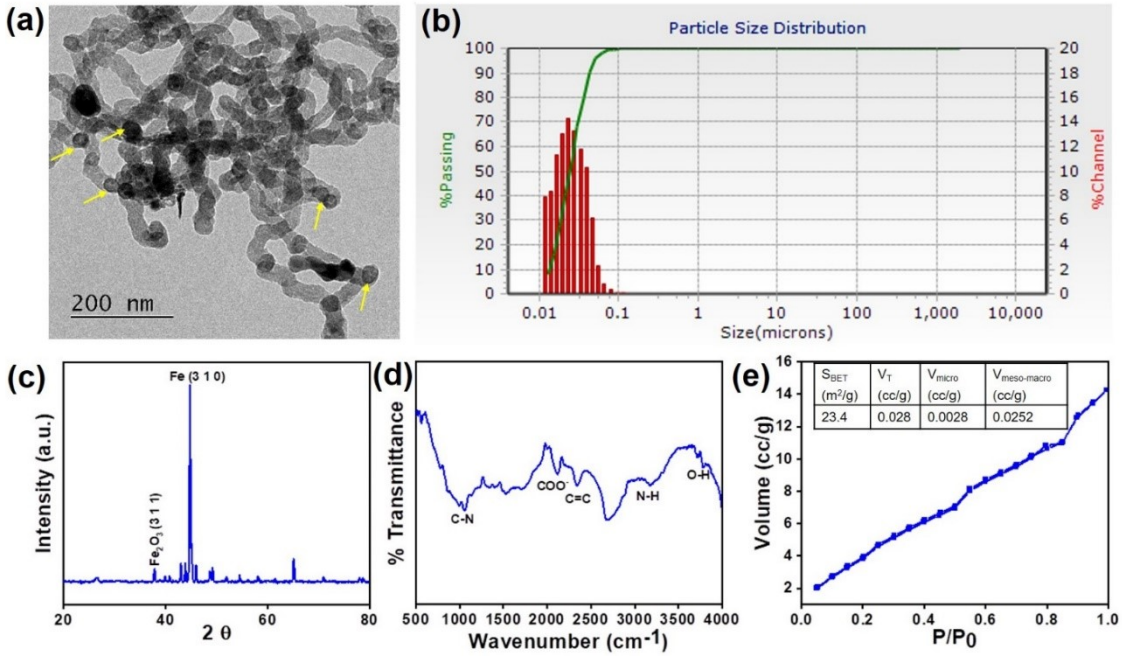

Fig. S2: Physicochemical properties of Fe-CNF. (a) Particle morphology determined using TEM, (b) Average hydrodynamic diameter in water. (c, d) XRD and FTIR spectra, and (e) the surface area and pore volume of Fe-CNF particles determined using BET analysis.

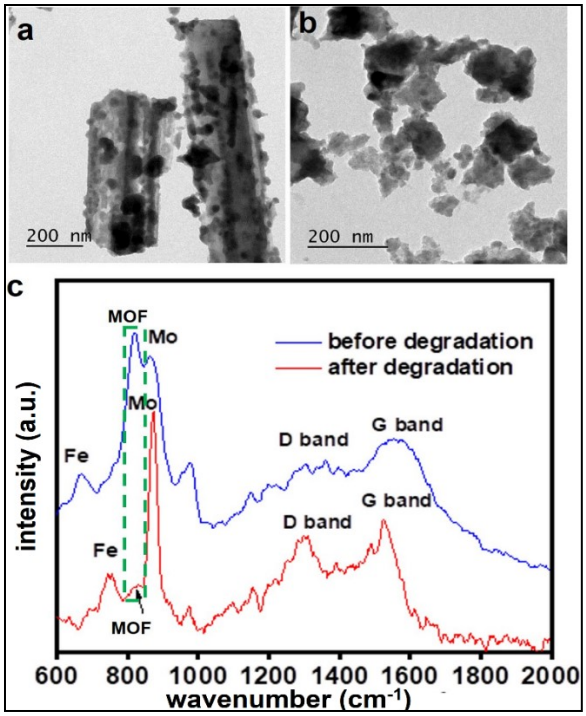

Fig. S3: Microbial-driven degradation of Fe-CNF/Mo-MOF in soil (220 mg L^{-1}) after 24 h. (a, b) TEM micrographs of Fe-CNF/Mo-MOF (a) before and (b) after degradation. RAMAN analysis of Fe-CNF/Mo-MOF reveals the disappearance of the Mo-MOF peak in the spectra, post bacterial incubation, corroborating the biodegradation of materials.

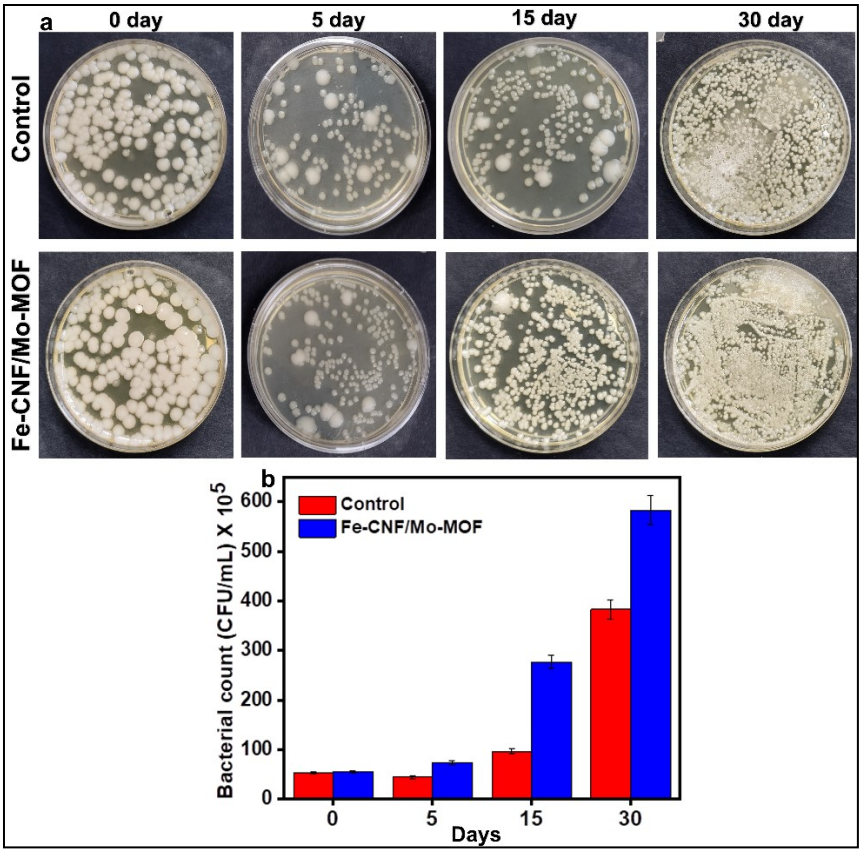

Fig. S4: Bacterial counts in the soil sample for 30 days after supplementing Fe-CNF/Mo-MOF (220 µg/cm^2). (a) Digital photomicrographs of bacterial colonies cultivated using soil at 0, 5, 15, and 15 days of plant growth, and (b) corresponding total bacterial count based on colony forming unit (CFU).

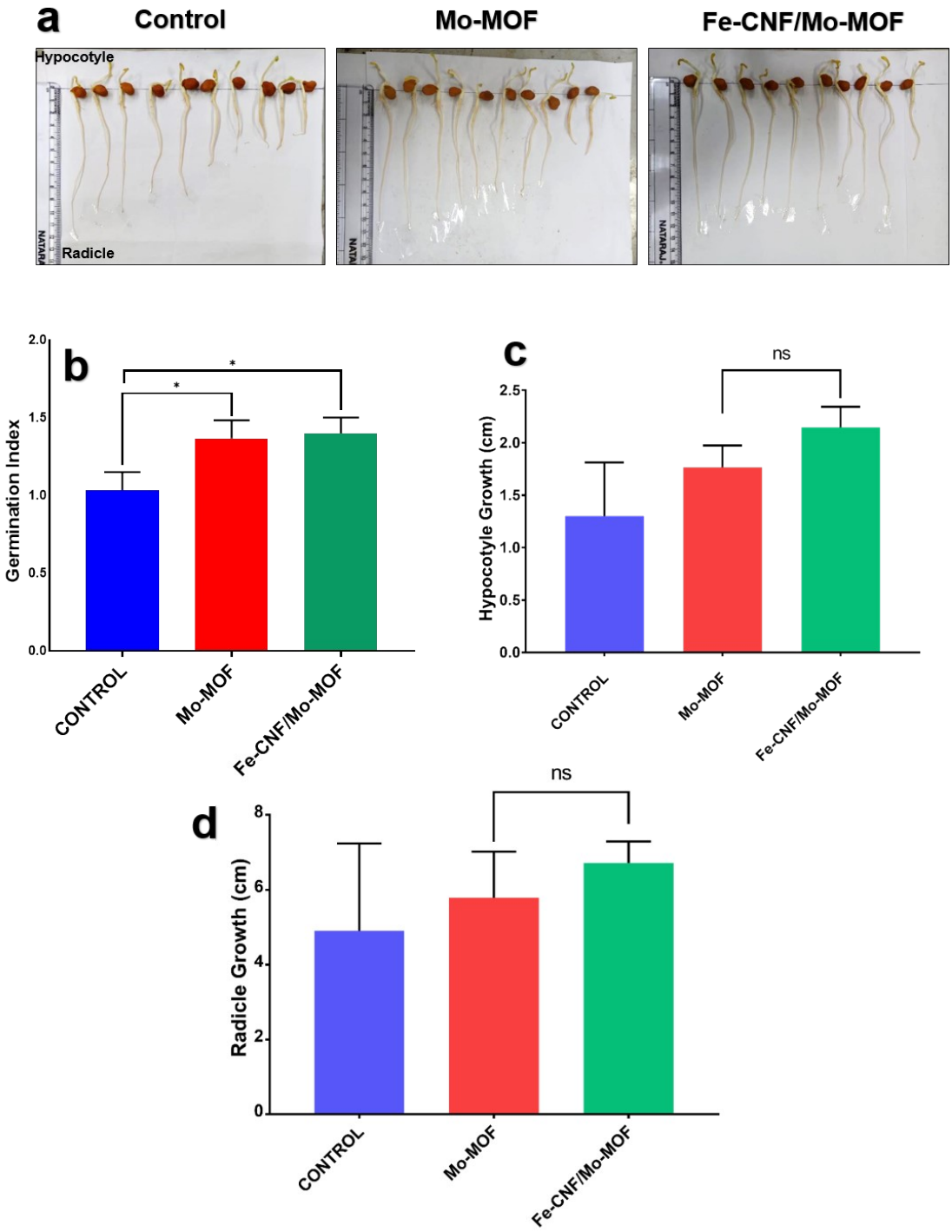

Fig. S5: Fe-CNF/Mo-MOF improved germination of chickpea seeds. (a) Optimal micrographs of germinated seeds represent hypocotyl and radicle growth after 6 days. (b-d) Germination index (b), hypocotyl growth (c), and radicle growth of chickpea seeds after 6 days. Data presented Mean ± SD (n = 3). Significance was calculated by applying One-way-ANOVA and Tukey`s post-hoc test. **p<0.01, ***p<0.001, and ****p<0.0001 indicate the statistical significance between control and treatments.

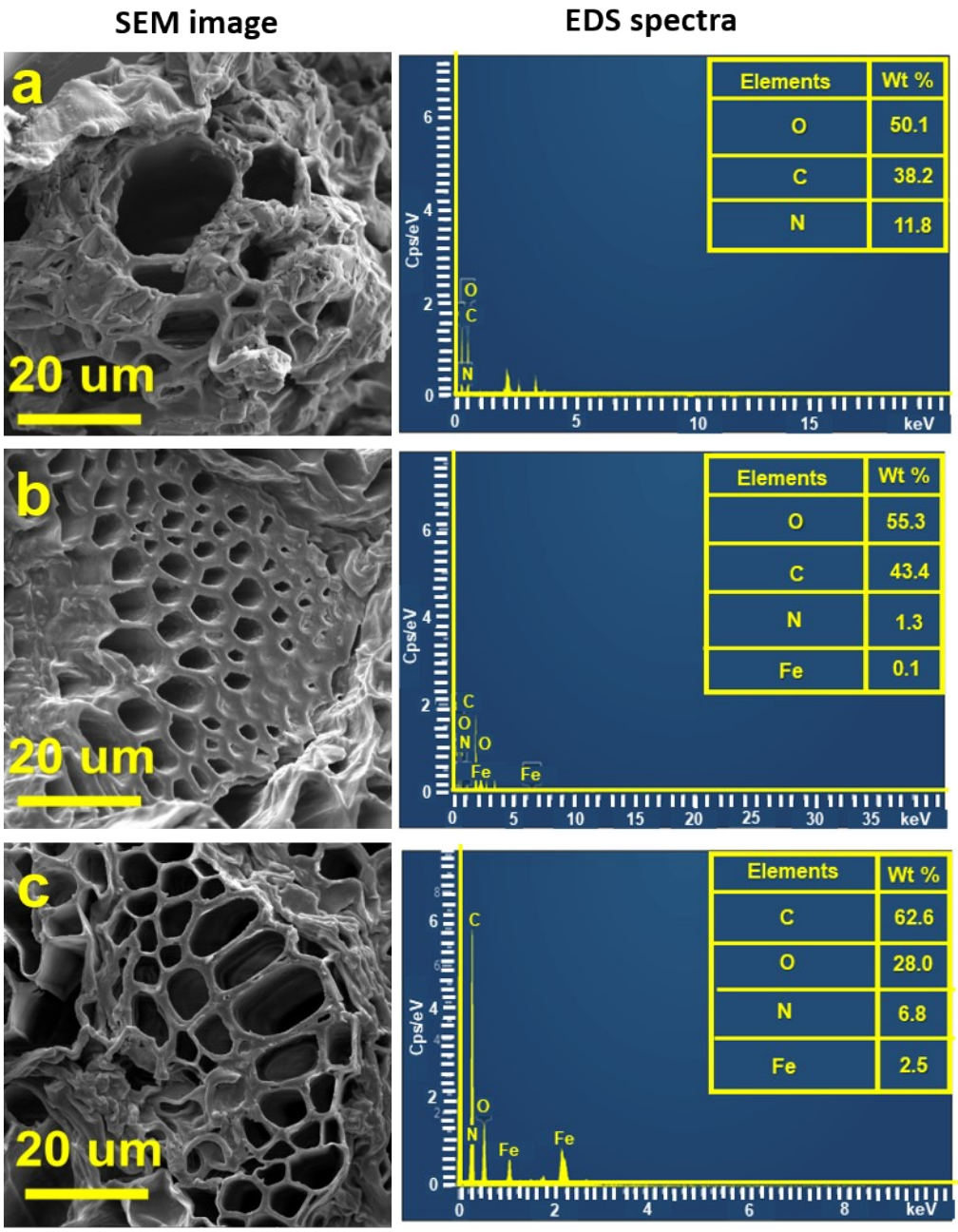

Fig. S6: Ultrastructural changes and elemental distribution in plants after 30 days of growth in the presence/absence of Fe-CNF/Mo-MOF (220 mg/kg). SEM and EDS analysis of cross sections of the plant shoot: (a) control, (b) Mo-MOF, and (c) Fe-CNF/Mo-MOF.

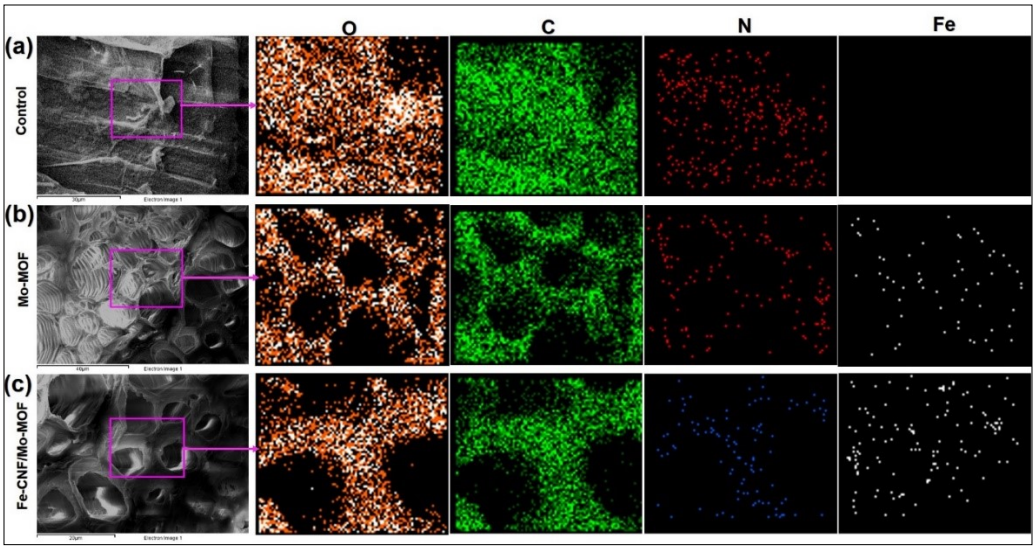

Fig. S7: Elemental distribution in plant tissue after 30 days of growth in the presence/absence of MOF (110 mg/kg) or Fe-CNF/Mo-MOF (220 mg/kg). Elemental mapping in the cross-sections of the plant shoot: (a) control, (b) Mo-MOF, and (c) Fe-CNF/Mo-MOF.

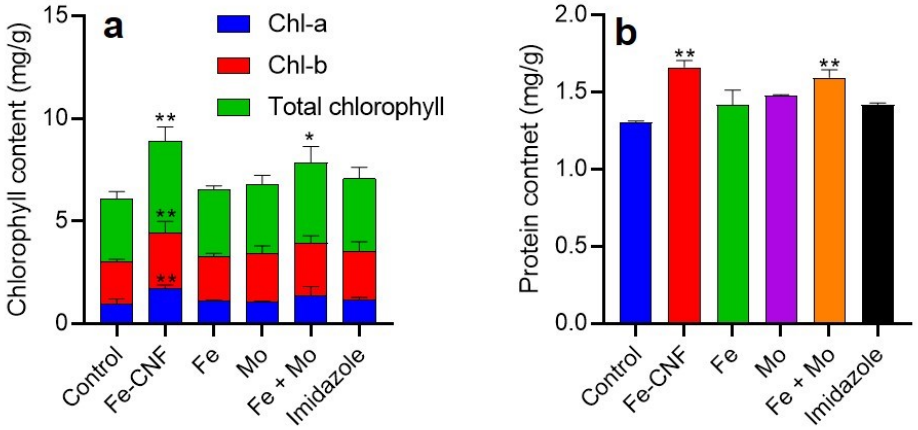

Fig. S8: Plants growth after individual treatment with Fe-CNF, Fe, Mo, Fe+Mo, and imidazole (220 mg/Kg of soil) for 30 days. (a) Chlorophyll content, (b) protein content. Data presented Mean ± SD (n = 3). Significance was calculated by applying One-way-ANOVA and Dunnett's post-hoc test. *p<0.05, **p<0.01 indicates the statistical significance between control and treatments.

Reference

1 R.A. Omar, N. Verma and P.K. Arora, Successive bacterial desulfurization and regeneration of liquid fuel over Ni-doped carbon beads using a single Enterococcus faecium strain isolated from an industrial wastewater, *Fuel*, 2022, 309, 122209.