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Abstract	:	Water contamination and scarcity pose critical global challenges. Existing water remediation technologies such as membrane technologies lack hydrophilic surface properties, prompting the need for novel, highly efficient supportive materials. Photocatalysis emerges as a promising solution for degrading organic pollutants in wastewater. However, existing photocatalysts such as titanium dioxide (TiO2) suffer from rapid recombination of photogenerated charge carriers and lower catalytic activity, hindering performance. Herein, a novel, high sorption capacity nZVI–SiO2–TiO2 nanocomposite material was synthesized via a combined chemical reduction approach. The influence of synthesis pH and the synergistic effects of nZVI, SiO2, and TiO2 on the physicochemical properties and overall performance of the nZVI–SiO2–TiO2 nanocomposite were investigated. Three sets of nZVI–SiO2–TiO2 nanocomposites were synthesized by varying synthesis pH from 2 to 4. MB dye degradation experiments and thermal analysis revealed that the nZVI–SiO2–TiO2 nanocomposite synthesized under pH 2 synthesis conditions exhibited the fastest dye degradation rate, highest removal efficiency (100%), and thermal stability. Characterization techniques, including FTIR, EDS (energy dispersive X-ray spectroscopy), SEM, BET (Brunauer–Emmett–Teller), XRD, TGA (thermogravimetric analysis), and DSC (differential scanning calorimetry), revealed that lower nZVI–SiO2–