## Abstract

Vanadium (V<sub>2</sub>O<sub>5</sub>) and carbon spheres (Cs)-doped NiO<sub>2</sub> nanostructures (NSs) were prepared using the co-precipitation approach. Several spectroscopic and microscopic techniques, including X-ray diffraction (XRD), UV-vis, FTIR, TEM, and HR-TEM investigations, were used to describe the as-synthesized NSs. The XRD pattern exhibited the hexagonal structure, and the crystallite size of pristine and doped NSs was calculated as 29.3, 32.8, 25.79, and 45.19 nm, respectively. The control sample (NiO<sub>2</sub>) showed maximum absorption at 330 nm, and upon doping, a redshift was observed, leading to decreased Eg energy from 3.75 to 3.59 eV. TEM of NiO<sub>2</sub> shows agglomerated nonuniform nanorods exhibited with various nanoparticles without a specific orientation; a higher agglomeration was observed upon doping. The (4 wt.%) V<sub>2</sub>O<sub>5</sub>/Csdoped NiO2 NSs served as superior catalysts with a 94.21% MB reduction in acidic media.