Hydrothermal assisted-green synthesis of Fe/N co-doped TiO2 nanocomposites using *Vernonia amygdalina* leaf extract

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Abstract: In this paper, pure TiO_2 and Fe/N co-doped TiO_2 nanocomposites were prepared via hydrothermal-assisted-green synthesis method using aqueous extract of bitter leaf, *Vernonia amygdalina*. The doping of pure TiO_2 was by wet impregnation of titanium isopropoxide with NH_4NO_3 and $FeCl_3.6H_2O$ such that the theoretical Fe/N doped Ti molar ratio was 4:1. The prepared nanocomposites were annealed at 450 ^oC and characterized by High resolution thermal electron microscopy (HRTEM), X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS) and Brunauer–Emmett–Teller (BET) measurements methods. The XRD patterns confirmed the formation of anatase for pure TiO_2 and rutile phase for Fe/N co-doped TiO_2 samples. The phase transformation from anatase to rutile was linked to electronic movement between 3d and 2p orbitals of Fe and N respectively. The increase in average crystallite size of 37.4 nm observed for Fe-N-TiO_2 was attributed to complete phase change from anatase to rutile. The HRTEM images of the as-synthesized Fe/N co-doped TiO_2 shows an agglomeration of small spherical shape nanoparticles with sizes in good agreement with the sizes obtained from XRD measurements. The co-doping effect of Fe and N was responsible for the increased surface area from 10.37 m_2/g for pure TiO_2 to 25.48 m_2/g for Fe/N co-doped TiO_2 nanocomposites respectively. This study demonstrated that the microstructural, textural, phase types and oxidation states of TiO_2 were influenced by Fe and N.

Keywords: TiO_2 nanocomposites, hydrothermal-assisted-green synthesis, *Vernonia amygdalina* crystallite size, anatase-rutile, surface area, impregnation

- http: DOI: 10.48187/stnanomat.2021.1.002