Abstract

The focus of present PhD research work is to explore the potentials of nickel cobaltite composite in photocatalytic and energy storage applications. The effect of multiwalled carbon nanotubes (MWCNTs) and reduced graphene oxide (rGO) in nickel cobaltite composite was also studied for supercapacitor and photocatalytic applications.

A brief overview of research work is given as follows:-

In the **first experiment**, hierarchical porous flower-like spinel nickel cobaltite ($NiCo_2O_4$) nanoflakes was synthesized by using simple and economical chemical method. The structure, morphology and optical properties of synthesized nanoflakes were analyzed by using XRD, FE-SEM and UV-visible. The $NiCo_2O_4$ nanoflakes showed 96% degradation of Toluidine Blue O (TBO) in 50 min and after 10 cycles of operation, the photocatalytic activity was enough for the 89% degradation of TBO under natural sun light. When compared with other photocatalysts materials, porous $NiCo_2O_4$ nanoflakes showed excellent thermal and chemical stability and offer exceptional electron transfer rate during photocatalytic reactions for real environmental pollution cleanup applications. The efficient photocatalytic efficiency and stability of $NiCo_2O_4$ nanoflakes is attributed to its inimitable structure with large surface area along with maximum active sites. This augments the efficiency of harvesting sun light, and spread active species over surface to decrease e^-/h^+ pair recombination. On the whole, this work demonstrates that the synthesis of nanoflakes $NiCo_2O_4$ can be used as potential candidate to remove organic pollutant and infections from water under natural sun light irradiations for industrial applications.

In the **second experiment**, Hierarchical spinel $NiCo_2O_4$ anchored with reduced graphene oxide ($NiCO_2O_4$-rGO) nanoflakes composites was prepared by using a chemical route for a high-performance photocatalyst. X-ray diffractometry and field emission scanning electron microscopy employed to study the structure and morphology. Fourier transform infrared spectroscopy used for functional group analysis. The UV-vis diffusive reflectance spectroscopy revealed the reduction of optical bandgap from 1.32 to 1.20 eV compared with pristine spinel $NiCo_2O_4$

nanoflakes. $NiCO_2O_4$-rGO nanoflake composites exhibited improved photocatalytic efficiency when compared with pristine $NiCo_2O_4$ nanoflakes. We verified that 100% degradation of MB in 40 mints was observed under natural sunlight irradiation. The photocatalytic degradation efficiency was found to retain 97% of its original value after 6 cycles. The superior photocatalytic efficiency of $NiCO_2O_4$-rGO nanoflakes composites over pristine $NiCo_2O_4$ nanoflakes is attributed to not only its enhanced surface area providing more active sites for light adsorption but also a hetero junction created between rGO and $NiCo_2O_4$. Addition of rGO greatly improves charge separation efficiency hence decreasing electron hole pair recombination. Overall, this work revealed that synthesis of $NiCo_2O_4$-rGO nanoflakes composites may be a promising candidate for the removal of inorganic pollutant from waste water at industrial level under natural sunlight irradiation.

In the **third experiment**, sun light driven hierarchal spinel nickel cobaltite nanoflakes ($NiCo_2O_4$) anchored multiwalled carbon nanotubes (MWCNTs) nanocomposite synthesized by using simple chemical route. The structural, morphological and functional group of as-prepared $NiCo_2O_4$ anchored MWCNTs studied using X-ray diffractometry, field emission scanning electron microscopy and Fourier transform infrared spectroscopy. The UV-vis diffusive reflectance spectroscopy results demonstrated decrease in optical bandgap from 1.32 to 1.16 eV compared with pristine spinel $NiCo_2O_4$ nanoflakes. MWCNTs anchored $NiCo_2O_4$ showed extremely good photocatalytic behavior and we verified 98% degradation of MB in 35 minutes under natural sun light. $NiCo_2O_4$ anchored MWCNTs also confirmed its excellent stability and reusability by retaining 96% of photocatalytic efficiency after 7 cycles of operation. Improved photocatalytic behavior of $NiCo_2O_4$ anchored MWCNTs nanocomposite in comparison to $NiCo_2O_4$ nanoflakes is mainly attributed to excellent electron storage ability of MWCNTs which made catalyst a great acceptor. Moreover, porous structure of MWCNTs not only provides large surface area with more active sites but also increases conductivity and decreases agglomerations on the surface of material which render e^-/h^+ pair recombination. Overall, this work shed new light for the synthesis of $NiCo_2O_4$ anchored MWCNTs with enhanced photocatalytic properties.

In the **forth experiment**, facile chemical route was used to synthesize hierarchal spinel nickel cobaltite nanoflowers anchored reduced graphene oxide

($NiCo_2O_4$-rGO) as high performance electrode material. $NiCo_2O_4$ anchored rGO demonstrated specific capacitance of 2695 Fg^{-1} at 1 Ag^{-1}, which is greater than pristine $NiCo_2O_4$ nanoflowers specific capacitance. $NiCo_2O_4$-rGO showed excellent stability and retention capability of 96% after 2500 cycles at 5 Ag^{-1}. Furthermore, $NiCo_2O_4$–rGO exhibited maximum energy density of 93.57 $WhKg^{-1}$ at power density of 250 WKg^{-1}. We have achieved specific capacitance and retention capability which is higher than previously reported results. This enhancement is mainly attributed to the spinel structure of $NiCo_2O_4$ and its robust structural affinity with rGO. Moreover, rGO possesses extended surface area provided ample of active sites and exceptional synergetic effect which helped to enhance the induction and consequently transportation of e^-/h^+. More importantly due to its special morphological effects, in future $NiCo_2O_4$ anchored rGO nanoflowers may open new avenue in research but also used as an efficient electrode material for the construction of high performance supercapacitors.

In the **fifth experiment**, we synthesized spinel nickel cobaltite anchored multiwalled carbon nanotubes and reduced graphene oxide ($NiCo_2O_4$- MWCNTs-rGO) hybrid nanocomposite by employing facile simple chemical method. $NiCo_2O_4$-MWCNTs-rGO hybrid nanocomposite showed specific capacitance 2901 Fg^{-1} at 1 Ag^{-1} higher than pristine $NiCo_2O_4$ nanoflakes. It also demonstrated retention capability and cyclic stability of 95% after 3000 cycles at 5 Ag^{-1}. The hybrid nanocomposite of $NiCo_2O_4$-MWCNTs-rGO exhibited promising energy density of 100.76 $WhKg^{-1}$ and power density 1250 WKg^{-1}. The enhanced electrochemical properties of $NiCo_2O_4$–MWCNTs-rGO nanocomposit is credited to the unique porous structure of $NiCo_2O_4$ and its empathy with MWCNTs-rGO. Anchoring of MWCNTs-rGO helped to improve the active surface area which consequently augmented the number of active sites. Moreover, synergetic effects of rGO and good electrical conductivity of MWCNTs supported the e^-/h^+ transportation. It is believed that $NiCo_2O_4$ anchored MWCNTs-rGO hybrid nanocomposite proves to be an exceptional electrode material at industrial level.