Synthesis of Nickel-Doped TiO_2 Nanoparticles For Enhanced Supercapacotor electrode Performance

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ABSTRACT

Nickel-doped TiO_2 nanoparticles for supercapacitor applications have been synthesized using a solution-based process. The nanoparticles were characterized using X-ray diffraction to confirm the phase and structural modifications induced by nickel doping. The morphology of the synthesized Ni-doped TiO_2 nanoparticles was investigated using a scanning electron microscope. Electrodes were prepared on stainless steel substrates via a drop-casting and drying process, incorporating the doped nanoparticles. The fabricated devices were subjected to electrochemical performance analysis, including cyclic voltammetry and electrochemical impedance spectroscopy. Electrochemical results indicate enhanced capacitive behavior due to nickel doping, highlighting the potential of Ni-doped TiO_2 nanoparticles as promising candidates for next-generation supercapacitor applications.

Keywords: Nanotechnology, Ni doped TiO_2 Nanoparticles, Chemical Method, Supercapacitor, Electrode Performance

1. INTRODUCTION

Metal oxide materials, including nickel-doped TiO_2 nanoparticles, have attracted a lot of interest as effective supercapacitor electrodes because of their superior electrochemical characteristics[1]. The performance of supercapacitors in terms of specific capacitance, energy and power density, cyclic stability, and long operating life has been the subject of extensive research[2]. A number of crucial elements must be taken care of in order to accomplish these gains, such as expanding the electrode material's surface area, refining the synthesis techniques, choosing complementary material combinations, and making use of cutting-edge, high-performance materials with exceptional chemical and physical characteristics[3]. Because nickel doping increases conductivity and active site availability, it is essential for optimizing these properties in TiO_2.

Pure nickel-doped TiO_2, composite materials, or hybrid structures that include many components for best performance can all be used in the electrode design[3]. Supercapacitors, sometimes referred to as ultracapacitors or electrochemical capacitors, have become highly promising energy storage devices for both present and future technology[4]. They perform better than traditional capacitors, especially when it comes to capacitance and energy density[5]. Typically, a supercapacitor is made up of two porous electrode materials deposited on a conductive substrate and separated by a non-conductive separator to avoid direct contact[6]. The electrical properties and shape of the electrode materials are the main determinants of a supercapacitor's capacitance[7].

The electrodes' shape and electrochemical characteristics can be successfully customized by maximizing the synthesis parameters[8]. Notwithstanding their benefits, supercapacitors have drawbacks that need to be further optimized, such as a lower energy density than batteries and variations in output voltage[9]. In order to overcome these constraints and improve their feasibility for a variety of uses, from small-scale to large-scale energy storage, efforts are constantly being made[10]. In order to create electrodes for supercapacitor applications, nickel-doped TiO_2 nanoparticles were created in this work utilizing a straightforward procedure. Because nickel doping improves TiO_2's mechanical and electrochemical characteristics, it is very promising for next-generation supercapacitor technology[11].

Because of its many uses, including its potential in supercapacitor energy storage systems, nickel-doped TiO_2 has garnered a lot of interest from researchers[12]. This substance has a high specific energy density, chemical stability, and an environmentally favorable nature in addition to being plentiful and reasonably priced[13]. Impressive performance is demonstrated by nickel-doped TiO_2 electrodes, which maintain a high energy density and cyclic efficiency above 95% even after 50,000 charge-discharge cycles[14]. The potential of TiO_2 nanostructures as electrode materials is highlighted by recent developments[15].

For instance, by boosting conductivity and adding more active sites, nickel doping improves the electrochemical characteristics even further[16]. Fe_2O_3 in combination with TiO_2 NTAs produced a volumetric capacitance of 608.2 F/cm³, while studies on modified TiO_2 systems, such as MnO_2-coated TiO_2 nanotube arrays (NTAs), have shown capacitance values as high as 1051 F/cm³. These materials have also demonstrated exceptional stability in asymmetric supercapacitors, maintaining approximately 91.7% of capacitance after 5000 cycles[17].

Nickel doping in TiO_2 enhances its potential for high-performance energy storage applications, building on previous advances. The hydrothermal process is a highly efficient way to produce metal oxide nanoparticles, making it a preferred alternative for supercapacitor applications[18]. Extensive research has been conducted on binary and ternary composites containing nickel-doped TiO_2 to improve their performance as electrode materials for supercapacitors. TiO_2 coupled with carbon nanotubes exhibits a specific capacitance of 345.7 F/g at a current density of 1 A/g. Ternary composites, as MnO_2-TiO_2/C, have enhanced performance even more[19].

This composite exhibited a specific capacitance of 580 F/g at a current density of 2.6 A/g, as well as outstanding rate and cycling stability[20]. Incorporating nickel into TiO_2 nanoparticles improves conductivity and electrochemical activity, making them a viable material[21].

Various approaches have been used to produce nickel-doped TiO_2 and combine it with other materials to improve supercapacitor performance[22].

This work found that nickel-doped TiO_2 nanoparticles may be produced on a large scale using a simple solution-based approach[23]. This method ensures the cleanliness of the electrode material because no new contaminants were added during manufacture[24]. Nickel-doped TiO_2 nanoparticles provide good electrochemical performance in an aqueous electrolyte, resulting in the high capacitance reported in this study[25]. The addition of nickel considerably improves capacitance behavior, indicating the material's potential for efficient supercapacitor applications[26].

2. EXPERIMENTAL DETAILS

2.1 MATERIALS

Nickel-doped titanium dioxide (Ni-TiO_2) nanoparticles require titanium precursors like TTIP or titanium dioxide powder, nickel precursors such nickel nitrate hexahydrate, and solvents like ethanol or deionized water. Additional reagents, such as acetic acid or ammonia, may be necessary to change pH during the synthesis. To achieve high purity and consistent findings, utilize analytical-grade chemicals. Stirring apparatus, a reaction vessel, and a calcination furnace are all required.

2.2 Synthesis

The synthesis is normally carried out using a sol-gel or hydrothermal procedure. In the sol-gel method, TTIP is dissolved in ethanol to produce a titanium precursor solution. Separately, a nickel nitrate solution is produced and gradually added to the titanium solution while constantly stirring. The pH of the mixture is adjusted to promote gel formation, and the liquid is further agitated to ensure homogeneity. After gelation, the product is aged and dried at moderate temperature before being calcined at high temperatures (400-600°C) to crystallize the nanoparticles and incorporate nickel ions into the TiO_2 lattice. Hydrothermal synthesis involves sealing the combined precursor solution in an autoclave and heating it at high temperatures and pressures to enhance nanoparticle formation. The finished product is characterized using techniques such as XRD and SEM[27].

3. RESULTS AND DISCUSSION

3.1 CharacterizationofNi-Doped TiO_2Nanoparticles

3.1.1 SEM

One essential method for examining the surface morphology and particle size of nickel-doped TiO_2 nanoparticles is scanning electron microscopy, or SEM. SEM pictures provide information about the homogeneity and agglomeration of the nanoparticles by displaying their distribution, surface texture, and structural characteristics[28]. SEM study of nickel-doped TiO_2 usually reveals uniformly distributed, spherical or irregularly shaped particles. In comparison to pure TiO_2, nickel doping may result in minor morphological and particle size alterations[29].

The high surface energy of nanoparticles may cause agglomeration, however this impact can be reduced by using controlled production techniques[30]. The elemental composition and the inclusion of nickel ions into the TiO_2 matrix are confirmed by Energy Dispersive X-ray Spectroscopy (EDS), which is frequently employed in conjunction with SEM[30]. A thorough grasp of the physical and compositional characteristics of the nanoparticles is possible thanks to the combination of SEM and EDS[31].

Important characteristics that affect the electrochemical performance of nickel-doped TiO_2 nanoparticles destined for usage as supercapacitor electrode materials are revealed by SEM investigation[32]. Usually, the pictures display a network structure that is porous and linked, which improves ion movement and expands the surface area that can be used to store charges. With diameters ranging from 10 to 50 nm, the nanoparticles frequently show a uniform size distribution, which raises their specific surface area. There is very little aggregation, which suggests effective synthesis and processing methods[33].

For supercapacitor applications, the porous morphology is particularly advantageous as it lowers charge-transfer resistance and makes electrolyte penetration easier[34]. By confirming the presence of titanium and nickel, EDS analysis validates effective doping, which improves TiO_2's pseudocapacitive behavior. Fast redox reactions are facilitated by this structural and compositional synergy, which enhances capacitance and cycling stability. Thus, the high electrochemical performance of nickel-doped TiO_2 as a prospective supercapacitor electrode material is highly correlated with the SEM data.

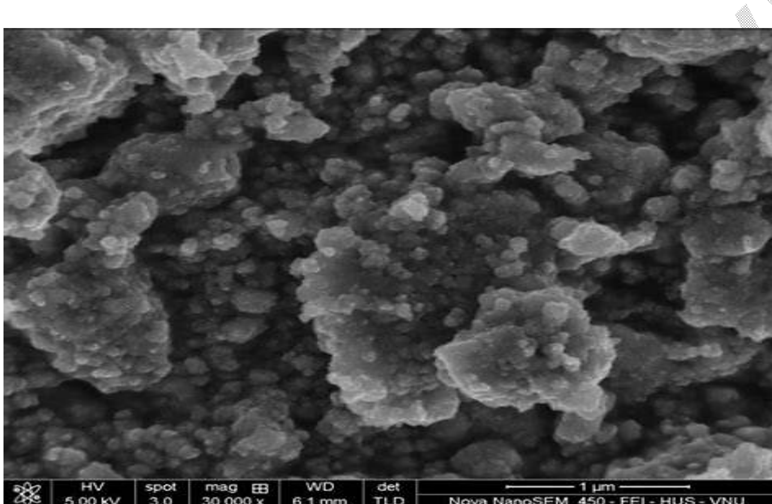

Fig. 1 SEM images of Ni-doped TiO_2 Nanoparticles at 1µm

3.1.2 EDX Analysis

An important analytical technique for verifying the elemental makeup of nickel-doped TiO_2 nanoparticles is energy dispersive X-ray spectroscopy (EDX). Strong peaks that represent titanium (Ti), oxygen (O), and nickel (Ni) are commonly seen in the EDX spectrum, confirming that nickel has been successfully incorporated into the TiO_2 lattice[35]. The doping concentration is indicated by the relative strengths of the nickel peaks, whereas homogeneous doping is suggested by the uniform distribution of nickel signals across the sample. The purity of the produced nanoparticles is shown by the spectrum's lack of impurity peaks. Additionally, the elemental mapping feature of EDX confirms the regularity and efficacy of the doping process by visualizing the spatial distribution of elements[36].

EDX analysis is essential for comprehending the elemental composition of supercapacitor electrode materials and how it affects electrochemical performance[37]. By adding more redox-active sites, nickel improves the pseudocapacitive characteristics of nickel-doped TiO_2, which is employed in supercapacitors. By verifying the addition of nickel without altering the TiO_2 stoichiometry, EDX ensures structural integrity[38]. For reliable performance during charge-discharge cycles, the electrode material's nickel must be uniformly distributed, as shown by elemental mapping. The electrochemical study and EDX results demonstrate how well nickel doping works to improve the cyclic stability, energy density, and capacitance of TiO_2-based supercapacitor electrodes[39].

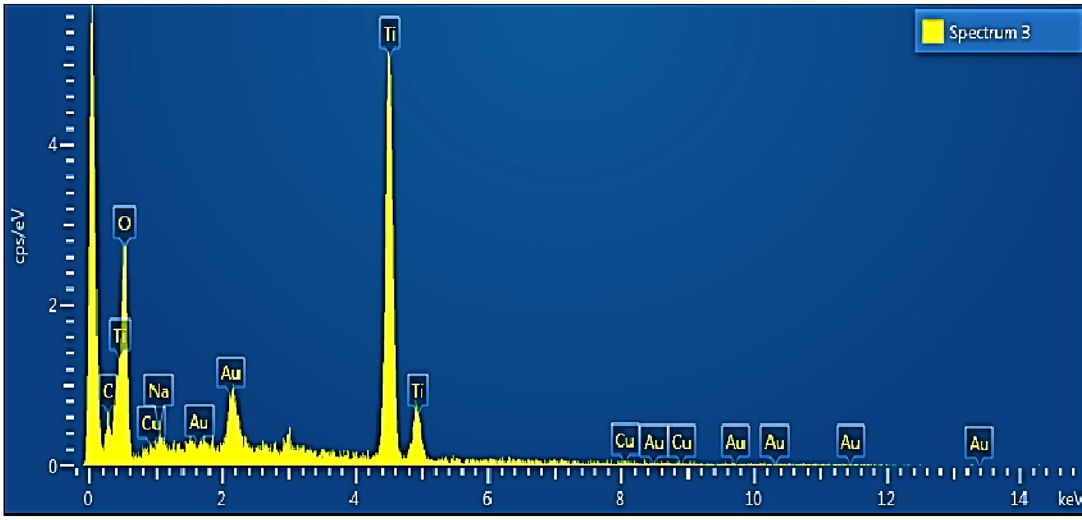

Fig. 2 EDX Spectra of TiO_2 Nanoparticles

3.1.3 FTIR Analysis

Fourier Transform Infrared (FTIR) spectroscopy is a valuable technique for characterizing Ni-doped TiO_2 nanoparticles, providing insights into their chemical composition and functional groups[40]. The FTIR spectrum typically reveals the presence of Ti-O-Ti bonds through strong absorption peaks around 400–700 cm^{-1}, indicative of the TiO_2 lattice structure. Doping with nickel can create small variations in these peaks because to alterations in the TiO_2 lattice generated by Ni inclusion, which influences bond lengths and strengths. Additionally, FTIR analysis may reveal surface hydroxyl groups and adsorbed water, with peaks emerging about 3200–3600 cm^{-1} for O-H stretching vibrations. Such knowledge is essential for comprehending the surface and structural characteristics of Ni-doped TiO_2 nanoparticles, which affect their optical, electrical, and photocatalytic activities[41].

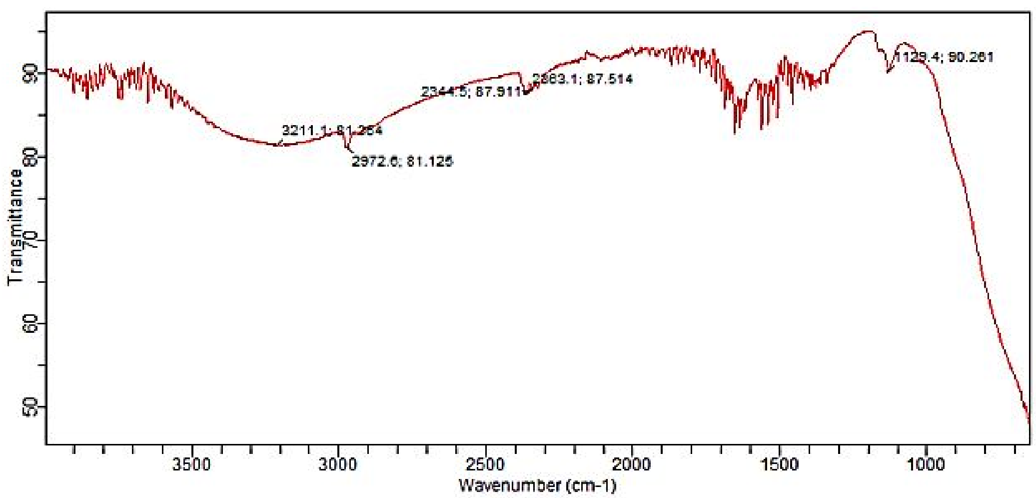

Fig. 3 FTIR Spectra of Ni-doped TiO_2 Nanoparticles

4. Experimental Details

4.1 Preparation of Electrode

Ni-doped TiO_2 nanoparticles must be prepared in a number of steps to ensure uniform coating and good substrate adhesion for best results[42]. This is a general process. For this aim, conductive substrate (e.g., fluorine-doped tin oxide (FTO) glass or titanium foil), Ethanol or isopropanol, Nafion solution or polyvinylidene fluoride (PVDF) binder, Glass rod or spin coater, Deionized water were utilized. These were weighted an adequate number of Ni-doped TiO_2 nanoparticles (~0.05–0.1 g) and then mixed the nanoparticles in ethanol or isopropanol to make a paste or colloidal solution. Adhesion was improved by adding a few drops of PVDF binder or Nafion solution. After that, a mortar and pestle or an ultrasonic

bath was employed to guarantee a uniform dispersion.The conductive substrate (FTO glass or titanium foil) is washed with soap and rinsed with deionized water before being completely dried using an oven or hot air blower[43].
A doctor blade, spin coating, or drop-casting technique are used to apply a suspension of nanoparticles to the cleaned substrate in order to coat it. For homogeneity, the film's thickness is kept constant, usually between 10 and 20 µm. To get rid of the solvent, the coated substrate is dried at 60 to 100°C. The electrode is anealed in a furnace at 400–500°C for 1-2 hours in air to enhance electrical conductivity, adhesion, and crystallinity. The electrode is left to cool to room temperature on its own.
Together with a counter electrode (such as platinum) and an electrolyte appropriate for the intended function, the prepared electrode is employed in the desired electrochemical configuration[44]. Depending on the performance characteristics of the Ni-doped TiO_2, this electrode can be employed in dye-sensitized solar cells, sensors, or photocatalysis. Using electrochemical impedance spectroscopy and cyclic voltammetry, the supercapacitor's performance was assessed.
Two electrode configurations were used in the CV experiment to examine the electrochemical behavior of the produced TiO_2 nanoparticles. The CV curves of the TiO_2 electrode, which were conducted at a scan rate of 100 mVs^{-1} in the 0-1 V range, are displayed in Fig. 4 (a). Supercapacitors are typically used in high power applications. To get the capacitance value with quick charging and discharging rates, a high scan rate is therefore selected in this case. Both the upper and bottom portions of the graph exhibit curve structure, and the curve's shape is symmetrical.
Capacitive behavior is shown by the curve's quasi-rectangular form. Additionally, a pseudo-type CV is noted. It may be concluded that TiO_2 exhibited both EDL and pseudo capacitive characteristics[45]. At a scan rate of 100 mVs^{-1}, the specific capacitance of the TiO_2 nanoparticles was estimated to be close to 25 Fg^{-1}. A certain quantity of TiO_2 nanoparticles is applied to the current collector in this work. The thickness or quantity of the electrode has been found to have a significant impact on the supercapacitor's performance[46]. Therefore, adjusting the thickness of the TiO_2 nanoparticles on the electrode may further increase the obtained value of the specific capacitance[47].

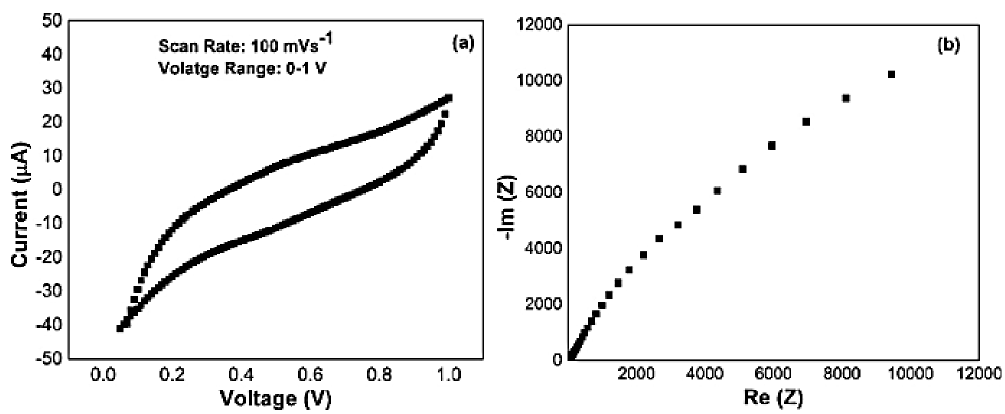

Fig. 4. (a). CV curve of TiO_2 electrodes in voltage range of 0–1 V at scan rate of 100 mVs^{-1} and (b) Nyquist plot
Further, the properties of TiO_2 based supercapacitor was examined using EIS. The EIS was performed to evaluate the series resistance. Fig. 4 (b) shows the Nyquist plot for TiO_2 nanoparticles. The frequency range of 100000–0.01 Hz was chosen for the EIS measurement. It shows that the TiO_2 has almost stable rate of charge and discharge. The series resistance was calculated to be about 50 X for device fabricated with TiO_2 nanoparticles as electrodes. The series resistance has to be minimized in order to have excellent ionic and electronic conduction, leading superior electrochemical performances. The value of series resistance may be reduced by mixing TiO_2 nanoparticles with a conductive material. The CV and EIS results show that TiO_2 nanoparticles synthesized by solution-based process may be utilized for supercapacitor application[48]. The electrochemical performance may also be enhanced by finding an optimum amount of mass of electrode and mixing them with carbon based conductive materials[49].

Conclusion
Nickel-doped TiO_2 nanoparticles exhibited remarkable capacitive behavior, underscoring their potential as advanced materials for supercapacitor applications. The synthesis process for Ni-doped TiO_2 nanoparticles and electrode preparation was designed to be efficient and straightforward, ensuring scalability and practicality. XRD analysis confirmed

the crystalline structure of the nanoparticles, with nickel doping significantly enhancing their electrochemical properties by improving charge storage and transport mechanisms. The Ni-doped TiO_2 nanoparticles achieved an impressive specific capacitance of 25 Fg^{-1} at 100mVs^{-1} demonstrating their superior energy storage capabilities. Further enhancement, such as integrating the doped nanoparticles into carbon-based nanocomposites, could unlock even greater electrochemical performance, paving the way for their use in high-efficiency energy storage devices.

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