**ABSTRACT**

The present work reports the preparation of olivine structured LiNiPO4 [nanoparticles](https://www.sciencedirect.com/topics/materials-science/nanoparticles%22%20%5Co%20%22Learn%20more%20about%20Nanoparticles%20from%20ScienceDirect%27s%20AI-generated%20Topic%20Pages) through [sol-gel](https://www.sciencedirect.com/topics/physics-and-astronomy/sol-gel) thermolysis method using three different chelating agents of [citric acid](https://www.sciencedirect.com/topics/physics-and-astronomy/citric-acid) (LiNi-C), l-ascorbic acid (LiNi-A) and D-sorbitol (LiNi-S). The flame temperature and [enthalpy](https://www.sciencedirect.com/topics/physics-and-astronomy/enthalpy) change of each reaction using the chelating agent is calculated. Further, the sharp X-ray diffraction peak reveals the phase pure and high crystalline nature of the prepared LiNiPO4 nanoparticles with the space group of Pnma (62) irrespective of the chelating agents. The four possible fundamental vibrations of phosphate anion PO43− are revealed through [Fourier Transform Infrared Spectroscopy](https://www.sciencedirect.com/topics/physics-and-astronomy/ftir-spectroscopy) (FTIR) studies. The presence of elements such as Ni, P and O and its valence state is identified through [X-Ray Photoelectron Spectroscopy](https://www.sciencedirect.com/topics/physics-and-astronomy/x-ray-spectroscopy) analysis. The spherical shape particles with the uniform size distribution of LiNi –S is observed than the LiNi-C, LiNi-A particles through FESEM analysis. The redox peaks and plateau regions in the [cyclic voltammetry](https://www.sciencedirect.com/topics/materials-science/cyclic-voltammetry) (CV) and Galvanostatic charge-discharge (GCD) profiles infer the dominance of battery-type charge process rather than a capacitive mechanism. As a result, LiNiPO4 exhibits a maximum specific capacitance of 417 F g−1 at 2 mV s−1 and 357 F g−1 at 1 mA cm−2 in 1 M LiOH, which enables as a suitable [cathode material](https://www.sciencedirect.com/topics/materials-science/cathode-material) for hybrid [supercapacitor](https://www.sciencedirect.com/topics/physics-and-astronomy/electrochemical-capacitors%22%20%5Co%20%22Learn%20more%20about%20Electrochemical%20Capacitors%20from%20ScienceDirect%27s%20AI-generated%20Topic%20Pages). Also, the assembled hybrid supercapacitor delivered a high [energy density](https://www.sciencedirect.com/topics/physics-and-astronomy/flux-density) of 12.5 Wh kg−1 at 200 W kg−1 as well as a longer cycle life of 89% at a current density of 1 mA cm−2 over 2000 cycles is noticed. These results infer that LiNiPO4 could be used as a novel [electrode](https://www.sciencedirect.com/topics/physics-and-astronomy/electrodes) material for hybrid supercapacitor application.