**Abstract**

Herein, discrete γ**‐**KCoPO4 nanocrystals were prepared by a facile, green and fast sol‐gel route. The lattice parameters as well as positional and displacement parameters of atoms in the average γ‐KCoPO4 structure with a space group of *P*21/*n* were calculated using full profile Rietveld refinement. Monodispersed quadrangular γ**‐**KCoPO4 nanocrystals with improved phase purity and crystallinity were found through XRD patterns and HRTEM images. Subsequently, the prepared γ‐KCoPO4 nanocrystals were tested as electrode material for supercapatteries in aqueous electrolytes. The γ**‐**KCoPO4 electrode shows superior specific charge capacity of 309 C g−1 at 1 mV s−1 in 1 M KOH, compared to 1 M NaOH (222 C g−1) and 1 M LiOH (77 C g−1). Further, it exhibits improved electrochemical activity by delivering an impressive specific charge capacity of 100 C g−1 at a current of 0.6 mA cm−2 in an aqueous electrolyte medium with acceptable capacity retention. A lab‐scale supercapattery was assembled resembling the commercial device. The fabricated device delivered an enhanced specific energy of 28 W h kg−1 and a specific power of 1600 W kg−1 and prolonged cycle life of about 5000 cycles.