

Calcination-Induced Structural Evolution of Nickel Phosphate and Its Impact on Charge-Storage Kinetics

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ABSTRACT

Supercapattery performance is governed by the intrinsic properties of its electrode materials, particularly the relationship between microstructure and electrochemical behaviour. In a previous study, amorphous nickel phosphate (NP) has shown superior electrochemical performance as a battery-type electrode despite the common expectation that crystalline materials perform better in electrochemical response. In this work, NP was synthesized via a sonochemical route and its crystallinity was modulated by varying calcination temperatures. At lower calcination temperatures (300–600 °C), NPs remain predominantly amorphous but turn crystalline when higher temperatures are applied. The thermal treatment also influenced the degree of hydration, particle morphology and pore structure. Among all temperatures, NP calcined at lower temperatures delivered the higher specific capacity and showed predominantly diffusion-influenced Faradaic kinetics. In contrast, NP calcined at higher temperatures showed lower specific capacity, but a stronger surface-control contribution to charge storage. These findings demonstrate that a high degree of crystallinity does not necessarily guarantee high charge-storage capacity and reveal a clear trade-off between active-site utilization and reaction kinetics during the amorphous to crystalline evolution of NP.

Keywords: Amorphous; calcination temperature; electrode; nickel phosphate; supercapattery

INTRODUCTION

The transition from fossil fuels towards renewable energy has increased the demand for efficient electrochemical energy storage systems. Supercapacitors offer rapid charging, high power density and excellent cycling stability but suffer from low energy density. In contrast, batteries can store high energy but often experience slower reaction kinetics and lower power capability [1]. These complementary characteristics create an energy and

power gap between the two technologies. Integrating supercapacitor and battery packs in a single system requires additional power-management components, increasing its mass, complexity and potential energy losses. Consequently, this leads to the idea of supercapattery, which combines capacitive and battery charge storage mechanisms in a single device. As with other energy storage devices, the performance of supercapattery depends on the intrinsic properties of the electrode materials such as crystal structure, surface chemistry and accessible electroactive sites [2,3].

Nickel phosphate (NP) is a promising battery-type electrode material due to its favorable redox activity, chemical stability and phosphate-based framework that provides multiple active sites for charge storage. In our previous study, NP was synthesized via a sonochemical process with post-calcination at 300, 600 and 900 °C. The amorphous structure exhibited higher specific capacity than its crystalline counterpart, which contradicts the established idea that the crystalline structure of electrode materials outperforms amorphous structures [4,5,6]. Recently, Wang et al. demonstrated that structurally disordered NP exhibit higher capacitance than crystalline counterparts because structural disorder promoted more efficient ion storage within nanopores [7] [Click or tap here to enter text..](#) The amorphous structure can accommodate the swelling during ion intercalation and deintercalation, highlighting the broader importance of structural disorder in determining ion accessibility and electrochemical performance [8,9].

Although crystalline and amorphous NP synthesized at different calcination temperatures have been reported, the correlation between the progressive structural evolution and electrochemical charge-storage kinetics remains insufficiently comprehended. Therefore, in the present study, the calcination temperature interval (300, 450, 600, 750 and 900 °C) has been narrowed to systematically correlate the structural evolution of NP from an amorphous to a more crystalline structure with its specific capacity, ion-transport behaviour and charge-storage kinetics. The findings provide further insight into calcination condition and corresponding crystal structure that provide the best electrochemical performance for NP electrodes.

MATERIALS AND METHODS

Nickel(II) acetate tetrahydrate ($\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$, 99%), anhydrous disodium hydrogen phosphate (Na_2HPO_4 , 99%) and acetylene black (100%) were purchased from Thermo Scientific, Germany), ethylene glycol (EG, 99.9%, Chemiz, Malaysia), polyvinylidene fluoride (PVdF, 99%, Sigma-Aldrich, USA), N-methyl-2-pyrrolidone (NMP, 99%, Sisco Research Laboratories, India) and potassium hydroxide (KOH, 85%, Sigma-Aldrich, USA) were used in this study. A platinum wire (99.99%, Sigma-Aldrich, USA) and an Hg/HgO electrode (Metrohm, Switzerland) were used as the counter and reference electrodes, respectively.

Preparation of synthesized NP

NP samples were synthesized using the sonochemical method and post-calcination treatment. Briefly, the samples were prepared by mixing nickel salt, EG and phosphate source and were kept stirring for a few minutes to ensure sufficient mixing of the reactants, followed by probe sonication for 30 min (350 watt, Fisherbrand™ Model 505

Sonic Dismembrator). After the samples were washed and dried under 50 °C for overnight, they were calcined at 300 °C for 3 h. The process was repeated for other samples, except the calcination temperature was varied at 450, 600, 750 and 900 °C (each sample was labelled as NP450, NP600, NP750 and NP900) to study its correlation with the structural evolution and electrochemical performance.

Characterizations

The synthesized samples were characterized using various physicochemical techniques. X-ray diffraction (XRD) analysis was performed using an X-ray diffractometer with Cu K α radiation ($\lambda = 1.5418 \text{ \AA}$) within the 2θ range of 5–80° at ambient temperature to determine the structural and phase properties of NP at different EG concentrations. Fourier-transform infrared spectroscopy (FTIR) analysis was carried out using a PerkinElmer Spectrum 400 spectrometer to identify the functional groups and vibrational characteristics of the synthesized materials. Field emission scanning electron microscopy (FESEM) analysis was performed using a ZEISS SUPRA 55VP microscope to investigate the surface morphology and particle size distribution of the samples, while transmission electron microscopy (TEM) was employed to examine the pore formation and internal structural features using a Talos L 120C with magnification up to 600 K. In addition, Brunauer–Emmett–Teller (BET) analysis was conducted to evaluate the specific surface area and pore size distribution of the synthesized NP samples.

Electrochemical characterization

The electrochemical measurements were carried out using an Autolab PGSTAT302N Metrohm in a three-electrode configuration with 1 M KOH as the electrolyte, while platinum wire and Hg/HgO were used as the counter and reference electrodes, respectively. The working electrode slurry was prepared by mixing calcined NP, acetylene black (AC) and PVDF in NMP at a weight ratio of 75:15:10 under continuous stirring overnight. The obtained slurry was then coated onto cleaned nickel foam (1 cm $\times$ 1 cm) using the doctor-blade technique which enables the slurry to be spread evenly across the substrate surface and dried at 60 °C overnight, resulting in an average active material loading of approximately $\sim 3.7 \text{ mg}$. Subsequently, cyclic voltammetry (CV), galvanostatic charge–discharge (GCD) and electrochemical impedance spectroscopy (EIS) measurements were conducted to evaluate the electrochemical performance of the prepared electrodes.

RESULTS AND DISCUSSION

Influence of EG Concentration on Nickel Phosphate Structure and Pore Formation

Figure 1(a) shows XRD diffractogram patterns of nickel phosphate calcined at different temperatures. NP300, NP450 and NP600 exhibit broad diffraction, indicating that the nickel phosphate framework remains largely amorphous at temperatures up to 600 °C. This amorphous structure stability stems from the bulky phosphate framework that forms strong covalent-like bonds with nickel ions which requires higher thermal energy for structural rearrangement. As the calcination temperature increased to 750 °C, a series of peaks were observed for NP750 and became more intense, as seen in NP900 when the calcination temperature reached 900 °C. No additional peaks appeared, which signifies

that NP has high thermal stability. This change in crystallinity can be ascribed to the increase in thermal energy that arrange the atoms into their preferred orientation, establishing long-range order of a monoclinic crystal lattice with $P2_1/a$ space group (PDF 35-0987) [10].

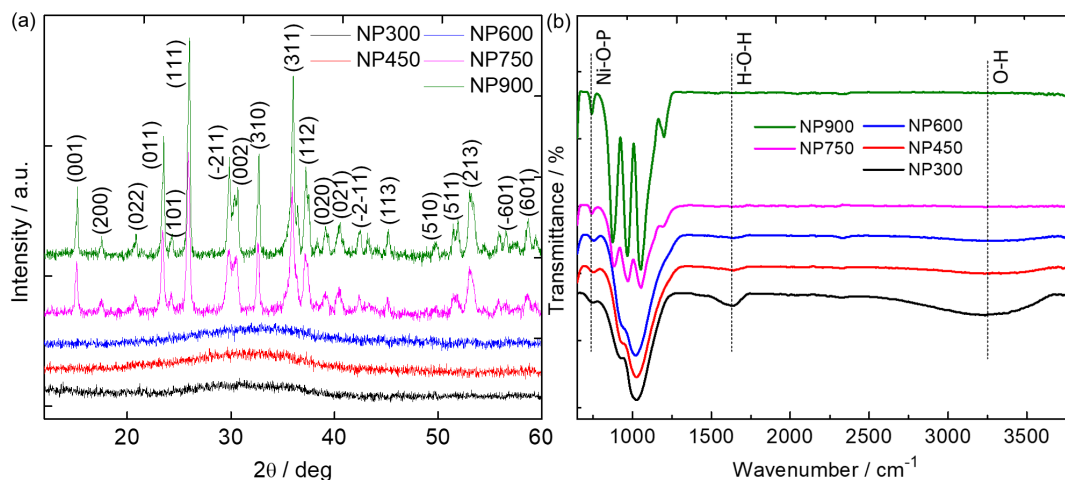


Figure 1. (a) XRD patterns, (b) FTIR spectra, (c) pore size distribution and (d) BET surface area of NP300, NP450, NP600, NP750 and NP900

The structural transformation observed in XRD is strongly supported by FTIR analysis (Figure 1(b)). All samples exhibit characteristic vibrational bands corresponding to phosphate groups, confirming the formation of nickel phosphate across all calcination temperatures. The bands at 752 cm^{-1} and 883 cm^{-1} are assigned to P–O–P bending vibrations, indicating the connectivity between phosphate units in the network structure. Meanwhile, the peaks at 920 cm^{-1} , 968 cm^{-1} , 1023 cm^{-1} , 1054 cm^{-1} and 1190 cm^{-1} correspond to P–O stretching vibrations within PO_4^{3-} tetrahedra, reflecting different local bonding environments of phosphate species¹¹. The presence of multiple overlapping P–O bands suggests a variation in structural symmetry, which becomes more defined as calcination temperature increases due to progressive ordering of the phosphate framework. In addition, the broad band at 3239 cm^{-1} is attributed to O–H stretching vibrations, while the peak at 1638 cm^{-1} corresponds to H–O–H bending vibrations from adsorbed water molecules. These hydroxyl-related features are more prominent in the NP300 sample, indicating the retention of surface –OH groups and physically adsorbed water. However, with increasing calcination temperature, the gradual weakening of O–H-related bands indicates dehydration and removal of surface hydroxyl groups, leading to reduced electrolyte affinity and lower ion accessibility [12].

The morphology evolution of the samples was investigated through FESEM (Figure 2(a–e)) and TEM images (Figure 2(f)–2(j)). NP300, NP 450 and NP600 show similar particle size $\sim 80\text{--}100 \text{ nm}$ with agglomeration. However, the difference can be seen in the size of the intraparticle pores, where NP300, NP 450 and NP600 possess 3, 6 and 4 nm, respectively. At 300°C , the formation of intraparticle pores is due to the partial dehydration and thermal decomposition of EG (boiling point $\sim 197^\circ\text{C}$), generating gaseous species that create internal voids within the NP framework. At 450°C , more absorbed water and EG were removed, resulting in the connection of adjacent small voids,

thus increasing intraparticle pore size. At 600 °C, atoms diffused and facilitated short-range arrangement of the Ni-O-P framework, slightly shrinking the intraparticle pores. The particle distribution becomes denser with increasing calcination temperature, where NP750 and NP900 exhibit a more compact morphology with the disappearance of intraparticle pores. The Ni-O-P framework arrangement extends over a sufficiently large distance to produce particle sizes (NP750: ~120 nm and NP900: 200-300 nm) with a crystalline structure.

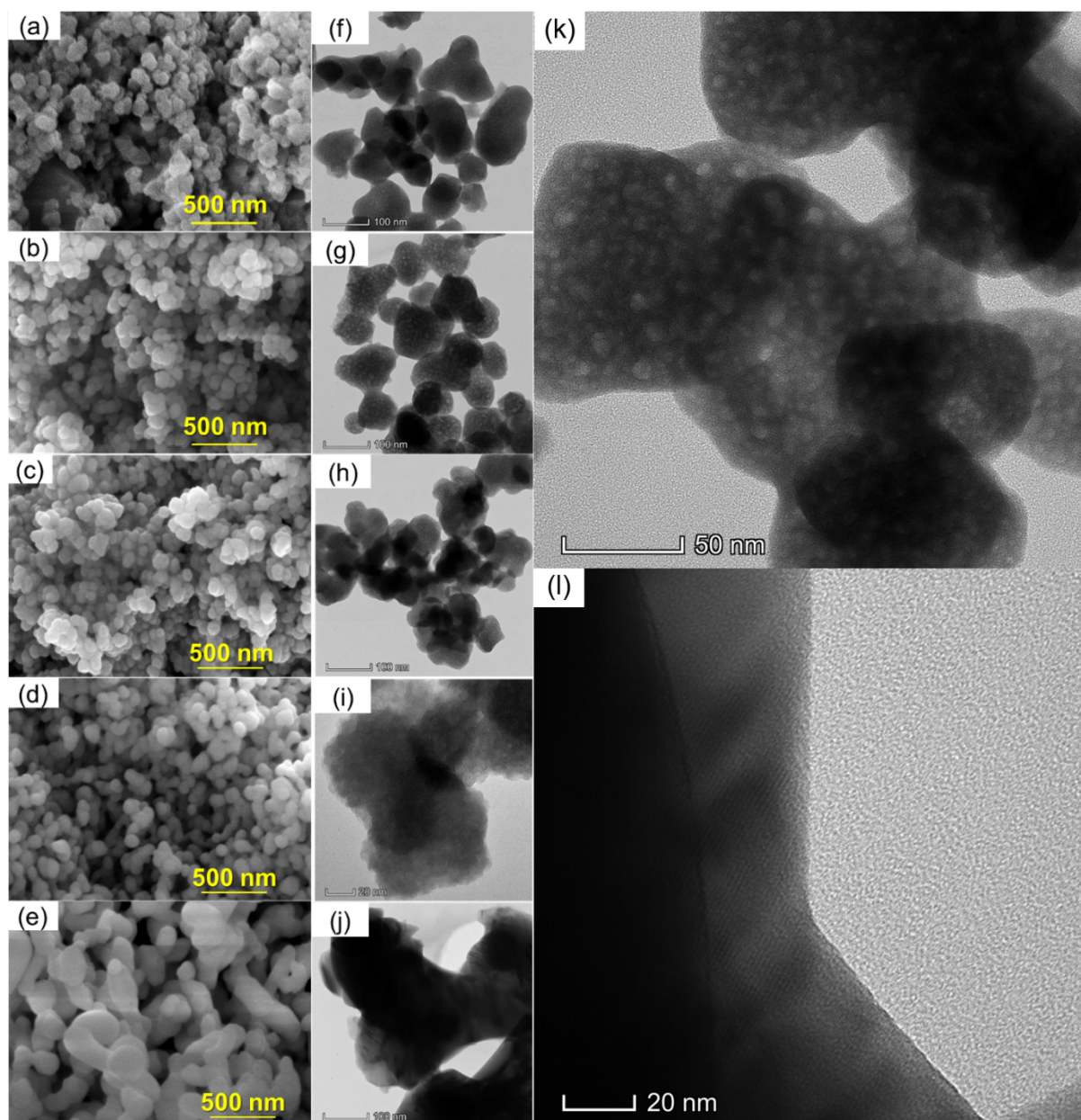


Figure 2. FESEM image of (a) NP300, (b) NP450, (c) NP600, (d) NP750, (e) NP900; TEM image of (f) NP300, (g) NP450, (h) NP600, (i) NP750, (j) NP900; High-magnification TEM image of (k) NP450 and (l) NP900

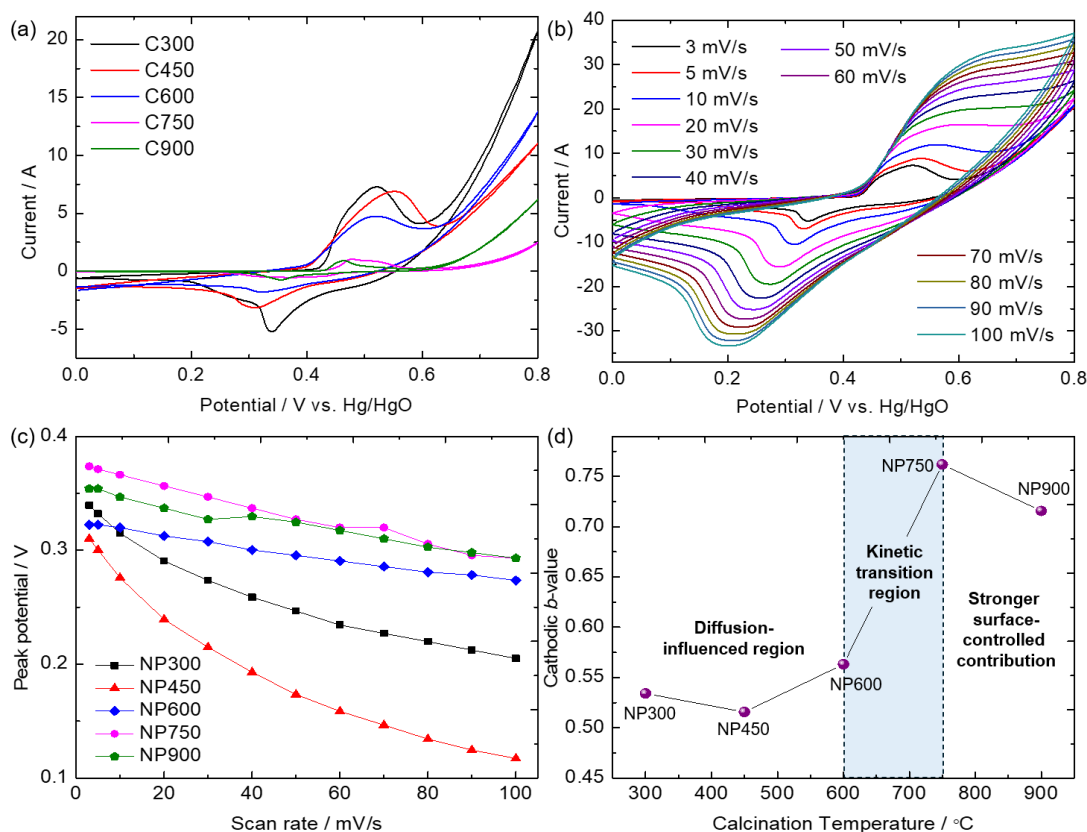
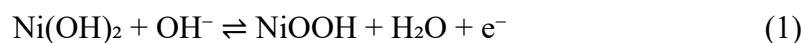


Figure 3. CV curves of (a) all electrodes recorded at 3 mV/s and (b) NP300 recorded at scan rates from 3 to 100 mV/s, (c) the cathodic peak potential with scan rate for all electrodes, (d) cathodic b -values as a function of calcination temperature, obtained from the slope of the $\log |i_p|$ versus $\log v$ relationship

The correlation between the microstructure of nickel phosphate calcined at different temperatures and its electrochemical performance is illustrated in Figure 3. Figure 3(a) shows the CV curves of NP300, NP450, NP600, NP750 and NP900 at 3 mV s⁻¹, where all samples exhibit well-defined redox peaks corresponding to the reversible Faradaic reactions between Ni²⁺/Ni³⁺ species and OH⁻ ions from the electrolyte [13]. The charge storage mechanism can be described as:



Among all samples, NP300 exhibits the largest integrated CV area, followed by NP450, NP600, NP750 and NP900, indicating that NP300 possesses the highest charge storage capability. This superior performance is attributed to its amorphous structure as confirmed by XRD, where the disordered Ni–O–P framework provides abundant defect sites and unsaturated coordination environments for redox reactions [14]. In addition, the presence of higher hydroxyl groups enhances its hydrophilicity, promoting efficient adsorption and diffusion of OH⁻ ions at the electrode–electrolyte interface, thereby accelerating redox activity [15]. Figure 3(b) and Figure S1 present the CV curves of NP300, NP450, NP600, NP750 and NP900 at different scan rates, where the anodic and cathodic peaks shift towards higher and lower potentials, respectively, with increasing

scan rate. This behaviour is attributed to polarization effects and diffusion limitations at higher scan rates, where insufficient time is available for complete ion diffusion and redox equilibration within the electrode structure [16]. However, the displacement of the peaks over increasing scan rates is different for each sample and thus, the cathodic peak displacement is plotted in Figure 3(c). At low calcination temperature, NP300 and NP450 exhibit stronger cathodic displacement, whereas at the higher calcination temperature, NP750 and NP900 maintain more stable cathodic peak potentials at increasing scan rate. Note that the cathodic peaks were selected instead of anodic peaks because the anodic peaks are not resolved for NP300, NP450 and NP600 at higher scan rates (>20 mV/s). This indicates that the samples calcined at higher temperatures have better high-rate redox reversibility than their counterparts. The charge storage mechanism and kinetics of the electrodes were studied via the power-law relationship $i_p = av^b$ as displayed in Figure 3(d). Generally, the b -value reveals how fast electrolyte ions and electrons move during the charging and discharging process. A value of $b \sim 0.5$ indicates a diffusion-controlled process where ion diffusion into the bulk electrode material limits the reaction rate. While $b \sim 1$ indicates a surface-controlled capacitive process, which is typically associated with the characteristic of electric double-layer capacitance or rapid surface Faradaic reactions [17]. NP300, NP450 and NP600 exhibit b -values of 0.53, 0.52 and 0.56, respectively, which are close to the theoretical value of 0.5 associated with diffusion-influenced Faradaic processes. These results suggest that the electrolyte ion transport toward the electroactive sites strongly governs the cathodic response of the lower-calcination temperature samples. In contrast, NP750 and NP900 exhibit higher b -values of 0.76 and 0.72, respectively, which signifies increasing the surface-controlled contribution to the overall current response. This coincides with the development of a more crystalline structure that facilitates more continuous electronic transport and promotes faster interfacial redox reactions. The slightly lower b -value of NP900 relative to NP750 further suggests that even though NP900 has a higher degree of crystallization, it does not continuously enhance the reaction kinetics. This might be due to reduced defect density or electrochemically accessible interfaces when the sample was calcined at the highest temperature. Between 600 and 750 °C, the b -value increases, indicating a shift from predominantly diffusion-influenced Faradaic behavior toward a stronger surface-controlled contribution. This matches the structural evolution as discussed under XRD data.

Figure 4(a) shows the GCD curves of all samples at 1 A g^{-1} . All electrodes exhibit quasi-symmetric non-linear profiles, confirming their Faradaic charge storage behaviour. Consistent with the CV results, NP300 displays the longest discharge time, indicating the highest specific capacity and the most efficient utilization of electroactive sites. The enhanced performance of NP300 is further associated with its highly porous and defect-rich structure, which facilitates deeper electrolyte penetration and efficient OH^- ion transport [18]. Figure 4(b) and Figure S2 illustrate the GCD curves of NP300 at different current densities, where the discharge time gradually decreases with increasing current density. This is due to the limited diffusion of OH^- ions and insufficient time for complete Faradaic redox reactions at higher current rates. The specific capacity values calculated from the discharge curves are presented in Figure 4(c). NP300 exhibits the highest specific capacity at all current densities, confirming its superior charge storage performance, with its maximum specific capacity of 186 C g^{-1} at 1 A g^{-1} , followed by NP

450, NP600, NP750 and NP900. This indicates that the particle size and internal voids provide additional pathways for electrolyte penetration and ion diffusion, which are favorable for electrochemical reactions. However, NP450 exhibited a lower specific capacity than NP300 even though its intraparticle pores are larger than those of NP450, suggesting that there is no correlation between interparticle pores and electrochemical performance [19].

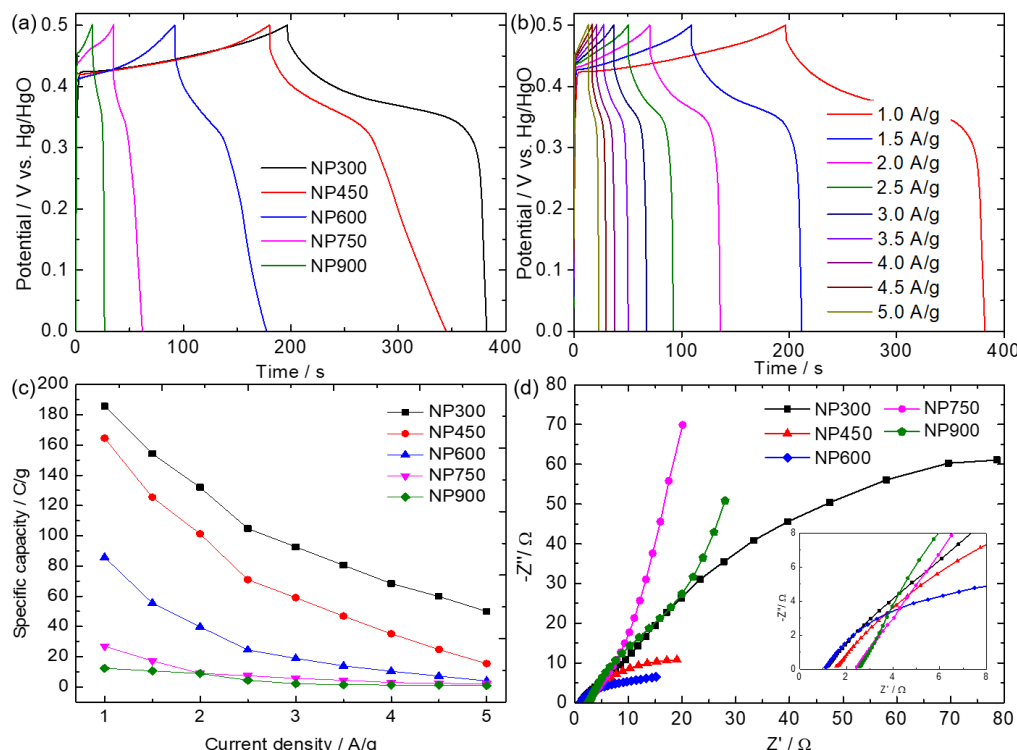


Figure 4. GCD curve of (a) all electrodes at 1 A/g and (b) NP300 at different current densities; (c) Specific capacity of all electrodes at different current densities; (d) Nyquist plot of all electrodes (inset: higher magnification)

Figure 4(d) presents the Nyquist plots and their higher magnification (inset) of all samples. The intercepts on the Z' axis in the higher frequency region represent the equivalent series resistance, R_s , which includes the electrolyte resistance, intrinsic electrode resistance and contact resistance [20]. All electrodes show a comparable value, indicating that the ohmic effect is comparable among all electrodes. More pronounced differences are observed in the low-frequency region, which agrees with the cathodic b-value analysis. NP300, NP450 and NP600 exhibit relatively inclined impedance curves, indicating a greater contribution from diffusion-controlled ion transport. In contrast, NP750 and NP900 display steeper curves, suggesting increasingly surface- or interface-controlled electrochemical behaviour [21].

CONCLUSIONS

NP was synthesized via a sonochemical route with post-calcination treatment by applying different calcination temperatures. Through XRD diffractograms and FTIR spectra, the crystallinity of NP was progressively changed from amorphous to crystalline as the calcination temperature increased and the adsorbed water reduced when the calcination temperature increased from 300-900 °C. In addition, from FESEM and HRTEM analysis, the particle size increases from 80 to 100 nm, with the presence of intraparticle pores at NP calcined at lower temperature range (300-600 °C). This ordering becomes more pronounced for NP calcined at higher temperature range (750-900 °C), where extensive crystallization leads to a highly compact structure with disappearance of intraparticle pores. Through the electrochemical studies, NP calcined at higher temperature has better high-rate redox reversibility than their counterparts contributed by its surface-controlled contribution to the overall current response. In addition, this also indicates that faster kinetics at the accessible sites do not necessarily correspond to greater active-material utilization.

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AUTHOR CONTRIBUTIONS

Investigation, writing-original draft preparation: Alia Rosli; Writing-review, resources, visualization, project administration and funding acquisition: F.S. Omar; Data curation, visualization: Alia Rosli; Investigation, experimental work: Aisyah Azizi; Writing-review: F.S. Omar and Aisyah Azizi. All authors read and approved the final manuscript.

DATA AVAILABILITY

The data supporting the findings of this study are available from the corresponding author upon reasonable request.

CONFLICT OF INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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