Flame spray-pyrolyzed vanadium oxide nanoparticles for lithium battery cathodes

See How Ng
*University of Wollongong, shn076@uow.edu.au*

T J. Patey
*Paul Scherrer Institut, Switzerland*

R Buchel
*Dep. Mechanical Process Eng. Switzerland*

F Krumeich
*Lab of Inorganic Chemistry, Switzerland*

Jiazhao Wang
*University of Wollongong, jiazhao@uow.edu.au*

*See next page for additional authors*

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**Recommended Citation**
Vanadium pentoxide (V$_2$O$_5$) nanoparticles (30–60 nm) were made by a one-step and scalable flame spray pyrolysis (FSP) process. Optimization of the FSP processing conditions (precursor concentration and injection rate) enhanced the electrochemical performance of these nanoparticles. Increasing the cut-off potential for discharging from 1.5 to 2.5 V vs. Li/Li$^+$ improved the cycle life of these V$_2$O$_5$ nanoparticles. Particles with the lowest specific surface area ($\sim$32 m$^2$ g$^{-1}$) and highest phase purity (up to 98 wt%) showed excellent cyclability between 2.5 and 4.0 V vs. Li/Li$^+$, retaining a specific charge of 110 mAh g$^{-1}$ beyond 100 cycles at a specific current of 100 mA g$^{-1}$, and also superior specific charge of 100 mAh g$^{-1}$ at specific current up to 20C rate (or 2000 mA g$^{-1}$).

Introduction

Vanadium pentoxide or vanadia (V$_2$O$_5$) is one of the few oxides having versatile redox-dependent properties due to the multiple valence state of vanadium, and therefore, finds wide applications in catalysis, electrochromism, and electrochemistry. Nanostructured forms of V$_2$O$_5$ have been employed in field-effect transistors (FETs), sensors, spintronic devices, and nanolithography templates. In the field of rechargeable lithium batteries, besides low cost and abundant source, it is argued that V$_2$O$_5$ is an attractive cathode material owing to its unique features such as high electrochemical activity, high energy density, and high rate cyclability towards lithium insertion. Electrochemical reduction of V$_2$O$_5$ can occur in a large potential window between 4.0 to 1.5 V vs. Li/Li$^+$, where approximately three moles of lithium per mole of V$_2$O$_5$ could be theoretically inserted, leading to a theoretical specific charge of approximately 442 mAh g$^{-1}$.

The reversible electrochemical lithium intercalation into V$_2$O$_5$ at room temperature was first reported by Whittingham in 1976. Depending on the amount of inserted lithium, several phase transitions of Li$_x$V$_2$O$_5$ in consecutive steps occur, namely $\alpha$ (for $x < 0.01$), $\varepsilon$ (0.35 < $x < 0.7$), $\delta$ (for $x = 1.0$) phases, respectively. For $x < 1$, the original V$_2$O$_5$ structure can be recovered upon lithium delithiation, and the phase transitions are fully reversible. However, as lithiation progresses ($x > 1$), a reconstruction occurs leading to a partially irreversible transformation from $\delta$-phase to $\gamma$-phase. This $\gamma$-phase can only be reversibly cycled in the stoichiometric range 0 < $x < 2$ without changing the $\gamma$-type structure. Upon further lithiation (up to $x = 3$), the $\gamma$-phase will be irreversibly transformed to the $\alpha$-phase with a rock-salt type structure. The Li$^+$ intercalation and deintercalation process can be expressed by the following overall equation:

$$V_2O_5 + xLi^+ + xe^- \rightleftharpoons Li_xV_2O_5 \quad (0 < x < 3)$$

In order to achieve a higher specific charge and better cyclability, extensive studies have been done on modifying the form and the structure of V$_2$O$_5$ as its electrochemical performance as V$_2$O$_5$ cathode depends on its degree of crystallinity and morphology. These studies showed that crystalline V$_2$O$_5$ has a high specific charge but has poor cycle life behaviour since its crystal structure is damaged by prolonged charge/discharge cycles. Meanwhile, amorphous and low crystallinity V$_2$O$_5$ allows faster lithium-ion diffusion and displays superior cyclability. Crystal deformation associated with lithiation may be relaxed in small crystallites with a high surface area that also leads to higher ionic conductivity. Therefore, nanocrystalline V$_2$O$_5$ has a promise as alternative cathode material in lithium batteries.
Vanadia nanoparticles can be obtained by various physical and chemical techniques. Such powders have been prepared mostly by dry processes, such as vacuum evaporation,\textsuperscript{26–27} and sputtering,\textsuperscript{28} although wet processes such as electrodeposition\textsuperscript{29} and sol-gel synthesis\textsuperscript{30} seem to be more advantageous in producing nanoparticles-based thin films on a large scale. Previously, we managed to obtain V$_2$O$_5$ nanoparticles by precipitation followed by heating in vacuum at 300 °C.\textsuperscript{25,25} In addition, we also made V$_2$O$_5$ nanoparticles by flame spray pyrolysis (FSP) as secondary composite materials with the TiO$_2$ and SiO$_2$ for use in catalysis,\textsuperscript{32–33} and even FSP-made LiV$_3$O$_8$ nanoparticles for lithium battery cathode materials.\textsuperscript{34} Here, crystalline V$_2$O$_5$ nanoparticles have been made by FSP to explore and evaluate their electrochemical performance for use as lithium battery cathodes focusing on the cut-off potential and the rate capability.

2. Experimental

2.1 Material synthesis

The experimental procedure for the FSP process is described in detail elsewhere.\textsuperscript{35} Here the precursor solution is prepared by first dissolving vanadium(V) oxytripropoxide (Aldrich, 98%) into 30 mL of diethylene glycol (DEG, Fluka). This solution was then stirred into 30 mL of toluene (Riedel de Haen) and 20 mL of tetrahydrofuran (THF, Fluka). By varying the amount of vanadium(V) oxytripropoxide in the precursors, the vanadium molar precursor concentrations ranged from 0.4 to 1.5 M. The precursor solution was injected at 3 to 6 mL min$^{-1}$ through the reactor nozzle and dispersed with 5.0 L min$^{-1}$ of oxygen into a fine spray while maintaining a constant pressure drop of 1.5 bar across the nozzle tip. A premixed flame fueled by 1.3 L min$^{-1}$ of methane and 3.0 L min$^{-1}$ of oxygen was maintained to ignite and support the combustion of the spray. A sheath gas of 5.0 L min$^{-1}$ of oxygen surrounding the flame was used to ensure complete combustion. The powder was collected by placing a glass fiber filter (GF/D Whatman, 257 mm in diameter) above the flame and drawing the gas streams with a vacuum pump. The reactor nozzle was cooled by water to prevent overheating and precursor evaporation within the liquid feed lines.

2.2 Material characterization

The powders were characterized by X-ray diffraction (XRD) using a Bruker AXS D8 Advance (40 kV, 40 mA) diffractometer and analyzed with the Topas 2 software. The XRD measurement was performed at a continuous scan between 2θ angles of 10 and 70° at a scan rate of 0.03° min$^{-1}$. Analysis by field-emission scanning electron microscopy (FE-SEM) was performed with a Zeiss Gemini 1530 operated at 1 kV. For the investigation by transmission electron microscopy (TEM), the material was deposited onto a holey carbon foil supported on a copper grid. TEM investigations were performed using a CM30ST microscope (Philips; LaB6 cathode, operated at 300 kV, point resolution $\sim$2 Å). The Brunauer–Emmett–Teller specific surface area ($S_{\text{BET}}$) of the FSP-produced nanoparticles was determined through a five-point nitrogen adsorption isotherm at 77 K (Tristar, Micrometrics Instruments Corp.) after degassing the powder samples with nitrogen at 150 °C. Assuming spherical, monodisperse primary particles with homogeneous density, the average particle size ($D_{\text{BET}}$) in microns is equalled to $6/(S_{\text{BET}} \cdot \rho_w)$, where $\rho_w$ is the average density of the flame-made VO$_2$ (based on the mass fractions of V$_2$O$_5$ and VO$_2$, respectively) nanoparticles with $\rho_{V_2O_5} = 3.4$ g cm$^{-3}$ and $\rho_{VO_2} = 4.7$ g cm$^{-3}$.

2.3 Electrode preparation and cell assembly

The cathode was prepared by mixing FSP-made nanocrystalline V$_2$O$_5$ or commercially available micro-sized V$_2$O$_5$ particles (Sigma-Aldrich) as electroactive materials with 20 wt% carbon black (Super P, TIMCAL, Belgium) and 10 wt% polyvinylidene fluoride (PVDF, Sigma-Aldrich) binder in N-methyl-2-pyrrolidinone (NMP, Merck GmbH) solvent to form a viscous slurry. Subsequently, the slurry was doctor-bladed at a thickness of 200 μm onto an aluminum foil and dried under vacuum at 110 °C overnight. Circular electrodes with a diameter of 13 mm were punched out and dried in a vacuum chamber at 120 °C overnight, each with a typical active material mass loading of 2–3 mg cm$^{-2}$. Hermetically sealed laboratory test cells were used in which the working and counter electrodes (metallic lithium, Aldrich, 99.9%) were slightly pressed together (at 2 kg cm$^{-2}$) against a glass fiber separator soaked with 500 μL of standard battery electrolyte [1 M LiPF$_6$ in ethylene carbonate (EC)/dimethyl carbonate (DMC) (1/1 by weight), provided by Ferro GmbH]. Test cells were assembled in an argon-filled glove box with oxygen and water content each less than 1 ppm.

2.4 Electrochemical measurements

Both the cyclic voltammetry (CV) and galvanostatic measurements were performed by a computer-controlled cell capture (CCCC) system (Astrol Electronics AG, Oberrohrdorf, Switzerland), by discharging (Li$^+$ insertion) first from the respective open-circuit potential. The CV was conducted between 1.5–4.0 V vs. Li/Li$^+$ at a potential scan rate of 0.1 mV s$^{-1}$. Meanwhile, for the galvanostatic measurements electrodes were cycled between 1.5–4.0 V vs. Li/Li$^+$ at specific currents ranging from 100 to 2000 mA g$^{-1}$ (based on the oxide weight). In order to promote complete discharge/charge at the respective potential limits, a potentiostatic step was included until the current was 10% of the current used in the galvanostatic step.

3. Results and discussion

3.1 Structure and morphology analysis of V$_2$O$_5$ nanoparticles

Vanadia nanoparticles can be obtained by various physical and chemical techniques. Such powders have been prepared mostly by dry processes, such as vacuum evaporation,\textsuperscript{26–27} and sputtering,\textsuperscript{28} although wet processes such as electrodeposition\textsuperscript{29} and sol-gel synthesis\textsuperscript{30} seem to be more advantageous in producing nanoparticles-based thin films on a large scale. Previously, we managed to obtain V$_2$O$_5$ nanoparticles by precipitation followed by heating in vacuum at 300 °C.\textsuperscript{25,25} In addition, we also made V$_2$O$_5$ nanoparticles by flame spray pyrolysis (FSP) as secondary composite materials with the TiO$_2$ and SiO$_2$ for use in catalysis,\textsuperscript{32–33} and even FSP-made LiV$_3$O$_8$ nanoparticles for lithium battery cathode materials.\textsuperscript{34} Here, crystalline V$_2$O$_5$ nanoparticles have been made by FSP to explore and evaluate their electrochemical performance for use as lithium battery cathodes focusing on the cut-off potential and the rate capability.
The processing conditions for the FSP were varied in order to investigate and optimize the physical properties of the flame spray pyrolyzed V2O5 nanoparticles, such as degree of crystallinity, average particle size (\(D_{\text{BET}}\)) and morphology, and final composition. The physical characteristics of 6 different V2O5 nanoparticles samples are summarized in Table 1.

### Table 1 FSP operating conditions and the corresponding physical properties of the V2O5 nanoparticles produced

<table>
<thead>
<tr>
<th>Sample</th>
<th>(C_F)/M</th>
<th>(Q_F)/mL min (^{-1})</th>
<th>(S_{\text{BET}})/m(^2) g (^{-1})</th>
<th>(D_{\text{BET}})/nm</th>
<th>(V_2O_5)/wt%</th>
<th>(VO_2)/wt%</th>
<th>(V_2O_3)/nm</th>
<th>(VO_2)/nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>FSP-1</td>
<td>0.40</td>
<td>5</td>
<td>60.5</td>
<td>29</td>
<td>87.6</td>
<td>12.4</td>
<td>28</td>
<td>31</td>
</tr>
<tr>
<td>FSP-2</td>
<td>0.75</td>
<td>5</td>
<td>46.1</td>
<td>39</td>
<td>97.5</td>
<td>2.5</td>
<td>24</td>
<td>34</td>
</tr>
<tr>
<td>FSP-3</td>
<td>1.50</td>
<td>5</td>
<td>40.7</td>
<td>44</td>
<td>97.6</td>
<td>2.4</td>
<td>23</td>
<td>35</td>
</tr>
<tr>
<td>FSP-4</td>
<td>1.50</td>
<td>3</td>
<td>53.3</td>
<td>33</td>
<td>93.4</td>
<td>6.6</td>
<td>28</td>
<td>32</td>
</tr>
<tr>
<td>FSP-5</td>
<td>1.50</td>
<td>4</td>
<td>44.3</td>
<td>41</td>
<td>97.5</td>
<td>2.5</td>
<td>20</td>
<td>41</td>
</tr>
<tr>
<td>FSP-6</td>
<td>1.50</td>
<td>6</td>
<td>31.9</td>
<td>56</td>
<td>97.8</td>
<td>2.2</td>
<td>26</td>
<td>39</td>
</tr>
<tr>
<td>Aldrich</td>
<td>N/A</td>
<td>N/A</td>
<td>4.1</td>
<td>440</td>
<td>100.0</td>
<td>0.0</td>
<td>&gt;100</td>
<td>N/A</td>
</tr>
</tbody>
</table>

\(a\) \(C_F\) is the precursor vanadium concentration. \(b\) \(Q_F\) is the precursor feed rate. \(c\) \(S_{\text{BET}}\) is the specific surface area measured with Brunauer-Emmett-Teller (BET) method. \(d\) \(D_{\text{BET}}\) is the average particle size calculated from \(S_{\text{BET}}\) measured. The respective compositions of the \(V_2O_5\) and the \(VO_2\) were calculated using the TOPAS 2 software from the XRD spectra. \(e\) \(D_{\text{XRD}}\) is the crystal size calculated based on the peak (001) for \(V_2O_5\) and the peak (110) for \(VO_2\).

As can be seen from Table 1, the increase in feed precursor’s molar concentration (\(C_F\)) from 0.4 to 0.75 M (at constant feed injection rate, \(Q_F\)) resulted in an increase in \(V_2O_5\) content from 87.6 to 97.5 wt\%. Further increase of \(C_F\) to 1.50 M increased the \(V_2O_5\) content only marginally (0.1 wt\%). This is consistent with Schimmoeller et al.\(^{37}\) who also observed increased \(V_2O_5\) content with increasing V-concentration in the precursor solution in their FSP-made \(V_2O_5\)–TiO\(_2\) particles. For FSP-1, the particle size (\(D_{\text{BET}}\)) is roughly equal to the crystal size (\(D_{\text{XRD}}\)), indicating that the powder is composed of monocrystalline particles. For FSP-2 and FSP-3, \(D_{\text{BET}}\) increases and \(D_{\text{XRD}}\) slightly decreases for increasing \(C_F\), suggesting these particles are composed of multiple crystals and are therefore polycrystalline. When \(Q_F\) was increased from 3 to 6 mL min \(^{-1}\) at a \(C_F\) of 1.5 M, polycrystallinity was increased further (caused by vanishing of the detected VO\(_2\) phase)\(^{37}\) as the \(S_{\text{BET}}\) was reduced even more to 31.9 m\(^2\) g \(^{-1}\) without significantly increasing the crystal size (\(D_{\text{XRD}}\approx 28\) nm). The reduction of \(S_{\text{BET}}\) from 53.3 m\(^2\) g \(^{-1}\) (FSP-4) to 31.9 m\(^2\) g \(^{-1}\) (FSP-6) corresponds to the growth of the particles with a \(D_{\text{BET}}\) from 33 nm (FSP-4) to 56 nm (FSP-6). As seen from the difference in particle (\(D_{\text{BET}}\)) and crystal size (\(D_{\text{XRD}}\)) at higher \(Q_F\) rates, particles are sintered rather than coagulated. This observation is consistent with the effect of precursor concentration in TiO\(_2\) produced by flame synthesis.\(^{38}\) These properties will be beneficial from the electrochemistry point of view since smaller crystals lead to faster solid-state diffusion kinetics. The higher specific surface area (m\(^2\) g \(^{-1}\)) of the smaller crystals permits more Li ions to be transferred per unit time from the electrolyte to the nanoparticles for the same mass of electroactive material. Meanwhile lower surface area will provide less unwanted surface reactions such as dissolution of the active transition metal and the irreversible charge loss from surface film formation during the first charge.

Typical XRD patterns for the 6 different \(V_2O_5\) nanoparticles powder samples produced by FSP and for the commercially available \(V_2O_5\) micro-sized particles are shown in Fig. 1. Most of the peak positions for the flame spray pyrolyzed \(V_2O_5\) nanoparticles agree well with those of the orthorhombic \(V_2O_5\) (JCPDS 41-1426; \(a = 11.5160\) Å, \(b = 3.5656\) Å, and \(c = 4.3727\) Å), except for the 2 peaks marked as “@” and “#” which correspond to the (110) and (011) peaks from the monoclinic VO\(_2\) (JCPDS 43-1051) impurities present, as observed also by Schimmoeller et al.\(^{37}\) Moreover, based on the fundamental parameter approach and the Rietveld method,\(^{39}\) \(D_{\text{XRD}}\) ranging from 20 to 28 nm was estimated from peak (001) of the \(V_2O_5\) phase (see Fig. 1a–f, also listed in Table 1). Note that Table 1 shows that the present flame-made \(V_2O_5\) nanoparticles had high \(S_{\text{BET}}\) (32–61 m\(^2\) g \(^{-1}\)) which is an order of magnitude higher than that of the commercially available \(V_2O_5\) (~4 m\(^2\) g \(^{-1}\)).
Fig. 1 X-Ray diffraction (XRD) patterns of FSP-made V$_2$O$_5$ nanoparticles (a)–(f) and V$_2$O$_5$ microparticles from Aldrich (g). The impurities phase VO$_2$ is indicated as “@” (110) and “#” (011).

Fig. 2 shows a typical field emission scanning electron microscope (FE-SEM) images of the flame spray pyrolyzed V$_2$O$_5$ nanoparticles, which is sample FSP-6 in this case. In the low-magnification image (Fig. 2a), sphere-like V$_2$O$_5$ nanoparticles are seen with a fairly homogeneous particle size distribution. In the high-magnification image (Fig. 2b), flame-made V$_2$O$_5$ nanoparticles are seen with diameters ranging from 30–60 nm. This is in good agreement with the calculations of $D_{XRD}$, which is the average size of the V$_2$O$_5$ crystals based on the weight fraction of the V$_2$O$_5$ phase. However, as seen in Fig. 2b, the majority of particles are approximately 30 nm in diameter, and therefore in the same size range of those estimated from the XRD patterns in Fig. 1.
Fig. 2 FE-SEM images of V$_2$O$_5$ nanoparticles (here FSP-6) at (a) low and (b) high magnifications. The spherical-shaped particles are almost monodisperse with sizes ranging from 30–60 nm.

Fig. 3a and 3b show typical transmission electron microscope (TEM) images of the present V$_2$O$_5$ nanoparticles indicating that the sphere-like V$_2$O$_5$ primary particles are connected to each other by sintered necks to form chain-like aggregates, seen in related work on LiV$_3$O$_8$. Furthermore, from the high-resolution TEM image in Fig. 3(c), the crystalline structure of the flame-made V$_2$O$_5$ nanoparticles is evidenced by their clearly visible lattice fringes. The measured distance of ca. 0.652 nm corresponds to half of the $a$-axis. The corresponding selected area electron diffraction (SAED) pattern for the V$_2$O$_5$ nanoparticles in Fig. 3b is shown in Fig. 3d, revealing the crystal lattice parameter of the V$_2$O$_5$ nanoparticles, in accord with the orthorhombic phase of V$_2$O$_5$ (JCPDS 41-1426).
3.2 Electrochemical performance of V$_2$O$_5$ nanoparticles

Fig. 4a–c show the cyclic voltammograms (CVs) of electrodes made from the FSP-6 V$_2$O$_5$ nanoparticles. The CVs were taken at a scan rate of 0.1 mV s$^{-1}$ with cycling at different lower (discharge) cut-off potentials. From Fig. 4a, it can be seen that during the cathodic scanning in the first cycle, four distinctive peaks are observed at 3.35, 3.15, 2.26, and 1.87 V vs. Li/Li$^+$, which corresponds to a complex multi-step lithium intercalation process.$^{17}$ As lithium ions are inserted into the layers of V$_2$O$_5$, the phase transformation occurs consecutively from $\alpha$-V$_2$O$_5$ to $\varepsilon$-Li$_{0.5}$V$_2$O$_5$ (3.35 V), $\delta$-LiV$_2$O$_5$ (3.15 V), $\gamma$-Li$_2$V$_2$O$_5$ (2.26 V), and $\omega$-Li$_3$V$_2$O$_5$ (1.87 V).$^{19,20,40}$ Among the various phases of Li$_x$V$_2$O$_5$, $\delta$-LiV$_2$O$_5$ can be restored to pristine V$_2$O$_5$ through lithium deintercalation, while $\gamma$-Li$_2$V$_2$O$_5$ and $\omega$-Li$_3$V$_2$O$_5$ (rock-salt type structure) are formed irreversibly. In the following anodic scanning, two broad peaks were observed at around 2.67 and 3.26 V vs. Li/Li$^+$, respectively, corresponding to the lithium extraction processes.$^{24,25}$
Fig. 4 Cyclic voltammograms (CVs) of the first 20 cycles of nanostructured V$_2$O$_5$ particles (here FSP-6) at different lower cut-off potentials vs. Li/Li$: (a) 1.5 V$, (b) $2.0 V$, and (c) $2.5 V$, at a scan rate of $0.1 \text{ mV s}^{-1}$. (d) Specific charge vs. cycle number from the CVs in plot (a) to (c).

From Fig. 4b and c, it can be seen that the reversibility of the redox kinetics with cycling improves when the discharge cut-off potential is limited to 2.5 V vs. Li/Li$. This can be explained by the fact that the ð-LiV$_2$O$_5$ phase can be reversibly cycled without destroying the crystal structure of the V$_2$O$_5$ nanoparticles. In addition, Fig. 4d shows the corresponding specific charge from the CV curves in Fig. 4a to c for the first 20 cycles. It revealed that the charge fading increases with a larger cycling potential window. Therefore, it is essential to investigate the effect of the discharge cut-off potentials.

After prolonged galvanostatic cycling at 100 cycles, it was found that the FSP-6 V$_2$O$_5$ electrode with a discharge cut-off potential of 2.5 V vs. Li/Li$^+$ retained the highest specific charge of approximately 110 mAh g$^{-1}$, when cycled at a specific current of 100 mA g$^{-1}$ (Fig. 5).
From the point of view of the entire battery, it is beneficial and practical to limit the cycling potential window of the positive electrode to 2.5 V vs. Li/Li⁺; the electrochemical performance of the 6 different V₂O₅ nanoparticle electrodes were compared accordingly. As can be seen from Fig. 6, the initial specific charge of all V₂O₅ electrodes, represented by the patterned columns, were above 140 mAh g⁻¹, except for samples FSP-1 and FSP-4. These high initial specific charge values are almost that of the theoretical value (148 mAh g⁻¹) and show that these nanoparticle-based electrodes are cycling well. In fact, one reason for the lower initial specific charge of the samples FSP-1 and FSP-4 is the higher wt% of VO₂ impurities in these samples, as shown in Table 1. However, when comparing the specific charge retained after 100 cycles, represented by the solid columns in Fig. 6, sample FSP-6 was clearly superior, retaining a specific charge of approximately 110 mAh g⁻¹, which was almost 80% of its initial charge. This excellent cycling behaviour could be due to the lower amount of surface reactions (vanadium dissolutions, passivation film formation, etc.) as sample FSP-6 has the lowest $S_{BET}$ of 32 m² g⁻¹.

Although a lower specific surface area is beneficial in reducing the side reactions during the electrochemical cycling, use of larger microparticles should not necessarily be the direction of future research. This is because nanosized particles have improved rate capability and, thus, the practical energy density at higher currents. Clearly there is a trade-off between the high rate capability of nanoparticles and the reduced...
amount of detrimental side reactions between microparticles and the electrolyte. To investigate the effect of V$_2$O$_5$ particle size further, the electrochemical performances of the FSP-6 (nanoparticles) and the Aldrich (microparticles) electrodes are compared (see Fig. 7). Cycling was performed between 2.5 and 4.0 V vs. Li/Li$^+$. As can be seen from Fig. 7a, when cycled at a low specific current of 100 mA g$^{-1}$ beyond 100 cycles, the nanostructured FSP-6 electrode retained a higher specific charge of 110 mAh g$^{-1}$ compared to 88 mAh g$^{-1}$ for the microstructured Aldrich oxide-based electrode. The power performance of battery electrodes depends on the size of the particles, making up the electrodes, and on the electrode surface area. As can be seen from Fig. 7b, the FSP-6 nanostructured electrodes have better rate capabilities when compared to the microstructured Aldrich oxide-based electrode, even up to the 20C rate (corresponding to 2000 mA g$^{-1}$), retaining a specific charge above 100 mAh g$^{-1}$. This is obviously due to the shorter Li$^+$ diffusion path lengths in the nanoparticles when compared to the microparticles. This effect delays the significant influence of the concentration polarization in the solid state to higher discharge currents, resulting in better rate capabilities and higher specific charge at high discharge rates.$^{21,25}$

![Graph](image.png)

Fig. 7 Galvanostatic cycling behaviour of V$_2$O$_5$ electrodes for nanoparticles (here FSP-6) and for microparticles (Aldrich): (a) cycling at a specific current of 100 mA g$^{-1}$, and (b) consecutive cycling at different specific currents. Electrodes were cycled between 2.5 and 4.0 V versus Li/Li$^+$. Here 1C-rate is set to 100 mA g$^{-1}$.

4. Conclusions

In this study, we have successfully synthesized crystalline, spherical-like V$_2$O$_5$ nanoparticles by flame spray pyrolysis and optimized the process.
conditions to obtain nanoparticles with improved electrochemical performance. Both the XRD patterns and SEM images revealed crystalline particles of approximately 30–60 nm in diameter. When the precursor’s concentration and injection rate were increased, higher V2O5 crystal purity (up to 98 wt%) and bigger particles with lower surface area (~32 m² g⁻¹) were made. In addition, it was found that the V2O5 nanostructures showed an improved cycling behaviour when the lower cut-off potential (for discharging) is increased from 1.5 to 2.5 V vs. Li/Li⁺. The significant charge “loss” when discharging to 1.5 V vs. Li/Li⁺ is most probably related to the structural changes upon cycling in the larger potential span.

Flame-made V2O5 nanoparticles with the lowest specific surface area and the highest purity show excellent cyclability when cycled between 2.5 and 4.0 V vs. Li/Li⁺, retaining a specific charge of 110 mAh g⁻¹ beyond 100 cycles at a specific current of 100 mA g⁻¹, and also a superior specific charge of 100 mAh g⁻¹ at a specific current up to 20C rate (or 2000 mA g⁻¹).

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