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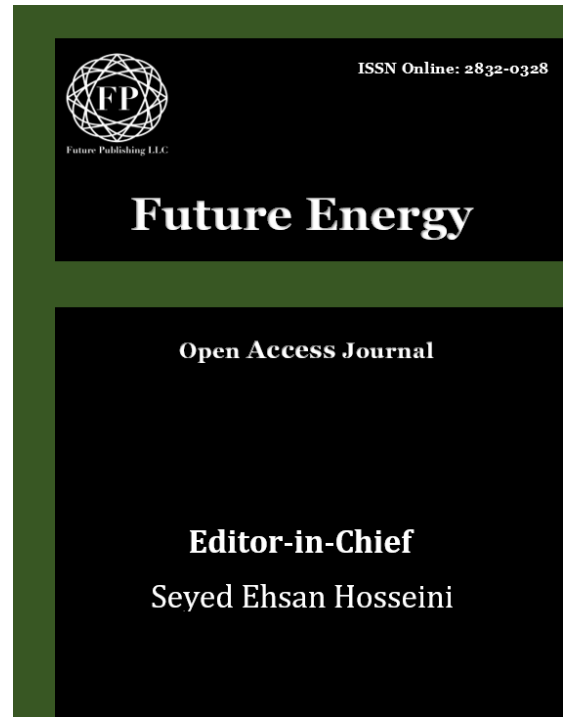
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Article

# Experimental investigation of cycling characteristics of anatase TiO<sub>2</sub> nanotubes as negative electrode of lithium-ion batteries

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## ABSTRACT

Anatase TiO<sub>2</sub> Nanotubes (NT-TiO<sub>2</sub>) is synthesized via electrochemical anodization of 99.9% pure titanium foils in a fluorine containing Ethylene Glycol (EG) electrolyte and used as the anode of lithium-ion batteries (LIBs). In the first cycle, the charge-discharge capacities are 550 mAhg<sup>-1</sup> and 400 mAhg<sup>-1</sup>, respectively, with coulombic efficiency of 75.75%. At 40<sup>th</sup> cycle, charge-discharge capacities are found to be 375 mAhg<sup>-1</sup> and 325 mAhg<sup>-1</sup>, respectively, with improved coulombic efficiency of 86%. The superior electrochemical performances of this type of battery originated from its high specific surface area and highly nanotubes structure. These advanced features of the nanotubes provide higher contact between electrodes and electrolytes, shorten the diffusion pathways for conductive ions.

## 1. Introduction

Lithium-ion batteries (LIBs) have emerged as a game-changing technology in the search for effective and sustainable energy storage solutions. They have revolutionized modern portable electronics and made it possible to electrify several industries, including grid energy storage and transportation [1]. LIBs have replaced traditional energy storage systems because of their superior features, including high energy density, extended cycle life, and lightweight design for greater portability. The field of LIBs has observed continuous advancements in response to the increasing demand for energy storage systems with better performance metrics, such as higher energy density, quicker charging times, improved safety, and longer lifespans. In addition, the field of batteries for energy has witnessed considerable interest in the domain of nanofabrication. A multitude of materials for anodes have been identified, therefore instigating continuous investigations aimed at ascertaining feasible alternatives. The utilization of TiO<sub>2</sub>, a transition oxide of metals, as a material for anodes in batteries powered by lithium ions offers a potentially advantageous substitute for traditional graphite [2, 3]. The investigation

into the use of TiO<sub>2</sub> substances for anode applications may be traced back to the identification of the capability of lithium titanates to conduct lithium insertion activities. Recently, there has been a growing interest in exploring the potential Li-insertion properties of titanite spinels. This has led to a heightened focus on investigating different nanostructures of TiO<sub>2</sub> polymorphs, specifically for their applicability in Li-ion battery systems. Rutile, which is considered the most thermally stable polymorph of TiO<sub>2</sub>, demonstrates a restricted ability to incorporate lithium ions, with a capacity of fewer than 0.1 lithium ions per unit of TiO<sub>2</sub> at room temperature [4]. Li-reactivity was higher at a temperature of 120°C when using a polymeric electrolyte instead of a liquid electrolyte. In these conditions, the first discharge reversible capacities were reported to be 0.5 Li [2] and 1 Li [5] per formula unit of TiO<sub>2</sub>. It is noted that the diffusion of Li in rutile exhibits a significant degree of anisotropy, characterized by rapid diffusion primarily along the channels aligned with the c-axis [6-10]. The utilization of a well-aligned and self-organized TiO<sub>2</sub> nanostructure array presents a promising opportunity for its application as a potential anode material in LIBs. It is noted that difficulties observed in the utilization

of traditional graphite as a material for anodes in LIBs, such as the formation of SEI layers, dendritic effects, exfoliation over cycling, toxicity, and structural collapse [11, 12]. The primary impetus behind this research stems from the need to address these limitations and explore alternative anode materials that possess desirable properties, with the goal of proposing a potential replacement for conventional graphite anodes. Anatase TiO<sub>2</sub> has a theoretical capacity of TiO<sub>2</sub> is slightly lower (330mAhg<sup>-1</sup>) compared to graphite's capacity of 372mAhg<sup>-1</sup>; the focus of research has primarily been on the structural stability of TiO<sub>2</sub>. This work highlights the usage of anatase TiO<sub>2</sub> nanotubes as the anode of LIBs. These nanotubes are created by anodizing pure Ti foils in a neutral fluoride solution, followed by calcination. Nanotube anatase structures with a significantly high specific area were created by carefully regulating the anodization and calcination processes. The primary cause of an interfacial process whereby lithium was stored on the surface of anatase particles was this sizable, exposed electrode area. The high specific surface area of anatase TiO<sub>2</sub> nanotubes electrodes exhibits the first cycle charge-discharge capacities are 550 mAhg<sup>-1</sup> and 400 mAhg<sup>-1</sup>, respectively, with columbic efficiency of 75.75%. However, this electrode exhibited improved electrochemical performances of 375 mAhg<sup>-1</sup> and 325 mAhg<sup>-1</sup>, respectively, with improved columbic efficiency of 86% after 40<sup>th</sup> charge-discharge cycle.

## 2. Experimental

### 2.1 Fabrication of anatase TiO<sub>2</sub> nanotubes

Using a Pt plate as the cathode, 99.9% pure Ti foil was oxidized to create TiO<sub>2</sub> nanotubes by the anodization process. The samples, particularly the Ti foils, were cleaned for 30 minutes using distilled water and detergent water prior to anodization. After that, the samples were cleaned for ten minutes in a pure ethanol solution. Following that, a final 20-minute acetone immersion rinse was performed. After that, the samples were dried for 24 hours at 105 °C. Two groups of all the Ti samples that needed to be anodized were created. One set of samples was cleaned with distilled water after being scraped with 0-grade emery paper. Both sets of Ti foils underwent anodization in a water-based solution that contained 0.5 wt.% NH<sub>4</sub>F + 1M (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> + 10% of ethylene glycol (EG). Anodization took place in a 100 ml solution, indicating that the electrolyte comprised 100 ml of distilled water, 0.5g NH<sub>4</sub>F (supplied by SUEN STUDIO, China), and 13.2g 1M (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> (supplied by SUEN STUDIO, China). The anode and cathode were connected to the positive and negative terminals of the DC power supply, respectively. The DC power supply's positive and negative terminals were linked to the anode and cathode, respectively. A DC power supply (DAZHENG brand, model PS-3050, China) provided a steady 32 V DC voltage for one hour and two hours, respectively, while maintaining a zero-current flow.

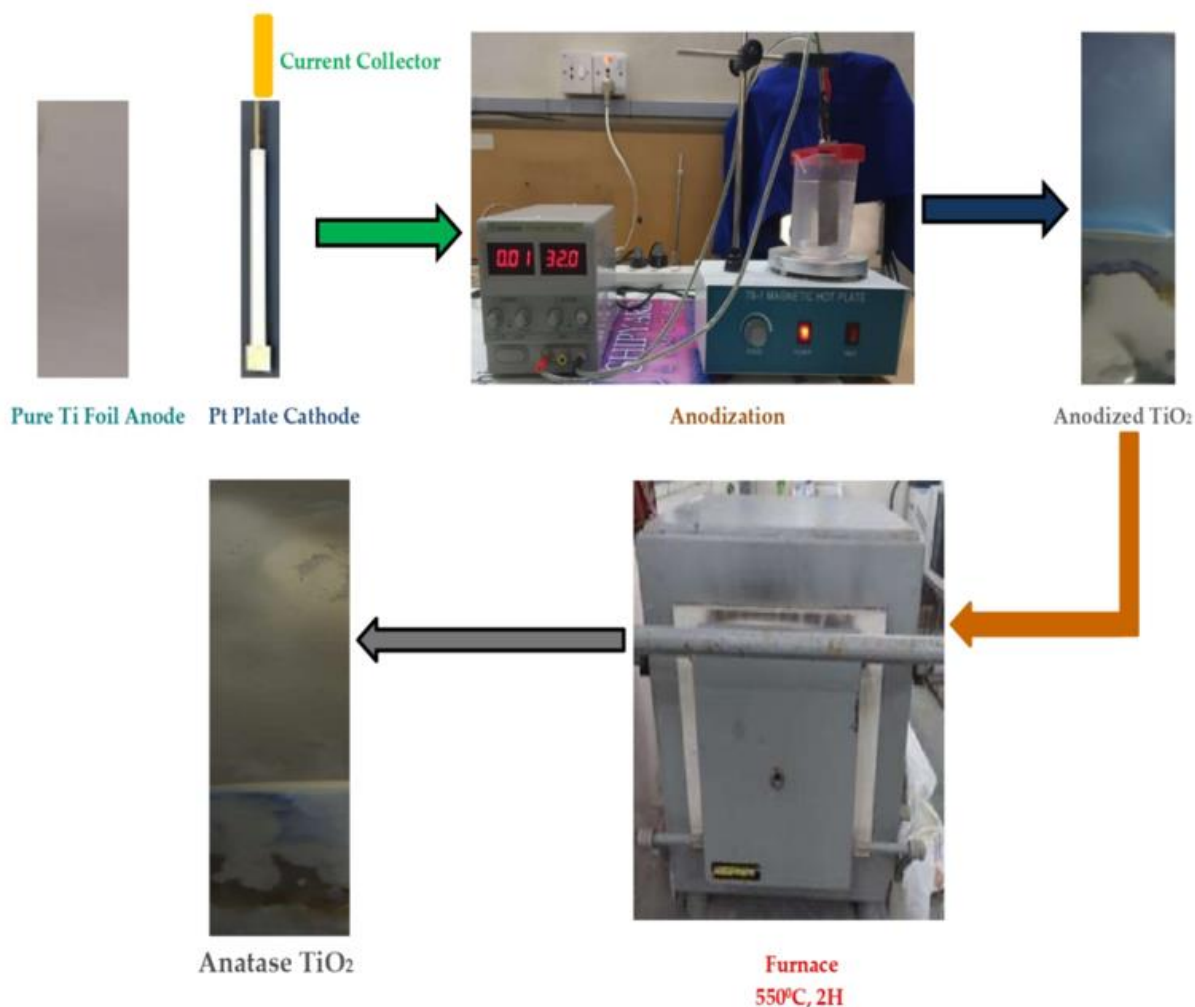


Figure 1. Visual representation of fabrication steps of anatase TiO<sub>2</sub> nanotubes

A magnetic stirrer hotplate ('78-1 Magnetic Stirrer Hotplate,' Yaeccc brand, China) at an average speed of 400 rpm during the anodization process verified that the solutes in solution were mixed uniformly and that the ions were moving properly. Following anodization, the anodized section of Ti foils changed from having a grey appearance to a bluish one. After being cleansed with distilled water, all samples were put in a furnace and calcined for two hours at 550°C. The full process is depicted in Figure 1.

## 2.2 Characterization

The NT-TiO<sub>2</sub> samples were analyzed using X-Ray Powder Diffraction (XRD) to obtain crystallographic information. To account for various scattering and random orientations, scans were conducted at different 2θ angles. Our analysis covered a 2θ range from 10° to 90° with a step size of 0.05°, utilizing Cu Kα radiation (Empyrean, Panalytical-Netherlands) with a wavelength of  $\lambda = 1.5406\text{Å}$ . The morphological and compositional characterization of anatase TiO<sub>2</sub> nanotubes was carried out using scanning electron microscopy and energy dispersive X-ray spectroscopy (SEM-EDX). For our experiment, SEM-EDX images were captured with a JSM 7600F from JEOL (Japan) at 5.0 KV and various magnifications.

## 2.3 Electrochemical performance test

Coin cell battery case sets CR2032 (provided by Lith Co. China) were utilized for assembly purposes. Anode samples measuring 18 mm in diameter and 0.1 mm in thickness (approximately 2 cm<sup>2</sup>) of NT-TiO<sub>2</sub>, along with LiCoO<sub>2</sub> coated aluminum sheet cathodes and a 20 mm battery grade PVC separator, fit perfectly into the cases. Both sides of the separator were saturated with a few drops of 1M LiPF<sub>6</sub> (from Ximen Tmax Battery Equipment Ltd., China) electrolyte, which was mixed in ethylene carbonate (EC) and diethylene carbonate (DEC) at a ratio of 3:7. Finally, crimping was performed at a pressure of about 100 psi using a battery crimping machine (Metrology Lab, CUET).

## 3. Results & discussion

### 3.1 XRD analysis

As illustrated in Figure 2, the X-ray diffraction (XRD) technique is used to analyze the phase purity and crystallinity of Ti and anatase TiO<sub>2</sub> nanotubes. TiO<sub>2</sub> has a tetragonal body-centered crystal structure from space group I41/amd, and the lattice parameters for samples and Ti foil are  $a = 3.79\text{Å}$ ,  $c = 9.51\text{Å}$ . The anatase phase of TiO<sub>2</sub> is confirmed by these data. Furthermore, the values match standard data (JCPDS Card no. 21-1272) exactly. At diffraction 2θ angles of 39.910 (101 plane), 52.770 (012 plane), and 70.4190 (013 plane), however, more noticeable anatase peaks are seen. The lack of any impurity phases within the detection range of the diffractometer indicates the purity of Ti. It is observed that no additional diffraction peaks related to the oxide phase were observed. Utilizing Scherrer's formula,  $D = 0.9\lambda / B \cos(\theta)$ , with  $\lambda$  representing the X-ray wavelength in nanometers, B representing the full width at half maximum (FWHM) of peaks at 2θ, and  $\theta$  representing the angle between the incident and diffracted beams in degrees, these diffraction data are entered. Table 1 displays the crystal sizes of Ti foil and the synthesized anatase NT-TiO<sub>2</sub> at various 2θ angles.

### 3.2 SEM & EDX analysis

The plan-view SEM images and EDX spectra of anatase NT-TiO<sub>2</sub> is shown in Figure 3. Non-uniform anatase NT-TiO<sub>2</sub> arrays is grown by anodization, which is observed clearly underneath the Ti substrates as shown in Figure 3(a). The nanotubes are compact in this area. For the elemental characterization of the obtained nanotube layers, energy dispersive X-ray analysis is conducted. The EDX spectrum indicates the presence of the TiKα peak at 4.508 keV and O peaks at 0.525 keV as well as C peak at 0.277 keV in the anodized sample as shown in Figure 3(b). The obtained mass percentages of Ti and O is observed to be 55.30% and 42.44%, respectively, and the atom percentage of Ti, O, and C is 28.93%, 66.81%, and 4.26%, respectively. It is noted that peak of carbon is found at 0.277 keV with the percentage of C is 2.04%.

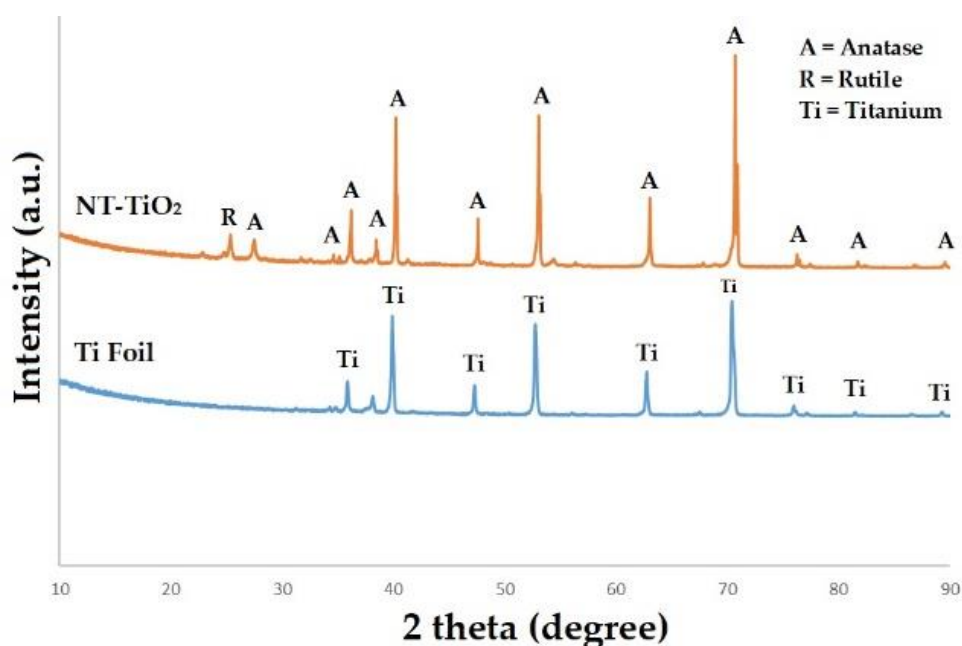
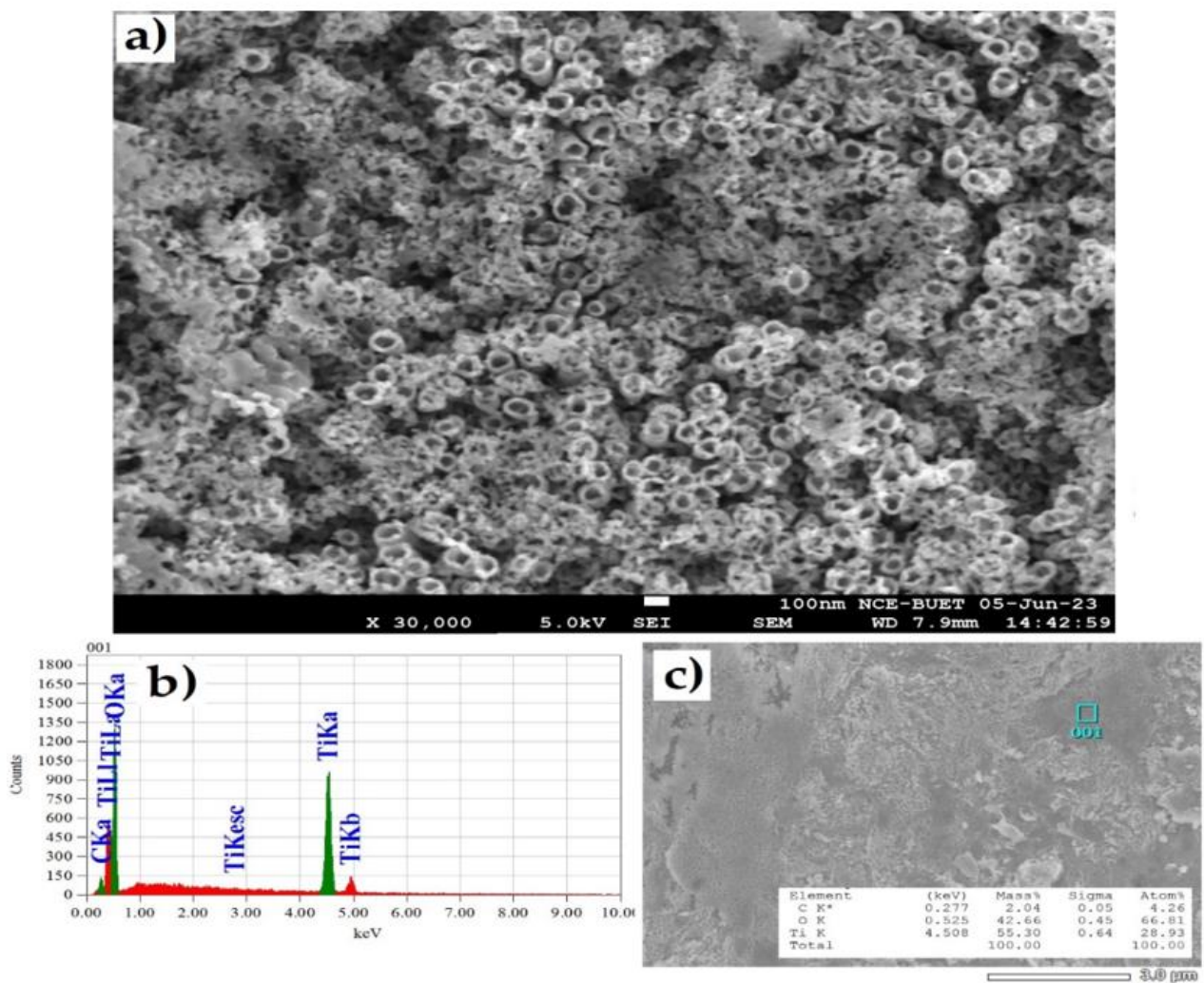


Figure 2. XRD patterns of different anatase NT-TiO<sub>2</sub> along with pure Ti

**Table 1.** Crystal Sizes Ti foil and as synthesized anatase NT-TiO<sub>2</sub>

Sample No.	Crystallographic Data for Pure Ti											Average Crystal Size (nm)
	2θ	35.90	38.14	39.92	47.30	52.77	62.78	70.42	-	-	-	
a	B	0.22	0.32	0.23	0.20	0.26	0.22	0.33	-	-	-	38.27
	D	40.42	28.01	38.76	46.31	36.18	46.02	32.16	-	-	-	
	Crystallographic Data of NT-TiO <sub>2</sub>											
b	2θ	22.87	25.37	27.51	36.21	38.47	40.23	47.61	53.06	63.07	70.71	65.66
	B	2.07	0.36	0.44	0.16	0.16	0.10	0.10	0.09	0.13	0.09	
	D	4.21	24.39	20.09	56.26	58.35	89.19	92.14	111.45	80.33	120.18	

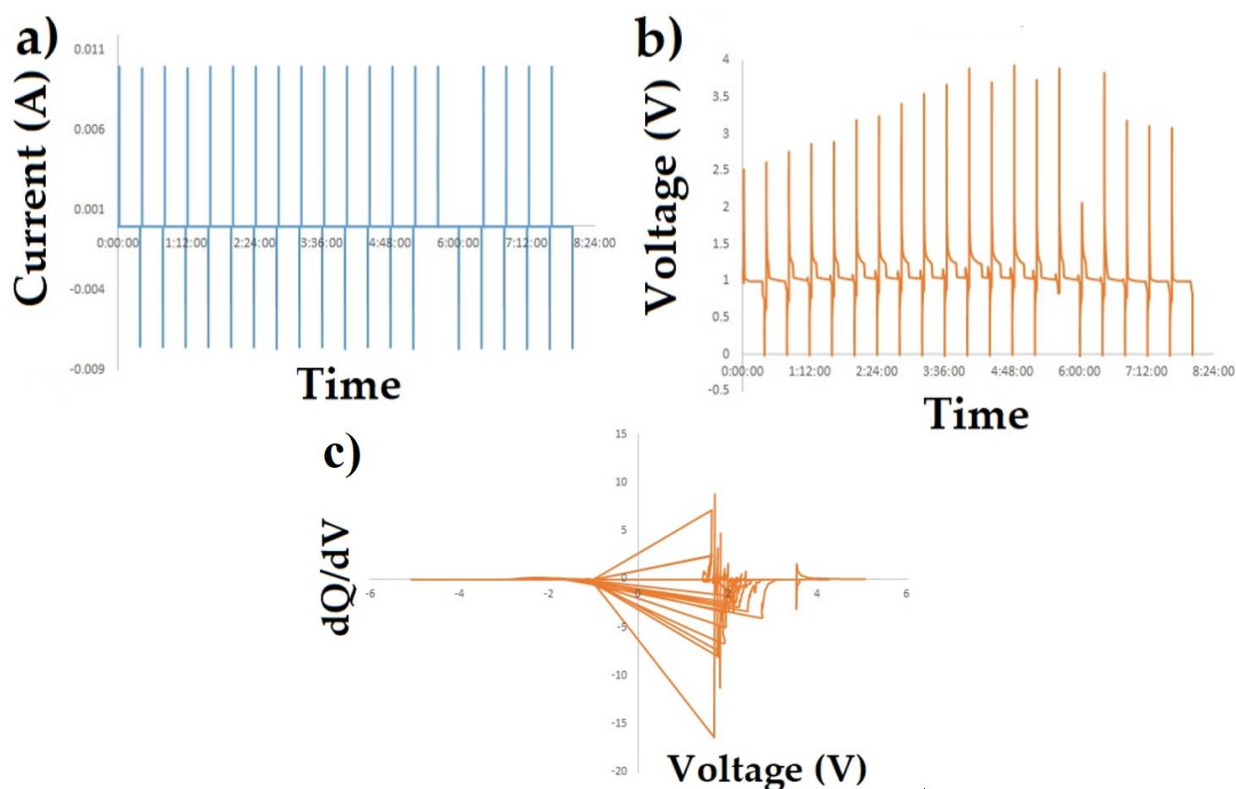


**Figure 3.** High magnification SEM image and EDX spectra of anatase NT-TiO<sub>2</sub>

### 3.3 Electrochemical performance of anatase NT-TiO<sub>2</sub> as anode of LIBs

The galvanostatic charge-discharge cycle is carried out to measure the electrochemical performance of the as-prepared anatase NT-TiO<sub>2</sub> anode. Figures 4(a & b) show the changes in current and voltage with respect to charge-discharge time. The cut-off voltage ranges are 4V-3.8V. It is noted that the voltages remain 2.5V at the beginning of the cycling, as shown in Figure 4(a). However, the voltage increases as the cycling moves forward, and after the 20th cycle, the voltage decreases. In contrast, the voltage and current both increase at the end of the 40th cycle of the charge-discharge process, where the voltage is found to be 4.2V, as shown in Figure 4(b). Figure 4(c) shows a variation in the rate of change in charge with a change in voltage (dQ/dV) till 40 charge-discharge cycles for incremental capacity analysis, which identifies and quantifies changes in the electrochemical properties of the cell based on voltage measurements under constant current charge or discharge. It is observed that the phase transitions in the active electrode material anatase NT-TiO<sub>2</sub> caused by the intercalation and deintercalation of lithium at about 2.5V correlate with the incremental capacity peaks. It is worth mentioning that dQ/dV analysis indicates the electrochemical performances of cell degradation over charge-discharge cycling, as shown in Figure 4(c), due to an increase in internal resistance [13]. Figure 5 shows the charge-discharge capacity and Columbic efficiency of LIB till 40 cycles. First cycle charge-discharge capacities are 550 mAhg<sup>-1</sup> and 400 mAhg<sup>-1</sup>, respectively, with columbic efficiency of 75.75%, as shown in Figure 5(a). Higher initial capacities are ascribed to gel-like SEI layer formation by electrolyte decomposition [14]. In addition, the low columbic efficiency is attributed to unstable SEI formation, low reversible capacity, and electrolyte decomposition.

However, in the subsequent 2<sup>nd</sup> cycle, the charge-discharge capacity comparably reduced to 500 mAhg<sup>-1</sup> and 360 mAhg<sup>-1</sup>, respectively, due to the amorphous Li<sub>2</sub>O formation, which required a huge amount of lithium and caused the loss of lithium [2]. The increased columbic efficiency of 75.4% in the 2<sup>nd</sup> cycle is attributed to stable SEI layer formation. It is noted that the cell shows instability with fluctuating columbic efficiency, which is attributed to the larger volume expansion and pulverization effect of the anode. In addition, at the 27th cycle, the columbic efficiency is as high as 120%, as shown in Figure 5(b). This phenomenon is typical for transition metal oxides due to the pseudo capacitance [15-17]. At the 40th cycle, charge-discharge capacities are found to be 375 mAhg<sup>-1</sup> and 325 mAhg<sup>-1</sup>, respectively, and the columbic efficiency is observed to be 80%, which is comparable to the theoretical capacity of TiO<sub>2</sub> 334 mAhg<sup>-1</sup>. Table 2 shows the comparison of charge and discharge capacities of this present work and previous work. It is noted that the charge-discharge capacities of this present work at 1<sup>st</sup> is observed to be 550 mAhg<sup>-1</sup> and 400 mAhg<sup>-1</sup>, respectively, which is higher compared to 290 mAhg<sup>-1</sup> and 239 mAhg<sup>-1</sup>, respectively. However, the observed Columbic efficiency is slightly lower, 75%, compared to the previous work of 82%. This deficiency could be ascribed to the impurities in the electrolytes, and/or our cell is assembled in ambient without using a glovebox [18]. However, the electrochemical performances and Columbic efficiency increased after the 40<sup>th</sup> charge-discharge cycle compared to the previous work. Here, we are anticipating that the Columbic efficiency will be improved over a large number of cycling of our synthesized material since Columbic efficiency rises and irreversible capacity falls after the irreversible Li insertion sites are filled and trace water has been used up in the first cycles [14].



**Figure 4.** Change of (a) Current (b) Voltage with respect to time from 1-20 Charge-Discharge cycles, c) Change of dQ/dV with respect to voltage from 1-40 cycles

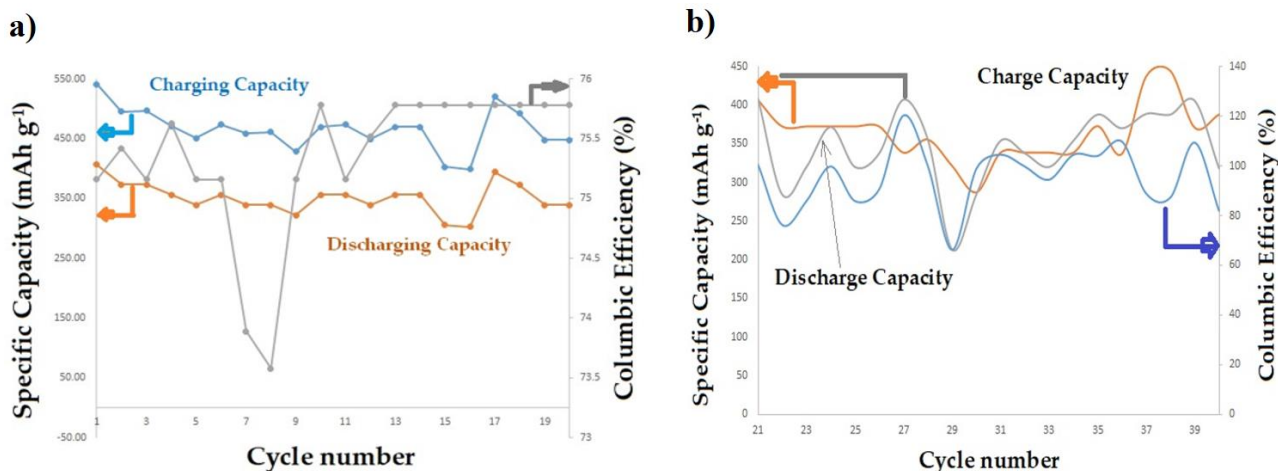


Figure 5. Change of Specific capacity and columbic efficiency during (a) 1-20 and (b) 21-40 charge-discharge cycles at 1C current rating

Table 2. Comparison of our work with previous work

Compare	Cycle No.	Unit	Our Work	Previous Work	Reference
Charge Capacity	1st	mAhg <sup>-1</sup>	550	290	[19, 20]
Discharge Capacity		mAhg <sup>-1</sup>	400	239	
Columbic Efficiency		%	75	82	
Charge Capacity	40 <sup>th</sup> /Last	mAhg <sup>-1</sup>	375	238	
Discharge Capacity		mAhg <sup>-1</sup>	325	198	
Columbic Efficiency		%	86	83	

4. Conclusion

Anatase TiO<sub>2</sub> nanotube has been considered as one of the most attractive anode materials for lithium-ion batteries (LIBs) due to comparable energy density and life cycle compared to graphite. The nanotube features of nanostructure provide higher contact between binder-less electrode TiO<sub>2</sub> and electrolytes, shorten the diffusion pathways for conductive ions and electrons that ensure faster kinetics. The work represents a facile fabrication of anatase NT-TiO<sub>2</sub> via electrochemical anodization of Ti foils. Anatase NT-TiO<sub>2</sub> is used as anode LIBs. Nanotubes provide a high surface area that allows for a higher electrode/electrolyte interface. Hence, lithium storage capacity is greatly enhanced compared to bulk amorphous TiO<sub>2</sub>. The cut-off voltage ranges are 4V-3.8V. It is noted that at the beginning of the cycling, the voltages remain at 2.5V. However, as the cycling moves forward, the voltage increases, and at the end of the 40<sup>th</sup> cycle of the charge-discharge process, the voltage is found to be 4.2V. The battery exhibits excellent 1<sup>st</sup> cycle charge-discharge capacity of 550 mAhg<sup>-1</sup> and 400 mAhg<sup>-1</sup>, respectively, with 75.75% columbic efficiency. At the 27<sup>th</sup> cycle, the columbic efficiency is as high as 120%. This phenomenon is typical for transition metal oxide due to the pseudo-capacitance. At the 40<sup>th</sup> cycle, charge-discharge capacities are found to be 375 mAhg<sup>-1</sup> and 325 mAhg<sup>-1</sup>, respectively, and the columbic efficiency is found to be 86%. The findings showed that anatase TiO<sub>2</sub> nanotubes' large surface area, short diffusion path, and quick kinetics make them promising electrode materials for LIB applications.

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Ethical issue

The authors are aware of and comply with best practices in publication ethics, specifically concerning authorship (avoidance of guest authorship), dual submission, manipulation of figures, competing interests, and compliance with policies on research ethics. The authors adhere to publication requirements that the submitted work is original and has not been published elsewhere in any language.

Data availability statement

The manuscript contains all the data. However, more data will be available upon request from the corresponding author.

Conflict of interest

The authors declare no potential conflict of interest.

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