

Article

High Performance of Salt-Modified-LTO Anode in LiFePO₄ Battery

Agus Purwanto ^{1,2,*} , Soraya Ulfa Muzayanha ^{1,3}, Cornelius Satria Yudha ^{1,2}, Hendri Widiyandari ^{2,4}, Arif Jumari ^{1,2}, Endah Retno Dyartanti ^{1,2}, Muhammad Nizam ⁵ and Muhamad Iqbal Putra ¹

¹ Department of Chemical Engineering, Faculty of Engineering, Universitas Sebelas Maret, Jl. Ir. Sutami 36 A, Surakarta, Central Java 57126, Indonesia; sorayamuzayanha@student.uns.ac.id (S.U.M.); cornelyudha@student.uns.ac.id (C.S.Y.); arifjumari_ft@staff.uns.ac.id (A.J.); endah_rd@staff.uns.ac.id (E.R.D.); iqtraaa@student.uns.ac.id (M.I.P.)

² Centre of Excellence for Electrical Energy Storage Technology, Universitas Sebelas Maret, Jl. Slamet Riyadi 435, Surakarta, Central Java 57146, Indonesia; hendriwidiyandari@staff.uns.ac.id

³ Research & Technology Center, PT. Pertamina (Persero), Jl Raya Bekasi km 20, Pulogadung, Jakarta 19320, Indonesia

⁴ Department of Physics, Faculty of Mathematic and Natural Science, Universitas Sebelas Maret, Jl. Ir. Sutami 36 A, Surakarta, Central Java 57126, Indonesia

⁵ Department of Electrical Engineering, Faculty of Engineering, Universitas Sebelas Maret, Jl. Ir. Sutami 36 A, Surakarta, Central Java 57126, Indonesia; muhammad.nizam@staff.uns.ac.id

* Correspondence: aguspurwanto@staff.uns.ac.id

Received: 23 September 2020; Accepted: 9 October 2020; Published: 13 October 2020



Abstract: Highly crystalline “zero-strain” Li₄Ti₅O₁₂ (LTO) has great potential as an alternative material for the anodes in a lithium ion battery. In this research, highly crystalline LTO with impressive electrochemical characteristics was synthesized via a salt-assisted solid-state reaction using TiO₂, LiOH, and various amounts of NaCl as a salt additive. The LTO particles exhibited a cubic spinel structure with homogenous micron-sized particles. The highest initial specific discharge capacity of LTO was 141.04 mAh/g with 4 wt % NaCl addition, which was tested in a full-cell (LTO/LiFePO₄) battery. The battery cell showed self-recovery ability during the cycling test at 10 C-rate, which can extend the cycle life of the cell. The salt-assisted process affected the crystallinity of the LTO particles, which has a favorable effect on its electrochemical performance as anodes.

Keywords: LTO; anode material; salt-assisted; solid-state; Li-ion battery

1. Introduction

Lithium titanate (Li₄Ti₅O₁₂), which is usually referred to as LTO, has become a promising candidate for anode material that could substitute for carbon. LTO operates on a stable voltage of ≈ 1.5 V vs. Li⁺/Li, which avoid electrolyte decomposition and the safety issues presented by the use of carbon [1–3] that tends to form lithium dendrite and causes the occurrence of a surface passivation film. The surface passivation film could cause an initial loss in capacity, safety issues, loss of critical electrode performance, and cell failure [4,5]. In the midst of many promising candidates for anodes such as Si, SiO_x, SiO₂, SnO₂, Fe₂O₃, Co₃O₄, etc. [6–13], LTO has superior structural stability with no structural or volume changes during the lithiation/de-lithiation process. Hence, LTO is classified as a zero-strain material that has 175 mAh/g of theoretical capacity [14–16].

As a lithium-intercalation material, the high crystallinity of LTO is a key for great capacity and cyclability [17]. Shen et al. 2013 studied the effect of crystallinity on the electrochemical performance

of LTO nanoparticles (NPs) in half-cells [18]. The results showed that the high crystallinity of LTO NPs gives them the highest rating for rate ability and long-term cyclability [18].

The many different techniques studied for the synthesis of LTO have included solid-state reactions [19–21], sol–gel methods [22–25], microwave-assisted synthesis [26,27], spray pyrolysis [28,29], and hydrothermal methods [30,31]. The sol–gel methods are used to obtain particles of LTO under low temperatures [32]. In order to achieve highly crystalline LTO, Yin et al. (2015) and Bai et al. (2008) used a molten salt method [33,34]. However, the finished product required washing to eliminate the salt, and hence, a large amount of lithium salt wastewater had to be discarded into the environment, which makes this process unattractive for adaptation to large-scale production. Other methods used in attempts to synthesize highly crystalline LTO have included a novel pulse flow supercritical reactor that was studied by Sha et al. (2013) [17]. Shen et al. (2013) used a hydrothermal method [18]. A novel continuous-flow hydrothermal method under supercritical conditions was proposed by Laumann et al. (2012) [35]. Nevertheless, these methods require high levels of temperature and pressure, which expends a large amount of energy.

An alternative way to improve the crystallinity of a material is by adding a small amount of salt during synthesis, which is referred to as a salt-assisted process. The addition of salt (NaCl, KCl) has succeeded in assisting in the crystal formation of materials such as ZnO, ZnFe_2O_4 , and strontium ferrite [36–38]. Our previous investigation focused on the effect of NaCl addition on the synthesis of fluorine-doped tin oxide for use in solar cells [39]. However, salt-assisted synthesis has never been demonstrated in the production of LTO material. In the present experiment, we proposed a systematic synthesis of highly crystalline LTO using a salt-assisted, solid-state reaction (various additions of NaCl) to increase the crystallinity and improve the specific discharge capacities of LiFePO_4 (LFP)/LTO battery electrochemical performance. An LFP/LTO battery was selected as the cell design because it is known for its fast charging feature, superior rate ability, and cyclability at high rates. Since it has zero cobalt, it is thermally stable with phenomenal safety features that are crucial and applicable for electric vehicle applications [40–42].

2. Materials and Methods

2.1. Materials $\text{Li}_4\text{Ti}_5\text{O}_{12}$

LiOH (Merck, Darmstadt, Germany) and TiO_2 (Merck, Darmstadt, Germany) were utilized as Li and Ti sources of $\text{Li}_4\text{Ti}_5\text{O}_{12}$. Analytical grade NaCl (Merck, Darmstadt, Germany) was used as the salt addition, while CH_3OH or methanol (Merck, Darmstadt, Germany) was used as the dispersant during solid-state mixing. All of the starting materials were used without any further purification steps.

2.2. $\text{Li}_4\text{Ti}_5\text{O}_{12}$ Preparation

The $\text{Li}_4\text{Ti}_5\text{O}_{12}$ (LTO) powders were prepared via solid-state reaction. TiO_2 and LiOH were used as titanium and lithium sources in a Li:Ti molar ratio of 4:5. In order to investigate the effect of salt addition on LTO powder, three different amounts of NaCl (0, 2, 4 and 6 wt %) were added. TiO_2 , LiOH , NaCl, and methanol were mixed in a ball mill for 4 h. Methanol acted as a dispersant to homogenize the precursors. The precursors were dried in an oven to evaporate the methanol and then sintered at 800 °C for 12 h under O_2 (Samator, Jakarta, Indonesia) atmosphere. After the sintering process, the LTO powders were ground and screened using a 100 mesh. The flowchart of Synthesis Process of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ Powder is explained in Figure A1.

2.3. $\text{Li}_4\text{Ti}_5\text{O}_{12}$ Characterization

The crystal structure of the LTO powders was investigated by X-ray diffractometer/XRD ((D2 Phaser Bruker, Germany) using Cu-K-alpha radiation ($\lambda = 1.5418 \text{ \AA}$) with a range of two theta (2θ) at 10°–70°. Scanning electron microscopy/SEM (Jeol JSM-6510LA, Tokyo, Japan) was employed to

identify the morphologies of the LTO powders. The LTO formation during sintering was analyzed using thermogravimetric and differential scanning calorimeter (TG-DSC) with a heating rate of 10 °C/min.

2.4. $\text{Li}_4\text{Ti}_5\text{O}_{12}/\text{LiFePO}_4$ Cylindrical Cell Assembly

The electrochemical characteristics were analyzed using a full cell (cylindrical battery of 18650-type), which is used in commercial $\text{LiFePO}_4/\text{LFP}$ (MTI, Richmond, CA, USA) as a cathode. The LTO powders were mixed with conductive carbon (acetylene black/AB, MTI, Richmond, CA, USA), and water-based binders (carboxymethyl cellulose (CMC) and styrene butadiene rubber (SBR)) at a weight ratio of 80:10:2:8 in a water to make a slurry that could be coated onto copper foil using the doctor's blade technique, followed by drying to evaporate the water. Then, the electrode was fabricated to the full-cell in a glove box under Ar atmosphere. LiPF_6 (in 1:1 *v/v* of EC/DMC) and cellgard (MTI, Richmond, CA, USA) were used as an electrolyte and separator (MTI, Richmond, CA, USA). The charge/discharge performance of the LTO/LFP cell were tested using MTI BST-CH8-3A battery analyzer (1.0 V to 3.0 V potential range). The anode was selected as the limiting electrode. Therefore, the calculation of the specific capacity was referred to the weight of the anode material. To assure the consistency of the results, four cells of each LTO sample with consistent results were assembled.

3. Results and Discussion

3.1. Structural Analysis of LTO

In order to investigate the structural properties of LTO materials samples obtained via the salt-assisted solid state method, the material was characterized using XRD. Figure 1 presents the XRD patterns of the LTO powders with various additions of NaCl with 2θ (deg.) range of 10–70°. The XRD patterns of all samples show that all peaks of LTO are detected and corresponding to the JCPDS Card No. 42-0207 with the structure of cubic spinel and the space group of $F\bar{4}3m$ [43–45]. Based on the XRD results, no slight impurities of NaCl were detected in the XRD pattern of LTO-2 wt % NaCl and LTO-4 wt % NaCl. However, with an addition of 6 wt % NaCl, tiny peaks of NaCl appear in the XRD pattern. The peaks of NaCl were easily detected at 2θ of 31° and 45° (JCPDS No. 5-0628).

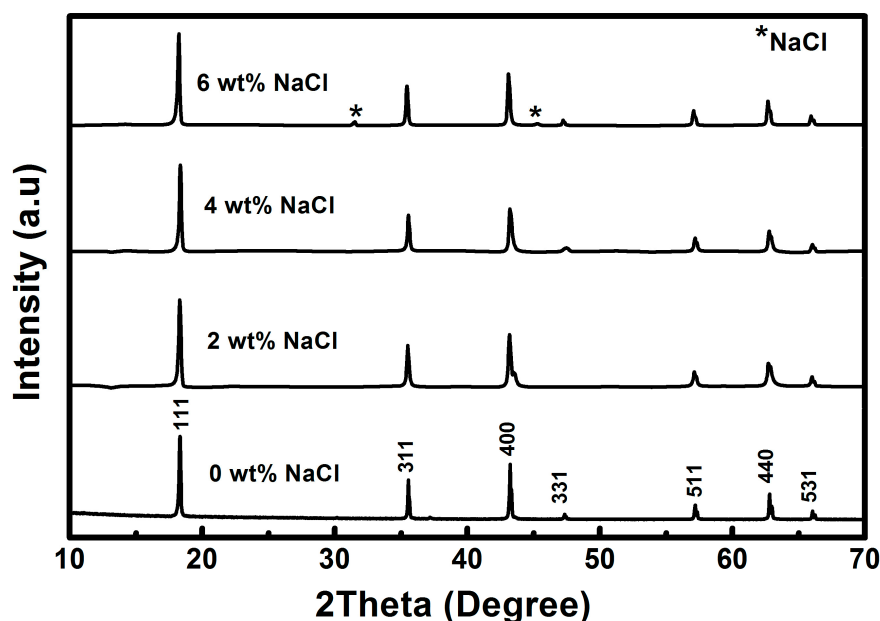


Figure 1. The XRD patterns of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ (LTO) powder in various NaCl additions.

For further analysis of the XRD patterns, the calculation of lattice constants, Full Width at Half Maximum (FWHM), and crystallite size were calculated and are presented in Table 1. The values of lattice constants were approximately 8.384–8.362 Å for the various samples of LTO, which correspond to those of the LTO sample reported in the literature (8.395–8.3538 Å), [34,46–48] and indicates that LTO is a zero-strain material with good structural stability [48,49]. Based on these results, it can be predicted that the as-obtained LTO exhibits no volume expansion during the lithiation/delithiation process. The lattice constant values are decreased with NaCl additions ranging from 0–4 wt % but increased with an NaCl addition of 6 wt %. Since LTO with 4 wt % NaCl has the lowest lattice constant, it is predicted that the sample will have better electrode performance compared to the other samples during electrochemical testing, as reported by Huang et al. [50].

Table 1. The structural parameters of LTO material.

LTO	Lattice Constant (Å)	FWHM (°)			Crystallite Size (nm)		
		(111)	(311)	(400)	(111)	(311)	(400)
0 wt % NaCl	8.377	0.321	0.266	0.272	26.94	32.72	32.80
2 wt % NaCl	8.373	0.197	0.140	0.170	42.66	62.17	52.48
4 wt % NaCl	8.362	0.154	0.137	0.168	54.57	63.53	53.11
6 wt % NaCl	8.384	0.151	0.135	0.146	55.66	64.47	61.11

Surprisingly, the XRD results revealed the effect of NaCl addition on the crystallinity of LTO material. The crystallinity of LTO can be evaluated based on the FWHM (β) of (111), (311), and (400) peaks. Table 1 shows the FWHM values of LTO samples decrease from the range of 0 to 6 wt % NaCl addition. It means that the higher NaCl addition will result in the sharper peak on XRD and smaller values of FWHM, indicating the higher crystallinity of the material [51]. Therefore, it can be concluded that the increasing salt addition during the solid-state reaction had a good influence on LTO crystal formation. Salt exerts a medium reaction that accelerates the mass transfer of a precursor during the sintering process. During the reaction process, the NaCl (salt) enlarges and provides heat flux, which improves crystal growth [39]. Therefore, a highly crystalline product can be produced rapidly.

The crystallite sizes of the samples with 0–6 wt % NaCl additions were calculated using the Debye–Scherrer equation [51–53]. The crystallite sizes of LTO on (111), (311), and (400) peaks increase as the NaCl additions increase, which is in agreement with a study reported by Cai et al. [51].

3.2. Morphology Analysis of LTO

In order to observe the morphology, the LTO materials were characterized using SEM. Figure 2 shows different magnifications of SEM images for LTO powder with various amounts of NaCl addition via the solid-state reaction. The LTO particles exhibit micro sizes ranging from 1.83 to 2.28 μm , and all samples display a cubic morphology with a smooth surface. Moreover, previous studies reported that at certain concentrations, salt can prevent the agglomeration of particles during the sintering process [37,38,54]. Based on SEM images, the agglomeration of LTO particles is occurred without the NaCl addition (Figure 2a). With the NaCl addition of 2–4 wt %, only a slight agglomeration is formed, indicating an effective inhibition of agglomeration using salt. However, the increasing NaCl addition to 6 wt % lead to more agglomeration of LTO particles as referred by Kong et al. [54]. Based on SEM images, it is predicted that LTO with a slight agglomeration will have better electrochemical performances.

3.3. Electrochemical Performance Measurement of LTO in LTO/LFP Battery

Then, for large-scale application, the electrochemical performance of LTO material is important to be analyzed using a full-cell battery (LTO vs. LFP). The LTO/LFP battery was tested using NEWARE Battery analyzer and BTS software. First, in order to investigate the best C-rate for the formation of an LTO-LFP battery, the various C-rates were used for testing. Figure A2 presents the charge–discharge formation curve at various C-rates of an LTO-0 wt % NaCl/LFP battery with a voltage range of 1.0–3.0 V.

The optimum initial specific capacity was operated at 0.3 C-rate, which obtained the specific discharge capacity of 121.1 mAh/g. Hence, the 0.3 C-rate was chosen for further testing of the LTO material with NaCl addition.

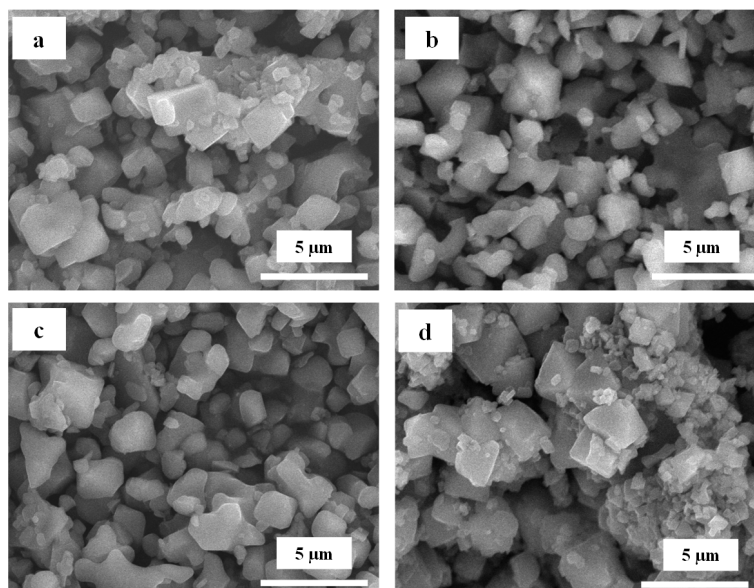


Figure 2. The 2000 $\times$ magnification SEM images of LTO with NaCl addition of: (a) 0 wt %, (b) 2 wt %, (c) 4 wt %, (d) 6 wt %.

Figure 3 presents the electrochemical performance (1.0–3.0 V) of LTO with various additions of NaCl at 0.3 C-rate. The LTO/LFP battery displayed a plateau voltage at ≈ 1.8 V, which agrees with the values found in the literature [42,55]. The initial specific discharge capacities at 0.3 C-rate of LTO with NaCl additions of 0, 2, 4, and 6 wt % were 121.1, 132.4, 145.6, and 113.5 mAh/g, respectively. The LTO-4 wt % NaCl shows the highest initial discharge capacity in this study, which confirms the predictions [50] that the highly crystalline LTO performs at great capacity [17]. However, LTO-6 wt % NaCl has the highest level of crystallinity, but it also has the lowest initial discharge capacity. It means that the addition of 6 wt % NaCl cannot be ignored on LTO material, which is confirmed by the appearance of NaCl peaks in the XRD pattern. In addition, based on the SEM result, the NaCl addition of 6 wt % can cause higher agglomeration on LTO morphology, resulting in the lowest initial specific discharge capacity in this study. It can be concluded that the higher NaCl addition (≥ 6 wt %) causes the NaCl to act as impurities on the LTO material, which will have a poor effect on the electrochemical performance. The initial Coulombic efficiency (CE) of LTO with NaCl additions of 0, 2, 4, and 6 wt % were 84.46%, 90.99%, 91.55%, and 97.84%, respectively. Based on this finding, it can be concluded that a high level of crystallinity results in better structural properties and caused better CE, which is consistent with previous research reported by Shen et al. [18].

In order to investigate the rate ability of LTO without and with NaCl addition, the LTO/LFP battery was charged and discharged at various higher current rates. Figure 4 compares the rate ability of LTO-0 wt % NaCl and LTO-4 wt % NaCl, as tested at 0.3, 1, 3, 5, 7, and 10 C charge–discharge rate. LTO-4 wt % NaCl shows a discharge capacity that ranged from 145.6 mAh/g at 0.3 C to 118.6 mAh/g at 10 C. Meanwhile, LTO-0 wt % NaCl showed a discharge capacity that ranged from 123.8 mAh/g at 0.3 C to 86.3 mAh/g at 10 C. It can be seen that the specific capacity of LTO-4 wt % only slightly decreases from 0.3 to 10 C-rate. Compared to LTO-0 wt % NaCl, LTO-4 wt % NaCl has a higher specific capacity at various higher current rates in this study. The capacity retentions of the LTO/LFP battery at 0.3–10 C-rate are presented in Table 2. The LTO-4 wt % NaCl has a smaller drop-specific capacity (18.6%) than LTO-0 wt % NaCl (30.8%) from 0.3 to 10 C-rate. It proves that the crystallinity of the LTO material can improve the rate ability performance compared with the previous studies [17,18].

However, both samples have a good reversibility while returning to the first rate at 0.3 C (99.9% for LTO-4 wt % NaCl and 99.7% for LTO-0 wt % NaCl).

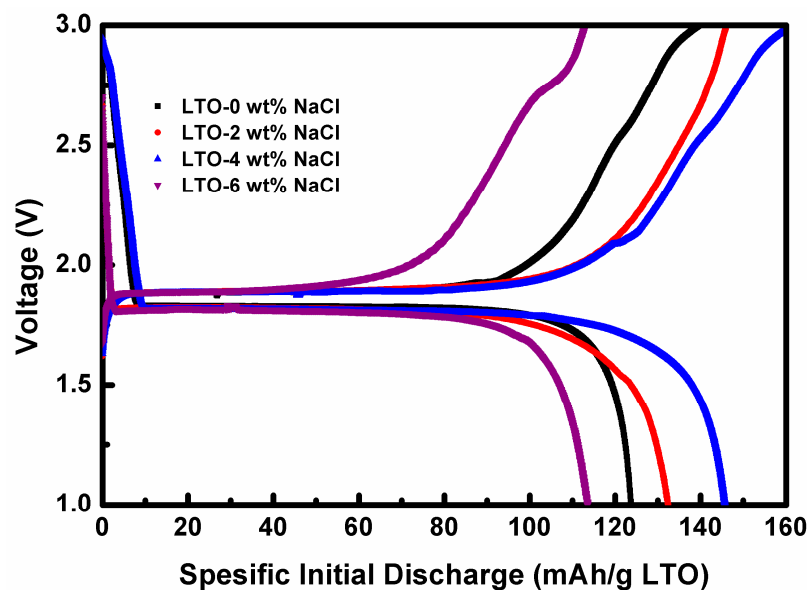


Figure 3. Specific initial discharge capacities of LTO with various NaCl additions at 0.3 C-rate.

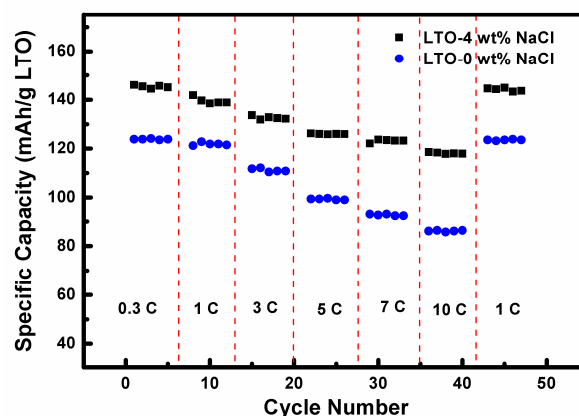


Figure 4. Rate ability of LTO-0 wt % NaCl and LTO-4 wt % NaCl.

Table 2. Capacity retention of LTO/LiFePO₄ batteries at various C-rates.

LTO	Capacity Retention (%)					
	0.3 C	1 C	3 C	5 C	7 C	10 C
LTO-0 wt % NaCl	100	97.1	90.3	80.3	75.0	69.2
LTO-4 wt % NaCl	100	97.4	91.8	86.6	84.9	81.4

Then, in order to study the performance stability of the LTO material, a cycle performance test at 10 C was conducted. Figure 5a shows the cycling test of LTO-0 wt % NaCl and LTO-4 wt % NaCl at the 10 C-rate. In the 1st cycle, the specific capacities of LTO-0 wt % NaCl and LTO-4 wt % NaCl were 86.3 and 118.6 mAh/g, respectively. After the 1065th cycle, the specific capacities of LTO-0 wt % NaCl and LTO-4 wt % NaCl decreased to 74.2 and 114.5 mAh/g, respectively. The cycling test shows that the drop capacity percentages of LTO-0 wt % NaCl and LTO-4 wt % NaCl after 1065 cycles are 13.9% and 3.4%, respectively. It exhibits that the LTO-4 wt % NaCl provides superior cyclability, which has only a 0.003% drop capacity per cycle. The result confirm that highly crystalline LTO shows not only good performance in rate ability but also cycling stability as reported by Sha et al.

and Shen et al. [17,18]. On comparing with previous studies on LTO synthesis, it was found that employing a simple modification of NaCl addition during the synthesis resulted in high capacity, good rate ability, and good cyclability. Different from other studies listed in Table 3, the electrochemical performance of LTO samples was tested in an LFP/LTO 18650 cylindrical cell that is often found in the market; thus, the result provides better proof for large-scale application.

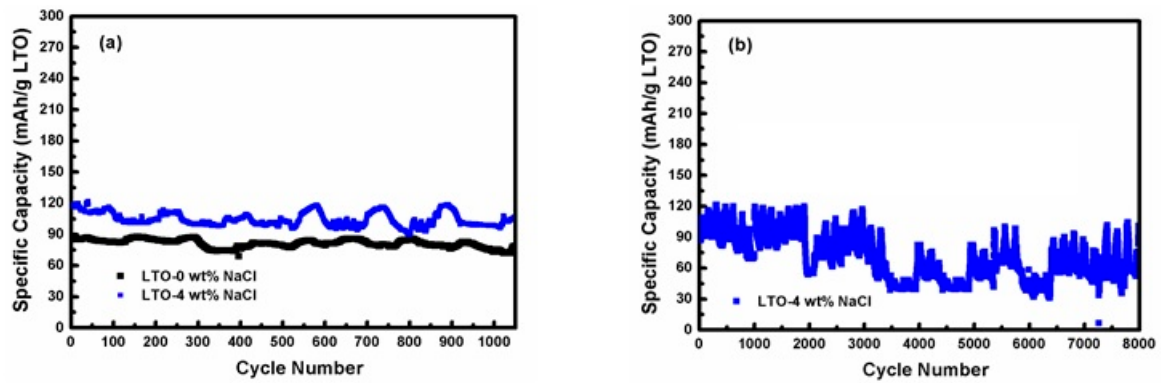


Figure 5. (a) Cycle performance of LTO material at 10 C-rate; (b) Further cycle analysis of LTO-4 wt % NaCl material at 10 C-rate.

Table 3. Summary of LTO performances from previous studies

LTO Precursors	Methods	Particles Size	Electrochemical Testing	Initial Specific Capacity (mAh/g)	Retention Capacity	Rate Performances	Ref.
TiCl ₄ + Li ₂ CO ₃	Sol-gel	5–20 µm	Half Cell	158 mAh/g at 0.1 C	75.95% after 50 cycles at 0.1 C	70 mAh/g at 1 C	[56]
LiNO ₃ + TTIP	Spray pyrolysis	1.5 µm	Half Cell	175 mAh/g at 0.1 C	81.14% after 30 cycles at 0.1 C	105 mAh/g at 1C	[57]
LiOH.H ₂ O + TTIP	Supercritical Hydrothermal	20–50 nm	Half Cell	149.6 mAh/g at 1 C	95.52% after 50 cycles at 1C	108 mAh/g at 8C	[31]
LiOH.H ₂ O + TiO ₂	Molten salt (LiCl-KCl)	4–5 µm	Half Cell	169 mAh/g at 0.2 C	93.49% after 50 cycles at 0.2 C	98 mAh/g at 10 C	[34]
Li ₂ CO ₃ + TiO ₂	Solid state (coated carbon)	1–3 µm	Full cell (LTO vs. LFP)	180 mAh/g at 0.1 C	97.77% after 400 cycles at 1 C charging and 3 C discharging	130 mAh/g at 5C	[55]
TiCl ₄ + LiOH.H ₂ O	Sol-gel	360 nm	Full Cell (LTO vs. NMC)	149 mAh/g at 1 C	93.29% after 150 cycles at 1 C	80 mAh/g at 0.2 C charging and 10 C discharging	[58]
Commercial LTO	-	2 µm	Full Cell (LTO vs. LFP)	147 mAh/g at 0.1 C	51% after 700 cycles at 0.5 C	40 mAh/g at 10 C	[42]
LiOH + TiO ₂ + NaCl	Salt-assisted solid state	1.83–2.28 µm	Full Cell (LTO vs. LFP)	145.6 mAh/g at 0.3 C	98% after 8000 cycles at 10C	9 mAh/g at 10 C	This study

Since an LTO/LFP battery has an impressive cyclability, we perform a further cycle analysis using different cells and extended the cycles to 8000 in Figure 5b. During high rate charge/discharge using a 10 C-rate current, the capacity drop can be observed; however, at a certain point, the specific discharge capacity increases to around ≈ 120 mAh/g. The phenomenon occurred constantly and consistently up to 8000 cycles and consistent with every cell testing, which can be seen in Figure A3. Technically, this can be caused by a continuous cycling process with no significant resting time [59,60] and due to the temperature fluctuation of the surrounding areas, which is not tightly controlled [18]. However, based on Figure 5a, the capacity fluctuation that occurred in an LTO 0 wt % NaCl is not significant compared to that of an LTO 4 wt % NaCl. Since the gradual decrease and increase of specific capacity significantly occurred in LTO 4 wt % NaCl, it can be concluded that the cell exhibits a self-recovery ability that can extend the cycle life of the cell due to the presence of NaCl. In addition, the cells were tested using a cylindrical (18,650 type) full cell, and it is safe to say that the results provide superior evidences for the mass production of LTO/LFP considerations with superior performance. The average capacity of LTO 4 wt % NaCl is 83.6 mAh/g, and based on 1st cycle and 8000th cycles, the capacity retention is 98%.

3.4. Post Cycle Analysis of LTO

The post cycle of the LTO structure was analyzed to evaluate the volume expansion of the LTO material after 1060 cycles. Figure 6a displays the XRD pattern of LTO-4 wt % NaCl after 1060 cycles compared to the initial XRD pattern. The XRD patterns demonstrate that there are no changes on the peaks of LTO before and after the cycling test. However, new peaks are observable at 2θ of 27.42° , 33.08° , and 36.15° , which strongly correspond to the peaks of residual acetylene black (AB) [61], Li_2CO_3 [62], and LiF [62,63], respectively. The presence of Li_2CO_3 originated from the side reaction between lithium and alkyl carbonates (electrolyte solvent) during the intercalation/de-intercalation process [62]. Then, the existence of LiF was due to the decomposition of LiPF_6 to LiF and PF_5 [62]. Moreover, the formation of PF_5 can also react with Li_2CO_3 to LiF_6 , as reported by He et al. [63].

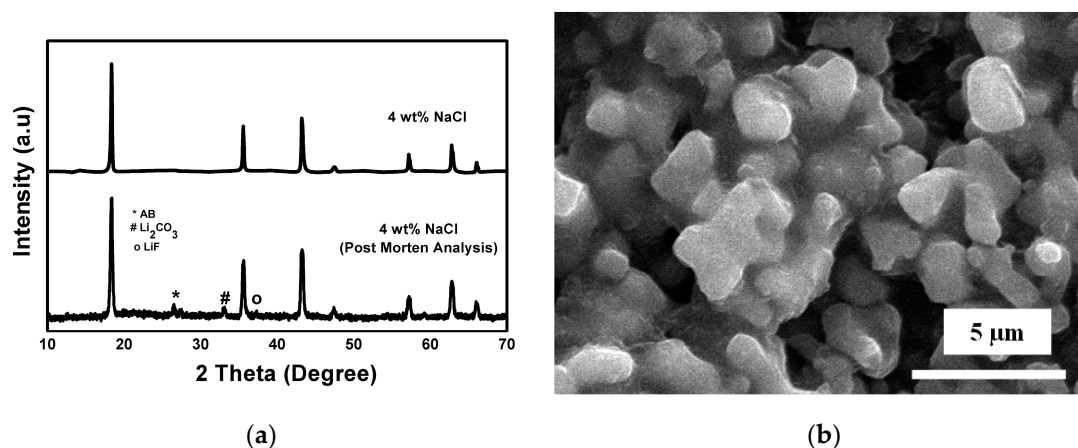


Figure 6. Post cycle of LTO-4 wt % NaCl after 1000 cycles by (a) XRD and (b) SEM.

Based on Figure 6b, the SEM image of LTO-4 wt % NaCl is still similar to the initial SEM image (Figure 2c) with cubic morphology. However, the cubic-shaped particles of LTO-4 wt % NaCl are covered by a thin film. It is predicted that the film consists of residual electrolyte and a solid electrolyte interface as referred by previous studies [62,63].

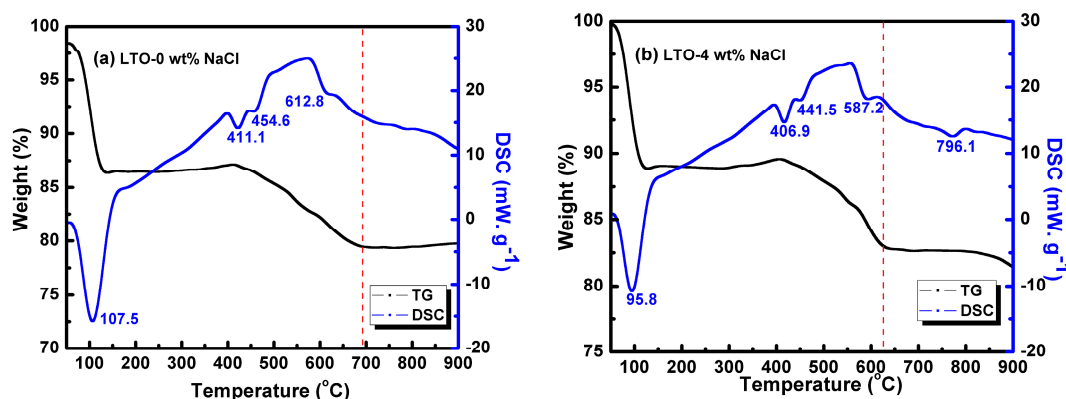
The structural parameter of LTO-4 wt % NaCl can be evaluated using XRD data, and the results are displayed in Table 4. The lattice constant of LTO-4 wt % NaCl after cycles is non-significantly different compared to the initial LTO-4 wt % NaCl, which provides strong evidence of a zero-strain material [48,49]. The FWHM analysis indicates that the crystallinity of LTO is slightly decreased after 1060 cycles, which is correlated with the bigger size of LTO crystallite.

Table 4. The structural parameters of LTO material before and after cycling.

LTO	Lattice Constant (Å)	FWHM			Crystallite Size (nm)		
4 wt % NaCl	8.362	0.154	0.137	0.168	54.57	63.53	53.11
4 wt % NaCl (post mortem)	8.357	0.161	0.142	0.175	52.23	59.22	48.05

3.5. TG-DSC Study of Salt-Assisted LTO Formation

The thermogravimetric and differential scanning calorimeter (TG-DSC) analysis of LTO-0 wt % NaCl and LTO-4 wt % NaCl are depicted in Figure 7. It is clearly observed that both curves show a similar trend. At temperatures between 0 and 100 °C, there are weight losses around 12.06 and 10.10 wt % of LTO-0 wt % NaCl and LTO-4 wt % NaCl, respectively, which indicated the removal of absorbed water in the LTO precursor, according to the endothermic peaks at 107.5 and 95.8 °C [64]. At temperatures between 100 and 415 °C, both curves show a flat straight line, which indicates that no mass change is occurred. However, at temperatures of 411.1 and 406.8 °C, the endothermic peak is detected, which indicates the melting point of LiOH [65]. Then, the small weight losses of LTO-0 wt % NaCl and LTO-4 wt % NaCl (4.35 and 4.11 wt %) that occurred at 454.6 and 441.5 °C, respectively, can be interpreted as the hydroxylation [66]. While the temperature increases to 612.8 and 587.2 °C of both samples, a small weight loss is observed in each curve (3.13 and 3.08 wt %) corresponding to tiny endothermic peaks at 612.8 and 587.2 °C, respectively, which is attributed the reaction of LTO. The formation of spinel LTO-0 wt % NaCl and LTO-4 wt % NaCl occurred completely at temperatures of 717.4 and 624.6 °C, respectively, indicating no mass change occurred. However, a new endothermic peak is observed at 796.1 °C in LTO-4 wt % NaCl, which corresponds to the melting point of NaCl [67]. This phenomenon confirmed the XRD analysis that the addition of NaCl alters the sintering behavior. The crystal growth of LTO-4 wt % NaCl initiates earlier at 624.6 °C, whereas LTO-0 wt % NaCl initiates at 717.4 °C, which clarifies the higher crystallite size of LTO-4 wt % NaCl than LTO-0 wt % NaCl.

**Figure 7.** Thermogravimetric and differential scanning calorimeter (TG-DSC) curve of LTO formation.

4. Conclusions

The synthesis of highly crystalline $\text{Li}_4\text{Ti}_5\text{O}_{12}$ material that shows high electrochemical performance was successful using a salt-assisted solid-state reaction with LTO made up of micron-sized, homogenous particles. The XRD pattern results showed that the NaCl addition enhanced the crystallinity of the LTO material. Increasing the NaCl addition from 0 to 6 wt % improved the crystal formation of the LTO material. However, the optimal amount of NaCl addition proved to be 4 wt %. The excellent charge–discharge capacity of LTO with addition of NaCl at 4 wt % was 145.6 mAh/g, and the compound showed good stability during a subsequent cycling test. The favorable benefits of this method are its facile synthesis and low cost for large-scale production, as well as a performance that features a high discharge capacity and good stability. This compound shows promise for use as an anode material in lithium ion batteries.

Author Contributions: Conceptualization, H.W., M.N., and A.J.; Data curation, S.U.M., C.S.Y.; Formal analysis, S.U.M., C.S.Y.; Investigation, M.I.P., E.R.D., H.W., and M.N.; Methodology, A.P., S.U.M., C.S.Y.; Project administration, A.P.; Supervision, A.P.; Validation, H.W., M.N., and A.J.; Visualization, S.U.M., E.R.D.; Writing—original draft, S.U.M., C.S.Y.; Writing—review and editing, A.P. All authors have read and agreed to the published version of the manuscript.

Funding: This research is financially supported by Indonesian Ministry of Education, Culture and Higher Learning through World Class Research scheme (WCR) with contract no. 112/UN27.21/HK/2020.

Acknowledgments: The authors thank Heni Triagline and Edwin Budi Setiawan for assistance in this research.

Conflicts of Interest: The authors declare no competing financial interests.

Appendix A

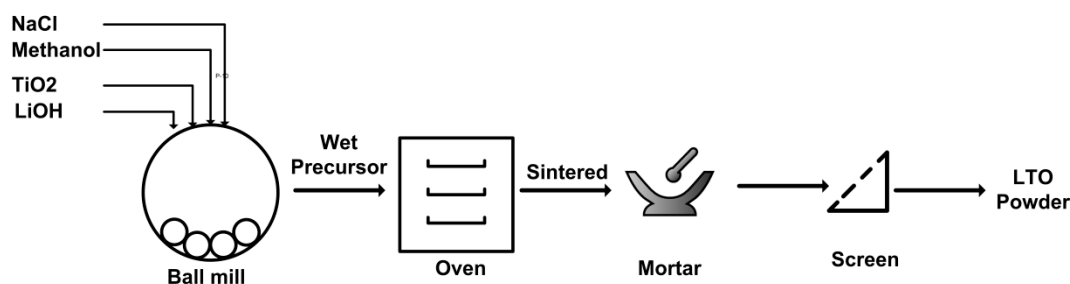


Figure A1. Synthesis process of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ powder.

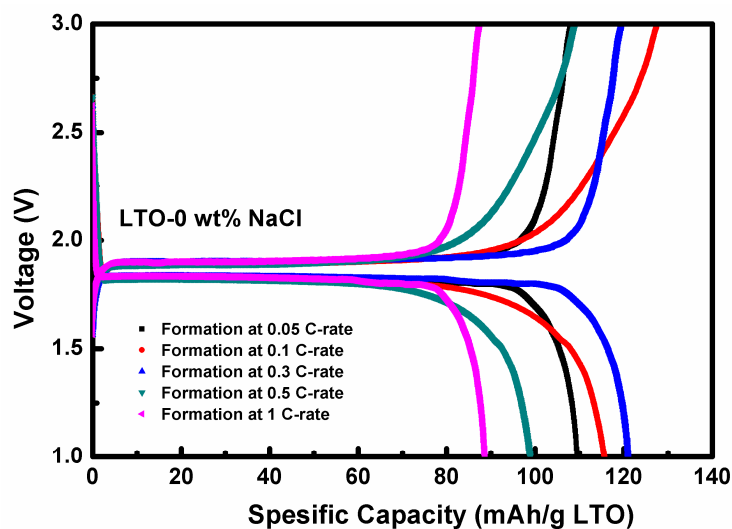


Figure A2. Charge-discharge formation curve at various C-rates (LTO vs. LFP).

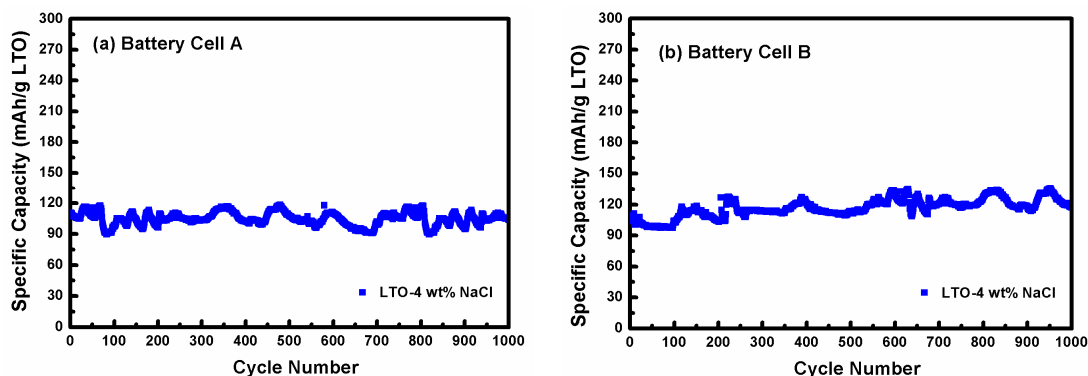


Figure A3. Cont.

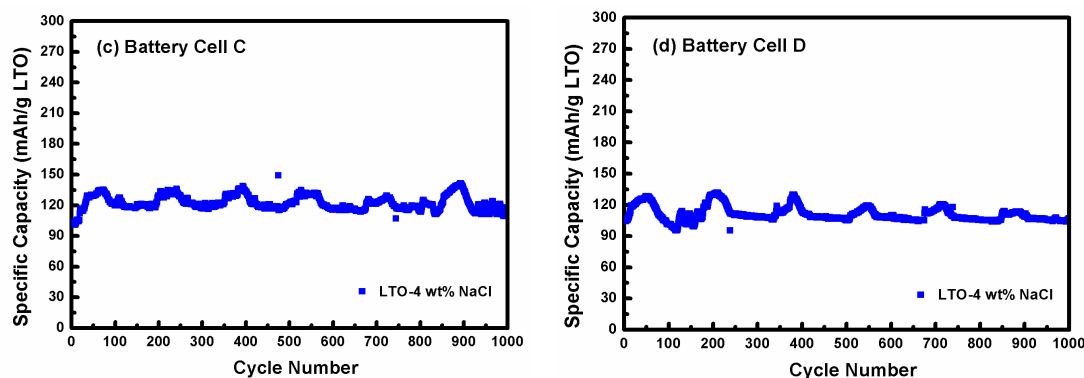


Figure A3. Cycle performances of various LTO-4 wt % NaCl/LFP battery cell at 10 C-rate.

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