

## ELECTROCHEMICAL PERFORMANCE OF $\text{Li}_{4-x}\text{Ti}_5\text{Cu}_x\text{O}_{12}$ FOR LITHIUM ION CAPACITOR APPLICATIONS

### PERFORMA ELEKTROKIMIA $\text{Li}_{4-x}\text{Ti}_5\text{Cu}_x\text{O}_{12}$ UNTUK APLIKASI KAPASITOR ION LITUM

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#### Abstrak

Senyawa litium titanat (LTO) menarik banyak perhatian karena memiliki karakteristik yang unik untuk aplikasi penyimpanan energi. LTO terdoping merupakan salah satu pendekatan untuk meningkatkan performa LTO. Hingga saat ini, kinerja LTO yang didoping dalam kapasitor litium ion jarang dikaji. Tujuan dari penelitian ini adalah menyintesis LTO yang didoping Cu melalui reaksi solid state dan milling energi tinggi serta mempelajari kinerja elektrokimia kapasitor ion litium. LTO terdoping Cu disintesis dengan menggunakan metode solid state berbasis high energy milling. Pola difraksi menunjukkan bahwa LTO yang didoping Cu telah berhasil disintesis, meskipun terdapat pengotor seperti Baddeleyite,  $\text{Zr}_4\text{O}_8$ , dan  $\text{Li}_2\text{O}$  (~50%) muncul di setiap sampel. Profil cyclic voltammetry dari LTO Cu-doped berbasis setengah sel menunjukkan bahwa puncak oksidasi dan reduksi menurun karena kandungan pengotornya. Resistansi listrik dari LTO dan LTO terdoping Cu menjadi lebih kecil karena meningkatnya kandungan dopan, sementara resistensi transfer muatan menjadi semakin meningkat seiring kandungan dopan. Performa sel penuh LIC menyatakan bahwa LIC berbasis LTO//karbon aktif secara umum menunjukkan kapasitansi yang lebih tinggi daripada LIC berbasis  $\text{LTOCu}$  0,025//karbon aktif.

**Kata kunci:** Litium titanat, Cu-doping, Performa elektrokimia, Kapasitor litium ion

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**Keywords:**

Lithium titanat  
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**Abstract**

Lithium titanate (LTO) has attracted considerable attention since it has unique characteristics for energy storage application. Doped LTO is one of the approaches to improve LTO performance. To date, doped LTO performance in full-cell lithium ion capacitor has rarely been discussed. This study is aimed to synthesize Cu-doped LTO via solid state reaction and high energy milling and investigate its electrochemical performance in full-cell of lithium ion capacitor. The diffraction patterns show that Cu-doped LTO has been successfully synthesized, even though the impurities such as Baddeleyite,  $Zr_4O_8$  and  $Li_2O$  (approximately 50%) appear in each sample. Cyclic voltammogram profile of half-cell based Cu-doped LTO shows that the oxidation and reduction peaks are declined due to its impure content. The electrical resistance of LTO and Cu-doped LTO decrease as doping content increases, while charge transfer resistance becomes higher. Full-cell performance of LIC represents that LIC based LTO//Activated carbon shows higher capacitance than that of LIC based LTOCu 0.025//Activated carbon.

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**INTRODUCTION**

Energy storage devices such as lithium ion battery (LIB) and electro double layer capacitor (EDLC) are widely developed in order to support renewable energy application in the future. These devices are chosen due to their natures, for instance high energy density (LIB) and power density (EDLC) (Lu et al. 2017; Shellikeri et al. 2018). Recently some researchers have discovered a new energy storage device named lithium ion capacitor (LIC). This device is ascribed as a promising device especially for hybrid electrical vehicle (HEV) application (Li et al. 2018). LIC theoretically owns high energy and power density donated from anode material of LIB and porous cathode of EDLC (Ding et al. 2018). Unfortunately, performance of LIC has to be explored in order to meet the desired properties of energy storage applications.

Spinel-lithium titanate ( $Li_4Ti_5O_{12}$ ), usually abbreviated as LTO, is one of the popular materials applied for negative electrode of energy devices due to its advantages. This material, which is classified into Fd3m group, is commonly called as “zero-strain” material (Sun, Radovanovicb, and Cui 2014). This is due

to the material property which may not suffer volume expansion during advanced charge-discharge process because of its compatibility with commercial electrolyte,  $LiPF_6$  (Sandhya, John, and Gouri 2014). LTO is also featured with excellent safety such as low volume expansion enabling to avoid explosion and good cycle stability performance. Therefore, this material is applied for lithium ion capacitor or hybrid supercapacitors in order to be installed in HEV applications (Ni et al. 2012; Zhao et al. 2016). Instead of that, LTO owns crucial drawbacks such as low electrical conductivity and inferior diffusion property, in which many researchers intensively pay attention to (Liu et al. 2013). To overcome these drawback, LTO has recently been modified through some approaches such as reducing size particles and substituting ion metal to the structure of LTO (Huang et al. 2005; Meng et al. 2018). Wang et al. (2013) have tried to insert certain amount of  $Cu^{3+}$  into Li site ( $Li_{4-x}Ti_5O_{12}$ ,  $x = 0, 0.1$ , and  $0.2$ ) for anode of LIB. This substitution may enhance the conductivity and stabilize the polarization degree. Cu-doped LTO composites were also successfully obtained and its performance can be characterized in

half-cell battery. This modification could stabilize LTO structure, which can retain the electrochemical performance at about 92.2% of its initial value after 500 cycles (Chen et al. 2015). To our knowledge, metallic-ion-doped LTO is intensively employed as negative electrode in LIB to enhance its electrical conductivity. However, due to its new development, the study of metal-ion-doped LTO for lithium ion capacitor, especially Cu-doped LTO for negative electrode of LIC, is still rare to find.

In this paper, LTO doped by low concentration of  $\text{Cu}^{3+}$  will be studied in a full-cell of LIC. Cu-doped LTO was successfully synthesized via solid state reaction followed by wet milling using High Energy Milling (HEM). The half-cell based Cu-doped LTO is evaluated by employing Cyclic Voltammetry (CV), Charge-Discharge (CD), and Electrochemical Impedance Spectroscopy (EIS) measurements. In addition, the full-cell LIC based LTO and Cu-doped LTO is also examined by CV and CD at various scan rate and current density to investigate its performance for lithium ion-capacitor application.

## METHODOLOGY

Initially,  $\text{Li}_{4-x}\text{Ti}_5\text{O}_{12}\text{Cu}_x$  ( $x = 0, 0.025, 0.05$ , and  $0.075$ ) was synthesized via wet-milling process and followed by sintering.  $\text{Li}_2\text{CO}_3$  (Merck, purity >98%),  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  (Merck, >90%), and  $\text{TiO}_2$  (Merck, Anatase, purity >90%) were used as raw materials for Li, Cu, and Ti sources, respectively. To obtain 5 g of  $\text{Li}_{4-x}\text{Ti}_5\text{O}_{12}\text{Cu}_x$ , the raw materials, mixed with 10 mL deionized water, were milled by High Energy Milling (HEM) featured by  $\text{ZrO}_2$  balls with ratio of 10:1 in weight (balls:raw materials) for 5 hours. The sludges were then sintered at  $900^\circ\text{C}$  for three hours with heating rate of  $5^\circ\text{C}/\text{min}$  in order to acquire the powder of

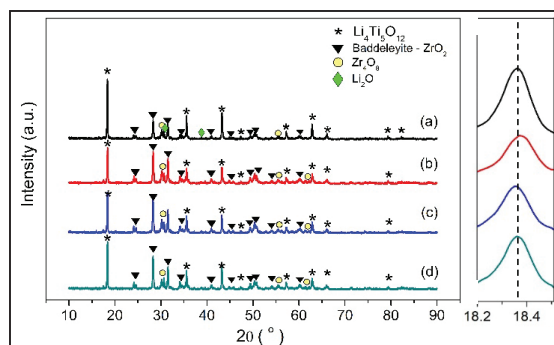
$\text{Li}_{4-x}\text{Ti}_5\text{O}_{12}\text{Cu}_x$ .  $\text{Li}_{4-x}\text{Ti}_5\text{O}_{12}\text{Cu}_x$  ( $x = 0.025, 0.05$ , and  $0.075$ ) and were named as LTO, LTOCu 0.025, LTOCu 0.05, and LTOCu 0.075 respectively.

To study the electrochemical performance, the sample was prepared into coin cell CR2032 type. The electrode contains active material, e.g. LTO or Commercial Food-Grade (FG), Activated Carbon (Technical Grade), Polyvinylidene Fluoride (PVDF, Technical grade) as binder and carbon black (Super-P, Technical grade) as conducting agents with ratio 85:10:5 in weight respectively. Initially, the materials were mixed in N,N-Dimethylacetamide (DMAc) at  $70^\circ\text{C}$  and the slurry was casted on copper foil and aluminium foil for anode and cathode respectively. The casted slurry was then heated at  $80^\circ\text{C}$  in order to evaporate the solvent and shaped into 16 mm-diameter disc. For half-cell, the LTO electrode was crimped with Metallic lithium, while for the full-cell capacitor, the LTO electrode was paired with a cathode based FG. The electrolyte and separator employed in this preparation were 1 M  $\text{LiPF}_6$  dissolved in ethylene-carbonate and ethyl-methyl-carbonate (EC:EMC) in ratio 3:7 v/v and Celgard separator.

The X-Ray Diffraction characterization (Rigaku SmartLab at Research Center for Physics, LIPI) was initially executed at 2-theta range  $10^\circ$  to  $90^\circ$  with Cu  $\text{K}\alpha$  with wave length of  $1.541862 \text{ \AA}$  to probe the phase formation of each samples. Electrochemical Impedance Spectroscopy (EIS) via EIS (HIOKI 5322-50 LCR HiTESTER) was also conducted to evaluate the impedance of each cell with frequency of 0.1 kHz-50 kHz. Cyclic voltammetry analysis was performed through both half-cell and full-cell capacitor at various scan rate under 3.0 volt. The full-cell capacitors was also measured by Charge-Discharge (CD) test using various current density of 10, 50, and  $100 \text{ mA g}^{-1}$  in the range of 0.5–3.0 volt.

## RESULT AND DISCUSSION

Figure 1 displays the diffraction pattern of LTO, LTOCu 0.025, LTOCu 0.05, and LTOCu 0.075. It can be seen that the spinel-LTO are definitely detected in each sample at 2-theta around 18.35°, 35.60°, 37.25°, 43.25°, 47.36°, 57.21°, 62.82°, 66.07°, 74.34°, 75.37°, 79.39°, and 82.35° (ICSD 98-016-9866) followed by higher crystallinity than other samples. The other phases are also observed at various 2-theta referring to Baddeleyite-ZrO<sub>2</sub> (ICSD 98-004-1010), Zirconium oxide (ICSD 98-016-4861), and Dilithium oxide (Li<sub>2</sub>O, ICSD 98-010-8886).



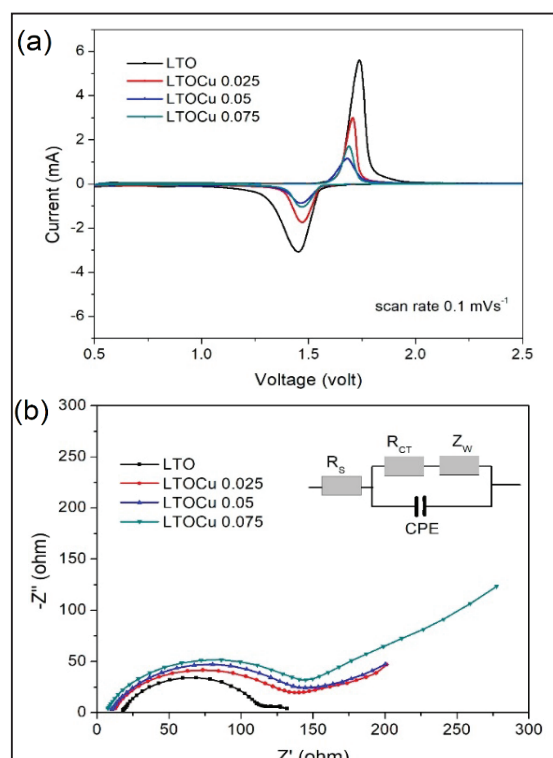
**Figure 1.** XRD-pattern of (a) LTO, (b) LTOCu 0.025, (c) LTOCu 0.05, and (d) LTOCu 0.075

**Table 1.** The percentage of impurities contained in each sample

| Samples<br>LTOCu | Impurity (%) |             |                                |                   |
|------------------|--------------|-------------|--------------------------------|-------------------|
|                  | Spinel-LTO   | Baddeleyite | Zr <sub>4</sub> O <sub>8</sub> | Li <sub>2</sub> O |
| 0                | 54.2         | 22.1        | 10.2                           | 13.5              |
| 0.025            | 41.1         | 40.5        | 18.4                           | -                 |
| 0.05             | 38.5         | 22.1        | 39.4                           | -                 |
| 0.075            | 50.5         | 38.8        | 10.7                           | -                 |

The graph is also featured by peak magnification at the highest peak of reflection index (111). It is obviously shown that the peaks appear shifted as it is substituted by Cu<sup>3+</sup>, especially for LTOCu 0.025, in which shifting peak is observed more significantly than those of LTOCu 0.05 dan LTOCu 0.075 samples. In addition,

the diffraction graph is analyzed using Rietveld refinement method to calculate the percentage of the impurities, as listed in Table 1. It is clearly shown that spinel-LTO contained in each sample seems to decrease as the doping increase. This phenomenon is due to the additional zirconium material suspected from the ZrO<sub>2</sub>-ball during high energy milling and the excessive Li source which unsuccessfully reacts with titanium oxide during milling process. The presence of impurities contained in each sample will affect the electrochemical performance in each cell.



**Figure 2.** (a) Cyclic Voltammogram and (b) Cole-cole plot of LTO, LTOCu 0.025, LTOCu 0.05, and LTOCu 0.075

To understand the electrochemical behavior of each sample, half-cells was initially characterized using CV and EIS. Figure 2a shows CV profile of each sample, which was assembled with lithium metallic into coin cell, at a voltage range of 0.5–2.5 volt with scan rate of 0.1 mVs<sup>-1</sup>. It is clearly

shown that oxidation and reduction peaks are detected at a voltage rate of 1.4–1.75 volt, in which the working voltage of LTO is located, namely 1.55 volt. In addition, other peaks are generally undetected in the voltage range, suggesting that the impurities observed by XRD characterization is ascribed as non-active materials to produce oxidation and reduction peak. Some reference also reported that the existence of these peaks can be an indication that the reaction inside of each cell is reversible (Linden and Reddy 2002). Furthermore, the peak pairs may relate to kinetic properties of LTO and Cu-doped LTO inside the cell, such as mobility of  $\text{Li}^+$ . Based on current response analysis, the peak pairs become dull as the LTO contained in the samples decreased. The cell based pristine LTO represent the highest peaks which can be affected by the LTO contained in the sample. This phenomenon is also supported by the voltage difference of redox peaks presented as 0.29 volt, 0.24 volt, 0.21 volt and 0.22 volt for sample LTO, LTOCu 0.025, LTOCu 0.05, and LTOCu 0.075 respectively. The difference of peak pairs voltage indicates the polarization degree of the ions in the electrode (Zhang et al. 2016; Song et al. 2014).

**Table 2.** Electrical and Charge Transfer Resistance of Each Samples

| LTOCu | Electrical Resistance (ohm) | Charge Transfer Resistance (ohm) |
|-------|-----------------------------|----------------------------------|
| 0     | 17.40                       | 114.84                           |
| 0.025 | 9.51                        | 142.37                           |
| 0.05  | 7.69                        | 155.28                           |
| 0.075 | 4.59                        | 163.12                           |

The sample of LTOCu 0.05 however possess sharper peak pairs than those of LTOCu 0.075 due to lower LTO content as shown in Table 2.

The Cole-cole plot of each sample is presented in Figure 2 (b) featured by the equivalent circuit as an inset in the graph. The semicircles of each sample are fitted using software Z-View in order to determine the electrical resistance ( $R_s$ ) and charge transfer resistance ( $R_{CT}$ ). Based on the equivalent circuit, the  $R_s$  and  $R_{CT}$  are defined as interception of the semicircle with  $Z'$  (X-axis) at high and low frequency respectively (Nuroniah et al. 2019).

As listed in Table 2, it can be seen that electrical resistance is declined. In contrast, the charge transfer resistance is elevated. The increasing  $R_{CT}$  may be due to the impurity of Baddeleyite and  $\text{Zr}_4\text{O}_8$  contributing to inferior conductance property of LTO (Wang et al. 2014). The  $R_s$  decreases as copper content increases due to the high electrical conductivity of copper suggesting fast electron transfer via external circuit.

Figure 3 (a) and (b) present the voltamogram of full-cell LIC based LTO//AC-FG and LTOCu 0.025//AC-FG respectively. The full-cell is performed in the voltage range of 1.0–4.0 volt at various sweep rate of 5–20  $\text{mVs}^{-1}$  at room temperature. These LIC cells are quite different from lithium ion battery and EDLC. Ion  $\text{Li}^+$  theoretically intercalate into LTO and LTOCu 0.025 structure, while the  $\text{PF}_6^-$  is adsorbed into carbon structure (Xu et al. 2018). None both of the redox peaks in LIC cell based on both LTO//AC-FG and LTOCu 0.025//AC-FG is detected, while the rectangular voltamogram shown suggesting capacitor performance (Chen and Chen 2017; Doloksaribu, Triyana, and Prihandoko 2017). It also reveals that current response is linearly escalated as the scan rate rises. This may be addressed to the ions kinetically which move faster and reach to the deepest structure of electrode at high scan rate (Zhao et al. 2015).

**Table 3.** Specific Capacitance of Full-Cell LIC Based LTO and LTOCu 0.025

| Specific Current<br>(mA g <sup>-1</sup> ) | Specific Capacitance<br>(F g <sup>-1</sup> ) |             |
|-------------------------------------------|----------------------------------------------|-------------|
|                                           | LTO                                          | LTOCu 0.025 |
| 10                                        | 5.45                                         | 0.43        |
| 50                                        | 2.63                                         | 0.16        |
| 100                                       | 3.55                                         | 0.08        |

To further understand the electrochemical performance, full-cell LIC based LTO//AC-FG and LTOCu 0.025//AC-FG are investigated by charge-discharge measurement as shown in Figure 3. The measurement was conducted at 1.0–3.0 volt with various current densities of 10, 50, and 100 mA g<sup>-1</sup> (c) and (d) respectively. However, the slope linearity of the presented curve is dissimilar with that of EDLC (Lee and Yoon 2013) which can

be caused by combination mechanism of electrode materials adopted from LIB and EDLC. By this measurement, the specific capacitance can also be obtained as listed in the Table 3. The specific capacitances were calculated via the following equation (Sun et al. 2017):

$$(F\ g^{-1}) = (mAh\ g^{-1}) \cdot 3.6 / \Delta V$$

$\Delta V$  represents the potential window of the measurements and (mAh g<sup>-1</sup>) specific capacity of the full-cell LIC. It can be seen that the specific capacitance of LTO//AC-FG is higher than that of LTOCu 0.025//AC-FG. This phenomenon may be due to the presence of impurity which affected the mobility of Li<sup>+</sup> to reach the structure. Declined as increasing current during the measurement for both of samples.

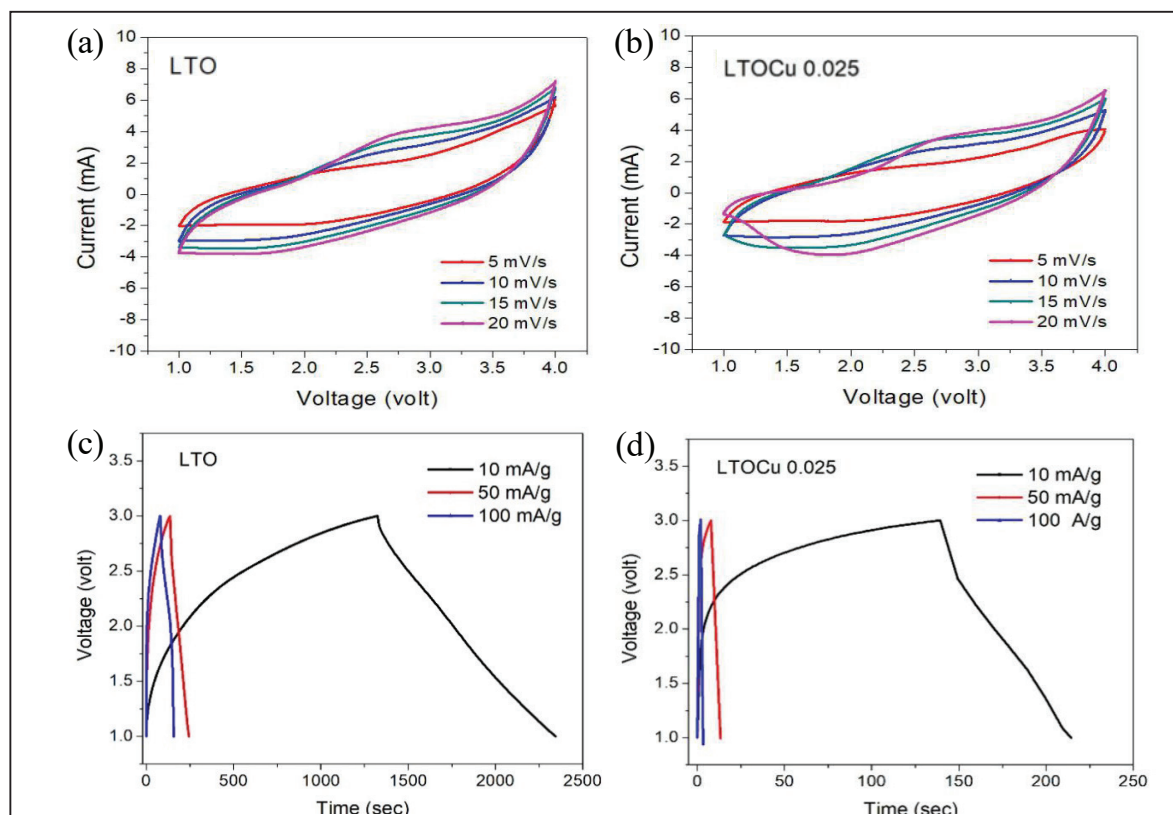


Figure 3 (a-b) Cyclic voltammetry of full cell LIC based (a) LTO//AC-FG and LTOCu 0.025 // AC-FG at various scan rate of 5–20 mVs<sup>-1</sup> and (c-d) Charge-discharge profile at current density 10 mA g<sup>-1</sup>, 50 mA g<sup>-1</sup>, and 100 mA g<sup>-1</sup>

## CONCLUSION

LTO and Cu-doped LTO are successfully conducted via high energy milling although each sample contains 50% impurities detected through XRD analysis. Electrochemical behavior of half-cell exhibit that undoped LTO perform as the highest redox peaks. As Cu increases, the electric conductivity decreases, while the charge transfer resistance increases to some extent. Full-cell performance of LIC represents that LTO//AC-FG shows higher capacitance than that of LTOCu 0.025//AC-FG. For further study, Cu-doped LTO can be synthesized via HEM with certain milling time and Ti-based balls in order to reduce the impurity content.

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