Fabrication And Surface Modification Of Li4ti5o12 Anode Doping Al0.03 With P123 (Pluronic) Template

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Abstract. Lithium Titanium Oxide (Li4Ti5O12) was used as the raw material for the anode of a lithium-ion battery. The active component LTO doped Al0.03 was produced using the sol gel technique and sintering at 850ºC for 4 hours in an airless atmosphere. Furthermore, the materials mixing in two different ways, each with a unique material ordering. In the first procedure, solutions A (lithium acetate), B (aluminum acetate), C (titanium tetrabutoxide), and D (ocean D) are combined. In the second metode, tetrabutoxide is added as the final step. The active components are crushed after the sintering process. The material is composed of anode sheets that are built into lithium battery cells in a glove box. As a result of the characterization, Lithium titanium oxide (LTO) and rutile (TiO2) phases for sequential phase percentage values in sample 1 (91%; 9%) and sample 2 (98%; 2%) with crystal size and lattice parameters, respectively sample 1 (8.3570 Å and 73.813 nm), The sample's surface morphology is non-porous and irregular, with particle sizes of (1.5799 μm and 1.5840 μm), with agglomeration in both samples. When P123 is added to an Al x=0.03 anode battery, both sample 1(109.4 $x10^{-5}$ S/cm) and sample 2 (89.22$x10^{-5}$ S/cm) exhibit strong conductivity and a high diffusion coefficient value.The capacity value of sample 1 is greater than sample 2 with a value of sample 1 (171.87-171.71 mAh/g) and sample 2 (75.79-77.33 mAh/g).

Keywords: lithium ion battery, sol-gel method, P123 (Pluronic).

1 Introduction

Lithium titanium oxide was first analyzed in 1983 by Murphy et al. for its lithium intercalation ability. Since then, Li4Ti5O12 has become one of the most prominent anode materials for lithium ion battery applications due to its unique “zero-strain” structure during charge/discharge processes [2] . With the rapid development of renewable energy power generation, hybrid electric vehicles and other energy storage applications, it has been regarded as the best candidate anodic material for energy storage due to some outstanding features, such as stable flat voltage of 1.5 V, high safety, long cycle performance and easy fabrication. Although Li4Ti5O12 is very superior in terms of safety and durability, there are still many challenges and shortcomings for EV energy storage. Considerable research efforts have been devoted

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to improving the electronic conductivity, ionic conductivity and specific capacity of Li4Ti5O12 by carbon coating , size reduction and structural doping strategies . Therefore, the power and stability of Li4Ti5O12 have been greatly improved , and it has been used in batteries in EVs and HEVs [1] .

On study This will done surface modification $Li_4Ti_5O_{12}$ Al with P123 as *a* template , the use of P123 material is shown so that the surface of the resulting $Li_4Ti_5O_{12}$ Al is porous so that it can increase the specific capacitance. The purpose of aluminum doping is to increase the ionic conductivity of lithium. Based on previous research, the optimum amount of Al doping used is 0.03 mol at this percentage, resulting in good performance with a value the highest diffusion coefficient is 7.31278×10^{-10} [11].

2 Metodology Research

The materials used in this study include Lithium Acetate ($C_2H_3O_2Li$), Aluminum Acetate $Al(CH_3CO_2)_3$, n-heptane (C_7H_{16}), Hydrochloric AC ID (HCL), Titanium Tetrabutoxide ($C_{16}H_{36}O_4$), Plunorium (P123), and Methanol (CH_3OH) as solvents. Research done with use sol method gel. To make electrode sheets, additional materials are used apart from material active Li4Ti5O12 that is PVDF And solvent DMAC. Meanwhile, to make coin cells, the materials used include: lithium metal, electrolyte $LiPF_6$, and one set coin cell. While the tools used in this study include digital scales, mortar and pestle, doctor blade, Furnace, hot Plate, Magnetic Stirer , 400 mesh sieve, CR2032 Coin Cell, Material characterization is done by testing, XRD, FeSEM, EIS, Cyclic Voltammetry, Charge discharge.

2.1 LTO Synthesis

The synthesis of Li4Ti5O12 was carried out by use method sole gel with reaction:

$$4C2H3LiO2 + (5\text{-}x)C_{16}H_{36}O_4Ti + XC_4H_7AlO_5 \rightarrow Li_4Ti_{(5-x)}Al_xO_{12}$$

The synthesis process is carried out using 2 methods, the first method l solutions are divided into four solutions, namely solutions A, B, C, and D, where each solution This in the rutkan during 30 minute with speed stirrer 250 rpm. Solution A (Methanol + lithium acetate), solution B (methanol + aluminum acetate + HCL), C solution (n-heptane + titanium tetrabutoxide), solution D (Methanol + P123). Solution B ,C And D in pour into the solution A with the respective sequences of solutions B , D and C so that they form one gel solution at room temperature , In the second method sequence l sequence B , D And C in pour into the solution A with the order of each solution B , D and C The raw materials are mixed homogeneously until they become a gel then dried at a temperature of 80°C and ground to produce a fine powder into an LTO precursor which will be sintered at a temperature of 850°C for 4 hours. The results of sintering are ground and sieved with a 400 mesh sieve then become the active material of LTO.

2.2 Anode Sheet Manufacturing

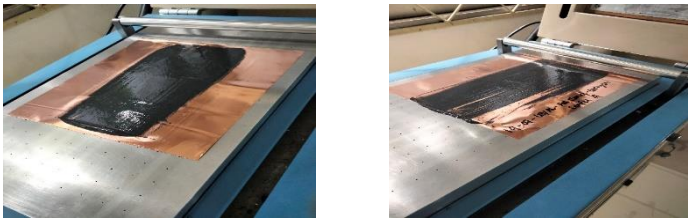
Li4Ti5O12 anode sheets using the doctor blade method . The active material powder (LTO anode) is mixed with additives, namely PVDF and Super P with a composition of 80:10:10 into the N,N-DMAC solution until a slurry is obtained . The coating process on Cu foil with a thickness of 100 μm, then cut with a diameter of 16 mm in the assembly to form a coin cell.

2.3 Battery Performance Test

Coin cell battery cells are subjected to X-Ray testing Diffraction (XRD) , field-scanning Electron Microscope (Fe-SEM), EIS and Cyclic Voltammetry with a voltage of 0.8-2.5 V and a scan rate of 100 mV/s and charge-discharge with a voltage of 1-3.5 V CD testing is only carried out at a speed of 0.1 C.

3 Results And Discussion

3.1 Sheet Analysis



(a) (b)

Fig. 1. Sheet Analysis (a) sample 1 (b) sample 2

The image it can be seen that the results of the LTOAl sheet with a diameter of 100 μm and 16 mm have differences. The second sample shows that the LTOAL sheet does not adhere well compared to the first sample, the sheet results have good adhesion and the sheet is smoother compared to the second sample, this is influenced by the sintering temperature can reduce the solid materials used in the research [5].

3.2 X-Ray Analysis Diffraction (XRD)

The results of the analysis using Highscore software showed that the sample had 2 phases, namely LTO (♦) and TiO2 rutile phase (•). The emergence of these 2 phases was due to the mixing of precursor solutions that were not homogeneous [4].

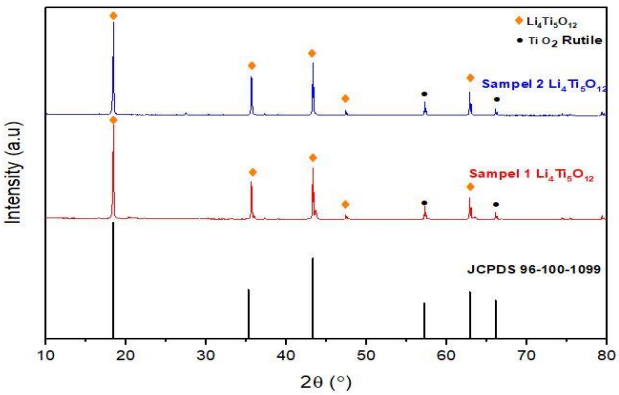


Fig. 2. XRD curve of LTOAl material.

Based on Figure 1, the LTOAl material curve shows that the XRD patterns of samples 1 and 2 are in accordance with the JCPDS 96-100-1099 database . The highest peaks of the Li 4 Ti 5 O 12 phase are at angles of 18, 35, 43, 47 and 62, which correspond to the planes (111), (131), (040), (133) and (044). Both samples have a cubic structure with the space group Fd3m. XRD pattern of the TiO 2 phase rutile in the sample also matches the JCPDS 96-900-9084 database . The highest peaks of the TiO 2 phase rutile is at angles 56 and 62 which correspond to the (220) and (002) planes. TiO 2 phase Rutile has a tetragonal structure with space group P42/mnm.The percentage of XRD phase results of LTOAl samples 1 and 2 can be seen in Table 1 with analysis method using Highscore software.

Table 1. Percentage of phases from XRD results using software High Score

Sample	$Li_4Ti_5O_{12}$ (%)	TiO_2 (%)
LTOAl Sample 1	98	2
LTOAl Sample 2	91	9

Based on the table, it shows that the percentage of the LTOAl phase of sample 2 decreases compared to sample 1. Meanwhile, the TiO2 phase rutile increases. To calculate the lattice parameters of LTOAl samples 1 and 2, the following equation can be used:

$$\frac{1}{d^2} = \frac{h^2+k^2+l^2}{a^2} \quad (1)$$

Table 2. Lattice parameters for LTOAl samples

Sample	Grid Parameter (Å)
LTOAl Sample 1	8.3570
LTOAl Sample 2	8.3568

LTO has a FCC cubic spinel crystal structure where the lattice parameter values of sample 1 and sample 2 are not much different or can be said to be the same. The lattice parameter values in Table 2 are close to the results of [8] which produced a lattice parameter value of a = 8.368 Å [7]. Al ion doping causes a decrease in lattice parameters because Al ions can enter the LTO spinel structure and substitute Ti^{4+}.

To calculate the crystal size of LTOAl samples 1 and 2, it was calculated using the Williamson-Hall method, as in equation :

$$\beta\cos\theta = \frac{K\lambda}{D} + 4\varepsilon \sin\theta \quad (2)$$

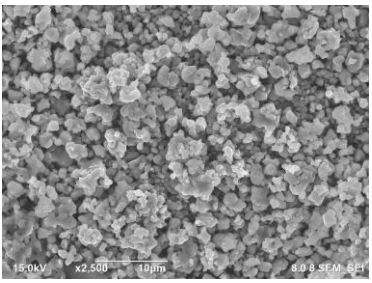
Table 3. Crystalline sizes of LTOAl samples

Sample	ε(*Gradient*)	Crystal Size (nm)
LTOAl Sample 1	0.000755	73.81312
LTOAl Sample 2	0.000844	83.59558

The crystallite size of the LTOAl sample 1 is 73.81312 nm. While the LTOAl sample 2 is 83.59558. The crystallite size value in Table 4.3 is close to the crystallite size value produced in [12], which is 81.11 nm [11], the crystallite size of sample 1 is smaller than that of sample 2, this indicates a high conductivity value in sample 1 because the smaller the particle size, the shorter the diffusion path.

3.3 Field Analysis - Scanning Electron Microscope (FE-SEM)

FE-SEM characterization was used to determine the morphology of the sample and average particle size distribution. Figures (a and b) show FE-SEM images. from L TO Al doping with a thickness of Alx 0.03 . Magnification Which used is 2.5 k Which relate with histogram size particle on picture (c and d).



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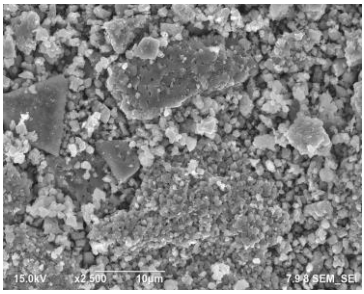

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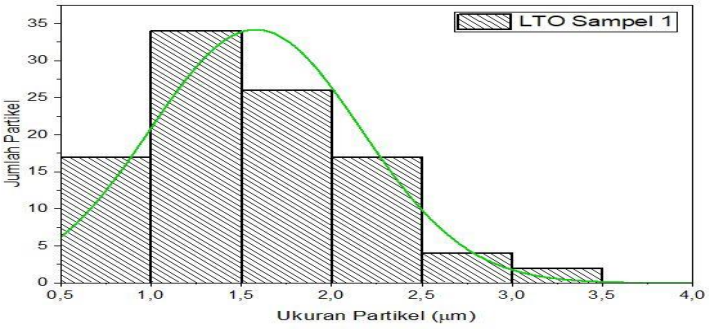

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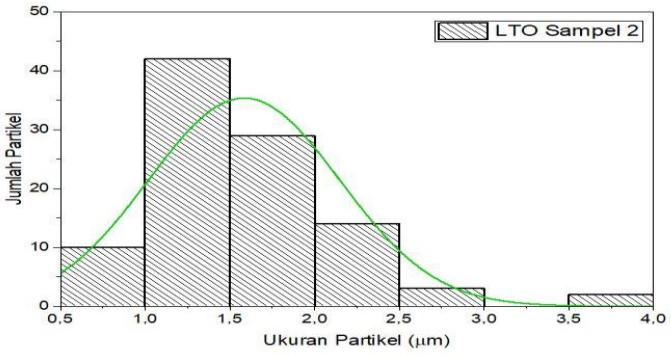
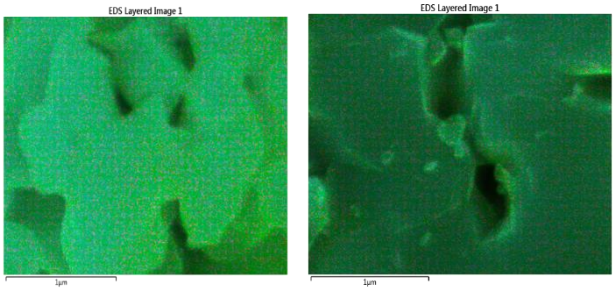


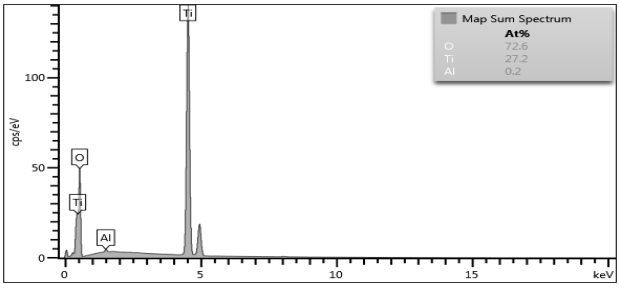
(d)

Fig. 3. Field Analysis-Scanning Electron Microscope (FE-SEM) Analysis (a) dsn (c) sample 1 (b) dan (d) sample 2

Processing data done using software image-j with use picture results testing with FE-SEM.). In images (a) and (b) show the surface morphology of the particles and images (c) and (d) show the number of particles against the particle size (μm). In general morphology, surface from every sample there is no difference in particle size, sample 1 has an average particle size value of 1.5799 μm while sample 2 has a value of 1.5840 μm . The size of both samples has a smaller value than the particle size of the research results of 3.938 μm [11], this shows that the homogeneity of samples 1 and 2 is better so that they have a greater particle distribution. However, the second samplehas higher agglomeration compared to sample 1. This is proven by the large percentage of the rutile phase in sample 2 compared to sample 1. This agglomeration is caused by the inhomogeneous mixing process which causes particles clumping , causing the resulting sheet to have poor adhesion and be rough as explained in the sheet analysis.



(a) (b)



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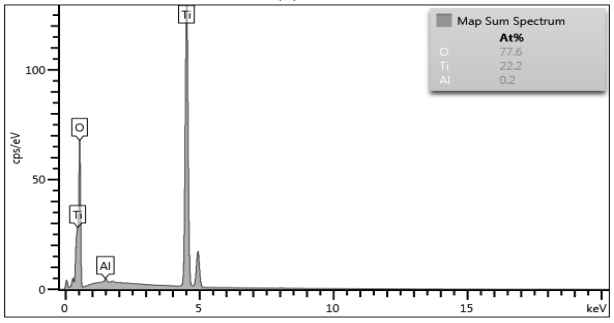


(b)

Fig. 4. Surface morphology EDS results of elemental content of LTOAl Samples 1 and 2

Figure 4 (a) and (b) show the surface morphology of the EDS particle results with a scale of 1 μm. In figure (a) there is a distribution of small particles that is slightly smaller than in figure (b), sample b has a large particle distribution due to agglomeration [10]. This agglomeration occurs due to the densification process (particle compaction) in the sintering process, resulting in grain growth. Figures (c) and (d) show the percentage of element content in the two samples shown in Table 4.

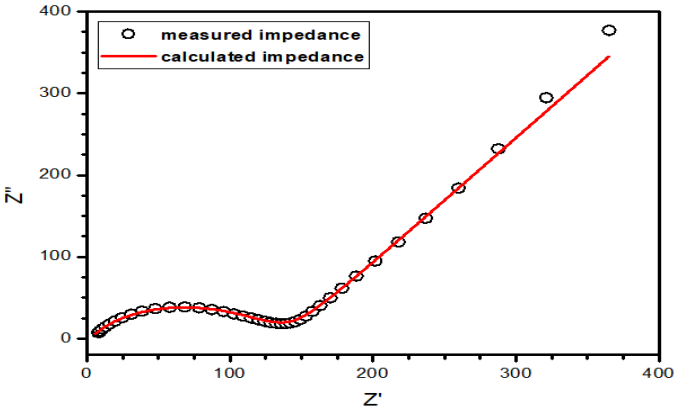
Table 4. EDS results of elemental content of LTOAl samples Samples 1 and 2

Sample	Ti (At%)	O (At%)	Al (At%)
LTOAl Sample 1	22.2	77.6	0.2
LTOAl Sample 2	27.2	72.6	0.2

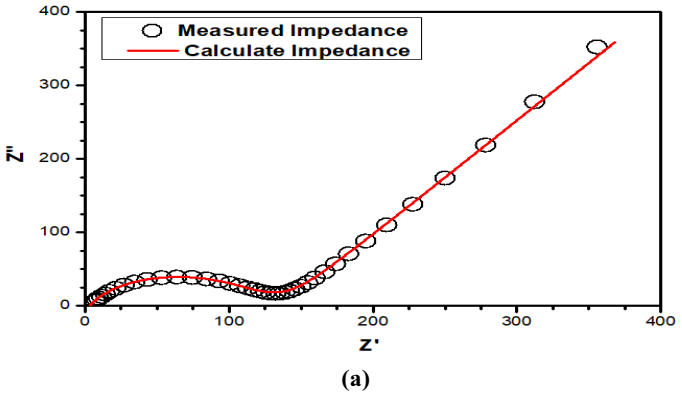
Based on Table 4, the content of the Aluminum element is the same, which is 0.2%, while the content of the Oxygen element in sample 1 is greater, which is 77.6%, than in sample 2, which is 72.6%. The content of the Titanium element is greater in sample 2, which is 27.2% compared to sample 1, which is 22.2%. In both samples, the Li element was not detected because the Li element is the lightest element.

3.4 Electrochemical Impedance Spectroscopy (EIS) Analysis

In the EIS , test it is carried out to determine the conductivity value of the complex impedance behavior of the Li4Ti5O12Al material . The test was carried out with AC current so that it produces the impedance value shown in graph 4.1 , it can be seen that the resulting impedance has two real and imaginary components, where the x-axis represents the real impedance (Z') and the y-axis represents the imaginary impedance (Z”). From both impedances, the electronic resistance (Re) and charge transfer resistance (Rct) values can be calculated, from the values of both resistances can be utilized to calculated the conductivity of LTOAl [9]. Anodes with good materials will form a semicircular arc pattern (semi cycle) called a nyquis plot and a straight line called the wargburg impedance which indicates the presence of diffusion/intercalation-



de-intercalation process of lithium.



(a)

(b)

Fig. 5. EIS results of elemental content of LTOAl Samples 1 and 2

From Figure 4 , it can be seen that the diameter of sample 1 is smaller than that of sample 2. This shows that the impedance value of sample 1 is greater than that of sample 2 and its electrical conductivity value is greater than that of sample 2. The value conductivity can counted with use equality asfollowing:

$$R = \rho \frac{l}{A} \quad (3)$$

Where R is resistance (Ω) , thick sample, l (cm), wide surfacesample, A (cm ^{2}) and ρ is resistivity. Conductivity calculation data Li4Ti5O12 can be seen on Table 5 as following :

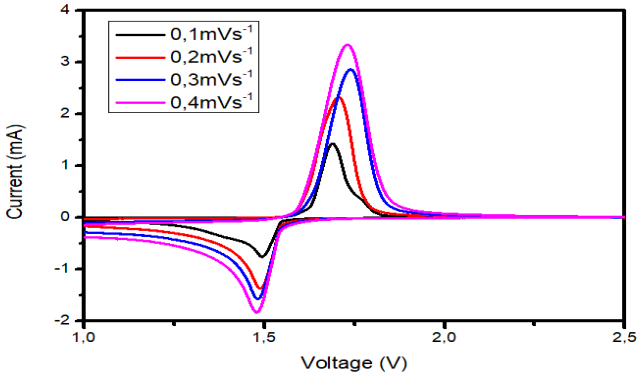
Table 5. Results calculation conductivity Li4Ti5O12

Sample	Sample 1	Sample 2
Thickness (cm)	0.0050	0.0058
A (cm^2)	2,0096	2,0096
R e (Ω)	2.31	3.33
Rct (Ω)	118	110
σ_e (S/cm)	107x10 $^{-5}$	86.6x10 $^{-5}$
σ_{ct} (S/cm)	2.10x10 $^{-5}$	2.62x10 $^{-5}$
σ_{total} (S/cm)	109.4x10 $^{-5}$	89.22x10 $^{-5}$

Based on Table 5, it shows that the electrical conductivity obtained is greater compared to previous research on sample 1, namely $109{,}4 \times 10^{-5}$ S/cm and sample 2, namely 89.22x10 $^{-5}$ S/cm and . In previous studies, the electrical conductivity obtained in lithium ion batteries with Al doped LTO (x = 0.025) was $9{,}48329 \times 10^{-6}$S/cm [6]. Then, in the same study, the electrical conductivity value was obtained at $3{,}185 \times 10^{-5}$ S/cm [3]. The addition of P123 (Pluronic) can increase electronic conductivity but does not increase ionic conductivity. This is because the LTOAl sample sample 1 has a smaller electronic resistance value than the LTOAl sample sample 2, which is 2.31 Ω. While the ionic resistance value in the LTOAl sample sample 1 is greater than the LTOAl sample sample 2, which is 118 Ω. The increase in conductivity values in both samples is due to the small particle size in both samples from the Fe-SEM characterization results.

3.5 *Cyclic Voltammetry* (CV) Analysis

Cyclic Voltammetry (CV) analysis using the WBCS3000, Automatic Battery Cycler Ver.3.2 tool conducted at mVs $^{-1}$. In this test, $Li_4Ti_5O_{12}$ plays a roleas a cathode paired with lithium metal Which play a role as anode because the voltage is lower compared to $Li_4Ti_5O_{12}$ [10]. The result data testing *Cyclic Voltammetry* in the form of curve.Which show connection between voltage (V) and current (I), the yield curve *Cyclic Voltammetry* testing can be seen in figure 4.2 . On the curve the there is peak with direction to on Which show process oxidation And peak with direction to lower shows the reduction process. The reduction process occurs on moment *discharging* , ion lithium move from anode going to cathode while the oxidation process occurs when the lithium ion *charging* moves towards Which opposite going to anode. Process the movement lithium from anode to cathode or on the contrary named as process intercalation And de- intercalation ion lithium.the Physics Research Center - LIPI. In the *Cyclic Voltammetry test* half cell testing was performed with a sample in the form of a coin cell at a voltage *range of* 1.0 -2.5 V by varying the *scan rate speed of* 0.1: 0.2: 0.3: 0.4



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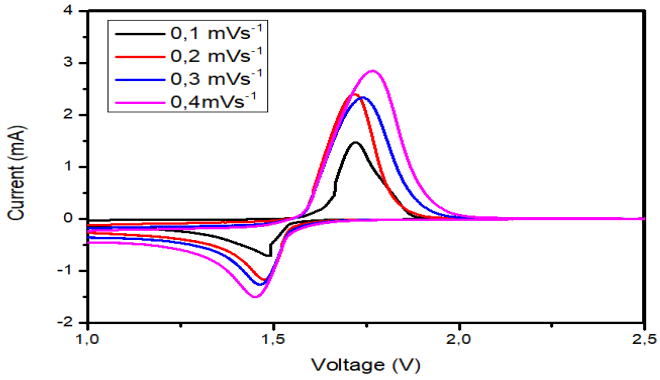


(b)

Fig. 6. Cyclic Voltammetry (CV) of LTOAl samples Samples 1 and 2

From curves a and b , it can be seen in sample 1 the peak is more tall And sharp from sample 2 this indicates that process intercalation And de-intercalation ion lithium takes place quickly. The higher the scan rate, the peak current also increases because more lithium ions flow from the anode to the cathode. At *a scan rate of* 0.1, it can be seen that during the *discharging process* the curve does not immediately decrease and there is a bulge indicating that there is another phase, namely rutile TiO2 . From the voltage and peak current data, the redox reaction can counted the magnitude coefficient diffusion ion Lee with use equality *Randles-Sevcik :*

$$i_p = 2{,}659 \times 10^5 n^{3/2} ACD^{1/2} v^{1/2} \quad (4)$$

Where n is the number of electrons per molecule, A is the surface area. (cm ^{2}), C concentration ion Li (mol/cm ^{3}), D coefficient diffusion ion Lee And v is*scan* speed

(V/s) and i_p is the peak current (A) [10]. Calculation results coefficient diffusion can seen in the table 6 as following :

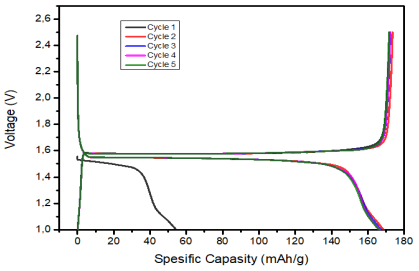
Table 6. Mark coefficient diffusion ion lithium on peak oxidation AndL TOAL material reduction

Lithium Ion Diffusion Coefficient		
Sample	Oxidation (cm^2/s)	Reduction (cm^2/s)
Sample 1	1.87×10^{-10}	1.79×10^{-9}
Sample 2	1.42×10^{-10}	1.23×10^{-9}

Based on Table 6 , the diffusion coefficient value of the two samples is greater than that of the study conducted [12], namely $7{,}31278 \times 10^{-10} cm^2/S$. This shows that the high level of homogeneity in both samples causes the quality of the sheets in both samples to be smoother so that the particle distribution is greater and produces a smaller particle size, with Al doping on LTO can increase the diffusion coefficient of a battery [11]. From Table 4.9, it can be seen that the diffusion coefficient value is very high compared to previous studies that used lower scant rate speed values. So that the greater the scant rate speed value , the lower the diffusion coefficient. Thus, at a scant rate speed *of 0.1* mV / s, the highest diffusion coefficient value is produced, the addition of Al ion doping causes the battery diffusion coefficient to increase. If the diffusion coefficient value is greater, the processintercalation And de-intercalation ion lithium Which in progress in a way fast.

3.6 Analysis Charge – Discharge (CD)

Half-cell batteries with LTO anode material that have been tested with cyclic voltammetry (CV) and then tested charge – discharge to find out discharging capacity on the LTO anode battery. Figure 6 is CD test results of each test sample with charge discharge speed 0.1 C . In *charge-discharge testing* , one cycle is equal to one *charge process* or the process of a redox reaction by releasing lithium ions and electrons to the anode, and one *discharge process* or the process of a reduction reaction by entering lithium ions and electrons to the cathode



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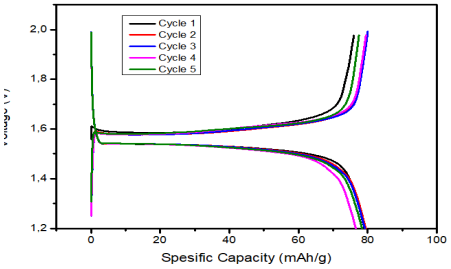

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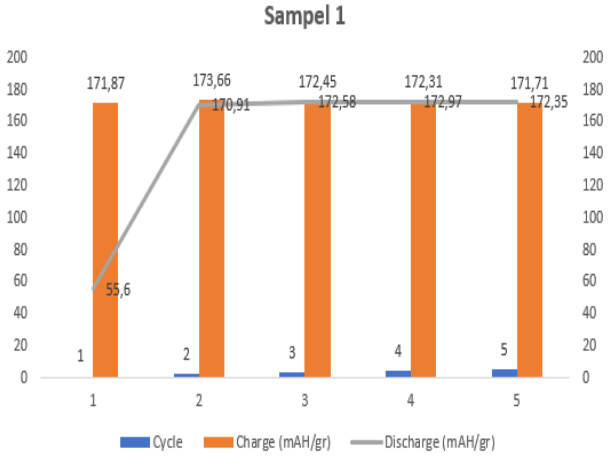

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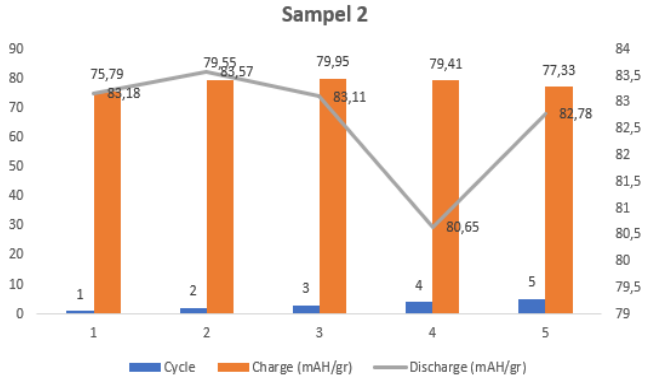

(d)

Fig. 7. *Charge-Discharge* (CD) Analysis (a) dsn (c) sample 1 (b) dan (d) sample 2

Based on Graphs c and d, it is known that the highest Coulomb efficiency value in Sample 1 occurs in *cycle* 4. While the lowest capacity occurs in *cycle* 1 to 1. In *the cycle,* the Coulomb efficiency value is below 50%, which is 32.35 %. This shows that in *cycle* 1 there is an imbalance between *charging* and *discharging so that the battery has* less power . While the Coulomb efficiency value in the next *cycle* has a value above 100%, which is 98.41% - 100.38%. This shows that in the *cycle range* 2-5, the battery has a large energy storage capacity because of its high Coulomb efficiency value. Then in sample 2 the coulomb efficiency value shows the equilibrium between *charging* and *discharging* so that it has greater power than sample 1. From previous research with the addition of Al to the sample [12] the capacity value obtained was low, namely with a capacity value at $Al_{0.03}$ of 156 mAh / g, but in this study the addition of Al succeeded in increasing the capacity value to a greater value. The addition of P123 to LTOAl can also increase the battery capacity so that the power it has is greater.

4 Conclusion

The synthesis of LTO with Al doping x=0.03 has been successfully carried out using the sol-gel method and P123 as a template , respectively showing the formation of the Lithium Titanium Oxide (Li4Ti5O12) phase as much as 91 % and 98% and the rutile phase . Titanium Oxide (TiO2) as much as 9% and 2%, lattice parameters and crystallite sizes of (8.3570 Å - 73.813nm) and (8.3568 Å - 83.595 nm), and morphologically the surface formed is irregular, non-porous, and there is agglomeration shown in the ED results. and the results of chemical performance characterization obtained the conductivity values of samples 1 and 2, respectively 109.4 $\times 10^{-5}$S / cm and $89{,}22 \times 10^{-5}$S / cm. So that it shows that the addition of P123 can increase electronic conductivity in EIS testing , from the addition of P123 also succeeded in increasing the value of the lithium diffusion coefficient in CV testing and the battery storage capacity in CD testing . The effect of the sequence of use of P123 material did not affect both samples because no porous sheets were formed in both samples.

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