



ENHANCED ELECTROCHEMICAL EFFICIENCY OF LITHIUM-ION BATTERY USING TITANIUM AND RHENIUM ADATOMS BY THE APPLICATION OF SQUARE WAVE POTENTIAL REGIME

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Abstract

A new approach for the modification of the Li-ion battery cathode by titanium (Ti^{3+}) and rhenium (Re^{3+}) monolayers using a square wave potential regime has been established. Cyclic voltammetry (CV), Energy-dispersive X-ray spectroscopy (EDX) and Chronoamperometric (CA) were used to analyze the electrochemical properties of these cathodes. The EDX indicates that the active material's powder parts have been fully coated with a thick and homogenous coating from titanium and rhenium after the application of square wave potential. The lower/upper limit potential allowing spontaneous monolayer coating of the lithium surface. This fundamental change has a significant impact on charge-discharge efficiency. Information about local Ti/Re and Li-ions arrangements was obtained from EDX spectra. The CV studies confirm the presence of the coating process in the studied samples and rule out the possibility of Ti^{3+}/Re^{3+} ion diffusion through the structure. Our research also reveals that while coating layers cannot help achieve optimal electrochemical characteristics, they can help to hinder power retention depending on the coating system and conditions. The constructed surfaces were exposed to 1×10^{-3} M of Ti^{3+}/Re^{3+} solution, causing Ti/Re atoms to be permanently adsorbed at the lithium surfaces, resulting in $Li_{sur}-Ti_{ad}/Li_{sur}-Re_{ad}$ surfaces. The lithium-ion surface modified by titanium and rhenium adatoms had greater capacity power than the lithium-ion surface pure. This demonstrates the synergistic effects of Ti^{3+} and Re^{3+} adatoms in imparting higher electrochemistry properties to lithium-ion batteries.

Keywords: Titanium, Rhenium, adatoms, Lithium ion, Battery, Modification, Square wave potential regime.

1. Introduction

At present, with huge petroleum consumption and renewable energy, requirements, because of its high safety, long life and environmentally friendly; Li-ion batteries are used in hybrid vehicles, laptop computers, and mobile phones [1–7]. Because cathode material is a key component of lithium-ion batteries, in recent years research has been dominated by efforts about the development of cathode materials with high energy density and low cost [8–12]. Regrettably, some disadvantages continue to

impede their future use, unsatisfying rate speed, including poor initial coulombic efficiency, decreasing voltage while charging, and rapid capacity fading [13–15]. In order to use such devices in stationary energy storage units and hybrid cars, it is necessary to combine high energy and power density with high protection and low costs. For this aim, the researchers focus on going from zero to 4.0V/Li and 1.5–5 V/Li in the electrochemical window for overcharge protection and safety. The lithium intercalation materials that can be used in the high voltage regime can be used to achieve improved characteristics [15]. A great deal of work was made to improve the electrochemical characteristics of Li-rich materials,

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including elemental doping, special surface treatment, particle nanosizing, and surface coating [16–21]. Nano sizing particles can succeed in enhancing rates, but will increase the material's special surface and lead to serious side effects with a solid-electrolyte interface. Element doping works to improve the structural stability and prevent oxygen escape of the cathode layer. However, the electrode cannot avoid electrolyte corrosion [22, 23]. The coating of the surface area is one of the effective methods for improving the electrochemical efficiencies of cathode materials. Such a layer of coating will serve as a layer of passivation that prevents direct interaction of the active material with electrolyte and suppresses metal ion dissolution in this manner. Many coating materials, such as LiFePO_4 , Li_2ZrO_3 , AlF_3 , Al_2O_3 and CoPO_4 can protect Li-rich materials and improve stability of the surface structures against electrolyte corrosion [22–25]. This could shield the cathode materials from HF attack, with the advantage of high thermal stability and higher electronic conductivity [26–28]. The so-called sol-gel process is mostly used for obtaining these coatings. This technique cannot however create a homogeneous and full layer of coating on the host materials. Thus, better and less secure areas are available against side reactions with the electrolyte [29]. The structured surface is benefiting from electrochemistry, electrocatalysis, and energy transfer among many fields [30–32]. On the other hand, adatoms on metal surfaces resemble cheap and reversible surface alloys. Previous adatom electrode experiments reveal that certain metal adatom electrodes have more catalytic characteristics than their respective plain metallic electrodes. Many of the adatom-substrates investigated demonstrated improved electrocatalytic properties for many important electrochemical processes [33–35]. Additionally, the preparation of adatoms surfaces is a straightforward operation involving exposure of metallic substrates in potential conditions or open circuits to solutions containing adatoms ions [36, 37]. Thus, ultra-thin layers that would be an ideal layer can be accomplished with this method. In comparison, other techniques are often rugged, unfinished or dense. This activates and simultaneously coats the surface [38]. The current study aims to modify the surface of a Li-battery cathode with a monolayer of titanium and rhenium adatoms using a square wave potential regime. Cyclic voltammetry (CV), Chronoamperometric (CA), and Energy-dispersive X-ray spectroscopy (EDX) were used to investigate the electrochemical and electrocatalytic properties of the obtained modification of Li-battery cathode materials.

2. EXPERIMENTAL

2.1 INSTRUMENTS, MATERIALS AND CELLS

A potentiostat (273A) interfaced to a computer have Echem® software was used for electronic control and data acquisition. A conventional electrochemical cell equipped with an inlet system for purging nitrogen to remove traces of oxygen in solution. Auxiliary electrodes were made from a lithium surface (Johnson Matthy, 99.99 %purity). The reference electrode had a composition of $\text{Ag}/\text{AgCl}/[\text{Cl}^-]=1.0\text{ M}$. The working electrode lithium-ion surface had a diameter of 1.0 mm (99.95% pure, Good fellow METALS). The National Gas Company supplied the purging nitrogen. Adatom was made from Titanium oxide (TiO_2 , LABORATORY CHEMICALS) and rhenium: (rhenium powder, 99.95%, England). Both reagents used were approved analytical reagent (A.R) chemicals with the highest purity. All solutions were made from the above-mentioned reagents dissolved in triply distilled water. EDX investigated the morphology of the surface coating material and the extent to which the coated particles had been covered.

2.2 PROCEDURES

The Li-battery cathode surface was modified by titanium and rhenium adatoms at the open circuit in a 0.5 M H_2SO_4 solution containing $1.0 \times 10^{-3}\text{ M}$ of Ti and Re ions by application of a square wave potential regime (concentration of the solution was $1.0 \times 10^{-4}\text{ M}$ while the deposition time was varied between 90, 120, and 180 s [39]. CV, CA and EDX were used to characterize and investigate the electrochemistry properties of the modified surfaces.

3. RESULTS AND DISCUSSION

3.1 LITHIUM BATTERY SURFACE MODIFIED BY TITANIUM AND RHENIUM

Figure 1 shows cyclical voltammograms with lithium-ion battery surface and Ti/Re adatoms modified of the surface obtained in 0.5 M H_2SO_4 over the potential range from 2.0 to 5.0 V at $100\text{ mV}\cdot\text{s}^{-1}$.

As shown in Figure 1-A, the peak located at 0.8V was assigned to the oxidation of the lithium surface. The lithium surface modified by titanium adatom improves the electrochemical properties of the lithium surface maximum oxidation current. Shows in Figure 1-B, the voltammograms of the lithium surface substrate were modified by adsorbed rhenium adatoms. The peak centered at 0.8V is indicative of the presence of rhenium adatom at the surface. Titanium and rhenium were irreversibly adsorbed in the oxidation region centered at 0.8V on Lithium-ion surface at the open circuit from 10^{-3} M (Ti^{+3} / Re^{+3}) solution.

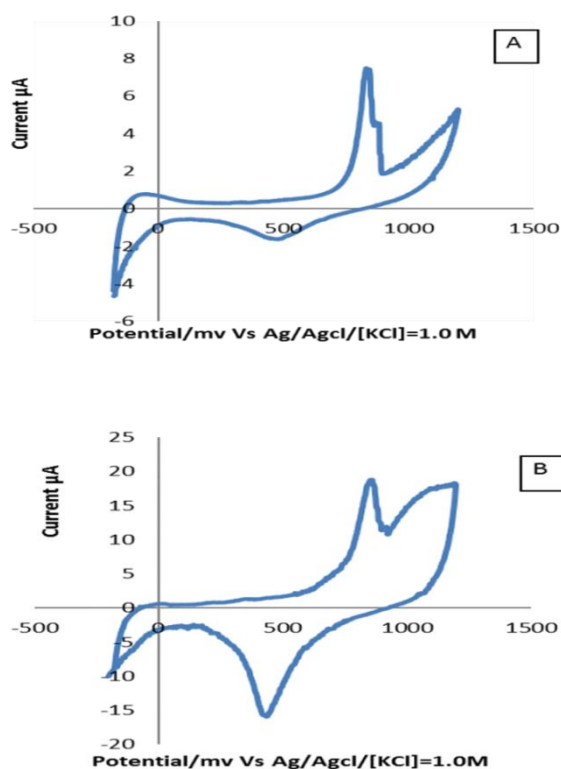


Figure 1. Cyclic voltammograms of Lithium-ion battery surface recorded at $100\text{mV}\cdot\text{s}^{-1}$, (A) Titanium-modified of the surface and (B) Rhenium-modified of the surface.

The apparent coverage of Ti/Re adatom (Θ) could be evaluated from the charge involved in the hydrogen desorption to the lithium-ion surface pure ($H_{\text{bare surface}}$) and the modified Ti/Re on the Lithium-ion battery surface ($H_{\text{modified surface}}$), by using the following relationship: [39].

$$\Theta = 1 - (H_{\text{modified surface}} / H_{\text{bare surface}}) \dots\dots\dots 1$$

The voltammograms in Figure 1 shows an area under the peak of the Ti-Li ion surface recorded at $100\text{mv}\cdot\text{s}^{-1}$ was $44\ \mu\text{C}$ and $120\ \mu\text{C}$ of Lithium-ion

battery, the coverage of Ti in the surface is 0.63. Re-Li recorded the area under the oxidation peak was calculated to be $37\ \mu\text{C}$ which coverage is 0.69. Additional proof of irreversible adsorption of Ti/Re to lithium surfaces. The EDX spectrum in Figure 2 shows two peaks for adatoms of Ti/Re adsorbed along in lithium-ion battery surface. The EDX spectrum revealed the existence of titanium atoms with an 11% atomic ratio compared to 89% for lithium-ion battery in (Figure 2-A) but in (Figure 2-B) atomic ratio of rhenium is 17% compared to 82% for lithium-ion battery nanoparticles. The EDX spectra for a sample prove the deposition of lithium-ion battery modified by adatoms of titanium/rhenium

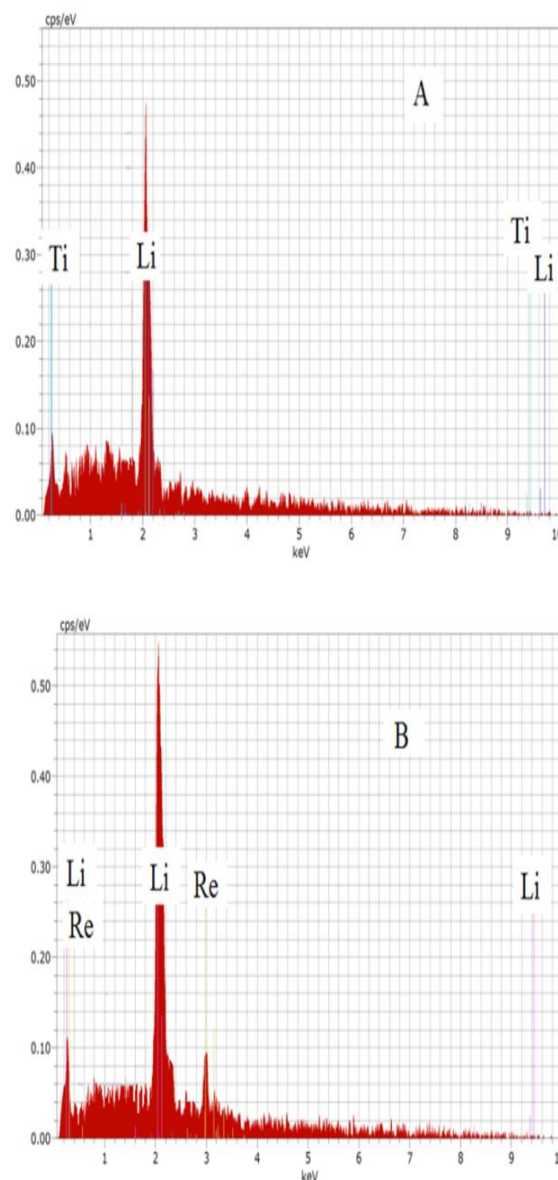


Figure 2. EDX spectrum of the modified Lithium ion battery A: Titanium and B: rhenium.

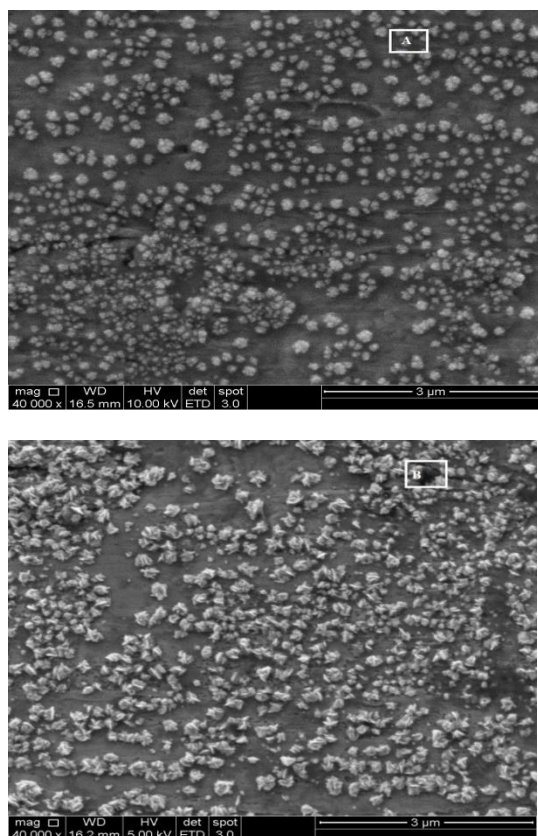


Figure 3. The SEM micrographs of the modified Lithium-ion battery A: Titanium and B: rhenium.

Figure 3 shows the SEM three-dimensional microstructures of the modified Lithium-ion battery Ti and Re by applying a square wave between -0.4 to 0.00 V at longer deposition times of 10 minutes and different frequencies, the SEM micrographs were analyzed in terms of the particle size and shape of the particles.

Cyclic voltammetry traced the stability of Ti/Re adatoms on lithium ion surfaces. As a test for the stability of the Ti/Re-adatom on the Lithium ion battery, the region was consistently below the time oxidation point. Figure 4 shows four voltammograms one recorded after one day while the second was recorded after two days, cyclic 3 after 3 days and four days. The voltammogram shows higher peak areas after one day than that obtained after two days, our impression is that the potential scan itself absorbs some of the Ti atoms at the surface. Repetitive cyclization of the electrode potential adsorbed Ti atoms from the surface and reduces the charge for voltammetric features in Figure 4-A. The voltammogram shows a decrease in peaks of Re atoms after two days and the same result after a change of potential range scan. The decrease in peak current is due to the desorption of Re-atoms in the

oxidation area from the surface when scanned in Figure 4-B.

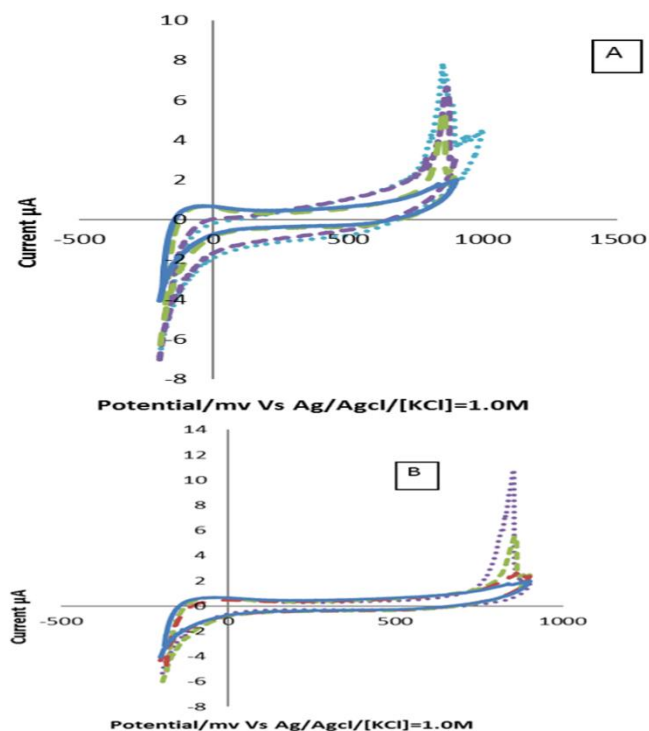


Figure 4. Cyclic voltammograms of Lithium ion battery modified by A: Titanium and B: Rhenium (.....) cycle one (---) cycle two (- -) cycle three (- . -.) Cycle four.

These findings can be explained by two competing pathways for modification lithium-ion battery substrate, particle growth and nucleation [40]. The findings of this study provide clear evidence that particle growth is the dominant process of metal modification at high concentrations and long deposition periods. Nucleation occurs selectively at high-energy surface sites during the early stages of deposition. The surface sites with high energy include surface faults, vacuums, grain borders or adatoms. Nucleation prevails with a low ion concentration due to the availability of high-energy sites. High surface energy sites, however, are relatively limited at high depositary ion concentrations, hence prevailing particle growth. Similarly, it can be clarified the impact of the deposition period. Due to the presence of high-energy surface sites, at short deposition periods, the nuclear process prevails while particle growth predominates as most of these sites are consumed at long deposition times. This would necessarily improve the formation in long deposition periods of 3-dimensional structures [41].

3.2 COULOMBIC EFFICIENCY OF TI, RE-ADATOM MODIFIED LITHIUM ION SURFACE

Figure 5 shows the cyclic voltammograms for the lithium-ion surface with Ti and Re atoms, recorded at different scan rates. The relationship between the coulombic efficiency and the peaks current indicates that these atoms are at the surface rather than in the bulk solution and irreversibly adsorb Ti and Re in Lithium-ion battery surface. The voltammograms show different values for a peak current of Ti and Re atoms at 0.8 mV as the scan rate change.

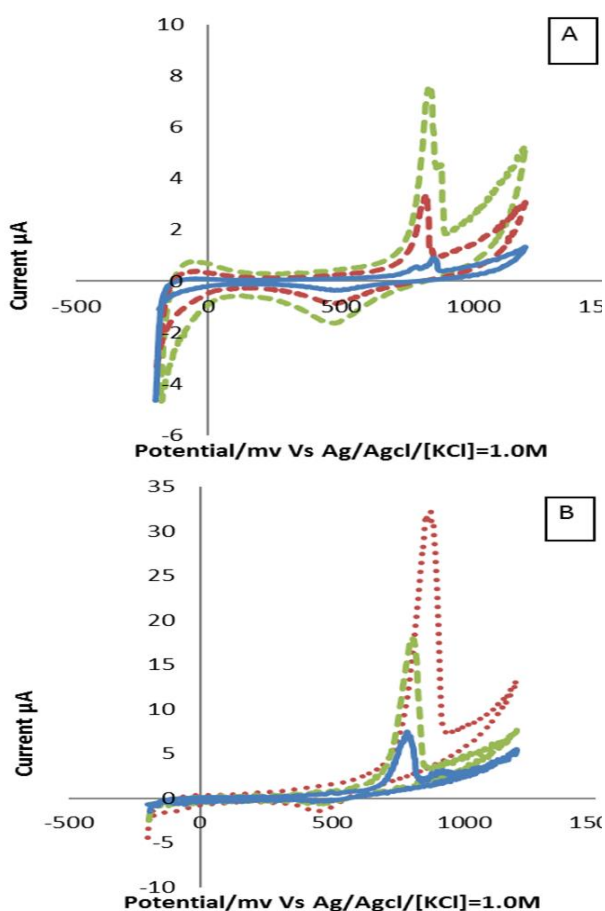


Figure 5. (A) Cyclic voltammograms of Titanium coating Lithium-ion surface (....) scan rate 100 mV/s (-----) scan rate 50 mV/s (—) scan rate 25 mV/s. (B) Rhenium of coating Lithium-ion surface

The experimentally determined relationship for the peak current assigned to Ti and Re atoms oxidation were, $i_p \propto v$ (R^2 of Ti = 0.998 and R^2 of Re = 0.991) where i_p is the adatoms oxidation peak current and v is the scan rate. This result shows that these pinnacles are related to a surface process instead of a bulk process [42]. The degradation of the

electrode/electrolyte boundary and any changes and potential accumulations of inactive material may be attributed to this behaviour.

The cathodic and anodic peaks display practically the same voltage profile for all surface substances after three cycles as shown in Figure 5. Furthermore, between the three cycles for Li-ion modified with space group, the average coulombic efficiency (CE) (Figure 4) is the largest (~87.4%). This sample seems to have a greater electronic conductivity, thereby improving its reversibility and reducing the voltage difference between the discharge and charge curves. Enhanced coulombic efficiency means that the sum of irreversible parasite reactions in the cell is reduced. The cover material used in this work has therefore a favourable effect and is useful for preventing the electrolytic from breaking down on the particle surface and can help to raise the ratio of "beneficial" load to parasite load (decomposition of electrolyte, etc.) [43].

3.3 ELECTROCHEMISTRY OF TI AND RE-MODIFIED LITHIUM ION SURFACE

Cyclic voltammograms for electrochemistry properties on the Lithium-ion (solid line) and Lithium-ion modified by Ti and Re (dashed line) were obtained in the potential range between -0.20 V to 1.2 V, at sweep rates of 100 mV/s are shown in Figure 6. This figure shows a large increase in the charge attributed to electrochemistry at the $Ti_{ad}-Li_{sub}$ and $Re_{ad}-Li_{sub}$ electrode compared to that observed for the Li-ion electrode. Peak resolution and the higher charge are evidence for the enhancement of the electrochemistry properties of the Ti and Re adatom-modified Li surface. The density of mass current (the onset potential) about 0.10 V, which was more negative than the Li-ion (0.30 V), was initially increased for the Ti-modified Li-ion surface. The greater the negative impact potential, the more positive the Ti-modified Li-surface was, the less their over-potential, was to encourage the coulombic efficiency. The current value observed during a positive scan has moved gradually from 0.40 to 0.80V for the Ti-modified Li-surface electrode to a more positive potential compared to the peak of the Li-surface. Titanium-modified Li-ion improved coulombic performance. In Re-modified Li-ion, the peak current was 400 μA , three times the current found in Li-ion under equivalent working conditions. Current peaks of approximately 0.90 V at Li-ion. The overall peak current of the Li-ion and Ti/Re-modified Li-ion increased several times compared to the response of the bulk Li surface. This improvement supported the assumption that the structure of the Li-ion had enough electrochemistry capacity. The bi-functional mechanism was used for this purpose. This result provides strong evidence for enhancement of

the electrochemistry properties by the presence of adatoms at the Lithium ion battery [44].

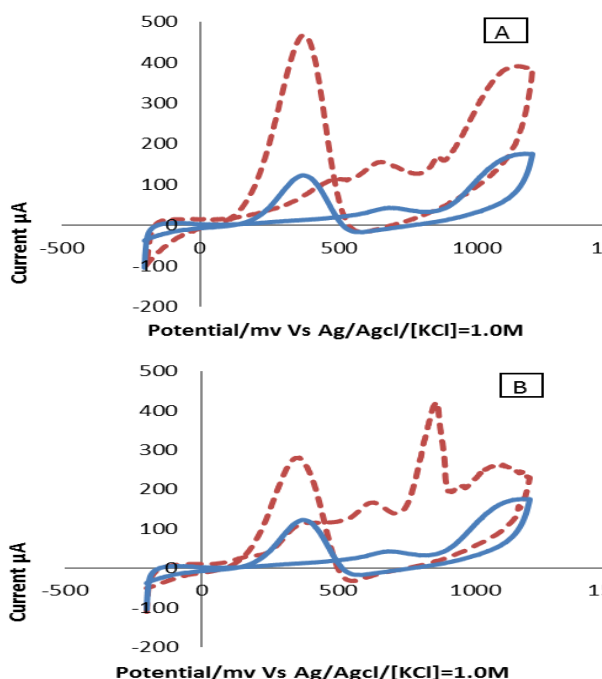


Figure 6. Coulombic efficiency of Li-ion (solid line) and Li-ion modified by (A) Titanium and (B) Rhenium (dashed line) at a sweep rate of $100 \text{ mV}\cdot\text{s}^{-1}$.

The cyclic voltammograms and coulombic efficiency for $\text{Li}_{\text{surface-Ti ad}}$ and Read show a huge increase in the charge attributed to the charge observed for the Li-ion surface (in Figure 7). Three peaks centred at 0.40 V, 0.65 V and 0.82 V. Important improvement of anodic current is apparent when Li-ion are covered by Ti^{+3} and Re^{+3} . Three current peaks on the positive-going scan were observed at 0.32V, 0.79V and 0.90V. Manifest the voltammetric features for electrochemistry. All of these features indicate an enhancement of electrochemistry properties by Ti and Re adatoms on the Li-ion electrode.

The represents a potential advantage of 300 mV in the electrode of the adatoms, which also shows higher coulombic properties for the modified Li-ion electrode. Enhancement factor in the anodic current appears in the potential region of 0.0 V - 1.0 V depending on Ti, Re coverage: roughly, 0.63 of Ti and Re around 0.69 on the Li-ion surface, the onset potential on the Ti, Re-modified Li-ion surface is more positive than that on the Li-ion surface pure. The changes in volumetric behavior can be attributed to the modified surface by the monolayer of adatoms.

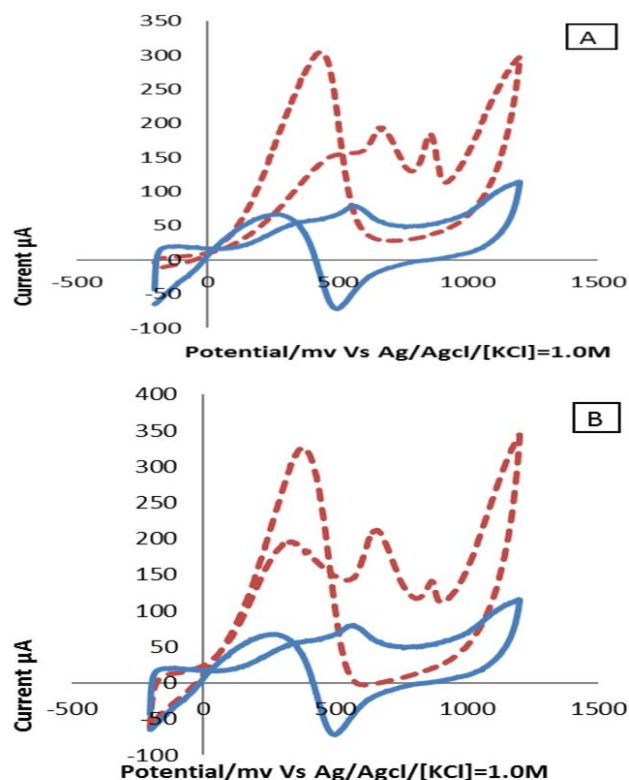


Figure 7. Cyclic voltammograms of Li-ion (solid line) and Li-ion modified by (A) Titanium and (B) Rhenium (dashed line) at a sweep rate of $100 \text{ mV}\cdot\text{s}^{-1}$.

As for battery charge, time in electronic devices depends on the rate of Li-ion removal and injection in the cathode material, rate performance is a significant property of the battery to be measured [45]. This small variation in a form in the electrochemical trials is well consistent with the variations found by cyclic voltammetry in transition metal orders. The rates of the Li-ion cathode were thus strengthened by the coating substance [45]. The electrical efficiency at high rates is known to be partly dependent on the electrical conductivity of the active ingredient. The high conductivity of the coated sheet can also be due to the increased electrochemical efficiency of Ti/Re on the surface at high rates. This effect implies that the layer coating of the adatoms does not prevent the intercalation and decomposition of the lithium ions, but enables the transfer of Li^+ to the host structure. The interfacial resistance between the cathode and the electrolyte can be decreased as a result. According to several literary studies, some adatoms materials showed very good properties as a surface protection layer of cathode content, however, the rates for the studied Li-ion battery were not enhanced [46, 47].

Apart from a protective layer, the covering layer of the adatom material considered here can also increase electrochemical efficiency at high cycling speeds. Ability differential from potential (dQ/dU compared with V) (extracted from capacity retention test). Figure 8 shows a comparison of differential power versus future curves derived from rates and retention tests. Results from the rate-power test show that the Ti^{+3}/Re^{+3} coated material is more reversible and stable (Figure 8).

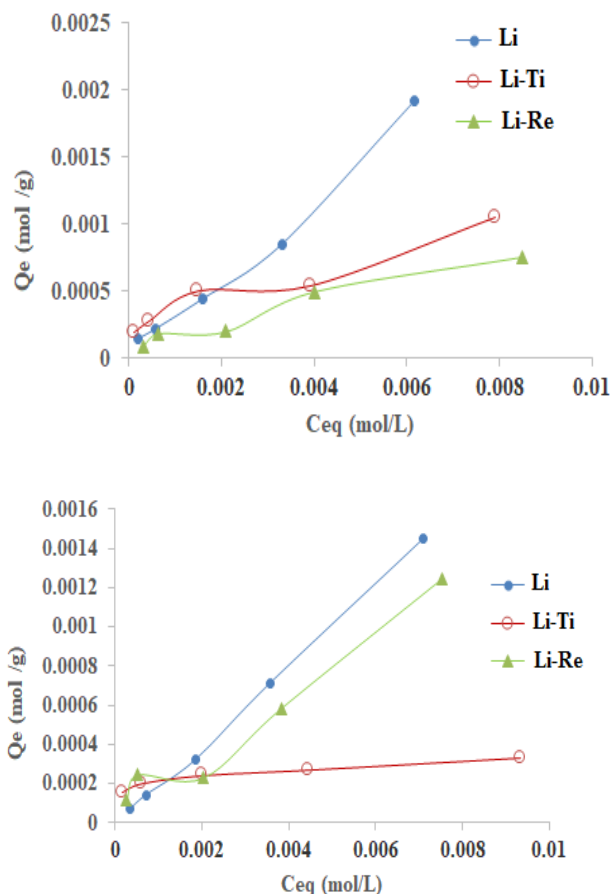


Figure 8. dQ/dU of Li-ion and Li-ion modified by Titanium and Rhenium.

The retention test differential ability vs. potential curves indicate comparable advancements but greatly reduced polarization and deterioration, especially for the bare material.

3.4 CHRONOAMPEROMETRIC OF TI AND RE-MODIFIED LITHIUM-ION SURFACE

Chronoamperometric technology is efficient in the evaluation, on both the Re/Ti-modified Lithium ion and the Lithium ion pure surface of the electrochemistry activity and current density–time responses at its fixed potential from 0.4 up to 0.8 V.

Figure 9-A/B of Re/Ti-modified Lithium surface. Equation 2 determined the forward reaction for formic acid oxidation[48]:

$$K_f = I_k/nFAC^* \dots\dots\dots 2$$

Where (K_f : the forward kinetically controlled reaction, n : no. of electrons transferred, C^* : concentration, F : Faraday constant, and A : the surface area). The titanium modified Lithium ion battery show the highest current density of steady-state (65 mA cm^{-2}) and Rhenium-modified Lithium ion battery shows a current density of about 10 mA cm^{-2} after 0.3 seconds. Such data indicates the greater operation and stability of the Ti, and Re-modified Lithium-ion compared to Lithium-ion. Such findings can also be clarified by the alteration of metal that can improve electrochemistry efficiency and the bifunctional mechanism or mechanism of ligand.

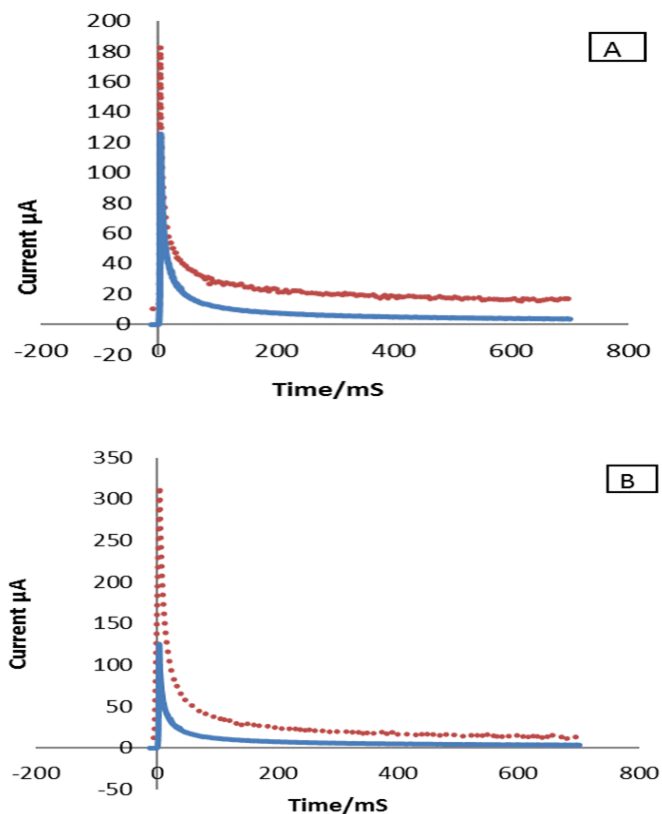


Figure 9. Chronoamperometric of Li-ion (solid line) and Li-ion modified by (A) Titanium and (B) Rhenium (dashed line).

4. CONCLUSIONS

The present work has shown that the square wave potential regime is applicable when the titanium and rhenium adatoms modify the lithium-ion battery. Synergistic electrochemistry effects of under potentially irreversibly adsorbed atoms on the Lithium ion battery surface has been proved by investigation of electrochemistry properties. An enhanced electrochemistry property was observed for the Ti/Re-modified Lithium ion battery. This shows that the modified lithium-ion imparted by adatoms has improved electrochemical properties. The Ti^{+3}/Re^{+3} modified Lithium-ion battery was improved in the oxidation charge for both test molecules. This shows that the modified electrode imparted by adatoms has enhanced electrochemistry properties. In stable state regions, the rhenium modified higher than titanium modified Li-ion, the titanium-modified Li-ion have the maximum current density in stationary (65 mA cm^{-2}), the rhenium-modified Li-ion (101 mA cm^{-2}), and the current oxidizing density inside Li-ion has been given after 0.3 seconds only 10 mA cm^{-2} of electrical density. The area of Ti-Li-ion and Re-Li-ion was $44 \mu\text{C}$ and $37 \mu\text{C}$, with a surface range of Ti and Re being 0.63 and 0.69.

COMPETING INTERESTS

Authors have declared that no competing interests exist. The products used for this research are commonly and predominantly use products in our area of research and country. There is absolutely no conflict of interest between the authors and producers of the products because we do not intend to use these products as an avenue for any litigation but for the advancement of knowledge. In addition, the research was not funded by the producing company rather it was funded by personal efforts of the authors.

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