

Competitive Performance by a New Non-Transitions Metal Doped Cathodic Material $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.09}\text{Mg}_{0.01}\text{O}_2$ for Lithium-Ion Batteries

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Graphical abstract

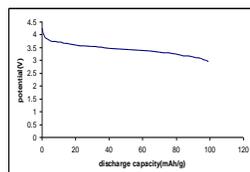


Figure 1. Potential versus discharge capacity curve of $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.09}\text{Mg}_{0.01}\text{O}_2$.

Abstract

Lithium cobalt nickel oxide cathodes had been doped with various metals in recent years to obtain a competitive high performance cathode material for lithium-ion batteries. Cathodes doped with Al and Mg were synthesized by solid-state reaction method. Structural investigation of this material was done using XRD. Galvanostatic charge/discharge and cyclic voltammetry were studied in order to outline the electrical performance of $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.09}\text{Mg}_{0.01}\text{O}_2$, $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.06}\text{Mg}_{0.04}\text{O}_2$ and $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.03}\text{Mg}_{0.07}\text{O}_2$ materials in lithium-ion batteries. Electrical impedance was done on all the materials and it gave decreasing conductivities with increasing temperature. The activation energies had negative values with increased magnesium content of the material. Larger conductivity variation with temperature was seen in the material with the higher magnesium content. Voltammographs of these materials showed good oxidation and reduction loops. Charge/discharge curve for $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.09}\text{Mg}_{0.01}\text{O}_2$ material showed about 96 mAh/g of discharge capacity for the first cycle.

Keywords: Cathode; lithium-ion batteries; aluminium; magnesium

Abstrak

Litium kobalt nikel oksida telah didopkan dengan pelbagai logam pada beberapa tahun ini untuk mendapatkan nilai kompetitif prestasi yang tinggi bagi bahan katod untuk litium-ion bateri. Katod didopkan dengan Al dan Mg disintesis dengan kaedah tindak balas keadaan pepejal. Kajian struktur bahan ini telah dilakukan dengan menggunakan XRD. Galvanostatik cas/nyahcas dan voltametri berkitar dikaji untuk menentukan prestasi elektrik bagi bahan $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.09}\text{Mg}_{0.01}\text{O}_2$, $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.06}\text{Mg}_{0.04}\text{O}_2$ and $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.03}\text{Mg}_{0.07}\text{O}_2$ di dalam bateri litium-ion. Impedans elektrik telah dilakukan ke atas semua bahan-bahan dan hasil menunjukkan konduktivitiannya bekurangan dengan peningkatan suhu. Keaktifan tenaga adalah menunjukkan nilai negatif dengan peningkatan kandungan magnesium dalam bahan serta meningkatkan kadar konduktiviti. Voltamograf bagi kesemua bahan ini menunjukkan pengoksidaan dan penurunan yang baik. Lengkung cas/nyahcas bagi bahan $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.09}\text{Mg}_{0.01}\text{O}_2$ menunjukkan nilai kapasiti sebanyak 96 mAh/g untuk kitaran pertama.

Kata kunci: Katod; bateri litium-ion; aluminium; magnesium

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1.0 INTRODUCTION

Technological improvements in rechargeable lithium ion batteries have long been boosted by an ever-growing demand for high-end electronic devices and attractive candidate for automotive application due to their energy and power density [1], e.g., cellular phones, notebook computers and hybrid electronic vehicles. Currently commercial lithium ion batteries based on conventional transition metal oxide cathode materials, such as LiCoO_2 and spinel LiMn_2O_4 , have been seriously prevented from extensive practical applications due to the underlying structural drawbacks [2]. Lithium transition metal oxides are amongst the most studied materials for, possessing high operating voltage, good reversibility and high capacity cathode in lithium ion

batteries as secondary batteries [3, 4]. Intercalation and deintercalation are the processes that occur in the rechargeable electrode in a lithium battery where the insertion and de-insertion of Li-ions into the electrode is topotactic and therefore, the structure of the host material was conserved during reaction. The reversible characteristic of insertion materials can be used for both cathodes and anodes for advanced lithium batteries in which these mobile cations move between two insertion materials. LiNiO_2 has been considered as a promising positive electrode material for high energy rechargeable batteries [5-9]. LiNiO_2 has layered structure similar to LiCoO_2 and is cheaper than LiCoO_2 [4, 10, 11].

LiNiO_2 has higher capacity of about 190-200mAh/g compared to LiCoO_2 and LiMn_2O_4 [23]. However, LiNiO_2 has

high theoretical capacity, it is difficult to synthesis in its tendency of non-stoichiometric phase, is not easy to prepare on large scale, [11, 12] due to its lower thermodynamic stability compare LiCoO₂ and the presence of excess nickel on Li sites. These anti sites in LiNiO₂ strongly affect the electrochemical properties of batteries [4]. In the case of doped LiNiO₂, there are still some possible improvement. For example, LiNi_{0.8}Co_{0.2}O₂ is promising cathode material with high capacity and medium cost for lithium ion secondary cells [11]. Presently, some lithium nickel oxide derivatives are considered by the battery manufacturers to be very promising for application in 4 V lithium-ion batteries. With the aim of finding new positive electrode materials for lithium-ion batteries, many research and development effort has been devoted towards LiNiO₂ and LiNiO₂ substituted materials. Several substituted LiNi_{1-y}MyO₂ systems (M : Ga [13], Mg and Ti [14]) as well as the LiCo_{1-y}Mg_yO₂ system [15] were recently shown exhibit very good reversible cycling behavior [16].

The new LiCoNiO₂ system presented in this paper are doped by Al and Mg were synthesized by solid-state reaction method to prepare the cathode materials. With the expectation to obtain a cathode material with high capacity and good cycling behavior we choose to study LiCo_{0.7}Ni_{0.2}Al_xMg_{0.1-x}O₂. Structural characterization and electrochemical behavior of the cathode material were studied.

2.0 EXPERIMENTAL

Powders were synthesized by using solid state reaction method. Mixture of LiNO₃, Co(NO₃)₂, Ni(NO₃)₂, Mg(NO₃)₂ and Al(NO₃)₃ according to the material ratio for the samples were mixed into a porcelain crucible to synthesize LiCo_{0.7}Ni_{0.2}Al_{0.09}Mg_{0.01}O₂, LiCo_{0.7}Ni_{0.2}Al_{0.06}Mg_{0.04}O₂ and LiCo_{0.7}Ni_{0.2}Al_{0.03}Mg_{0.07}O₂ materials. The crucible then was put into a furnace and the mixture was annealed at 800°C for 18 hours. After heating the black colored mixtures were left to cool down for few hours until it reached room temperature. Materials in the crucible were then ground with a mortar and pestle several times until it turned into a fine powder.

2.1 Structural Characterization

Structural characterizations were performed on the material in order to determine the structure and the grain size of the material. X-Ray diffraction analysis (XRD) method was used to obtain the XRD patterns produced which were used to identify crystalline phase of the prepared materials. PHILIPS PW 1840 diffractometer instrument which was equipped with a diffracted beam curved graphite monochromator was used to perform the X-Ray diffraction. The scan data were collected in the 2θ range of 10° < 2θ < 180°.

2.2 Electrochemical Studies

Electrochemical study of the cathodes LiCo_{0.7}Ni_{0.2}Al_{0.09}Mg_{0.01}O₂, LiCo_{0.7}Ni_{0.2}Al_{0.06}Mg_{0.04}O₂ and LiCo_{0.7}Ni_{0.2}Al_{0.03}Mg_{0.07}O₂ were obtained by galvanostatic charge/discharge method using Arbin instrument. Cathode layers were prepared by using several chemicals. Suitable binding agents were mixed to produce a good layer for all the materials. After that a certain volume of acetone was poured into the mixture and was stirred until it was homogeneous. Then, this mixture was then heated with a temperature of 40°C for more than one hour. The homogenous mixture then was mixed together with the electrode material and activated carbon (super-p) in the blender and blended for several

minutes until it turned into a form of slurry. The composite electrode would be poured into a rig which was placed on a glass surface. Finally the compound was cast onto the glass surface.

After it was dried, the material would be in a form of sheet. The material would be attached to a current collector aluminum. The material would be cut into square shapes and then used as a cathode in the cell. Meso carbon micro beads (MCMB) were used as anode which was prepared using same procedure and a LiPF₆ salt in equal volume of ethylene carbonate (EC) and propylene carbonate (PC) was used as an electrolyte.

Galvanostatic charge/discharge was performed to the cells containing LiCo_{0.7}Ni_{0.2}Al_{0.09}Mg_{0.01}O₂, LiCo_{0.7}Ni_{0.2}Al_{0.06}Mg_{0.04}O₂ and LiCo_{0.7}Ni_{0.2}Al_{0.03}Mg_{0.07}O₂ as the cathodes. Several charge and discharge currents were used to charge and discharge the cell and cell potential was monitored in a range of 3.0 – 4.2V. Cyclic voltammetry was performed using AutoLab potentiostat /galvanostat instrument using lithium metal as the counter and the reference electrode in cell assembled in a dry box under argon atmosphere.

3.0 RESULT AND DISCUSSION

X-Ray powder diffraction (XRD), is an instrumental technique that is used to identify minerals, as well as other crystalline materials. XRD provides the researcher with a fast and reliable tool for routine material identification. XRD patterns of the materials synthesized at 800°C for 18 hours shown in Figure 1-3. As can be seen the pattern which was synthesized with 800°C for 18 hours have a similar pattern to hexagonal LiNiO₂ commercialized cathode material [17]. As can be seen in the patterns, the width of the peaks of the materials had increased when the content of Mg was increased.

The ratio between (104) peak and (003) peak in the patterns confirms the crystallinity of the material. It has been reported that the intensity ratio of (104) and (003) peaks is a key parameter that indicates the degree of displacement of nickel and lithium ions. Ohzuku *et al.* [18] reported that the intensity ratio of (104) and (003) peaks should be more than 1.0 to enhance the electrode performance of LiNiO₂. The I₍₀₀₃₎/I₍₁₀₄₎ ratio of LiCo_{0.7}Ni_{0.2}Al_{0.09}Mg_{0.01}O₂ material is about 2.0. The diffracted lines are sharp which indicates that, this cathode is a well crystalline material. This evidence strongly supports the fact that LiCo_{0.7}Ni_{0.2}Al_{0.09}Mg_{0.01}O₂ is a potentially good cathode. The I₍₀₀₃₎/I₍₁₀₄₎ ratio of LiCo_{0.7}Ni_{0.2}Al_{0.06}Mg_{0.04}O₂ and LiCo_{0.7}Ni_{0.2}Al_{0.03}Mg_{0.07}O₂ are 0.73 and 0.88.

Nitta *et al.* [19] estimated the electrochemical characterization of LiNiO₂ cathode material on the basis of the intensity ratio of (006) and (101) peaks. They proposed that the intensity ratio should be lower than 1.0 and high discharge can be obtained at the intensity ratios of 0.34 - 0.4. The intensity ratio of (006) and (101) peaks of LiCo_{0.7}Ni_{0.2}Al_{0.09}Mg_{0.01}O₂ material is 0.33. The intensity ratio of (101) and (006) for LiCo_{0.7}Ni_{0.2}Al_{0.06}Mg_{0.04}O₂ and LiCo_{0.7}Ni_{0.2}Al_{0.03}Mg_{0.07}O₂ is 0.41 and 0.50 respectively. The width of all the peaks for LiCo_{0.7}Ni_{0.2}Al_{0.06}Mg_{0.04}O₂ and LiCo_{0.7}Ni_{0.2}Al_{0.03}Mg_{0.07}O₂ materials are bigger compared LiCo_{0.7}Ni_{0.2}Al_{0.09}Mg_{0.01}O₂ material. It shows that when the content of Mg is higher, the width of the XRD peak is bigger thus the electrical performance will decrease as reported in the journals previously. From these XRD pattern of the all material showed Mg has an effect on arrangement of the atoms in the structure when it is used in higher quantity compared to Al. It further proves that higher Al content gives a better discharge capacity.

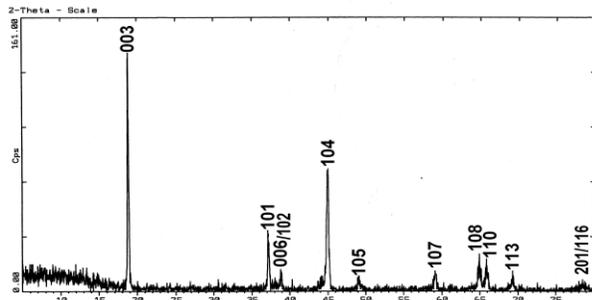


Figure 1 X-ray diffractogram of $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.09}\text{Mg}_{0.01}\text{O}_2$ material

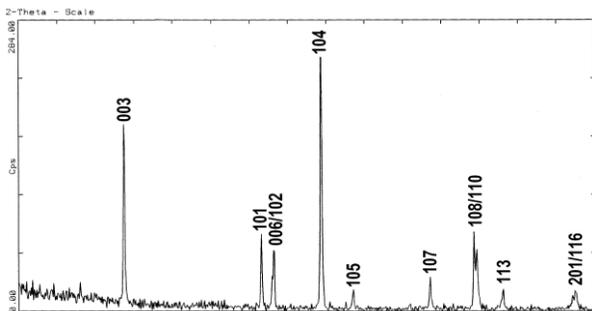


Figure 2 X-Ray diffractogram of $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.06}\text{Mg}_{0.04}\text{O}_2$ material

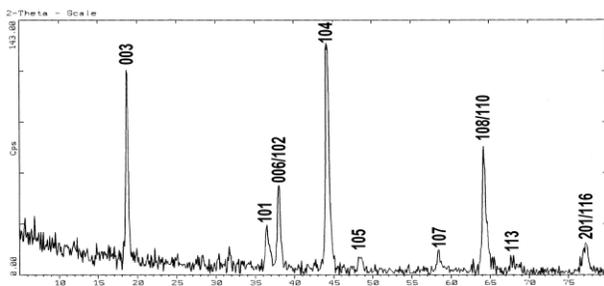


Figure 3 X-Ray diffractogram of $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.03}\text{Mg}_{0.07}\text{O}_2$ material

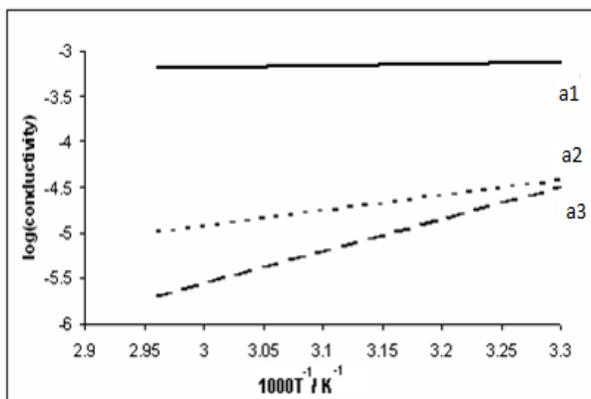


Figure 4 Arrhenius plots of $\log k$ vs. $1000T^{-1} / K^{-1}$ a1 is $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.09}\text{Mg}_{0.01}\text{O}_2$, a2 is $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.06}\text{Mg}_{0.04}\text{O}_2$ and a3 is $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.03}\text{Mg}_{0.07}\text{O}_2$

Figure 4 showed Arrhenius plot of $\log k / \text{S cm}^{-1}$ vs $10^3 T^{-1} / K^{-1}$ for all three cathode materials. All three materials have negative activation energy. The activation energy which can be

estimated from the Arrhenius equation, $\log k = \log A - Ea/(2.303RT)$, gave $-0.25 \text{ kJ mol}^{-1}$, -1.5 kJ mol^{-1} and -4.0 kJ mol^{-1} for $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.09}\text{Mg}_{0.01}\text{O}_2$, $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.06}\text{Mg}_{0.04}\text{O}_2$ and $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.03}\text{Mg}_{0.07}\text{O}_2$ respectively.

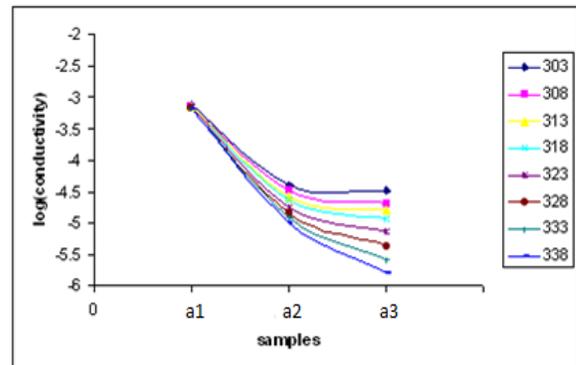


Figure 5 Log (conductivity) variations for all three materials at different temperatures

Figure 5 shows the log Conductivity for all three materials for various temperatures. It can be seen that the conductivity variation with temperature was the highest for the material with the highest content of magnesium $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.03}\text{Mg}_{0.07}\text{O}_2$. Researchers had reported negative activation energy with LiFePO_4 cathode material mixed with graphite [20], and on the other hand positive activation energy was also reported by Luo S. 2007 with the LiFePO_4 with graphite material [21].

The effect of doping non-transition metals onto lithium transition metal oxide material is still not clear. AC Impedance on $\text{LiNi}_{0.8}\text{Co}_{0.2}\text{O}_2$ and the Al doped $\text{LiNi}_{0.8}\text{Co}_{0.15}\text{Al}_{0.05}\text{O}_2$, gave higher resistance for the $\text{LiNi}_{0.8}\text{Co}_{0.2}\text{O}_2$ than the $\text{LiNi}_{0.8}\text{Co}_{0.15}\text{Al}_{0.05}\text{O}_2$ at 50°C [14].

Others reported increasing resistance with the doped materials. A silicon doped LiCoO_2 , $\text{LiCo}_{0.99}\text{Si}_{0.01}\text{O}_2$ gave higher resistance than LiCoO_2 at lower cycling numbers, but the effect reversed at higher cycling numbers [22].

Figure 6 is showing the cyclic voltammograms of $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.09}\text{Mg}_{0.01}\text{O}_2$. Cyclic voltammogram of the material shows a redox pair of peaks at around 5.2 V or oxidation and 5.0 V vs. Li^+ / Li for reduction. Many peaks at the voltammogram shows that more than electrons are intercalating and de-intercalating at different potentials. The voltammogram in Figure 7 for $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.06}\text{Mg}_{0.04}\text{O}_2$ material showing loops when scanned at scan rate 0.1 mVs^{-1} . The oxidation loop is at 4.2 V and the reduction loop is at the 2.5 V. Voltammogram for $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.03}\text{Mg}_{0.07}\text{O}_2$ material for the scan rate of 0.1 mVs^{-1} shows oxidation and reduction curves (Figure 8) and also only oxidation loop at 4.2 V.

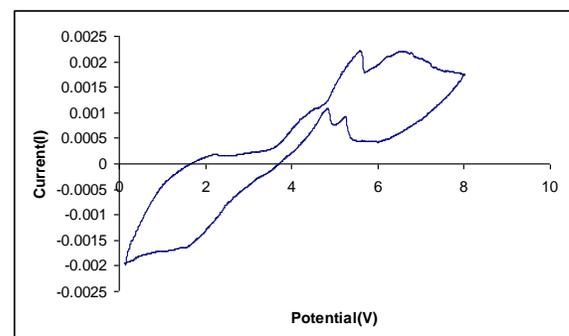


Figure 6 Cyclic voltammogram I/V of $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.09}\text{Mg}_{0.01}\text{O}_2$

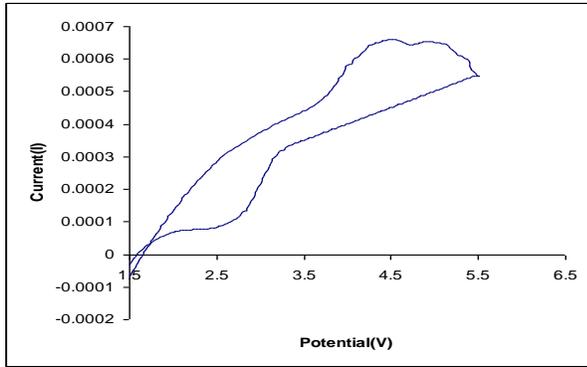


Figure 7 Cyclic voltammogram I/V of $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.06}\text{Mg}_{0.04}\text{O}_2$

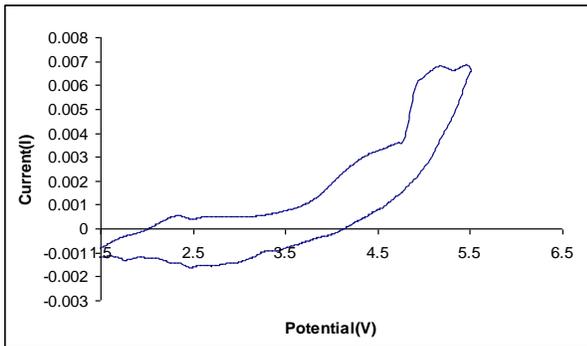


Figure 8 Cyclic voltammogram I/V of $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.03}\text{Mg}_{0.07}\text{O}_2$

$\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.09}\text{Mg}_{0.01}\text{O}_2$, $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.06}\text{Mg}_{0.04}\text{O}_2$ and $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.03}\text{Mg}_{0.07}\text{O}_2$ were used as cathodes with meso carbon micro beads (MCMB) as an anode and LiPF_6 as salt with the electrolyte EC. PC in a cell to carry out the electrochemical studies. Galvanostatic charge/discharge was done to the material which exhibits a good performance. The potential versus discharge capacity shown in Figure 9 was done with constant currents of 40mA. As can be seen the material produced a discharge capacity of about 96 mAh/g for $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.09}\text{Mg}_{0.01}\text{O}_2$ material.

$\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.06}\text{Mg}_{0.04}\text{O}_2$ has a higher percentage of Mg and Al is reduced. This cathode exhibit a very low discharge capacity compares to $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.09}\text{Mg}_{0.01}\text{O}_2$ material as can be seen in Figure 10. It produced a discharge capacity about 42mAh/g for the first discharge. The third cathode $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.03}\text{Mg}_{0.07}\text{O}_2$ with the highest Mg content exhibited even lower discharge capacity. Galvanostatic discharge curve shown in Figure 11 appears to have 30 mAh/g as the highest discharge capacity.

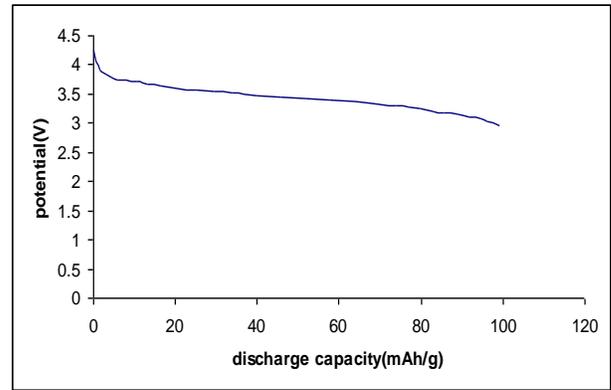


Figure 9 Potential versus discharge capacity curve of $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.09}\text{Mg}_{0.01}\text{O}_2$

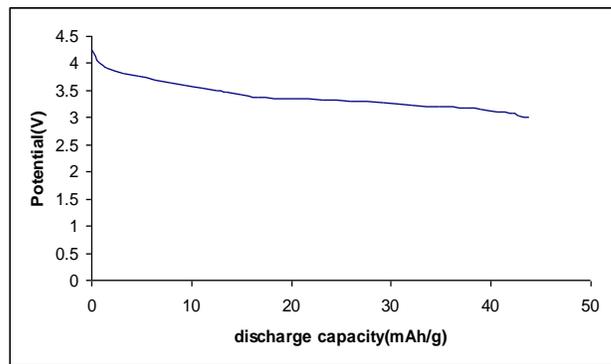


Figure 10 Potential versus discharge capacity curve of $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.06}\text{Mg}_{0.04}\text{O}_2$

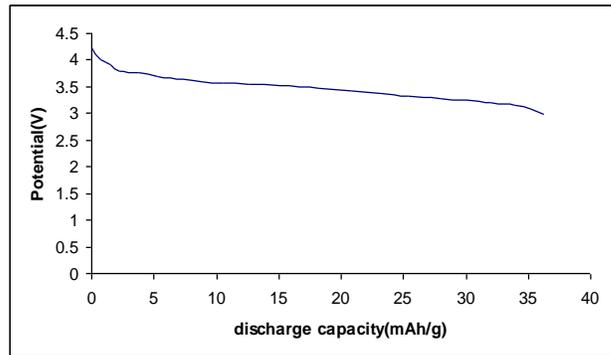


Figure 11 Potential versus discharge capacity curve of $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.03}\text{Mg}_{0.07}\text{O}_2$

4.0 CONCLUSION

Aluminium and magnesium were successfully substituted for nickel in lithium cobalt nickel oxide. The structural characteristic of $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.09}\text{Mg}_{0.01}\text{O}_2$, $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.06}\text{Mg}_{0.04}\text{O}_2$ and $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.03}\text{Mg}_{0.07}\text{O}_2$ have been investigated and shown by XRD to be isostructural with hexagonal LiCoNiO_2 . Electrochemical studies of $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.09}\text{Mg}_{0.01}\text{O}_2$ showed that the material produced over 96 mAh/g of discharge capacity. The above results, based on structural and electrochemical data, suggest that intercalation and de-intercalation from the compound proceed reversibly and without a small drop in discharge

capacity. $\text{LiCo}_{0.7}\text{Ni}_{0.2}\text{Al}_{0.09}\text{Mg}_{0.01}\text{O}_2$ was introduced as a promising cathode electrode for Li-ion battery.

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