

## Electrochemical behaviour of $\text{LiMn}_2\text{O}_4$ and $\text{LiCoO}_2$ in water electrolyte

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Li-Ion Batteries can afford quite large energy densities based on organic electrolytes with highly toxic and flammable solvents and its preparation is complicated and expensive, partly because the electrolyte is moisture and air sensitive. Utilization of aqueous electrolytes in rechargeable lithium batteries will increase their safety, economical, and environmental issues. The electrochemical properties of  $\text{LiMn}_2\text{O}_4$  and  $\text{LiCoO}_2$  as positive electrode materials for Li-ion battery with water electrolytes against  $\text{LiV}_3\text{O}_8$  as negative electrode were investigated. The used salts are 6M  $\text{LiNO}_3$  and 2M  $\text{Li}_2\text{SO}_4$  dissolved in distilled water.  $\text{LiMn}_2\text{O}_4$  was prepared by conventional solid state reaction heating stoichiometric mixtures of  $\text{LiNO}_3$  and  $\text{MnO}_2$  at temperature under  $650^\circ\text{C}$  for 24 h.  $\text{LiCoO}_2$  was obtained via sol-gel method with final thermal treatment at  $800^\circ\text{C}$  for 24 h. Lithium vanadate was prepared via solid state reaction. All materials are stable in the aqueous solution and intercalation/deintercalation of lithium ions occurs within the window of electrochemical stability of the water, which was determined using slow cycling voltammetry. Manganese spinel exhibit specific capacity of  $99\text{mAh.g}^{-1}$  in 6M  $\text{LiNO}_3$  and  $94\text{mAh.g}^{-1}$  in 2M  $\text{Li}_2\text{SO}_4$  water electrolyte, while its capacity in organic electrolyte is  $135\text{mAh.g}^{-1}$ . The obtained capacities from lithium cobaltate in water electrolytes were  $99\text{mAh.g}^{-1}$  and  $79\text{mAh.g}^{-1}$  respectively while that in organic electrolyte is  $145\text{mAh.g}^{-1}$ . The cycling behavior and specific capacity of these active compounds in electrochemical cell with vanadate as a negative active mass is not as stable as in organic electrolytes.

**Key words:** Manganese spinel, Lithium cobaltate, Water electrolyte, Li-ion Battery

### INTRODUCTION

The present commercially available lithium ion batteries contain flammable organic liquid electrolyte, which are major safety problem [1]. In recent years, spinel  $\text{LiMn}_2\text{O}_4$  has been intensively investigated as a promising candidate for positive materials for lithium-ion batteries (LIBs) due to its low cost, non-toxicity, environmental friendliness, easy preparation, excellent voltage profile, and operating safety characteristics [2,3]. The  $\text{LiCoO}_2$  has provided a good balance between high energy density and good cyclability. The specific capacity of  $\text{LiCoO}_2$  remains  $160\text{mAh.g}^{-1}$  or less when it is charged up to 4.2 V, which is 60% at of the theoretical capacity of  $274\text{mAh.g}^{-1}$  for one electron reaction. Elevating the charging voltage beyond 4.2 V can be one promising approach to utilize more lithium ions in the  $\text{LiCoO}_2$  and to increase the capacity and energy density of the lithium-ion batteries [4]. Aqueous Rechargeable Lithium Batteries (ARLB) can solve the safety problems in some extents, which can assemble the battery without gas protection and humidity control. The inorganic electrolyte is cheaper; the ion conductivity is two orders of magnitude higher than in the organic electrolyte. The capacity of  $\text{LiCoO}_2$  and  $\text{LiMn}_2\text{O}_4$  are well known and shown in our

pervious papers [5-7]. Especially, the ARLB is the green environmental protection battery indeed [8-13]. The manganese spinel  $\text{LiMn}_2\text{O}_4$  is cited in the literature as suitable for application as active material in li-ion battery with  $\text{LiNO}_3$  aqueous solution electrolyte [14-17]. The lithium cobaltate  $\text{LiCoO}_2$  show also reversible Li intercalation in  $\text{LiNO}_3$  water electrolyte [17]. In the literature  $\text{LiNO}_3$  and  $\text{Li}_2\text{SO}_4$  are cited as promising conducting salts for li-ion battery with water electrolyte [18, 19].

That preliminary information gives rise to the following questions:

-What is the better conducting salt for water electrolyte?

-What is the difference of capacity in water electrolyte and organic one?

-Is it possible to use  $\text{LiMn}_2\text{O}_4$  as a negative active material?

The aim of the present work is to study the impact of synthesis methods, physicochemical characteristics, and electrochemical behaviour of  $\text{LiMn}_2\text{O}_4$  and  $\text{LiCoO}_2$  in  $\text{LiNO}_3$  and  $\text{Li}_2\text{SO}_4$  water electrolyte.

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## EXPERIMENTAL

### Preparation of active compounds, electrodes and electrolyte

Samples studied of  $\text{LiMn}_2\text{O}_4$  were prepared by heating of stoichiometric mixture of  $\text{MnO}_2$  and  $\text{LiNO}_3$  in a nickel crucible for 12 to 48 h in the temperature range of 450-850 °C. The Li salt used is  $\text{LiNO}_3$ , commercial product of Fluka, the manganese dioxide is CMD Faradizer M supplied by Sedema. The precursors were mixed, dried at 80°C for 12 h and milled. After that, the obtained mixture was preheated at 450°C for 12h and then heated finally on 650°C for 24h. The  $\text{LiCoO}_2$  was prepared by sol-gel method by stoichiometric mixture of  $\text{Co}(\text{NO}_3)_2$  and  $\text{LiNO}_3$ . Appropriated amount of lithium and cobalt nitrate (Fluka) salts with the cationic ratio of Li:Co 1:1 were dissolved in distilled water and mixed well with an aqueous solution of citric acid (Fluka). Citric acid was used as a chelating agent in making a gel. The resultant solution was evaporated at 80°C with magnetic stirring until a transparent pink sol was formed. As water evaporated further, the sol turned into a viscous transparent pink gel. The gel obtained was heated at 800°C for 8 h. The inorganic electrolyte, which was used, was neutral saturated solution of  $\text{LiNO}_3$  or  $\text{Li}_2\text{SO}_4$  (Fluka) in distilled water.

Electrochemical measurements were performed in a three-electrode glass test cell containing 6 M  $\text{LiNO}_3$  or 2M  $\text{Li}_2\text{SO}_4$  water electrolyte. The active mass of the test electrodes was a mixture of tested active electrode materials studied and teflonized acetylene black at 1:1 ratio by weight. After that test, electrodes were formed by pressing 25  $\text{mg}\cdot\text{cm}^{-2}$  of thus prepared mixture onto an expanded nickel grid at a pressure of 3t. $\text{cm}^{-2}$ .

### Characterization techniques

Cyclic voltammetry (CV) was performed with an Arbin 320 potentiostat/galvanostat (Arbin Instruments Co.) in a three-electrode glass cell at 200 and 50  $\mu\text{V s}^{-1}$  scan rate in various voltage ranges, using  $\text{LiMn}_2\text{O}_4$  or  $\text{LiCoO}_2$  as working electrode, counter electrode were  $\text{LiV}_3\text{O}_8$ ,  $\text{LiCoO}_2$  and  $\text{LiMn}_2\text{O}_4$ , and reference electrodes  $\text{Ag}/\text{AgCl}$ , respectively. X-ray analysis was carried out on a Philips APD 15 diffractometer with  $\text{Cu K}\alpha$  radiation.

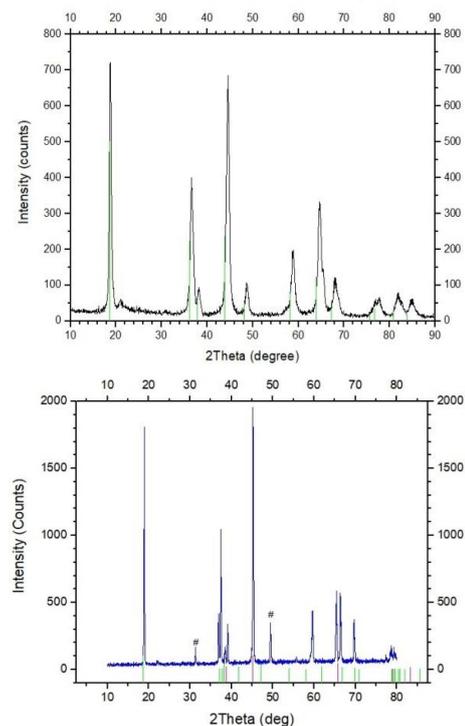
## RESULTS AND DISCUSSIONS

Fig. 1(a) shows the XRD patterns of  $\text{LiMn}_2\text{O}_4$  samples having the spinel structure without any impurity phases, which belongs to Fd3m face cubic crystal system (ICSD, no. 087775). In this crystal structure, lithium atom occupies the tetragonal 8a

position, manganese atom occupies 16d position, and oxygen atom is in the position of 32e.

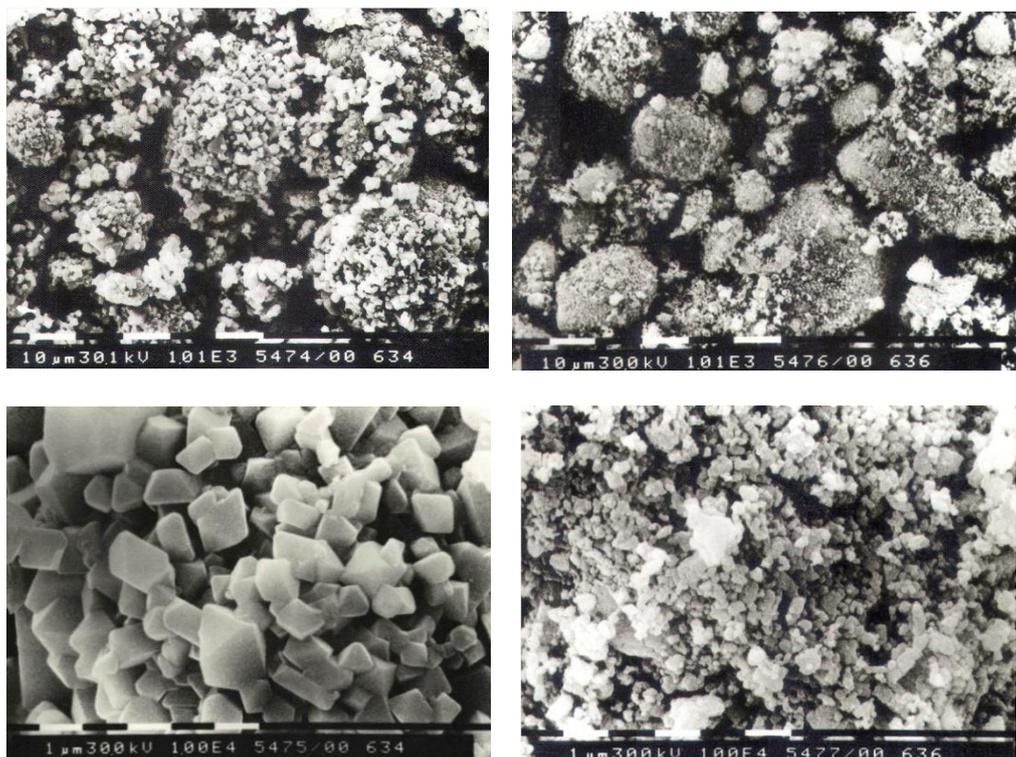
In Fig. 1(b) X-ray diffraction patterns for  $\text{LiCoO}_2$  obtained by nitrate gel treated at 800°C for 24h as described above. The peaks marked as (#) correspond to  $\text{Co}_3\text{O}_4$  impurity.

The diffraction pattern shows clear (006)/(102) peaks and (018)/(110) split peaks indicating a perfect layered-structure for  $\text{LiCoO}_2$  [19]. The pattern of  $\text{LiCoO}_2$  prepared at 800°C comprises a major  $\text{LiCoO}_2$  phase crystallizing in rhombohedral structure with a small amount of  $\text{Co}_3\text{O}_4$  as impurity.



**Fig. 1.** The X-ray diffraction pattern of the materials. (a)  $\text{LiMn}_2\text{O}_4$  and (b)  $\text{LiCoO}_2$

Fig.2 shows the SEM micrographs of the  $\text{LiMn}_2\text{O}_4$  sample synthesized by solid-state reaction from  $\text{MnO}_2$  Faradizer M (CMD) and  $\text{LiNO}_3$  heated at 850°C a) and b) and heated at 650°C c) and d) for 24h. From Fig. 2a and 2b it is seen that the resultant  $\text{LiMn}_2\text{O}_4$  sample mainly consists of large quantities of particles with a size about 1 $\mu\text{m}$ . Fig.2b gives a high-magnification image of several selected  $\text{LiMn}_2\text{O}_4$  particles. It is found that the prepared  $\text{LiMn}_2\text{O}_4$  sample is composed of particles with well-developed octahedral shapes [20, 21]. In addition, very few quasi-spherical particles can also be observed in Fig. 2c. However, a high-magnification micrograph in Fig. 2d indicates the outer part of this quasi-spherical  $\text{LiMn}_2\text{O}_4$  particle has been evolved to many small polyhedral particles, suggesting better electrochemical performance.



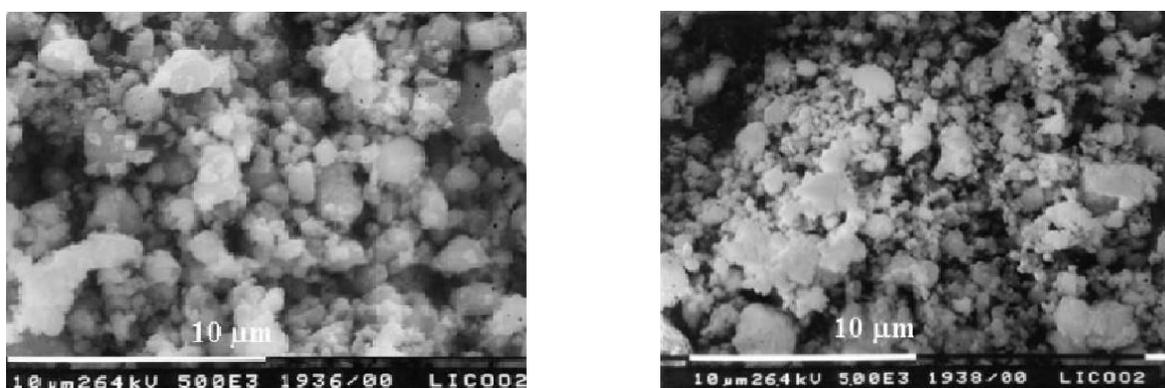
**Fig.2** EM images of the  $\text{LiMn}_2\text{O}_4$  synthesized by the solid-state reaction at  $850^\circ\text{C}$  a), b) and at  $650^\circ\text{C}$  c), d) for 24h from  $-0.6$  to  $0.02\text{V}$  vs.  $\text{Ag}/\text{AgCl}$  using two different aqueous electrolytes,  $6\text{M}$

$\text{LiNO}_3$  and  $2\text{M}$   $\text{Li}_2\text{SO}_4$ . This involved the use of a  $\text{LiV}_3\text{O}_8$  as a counter electrode. The results are shown on Fig.4, Fig.5 and Fig.6. The scan rates were rather low,  $50\mu\text{V}\cdot\text{s}^{-1}$ . In all cases, an electrochemical process was clearly observed. The electrochemical behavior of  $\text{LiMn}_2\text{O}_4$  in  $6\text{M}$   $\text{LiNO}_3$  and in  $2\text{M}$   $\text{Li}_2\text{SO}_4$  is very close. The specific capacities in two water electrolytes are almost the same as the nitrate is slightly higher, respectively,  $99\text{mAh}\cdot\text{g}^{-1}$  and  $94\text{mAh}\cdot\text{g}^{-1}$ , which correspond to 80% of the theoretical capacity of the material. The material works in the range of electrochemical stability of water, the evolution of  $\text{O}_2$  starting after  $1.15\text{V}$  (Fig.4).

Fig.3 shows SEM images of the  $\text{LiCoO}_2$  synthesized by sol-gel method a) and by solid state reaction - SSR b). It is clear that both materials are characterized by good crystallinity and have similar morphology. As can be seen from Fig.3, the resulting materials are not monodispersed and consist of both particles of less than  $1\mu\text{m}$  and agglomerates of about  $1\text{-}2\mu\text{m}$ . Better

dispersion of the precursor particles in the sample sol-gel method results in particle size reduction (Figure 3b).

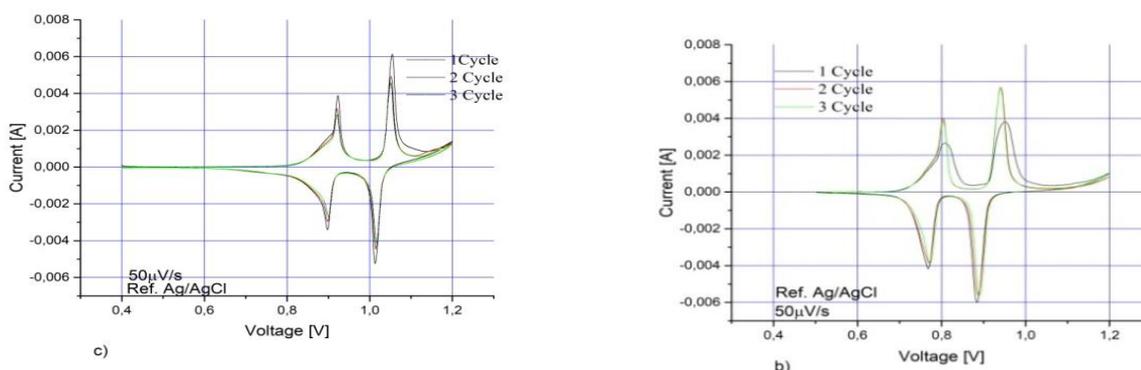
To understand the electrochemical behavior of  $\text{LiCoO}_2$  and  $\text{LiMn}_2\text{O}_4$  in aqueous solutions, we performed voltammograms from  $-0.2$  to  $1.6\text{V}$  and



**Fig. 3.** SEM images of the  $\text{LiCoO}_2$  synthesized by sol-gel method treated at  $800^\circ\text{C}$  a) and SSR at  $650^\circ\text{C}$  b) for 24h

Using 6M  $\text{LiNO}_3$  as electrolyte,  $\text{Ag}/\text{AgCl}$  as reference electrode and scan rate of  $50 \mu\text{V}\cdot\text{s}^{-1}$  the first peak of  $\text{Li}^+$  ions extraction appears at 0.92V and the corresponding insertion peak is located at 0.9V. The second peak of extraction, was at 1.05V and peak of insertion was at 1.01V and the specific capacity of  $99 \text{ mAh g}^{-1}$ . Observed difference between both couple of peaks is only 20-30mV that means the processes are in quasi equilibrium (Fig.4a). The CV carried out in the same conditions but with  $200 \mu\text{V}\cdot\text{s}^{-1}$  showed difference of 50mV and almost the same specific capacity ( $95 \text{ mAh}\cdot\text{g}^{-1}$ ) which indicate a fast electrochemical process for this material. On the Fig. 4b are shown the curves

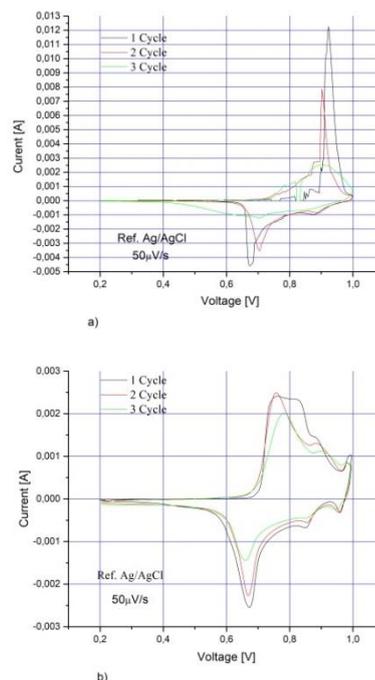
of works of  $\text{LiMn}_2\text{O}_4$  in  $\text{Li}_2\text{SO}_4$  with scan rate  $50 \mu\text{V}\cdot\text{s}^{-1}$ . The specific capacity is  $94 \text{ mAh g}^{-1}$  and all peaks are shifted with 100 mV to the potential of reference electrode in comparison with this in  $\text{LiNO}_3$ , this is most probably due to the nature of electrolyte. This shifting to the lower potentials reduces probability of  $\text{O}_2$  evolution. The last electrochemical system realized in real cell will work more stable and safety. The capacity of delithiation (lithium extraction) is little higher using  $\text{LiNO}_3$  (in both rates) than those of delithiation in  $\text{Li}_2\text{SO}_4$  cause of proximity of peaks to the evolution of  $\text{O}_2$  in  $\text{LiNO}_3$  electrolyte.



**Fig.4.** Cyclic voltammograms of  $\text{LiMn}_2\text{O}_4$  synthesized by SSR method at  $650^\circ\text{C}$  in 6M saturated aqueous  $\text{LiNO}_3$ , a), in 2M  $\text{Li}_2\text{SO}_4$  b) scan rate  $50 \mu\text{V}\cdot\text{s}^{-1}$ .

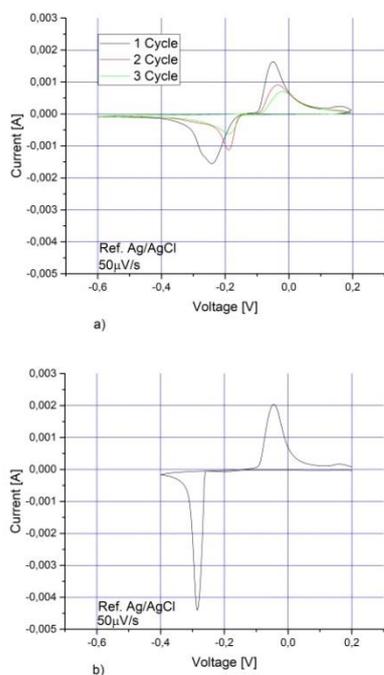
Fig.5 shows cyclic voltammograms of  $\text{LiCoO}_2$ . In the first cycle of the sol-gel-synthesized  $\text{LiCoO}_2$ , a deintercalation peak at 0.92V and intercalation peak at 0.67V were observed (Fig.5a). These potency values are typical of the  $\text{LiCoO}_2$  presented. In the second cycle, the peak potential values of deintercalation/intercalation are respectively 0.7 and 0.9V. The value of the second peak of the potential is higher than that of the first. This polarization difference is most likely due to rearrangement of the structure after the first cycle. The voltammogram in  $\text{Li}_2\text{SO}_4$  shows that deintercalation peaks were at 0.76V and the intercalation were at 0.66V. The difference is about 50 mV. The process of extraction of  $\text{Li}^+$  in  $\text{Li}_2\text{SO}_4$  is easier compared to process in  $\text{LiNO}_3$  (Fig.5b). The specific capacities in the two water electrolytes are  $99\text{mAh}\cdot\text{g}^{-1}$  and  $79\text{mAh}\cdot\text{g}^{-1}$  respectively for  $\text{LiNO}_3$  and  $\text{Li}_2\text{SO}_4$ , which is 68% of the theoretical capacity of the material.

The manganese spinel  $\text{LiMn}_2\text{O}_4$  has two voltage plateaus at 3 and 4 V vs. Li, both of them located in the electrochemical window of aqueous electrolyte.



**Fig.5** Cyclic voltammograms of  $\text{LiCoO}_2$  as synthesized by sol-gel method and treated at temperature  $800^\circ\text{C}$  in 6M saturated aqueous  $\text{LiNO}_3$  a) and 2M  $\text{Li}_2\text{SO}_4$ ; scan rate was  $50 \mu\text{V}\cdot\text{s}^{-1}$ .

Therefore,  $\text{LiMn}_2\text{O}_4$  could be utilized as both the cathode and anode in such an aqueous cell. We focused on the performance of the aqueous negative active material. The results are shown in Fig. 6. We present voltammograms within 0.2 to -0.6V vs. Ag/AgCl in the lithium nitrate aqueous solutions. This include, the use of a  $\text{LiCoO}_2$  (Fig.6 a)) and  $\text{LiMn}_2\text{O}_4$  (Fig.6 b) as counter electrode.



**Fig. 6** Cyclic voltammograms of  $\text{LiMn}_2\text{O}_4$  for negative electrode in 6M saturated aqueous  $\text{LiNO}_3$  vs.  $\text{LiCoO}_2$  as counter electrode a) and vs.  $\text{LiMn}_2\text{O}_4$  as counter electrode b); scan rate was  $50 \mu\text{Vs}^{-1}$

Fig.6 a) shows use of spinel  $\text{LiMn}_2\text{O}_4$  as anode material. In the first cycle an intercalation peak at -0.25V and deintercalation peak at -0.048V were observed. In second cycle, the peaks are shifted to -0.19 and -0.035V and in the third cycle the peaks position were 0.19 and -0.14V. In the beginning is observed polarization of electrode, but in the next cycles the process of charge/discharge becomes stable. Charge capacity in the first cycle was  $40\text{mAh.g}^{-1}$  and the discharge capacity -  $32\text{mAh.g}^{-1}$ . In second cycle the values were  $26 \text{mAh.g}^{-1}$  and  $21 \text{mAh.g}^{-1}$  and in the third cycle -  $21\text{mAh.g}^{-1}$  and  $16\text{mAh.g}^{-1}$ . The efficiency is about 80% from the theoretical one that means the material is stable and work good.

Fig.6 b) shows the rechargeable lithium-ion cell with  $\text{LiMn}_2\text{O}_4$  as a use of spinel  $\text{LiMn}_2\text{O}_4$  as anode material, working and counter electrodes. In this way, we have fully symmetric cell with same active electrode materials as cathode and anode. The intercalation peak appears at -0.28 V and the deintercalation is at -0.043V. The charge capacity is

$49 \text{mAh.g}^{-1}$  and the discharge is  $41 \text{mAh.g}^{-1}$ , values very close each other. In reality, the discharge capacity is exactly 84% from the charge capacity.

## CONCLUSION

The electrochemical characteristics of  $\text{LiMn}_2\text{O}_4$  and  $\text{LiCoO}_2$  used as cathode were studied in aqueous electrolyte containing lithium nitrate or sulfate with respect to its use as a new kind of rechargeable battery system. Cyclic voltammetry showed that spinel  $\text{LiMn}_2\text{O}_4$  and  $\text{LiCoO}_2$  reversibly intercalated/deintercalated  $\text{Li}^+$  ions at potentials lower than the potentials of hydrogen or oxygen evolution in present aqueous solution with neutral pH.

The use of spinel and cobaltate as a positive active material for rechargeable battery delivers  $95\mu\text{mAh.g}^{-1}$  and  $99\mu\text{mAh.g}^{-1}$  discharge capacity. The capacity of cobaltate is lower in sulfate solution -  $79 \text{mAh.g}^{-1}$ . The use of manganese spinel  $\text{LiMn}_2\text{O}_4$  as anode were investigate against  $\text{LiCoO}_2$  as counter electrode in nitrate solution. This electrochemical system is stabile but the discharge capacity was very low -  $32 \text{mAh.g}^{-1}$ .

The experiments show that the capacity in lithium nitrate is higher but the process of extraction of  $\text{Li}^+$  is easier in sulfate. A symmetric electrochemical system based on manganese spinel in  $\text{LiNO}_3$  electrolyte was tested for the first time and shows very promising results for large-scale energy storage and energy conversion.

The cost of the aqueous cell will be lower than that of conventional Li-ion battery cell, because electrode and electrolyte materials are not expensive and toxic compared with those used in conventional lithium ion batteries. The assembling process is simple, environmental friendly and trouble-free.

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## ЕЛЕКТРОХИМИЧНИ СВОЙСТВА НА $\text{LiMn}_2\text{O}_4$ И $\text{LiCoO}_2$ ВЪВ ВОДНИ ЕЛЕКТРОЛИТИ

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(Резюме)

Литиево-йонните батерии базирани на органичен електролит дават възможност за постигане на големи енергийни плътности. Тези електролити представляват силно токсични и запалими разтворители, а подготовка им е сложна и скъпа, отчасти защото са чувствителни към влагата от въздуха. Използването на водни електролити в акумулаторни литиево-йонни батерии ще увеличи безопасността, екологичността им и ще намали стойността им. Разгледани са електрохимичните свойства на  $\text{LiMn}_2\text{O}_4$  и  $\text{LiCoO}_2$  като положителни електроди за литиево-йонна батерия с воден електролит срещу  $\text{LiV}_3\text{O}_8$  като отрицателен електрод. Използваните електролити са 6M  $\text{LiNO}_3$  и 2M  $\text{Li}_2\text{SO}_4$ , разтворени в дестилирана вода.  $\text{LiMn}_2\text{O}_4$  е получен чрез приготвяне на стехиометрична смес от  $\text{LiNO}_3$  и  $\text{MnO}_2$  в твърдо състояние и нагряване при температура 650°C в продължение на 24 часа.  $\text{LiCoO}_2$  се получава чрез зол-гел метод с крайно термично третиране при 800°C в продължение на 24 часа. Литиевият ванадат се получава чрез твърдофазен синтез. Всички материали са стабилни във водния разтвор и интеркалацията/деинтеркалацията на литиевите йони се извършва в прозореца на електрохимична стабилност на водата, който се определя при използване на бавна циклична волтаметрия. Манганоксидният шпинел има специфичен капацитет от 99  $\text{mAh.g}^{-1}$  в 6M  $\text{LiNO}_3$  и 94  $\text{mAh.g}^{-1}$  в 2M  $\text{Li}_2\text{SO}_4$  воден електролит, докато неговият капацитет в органичния електролит е 135  $\text{mAh.g}^{-1}$ . Полученият капацитет от литиев кобалтат във водните електролити е съответно 99  $\text{mAh.g}^{-1}$  и 92  $\text{mAh.g}^{-1}$ , докато този в органичния електролит е 145  $\text{mAh.g}^{-1}$ . Цикличното поведение и специфичният капацитет на тези активни материали са изследвани в електрохимичните клетки и като отрицателна активна маса.

**Ключови думи:** манганов шпинел, литиев кобалтат, воден електролит, литиево-йонна батерия