



Synthesis of LiCoO_2 powders for lithium-ion batteries from precursors derived by rotary evaporation

P.N. Kumta^{a,*}, D. Gallet^a, A. Waghray^a, G.E. Blomgren^b, M.P. Setter^b

^a Department of Materials Science and Engineering, Carnegie Mellon University, Pittsburgh, PA 15213, USA

^b Eveready Battery, Westlake, OH 44145, USA

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Abstract

Lithium cobalt oxide (LiCoO_2) has received considerable attention in the last few years and is a well-known cathode material for high voltage (4 V) rechargeable Li-ion batteries. A simple chemical approach based on aqueous solution chemistry has been developed to synthesize molecularly mixed amorphous precursors. The precursors begin to transform to form LiCoO_2 upon heat treatment at temperatures as low as 400°C. Strong peaks characteristic of the desired high temperature (HT) phase of LiCoO_2 evolve when the precursors are heat treated to 800°C for 2 h. The morphology and particle size of the powders have been examined using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Cathodes fabricated from the oxide powders have been analyzed for their electrochemical performance using 'hockey-puck' type test cells. The cells exhibit initial discharge specific capacity as high as 165 mAh/g and an average specific capacity of about 161 mAh/g with a reversible range close to 0.6 Li-ions. © 1998 Elsevier Science S.A. All rights reserved.

Keywords: Lithium-ion battery; Rotary evaporation; Lithium cobalt oxide

1. Introduction

Li-ion batteries have recently received considerable interest as rechargeable power sources for room temperature consumer applications since the commercialization of Sony's lithium-ion cell in 1990 [1]. Interest in these types of batteries have arisen mainly because of the identification of new materials capable of intercalating lithium-ions [2–4]. The concept of intercalation has opened up a new window of opportunities for electrode materials eliminating the use of lithium metal and thereby providing alternative materials that are quite efficient and more importantly, show the potential for safe use as electrodes in rechargeable lithium batteries. Table 1 shows the three most popular materials that have been studied intensely for use as cathodes in high voltage rechargeable batteries namely, LiMn_2O_4 , LiCoO_2 and LiNiO_2 . In addition to the three common oxides, there has also been activity in the mixed oxides of these three systems [5,6]. Among the three materials shown in Table 1, LiCoO_2 and LiNiO_2 possess

the highest theoretical specific discharge capacity of 274 mAh/g, but structural instabilities in the former and cation disorder in the latter have limited the practical capacity to only 50% of the theoretical value. However, LiCoO_2 was one of the first materials that was tested as a cathode in rechargeable batteries and with the development of Sony's Li-ion battery, it has received considerable interest. Consequently, this material has been selected for the present study.

The oxide was first synthesized by Mizushima et al. [7,8] in 1980 who demonstrated its potential as a cathode material by electrochemical extraction of lithium from the ordered rock-salt structure of the parent LiCoO_2 crystal. Following their work, Gummow and Thackeray [9] and Gummow et al. [10] and a number of other workers have synthesized this material by solid state processes involving prolonged heat treatment of the lithium and cobalt oxides or carbonates at 900°C [11,12]. Typically, the procedure consists of forming pellets of the mixture of lithium carbonate and cobalt carbonate and heat treating these pellets in air at 900°C for 20 h followed by two further heat treatments for 24 h at the same temperature. Some other approaches that have been described in the literature are

* Corresponding author.

Table 1
Properties of some important cathode materials for rechargeable batteries [3]

	Cathode material	Molecular weight	Density (g/cm ³)	Reversible range (Δx)	Specific capacity (AH/kg)	Capacity density (AH/l)
Charged	Li _{1-x} TiS ₂	112.0	3.27	1.0	239	782
	Li _{1-x} MoS ₂	160.1	5.06	0.8	134	678
	Li _{1-x} V ₂ O ₅	181.9	3.36	1.0	147	495
	Li _{1-x} V ₆ O ₁₃	513.6	3.91	3.6	188	734
	Li _{1-x} MnO ₂	86.9	5.03	0.5	154	775
	Li _{1-x} NbSe ₃	329.8	8.70	3.0	244	2121
Discharged	Li _x CoO ₂	97.9	5.16	0.5	137	706
	Li _x NiO ₂	97.6	4.78	0.7	192	919
	Li _x Mn ₂ O ₄	180.8	4.28	1.0	148	634

chemical lithiation of Co₃O₄ using a solution of *n*-butyl lithium. The lithiated oxide is then heat treated at high temperatures to form Co₃O₄ and Li₂O. These mixed phases then undergo solid state reactions very similar to those described in the earlier process to form LiCoO₂ [13].

All these solid state processes normally require prolonged heat treatments at high temperatures to form the desired phase of the material without the presence of any unwanted secondary phases. These long heat treatments at high temperatures normally do not provide much control of the microstructure since grain growth and overall agglomeration of the particles are always inevitable in these treatments. In addition, the risk of contamination due to possible reaction with the crucible containers and the possible volatilization of lithium itself is an inherent problem, which tends to be accentuated due to the long heat treatment cycle. Thus, although solid state processes are very convenient and still constitute by far, the most widely accepted approaches for synthesizing electrochemical materials, they are not very amenable for controlling many of the material characteristics. This is particularly related to the microstructure which includes the grain size, particle size and morphology. These factors affect the diffusion of lithium, which ultimately relate to the intercalation efficiency, the cyclability of the cells, and the specific capacity.

Chemical processes offer much improvement and potential in terms of synthesizing materials of high purity, and controlling their microstructure and morphology. Interest in these processes have emerged following the advent of the sol-gel process in 1960 and over the years, several techniques have evolved. The most common chemical processing techniques in addition to the sol-gel process include chemical precipitation, spray drying, sol evaporation and decomposition. These approaches offer significant advantages over the conventional techniques. These advantages stem from conducting the reactions in solution containing either dissociated species or solvated ions or complexes which lead to very good mixing of the individual reacting components at a molecular level. Mixing of the species at a molecular level contributes to a reduction in

the diffusion distances thereby leading to the formation of the desired material with significant reduction in time and temperature in comparison to the solid state mix and fire approaches. The homogeneity achieved at a molecular level also provide good control of the stoichiometry of the synthesized material. In addition, for example, in the sol-gel process, the reactions in solution lead to the formation of solid precursors at room temperature that contain metal-oxygen bonds. The formation of these bonds creates an initial structural framework that significantly lowers the time and temperature of the subsequent heat treatments required to form the desired crystalline oxide material. Furthermore, these combined attributes of solution based chemical processes also enables the formation of stoichiometric materials containing multicomponents. There is significant information available in the literature on chemical synthesis of a myriad of oxide and non-oxide ceramics and glasses [14–16]. Following the success of these approaches for synthesizing ceramics there has been some work on the use of solution techniques for synthesizing LiCoO₂. Bach et al. [17] adopted a solution based redox reaction, while the recent work of Rossen et al. [18] and Garcia et al. [19,20] is based on chemical precipitation of amorphous hydroxides. Yazami et al. [21] on the other hand, utilized a solution based technique for synthesizing LiCoO₂ based on the use of a suspension of a metallorganic Co salt. Similarly, Zhecheva et al. [22] employed citrate precursors while Sun et al. [23] used polyacrylic acid as a chelating agent in a solution containing nitrate ions to form the oxide.

In this paper we report a simple aqueous solution based process for synthesizing lithium cobalt oxide employing metallo-organic precursors for lithium and cobalt. The paper describes the process used for synthesizing the precursors and also discusses the transformation of these precursors to form the oxide phase. Results of the characterization of the emerging lithiated oxide phase using X-ray diffraction, scanning electron and transmission electron microscopy are also presented. In addition, the paper discusses results of the electrochemical tests conducted in prototype test cells fabricated using the synthesized oxide

powder as the cathode. These results are discussed with regards to the amorphous precursor, the evolved oxide phase and microstructure of the crystalline powder.

2. Experimental procedure

2.1. Materials synthesis and characterization

An aqueous solution based process was used for synthesizing LiCoO_2 powders. Equimolar amounts of the corresponding acetate salts, $\text{LiCH}_3\text{COO} \cdot 2\text{H}_2\text{O}$ and $\text{Co}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ procured from Aldrich were dissolved in doubly distilled deionized water. The salts were dissolved for 1 h in the aqueous solution. The liquid phase obtained was then dried in the temperature range of 100°C – 180°C using a Büchi rotary evaporator employing an envelope of N_2 gas. The overall drying time varied from 1 h 30 min to 4 h 30 min. At the end of the drying stage, the product is essentially a ‘glassy’ solid. This dried xerogel product obtained was then ground into a fine powder using an alumina mortar and pestle. The powders were then heat treated in a box furnace using a nickel crucible to three different temperatures, 400°C , 600°C , and 800°C respectively. The heating rate was set at $10^\circ\text{C}/\text{min}$ for all the temperature settings and the samples were soaked for a period of 2 h at each of these settings after which they were allowed to furnace cool. The heat treated product was ground in an agate mortar and pestle and the fine powders were then analyzed for their structure and phase evolution characteristics using X-ray diffraction (Rigaku θ/θ diffractometer employing $\text{Cu-K}\alpha$ radiation). The morphology and microstructure of the powders were examined using a scanning electron microscope (CamScan Series IV), while the particle sizes were observed using a JEOL 120CX transmission electron microscope. Fig. 1 is a schematic flow sheet showing the procedure used to synthesize these powders.

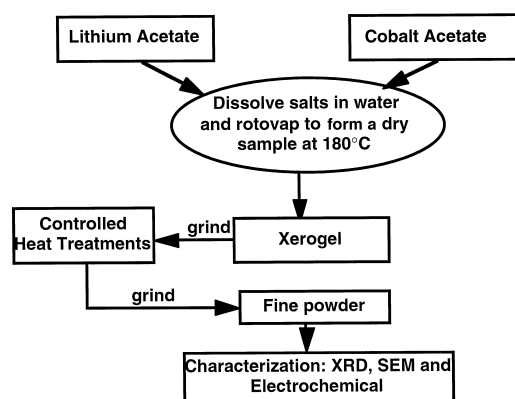


Fig. 1. Experimental flow sheet showing the procedure used to synthesize LiCoO_2 .

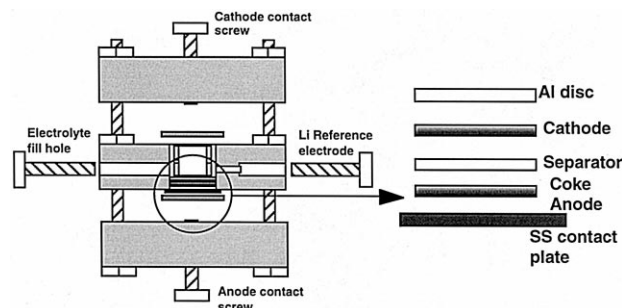


Fig. 2. Schematic of the experimental test cell used to evaluate the electrochemical performance of LiCoO_2 cathodes.

2.2. Electrochemical characterization

A three electrode experimental cell design was used to evaluate the electrochemical performance of these cathodes. The cathode mixture was prepared by dissolving the ethylene/propylene copolymer binder (5.34%) in trichloroethylene to which was added a physical mixture of LiCoO_2 (87.06%) and Super S (7.59%) acetylene black powder (to provide the required electrical conductivity). This cathode mixture having a consistency of a pancake-mix was tape cast onto a 1 mil aluminium foil and then air dried before punching 1 cm^2 cathode disks. These disks were dried in a vacuum oven at 160°C for 16 h before use. The cathodes were tested using lithium metal as the reference electrode. The coke anode was initially titrated with the lithium reference to ensure the availability of excess lithium before charging the cathode. The electrolyte used was a 1 M LiPF_6 solution in 66 wt.% ethylene carbonate and 33 wt.% dimethyl carbonate that was soaked onto a Whatman DR2 separator (predried under vacuum at 250°C for 16 h). The entire cell was assembled in an argon filled glove box (Vacuum Atmospheres, CA). The cells were tested using a constant current of 0.25 mA in the voltage range of 4.35 and 2.9 V. An initial setting of ten cycles was used to test the fabricated cells. Two cells were tested for consistency. Fig. 2 shows a schematic of the experimental cell design used for evaluating the fabricated test cells.

3. Results and discussion

As indicated above, LiCoO_2 powders were synthesized by heat-treating precursors obtained from an aqueous solution containing lithium and cobalt acetates. The clear neutral solution is a good medium for providing good mixing of the Co and Li-ions without inducing precipitation of any cobalt hydroxide. Rotary evaporation of the solution in the Büchi set-up operated under the conditions described above results in a xerogel of the acetates that appeared slightly pink in color. The precursors were analyzed for their crystalline nature using X-ray diffraction. The XRD patterns collected on the precursors are shown in

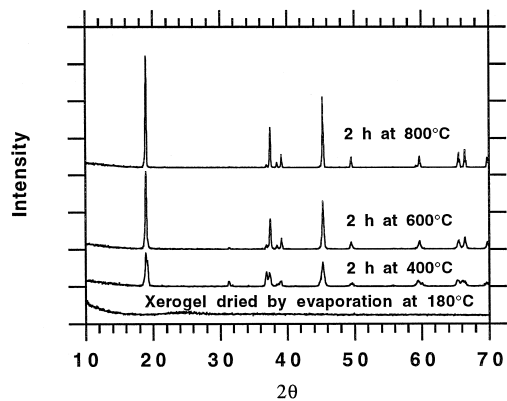


Fig. 3. XRD patterns showing the phase evolution of LiCoO_2 upon heat treatment of the precursors.

Fig. 3. As can be seen the as-prepared precursors are amorphous. Infrared spectroscopic analysis of the xerogel indicate that the precursor is mainly comprised of a molecular mixture of the hydroxy acetate complex of cobalt and the dried hydroxide of lithium [24].

3.1. Phase evolution of LiCoO_2

The amorphous precursors were then heat treated in air at 400°C, 600°C and 800°C respectively for 2 h. The phase evolution of the precursors upon heat treatment is shown in Fig. 3. The pattern at 400°C indicates the formation of LiCoO_2 although significant amounts of the spinel phase of cobalt, Co_3O_4 , belonging to the space group $Fd\bar{3}m$ is also seen. This is indicative of the fact that the amorphous precursor particles are very reactive and contain molecular units which by and large favor the formation of LiCoO_2 . Heat treatment at 400°C leads to decomposition of the acetate and hydroxy groups leading to a generation of intimately mixed oxides which then react to form LiCoO_2 . However, heat treatment at 400°C for 2 h is not adequate to consume all of the Co_3O_4 phase. Prolonged heat treatment at this stage would probably result in completion of the reaction and lead to the formation of largely LiCoO_2 as observed by Garcia et al. [19,20] in the precipitation approach developed by them. This heat treatment was not conducted in the present study due to the possibility of formation of the low temperature (LT) polymorph of LiCoO_2 that is known to exhibit poor electrochemical behavior [18]. It was therefore decided to heat treat the precursor to a higher temperature in order to accelerate the formation of the oxide and the phase transformation reaction to induce the conversion of the LT-phase to the desired high temperature (HT) phase. Correspondingly, it can be seen from Fig. 3 that the secondary oxide phase decreases rapidly on subsequent heat treatment of the powders to higher temperatures of 600°C and 800°C. In fact, heat treatment of the precursors directly to 800°C for 2 h results in strong intense peaks of HT- LiCoO_2 and almost complete elimination of the Co_3O_4 phase. The peaks in the X-ray diffraction pattern have been indexed to

the hexagonal form of LiCoO_2 which is the high temperature (HT) polytype belonging to the space group $R\bar{3}m$.

The HT-polytype of LiCoO_2 has a layered structure as is well known in which the lithium and the cobalt ions occupy all the octahedral sites in the successive layers along the (0001) direction [18]. The existence of a low temperature polymorph of LiCoO_2 has also been reported in the literature. The structure of the low temperature phase (LT) of LiCoO_2 remained an enigma since both the polymorphs apparently exhibit identical X-ray patterns although belonging to two different space groups. Recently, Garcia et al. [19] have performed high resolution electron microscopy on the LT-phase and have shown that it can be indexed on a cubic unit cell with a lattice constant of $\approx 8.00 \text{ \AA}$ having a structure similar to the spinel related structure of $\text{Li}_2\text{Ti}_2\text{O}_4$. Their studies indicate that it is more appropriate to describe the structure of this phase as an ordered cubic rock salt structure. This phase has been reported to be synthesized by reacting the corresponding carbonates of lithium and cobalt at 400°C for a week to ten days. In this structure, the oxygen framework is identical to that observed in HT- LiCoO_2 provided the c/a ratio of the layered structure is chosen to be 4.9 and that the oxygen z coordinate (6c sites) is 0.25. The cation sites are also known to be in the same positions in the two structures. However, the HT- LiCoO_2 structure contains the cations which are grouped into layers of Li alternating with layers of Co perpendicular to only one of the four (equivalent) cubic (111) directions. On the other hand, in the ordered rock salt structure belonging to $Fd\bar{3}m$ space group, the cations are in the octahedral 16c and 16d sites respectively, while the oxygen ions are in the 32e sites with the oxygen coordinate $z = 0.25$ [18]. The cations are reported to be arranged in alternating layers of composition (0.75 Co, 0.25 Li) and (0.75 Li, 0.25 Co) perpendicular to each of the four cubic (111) directions. Fig. 4 shows the two different structure types. Based on their results, it appears that the phase seen on heat treatment of the amorphous precursors at 400°C is the LT-phase of LiCoO_2 . However, it is clear that on heat treatment to 600°C and 800°C the dominant phase does correspond to the desired HT- LiCoO_2 .

The above results indicate that the amorphous xerogel represents an intimately mixed state of cobalt and lithium. This mixing of the molecular species certainly helps to accelerate the kinetics of the reaction which leads to the formation of LiCoO_2 at temperatures as low as 400°C and, more importantly, in a reaction time of only 2 h. This is in significant contrast to the long anneal time of 96 h and more required in the conventional solid-state processes involving mechanical mixtures of crystalline solids or aqueous suspensions of insoluble solids [11,12,21]. As discussed above, XRD indicates the phase obtained after the short heat treatment at 800°C to be indeed LiCoO_2 . Chemical analysis was not conducted to ascertain the chemical composition of the oxide since negligible loss in

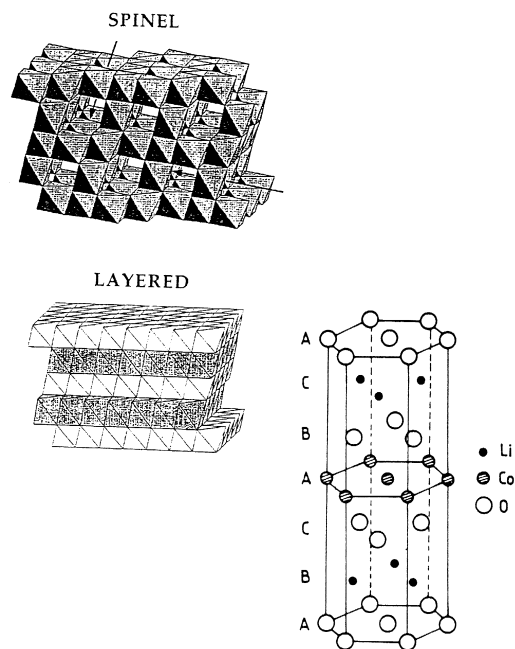


Fig. 4. Schematic of the two possible structures of LiCoO_2 [14]. Co atoms are at the center of each shaded oxygen octahedral and Li atoms are in the center of all other vacant octahedral sites. The ABCABC stacking of the layered structure is shown on the right.

lithium has been reported in the case of the oxide synthesized by the solid-state methods involving extended heat treatments at high temperatures. In light of these reports, it is likely that there is no loss in lithium under the short heat treatment conditions employed in the present case.

3.2. Morphology of the heat treated LiCoO_2 powders

The morphology of all the heat treated powders derived using the aqueous solution of the acetates were observed under scanning electron microscopy (SEM). Figs. 5–7



Fig. 5. SEM micrograph of LiCoO_2 obtained by heat treating the precursors to 400°C for 2 h. Note the presence of agglomerated clusters of particles whose primary particles are in the range of ≈ 70 nm.

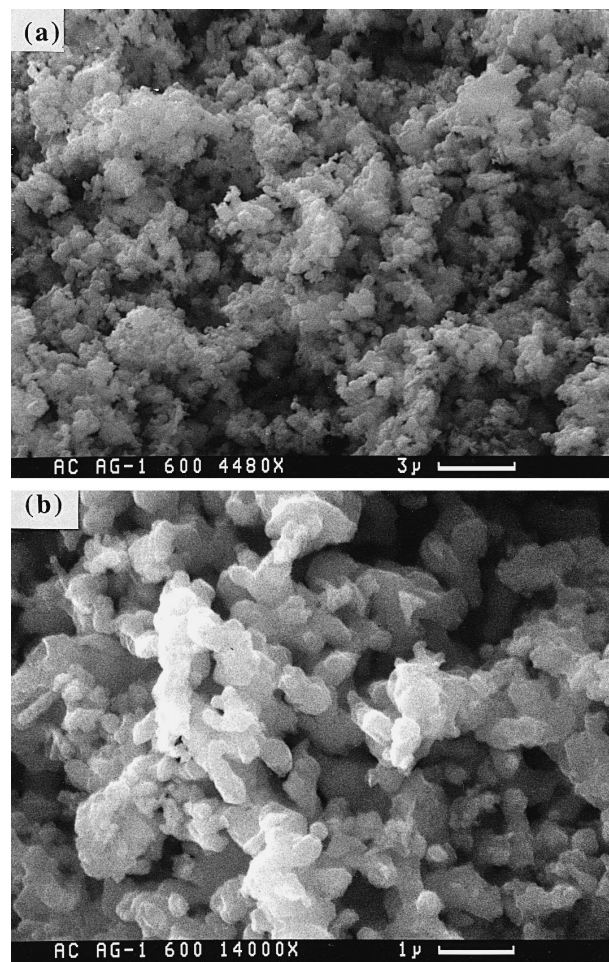


Fig. 6. SEM micrographs (a) and (b) of LiCoO_2 obtained by heat treating the precursors to 600°C for 2 h. Note the growth of the primary particles to form agglomerates $> 3 \mu\text{m}$ in size. Also note the cuboidal nature of the particles in (b).

show the SEM micrographs of each of the powders obtained at 400°C , 600°C , and 800°C respectively, after heat treatment for 2 h. Fig. 5 shows the morphology of the powders obtained after heat treatment of the molecularly mixed acetates using the aqueous process at 400°C . The micrograph reveals clusters of particles ranging in size from one to several microns with primary particles being in the range of ≈ 70 nm. The particles were also observed under transmission electron microscopy (TEM) to identify the size of the individual crystallites. The TEM analysis indicated that the primary particles were in the range of 20–40 nm confirming the SEM analysis. On heat treatment to 600°C , it can be seen that the particles have expectedly grown in size to $\approx 0.3 \mu\text{m}$ (Fig. 6a) and have undergone necking to form agglomerates that range in size from $3 \mu\text{m}$ to several microns. It can also be seen that the particles have begun to attain a definite cuboidal shape (Fig. 6b). Subsequent heat treatment of the precursor to 800°C results in further growth of the particles and it can be seen that the microstructure of the powders is now comprised of

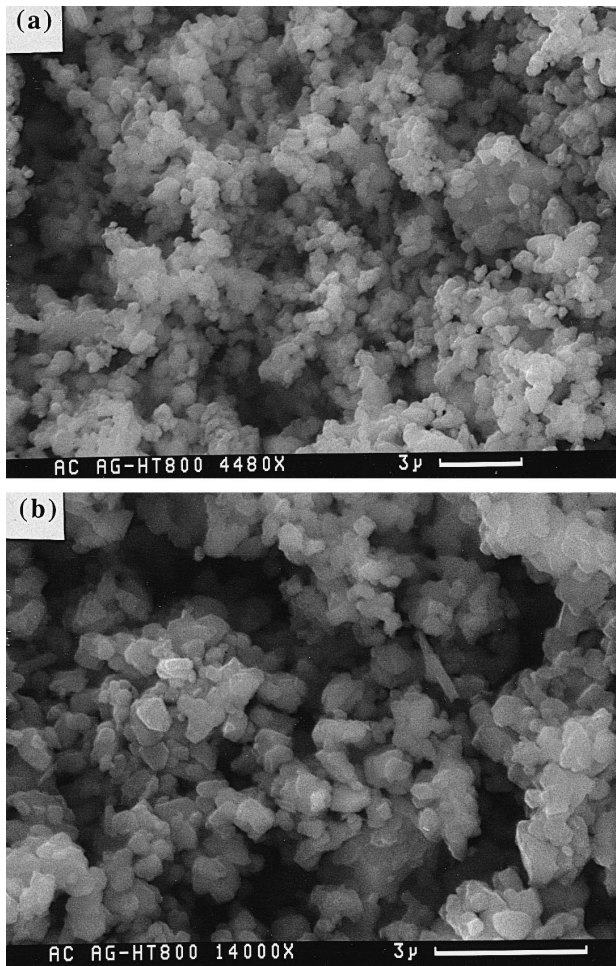


Fig. 7. SEM micrographs (a) and (b) of LiCoO_2 obtained by heat treating the precursors to 800°C for 2 h. Note the well defined faceted cuboidal shape of the primary particles, about $0.5 \mu\text{m}$ in size in (b).

large clusters several microns in size (Fig. 7a). The primary particles are correspondingly much smaller and extend to less than half a micron. An important aspect to note is that the heat treatment to 800°C has resulted in more faceting and the particles exhibit a definite regular cuboidal shape (Fig. 7b) as opposed to the heat treatments conducted at 400°C and 600°C .

In order to ascertain the crystallite size, the powders obtained after heat treatment in air at 800°C were floated on a holey carbon grid and observed under TEM. Images from several regions were taken to estimate the average crystallite size. A dark-field TEM image obtained from a representative region is shown in Fig. 8. As shown, the crystallites range in size from about 0.4 to $0.6 \mu\text{m}$ and exhibit the same cuboidal features seen in the SEM image. This shows that the primary particles are essentially sub-micron and tend to undergo significant agglomeration that is characteristic of fine particles. In addition, it can be seen that the heat treatment conditions also seem to affect the morphology of the particles. Gradual exposure of the precursors to temperatures of 400°C , 600°C and 800°C

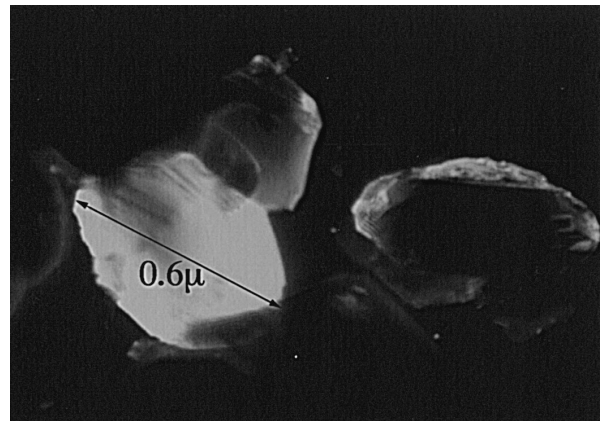


Fig. 8. TEM dark field image of 800°C calcined LiCoO_2 powders showing crystallites $\approx 0.6 \mu\text{m}$ in size.

results in the formation of the oxide phase and growth of the crystallites with the definite cuboidal facets.

3.3. Electrochemical characteristics

In order to test the electrochemical performance of the synthesized oxide materials, cathodes were fabricated using the precursor powders heat treated to 800°C according to the procedure described earlier. These powders were selected for the electrochemical characterization since X-ray analysis of these powders indicated strong peaks characteristic of the well crystallized high temperature phase of LiCoO_2 . The charge–discharge curves obtained for the first four cycles for these cathodes are shown in Fig. 9. As can be seen, the results indicate the profiles that were obtained for tests conducted in the voltage range of 4.35 and 2.9 V . A typical test for LiCoO_2 is normally conducted between the voltage range of 4.2 and 3.3 V . However, in the present tests the cells were discharged to a lower voltage to observe their capacity to intercalate lithium. As is evident from the profile, this nevertheless affects the amount of recoverable lithium. In any case, it can be seen that the cells cycled from LiCoO_2 to $\text{Li}_{0.4}\text{CoO}_2$

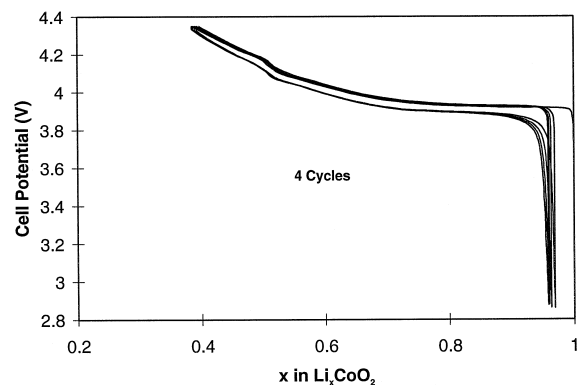


Fig. 9. Charge–discharge profiles corresponding to the first four cycles for cathode fabricated from LiCoO_2 (heat treated at 800°C) powders.

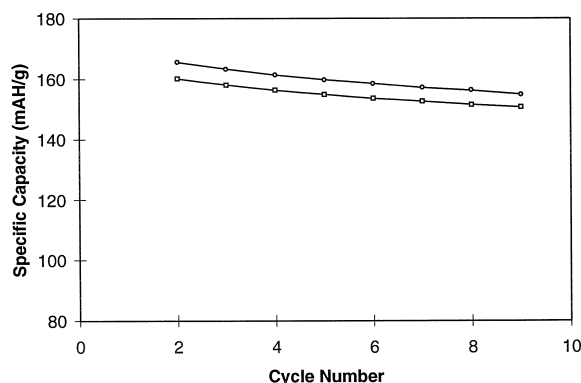


Fig. 10. Specific capacities of two cells fabricated from the synthesized LiCoO_2 (heat treated at 800°C) powders as a function of the number of cycles.

exhibiting discharge capacities as high as ≈ 165 mAh/g at the end of the second cycle and ≈ 156 mAh/g at the end of the ninth cycle. The cells therefore exhibited an average cathode utilization of about 59% yielding an average specific capacity of about 161 mAh/g as shown in Fig. 10. The cell efficiencies are therefore representative of LiCoO_2 despite the higher voltage employed during charging [25].

It is known that the HT-phase undergoes irreversible transformation to the monoclinic phase with the removal of more than 0.5 Li-ions [25,26]. It is possible that this may be the cause for a drop in the capacity during the subsequent cycles. The use of higher voltage during charging could cause for a certain fraction of the material to undergo the irreversible transformation thereby leading to a drop in the capacity. The use of a lower range of the voltage during testing could result in slightly lower but stable capacities during each cycle. It is clear therefore that the powders obtained at 800°C employing the aqueous solution processes certainly perform very well and the efficiencies are within the acceptable test limits expected for LiCoO_2 [1,27].

Based on the present results it appears that the presence of well defined fine sub-micron ($< 0.5 \mu\text{m}$) cuboidal particles do indicate good electrochemical characteristics. These fine particles could perhaps provide easier diffusion paths for the deintercalation of lithium which is the cause for the high capacity during the first charge. Subsequently, there is a $\approx 3\%$ loss in the specific discharge capacity at the end of the first cycle as shown in the charge–discharge plot of Fig. 9. This is an irreversible loss that occurs, the cause for which is at present unknown. However, based on reports in the literature it is possible that a combination of the high voltage of 4.35 V and the removal of more than 0.5 Li-ions, could induce a structural change which prevents the intercalation of all the Li-ions. Subsequent cycling of the cells between these two voltage points could cause for two changes to occur. A change in the crystal structure of the rhombohedral LiCoO_2 phase, which could

induce expansion and contraction of the particles thereby causing fragmentation and separation of the particles in contact. This could lead to a drop in the capacity during cycling as is displayed in Figs. 9 and 10. Another change that can be anticipated is the dissolution of the particles in the electrolyte at the high voltage of 4.35 V as reported by Amatucci [28]. More detailed studies are of course warranted to support these proposed reasons. Control of the particle size and shape could help in optimizing these reactions and changes occurring during cycling. These can be made possible by varying the chemical reactions in solutions. Accordingly, other variations of the present chemical process involving the use of chelating agents have also been attempted. These processes are similar to the solution sol–gel process except that particles are trapped within the gel matrix to yield a particulate gel. The LiCoO_2 obtained from all these processes exhibit very different morphologies while also exhibiting vastly different capacities. These results will be reported in subsequent publications.

The above results clearly demonstrate that the simple aqueous based chemical process developed in this work provides an opportunity to synthesize LiCoO_2 powders at 800°C in only 2 h. The oxide phase is actually seen to evolve at 400°C and the transformation to the HT-phase is complete after 2 h treatment at 800°C . The fine sub-micron cuboidal particles display discharge capacity as high as ≈ 165 mAh/g suggesting that approaches based on solution chemistry are viable processes for synthesizing good quality electrode materials.

4. Conclusions

An aqueous solution based process has been developed for synthesizing precursors that yield LiCoO_2 powders. The as-prepared xerogels obtained by drying the aqueous solutions are amorphous and indicate the formation of high temperature (HT) phase of LiCoO_2 upon heat treatment at 800°C for 2 h. The oxide begins to evolve at a temperature as low as 400°C with some Co_3O_4 as the secondary phase. Further heat treatments to 600°C and 800°C for 2 h result in almost complete elimination of the secondary phase and lead to the formation of the desired HT phase of LiCoO_2 . The microstructure of the powders synthesized at low temperature consist of very fine (≈ 70 nm), irregularly shaped cluster of particles ranging in size from $1 \mu\text{m}$ to several microns. The particle morphology begins to evolve upon heat treatment beyond 400°C . Heat treatment to 800°C results in well-faceted cuboidal particles that are in the sub-micron ($< 0.5 \mu\text{m}$) size range. Transmission electron microscopy reveals the primary LiCoO_2 crystallites to be ≈ 0.4 – $0.6 \mu\text{m}$ in size. The LiCoO_2 powders obtained at 800°C were tested for their electrochemical performance. The cells tested very well, exhibiting initial dis-

charge capacity as high as ≈ 165 mAh/g. The cells tend to fade exhibiting an average capacity of ≈ 161 mAh/g at the end of nine cycles. These preliminary findings demonstrate the potential of simple aqueous chemical processes for synthesizing cathode materials which display good electrochemical behavior.

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