

## Research Article

# Powder Characterization and Electrochemical Properties of $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ Cathode Materials Produced by Large Spray Pyrolysis Using Flame Combustion

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$\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode materials were produced by spray pyrolysis apparatus using the flame combustion. SEM revealed that as-prepared powders had spherical morphology with porous microstructure which had an average diameter of about  $2\ \mu\text{m}$  with broad size distribution. After the calcination,  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders with polygonal morphology and narrow particle size distribution were obtained. XRD showed that  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  was well crystallized after the calcination at  $900^\circ\text{C}$ . Rechargeable measurement of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode showed that the long plateau was observed at  $4.7\ \text{V}$  in discharge curve of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode and its discharge capacity was  $145\ \text{mAh/g}$  at  $1\ \text{C}$ . The capacity retention of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode were  $95\%$  at  $1\ \text{C}$  after 100 cycles. The discharge capacity and capacity retention of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode were  $125\ \text{mAh/g}$  and  $88\%$  at  $20\ \text{C}$ .  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode exhibited also stable cycle performance at  $50^\circ\text{C}$ .

## 1. Introduction

Lithium ion batteries have been extensively used as energy storage devices for portable electronics. Recently, these are well noted as the power sources for the vehicles such as EV and HEV [1]. Both layered type  $\text{LiCoO}_2$  and spinel type  $\text{LiMn}_2\text{O}_4$  is the most important cathode materials because of their high operating voltage at  $4\ \text{V}$  [2, 3].  $\text{LiCoO}_2$  have been mostly used as cathode material of commercial lithium ion batteries. However,  $\text{LiCoO}_2$  has a problem related to capacity fading due to the instability in rechargeable cycles. Cobalt is also expensive and its resource is not sufficient. Furthermore, the thermal stability is very low in the rechargeable process. Therefore,  $\text{LiCoO}_2$  cathode material is not suitable as a lithium ion battery for EV and HEV. On the other hand,  $\text{LiMn}_2\text{O}_4$  cathode material is suitable due to their advantages such as low cost, abundance, nontoxicity, and thermally stable [4]. It was known that Ni-substitute lithium manganese oxide spinel ( $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ ) was exhibited rechargeable behavior at about  $5\ \text{V}$  [5–7].

$\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  has attracted significant attention as a cathode material with high energy density. It was important

to control the chemical composition to obtain homogeneous  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders. So far,  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders have been prepared via the solution techniques such as coprecipitation [8, 9], spray drying [10], sol-gel [11, 12], polymer gel [13] and chemical wet process [14].  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders have been also prepared via the improved solid state reaction [15–18] and molten salt reaction [19, 20].

We have noted spray pyrolysis in order to prepare a homogeneous  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powder. It was well known that the spray pyrolysis was an effective process for the rapid synthesis of homogeneous multicomponent oxide powders [21]. We have tried to synthesize various type cathode materials for lithium ion battery by spray pyrolysis [22–24]. The rechargeable capacity and cycle performance of lithium ion battery were improved by using the cathode materials for lithium ion battery derived from spray pyrolysis. Park and Sun [25] have reported that homogeneous  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders can be obtained by ultrasonic spray pyrolysis. However, spray pyrolysis has not been applied as an industrial process because it is difficult to homogeneously pyrolyze the mist of inorganic salts in the electrical furnace that the scale-up was done. The difference of pyrolysis temperature

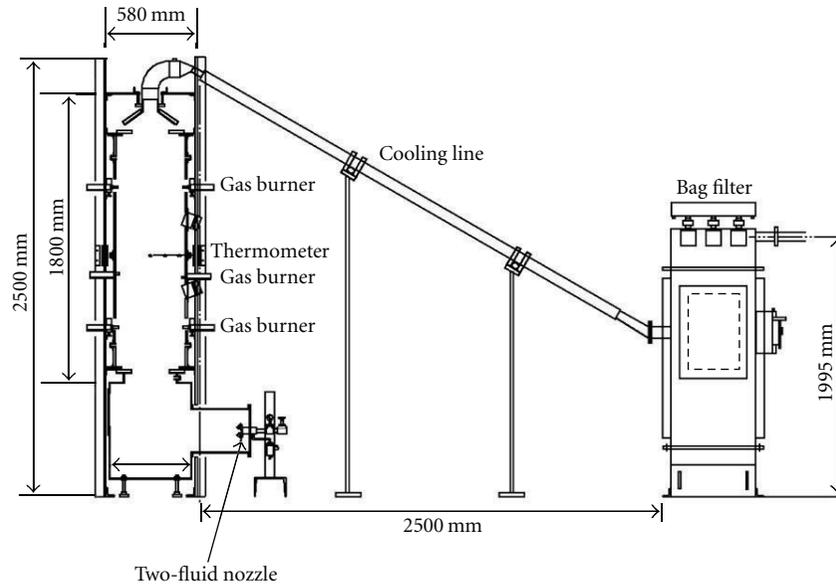


FIGURE 1: Schematic diagram of flame spray pyrolysis apparatus.

inside and outside of the electrical furnace increases with increasing the dimension of electrical furnace. So far, we have also offered the two types of spray pyrolysis apparatuses by using gas burner to produce cathode materials [26, 27]. The advantage of these apparatuses is that it is possible to pyrolyze a large amount of mist during the short time compared with that of electrical furnace. The spray pyrolysis using the flame combustion has also the high effect of energy saving compared with that using the heating of conventional electric furnace. In this work, we modified the flame combustion type spray pyrolysis apparatus [26] in which the mist flowed from the bottom to the top and then tried to produce  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders. In this paper, the powder characterization and electrochemical properties of the  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders produced by large spray pyrolysis apparatus using the flame combustion were described.

## 2. Experimental

**2.1. Powder Preparation.**  $\text{LiNO}_3$ ,  $\text{Mn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , and  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  were used as starting reagents. They were weighted out to attain the molar ratio of metal component (Li:Mn:Ni) of 2:3:1 and were dissolved in distilled water to prepare the starting solution. The concentration of the starting solution was  $1 \text{ mol/dm}^3$ . Figure 1 shows schematic diagram of flame spray pyrolysis apparatus used in this work. The apparatus is a prototype developed in order to verify the industrial production of cathode materials by the spray pyrolysis. This consisted of a two-fluid nozzle atomizer (the diameter of nozzle was  $10 \mu\text{m}$ ), a furnace ( $1800 \text{ mm} \times 580 \text{ mm}$ ) with six gas burners, a cooling line, and a bag filter. In comparison with the spray pyrolysis apparatus developed in a past study [26], the scale-up of the furnace was done. The flame combustion was generated using the liquefied petroleum gas and then the temperature of it was maintained

at  $500^\circ\text{C}$ . The mist of the starting solution was continuously generated by two-fluid nozzle atomizer with the flow rate of  $10 \text{ dm}^3/\text{h}$ . The mist was introduced to the furnace by using the air carrier gas and pyrolyzed in the furnace at  $500^\circ\text{C}$ . The temperature in the furnace was monitored by the thermometer using Pt thermocouple. The thermometer was set in the centre of the furnace. The cooling line was naturally cooled by the radiation of heat using air. The potential of powders production increased up to  $5 \text{ kg/day}$  compared with the spray pyrolysis apparatus ( $3 \text{ kg/day}$ ) developed in a past. Both as-prepared  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders and outgas were cooled less than  $100^\circ\text{C}$  by a cooling line before the powder collection in the bag filter because the temperature of outgas was more than  $200^\circ\text{C}$ .  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders were continuously produced for 5 h at the rate of  $200 \text{ g/h}$ . As-prepared  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders were calcined from  $700^\circ\text{C}$  to  $1000^\circ\text{C}$  for 2 h under the air atmosphere.

**2.2. Characterization of Powders.** The crystal phases of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders were identified with powder X-Ray Diffraction (XRD-6100, Shimadzu). The particle morphology, microstructure, and state of aggregation of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders were observed with a scanning electron microscope (SEM, JSM-6390, JEOL). The chemical component of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders was determined by induced coupled plasma analysis (ICP, SII, SPS-7800). Specific surface area of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders was measured by BET method (SSA, BEL Japan, BELSORP-miniII). The particle density of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders was determined with the pycnometer (Pycnomatic, Thermoelectron) using He gas.

**2.3. Electrochemical Measurement.** Cathode was prepared using 80 wt%  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders, 10 wt% acetylene black as a conductive agent, and 10 wt% polyvinylidene fluoride resin as a binder. They were homogeneously mixed

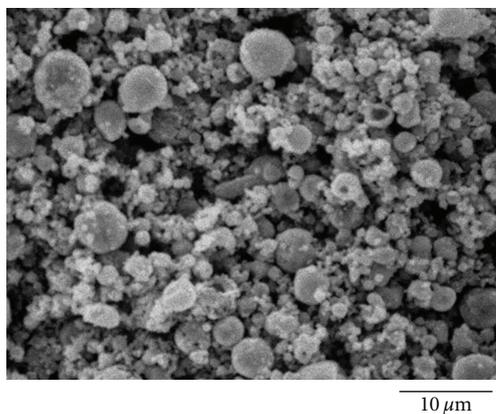


FIGURE 2: SEM photograph of as-prepared  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders.

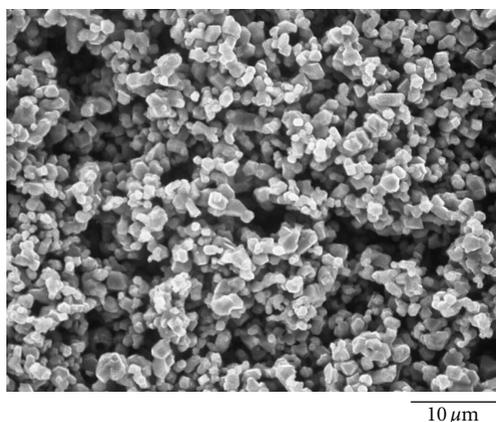


FIGURE 3: SEM photograph of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders calcined at  $800^\circ\text{C}$ .

with *N*-methyl-2-pyrrolidone. They were dried at  $120^\circ\text{C}$  for 24 h in a vacuum oven. Metal lithium sheet was used as the anode. Microporous polypropylene membrane was used as a separator.  $1\text{ mol/dm}^3$   $\text{LiPF}_6$  in ethylene carbonate/diethyl carbonate (EC:DEC = 1:1 in volume ratio) was used as the electrolyte. The coin type cell (CR2032) was assembled in a glove box filled with an argon gas. The rechargeable capacity and cycle stability of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode were measured by the rechargeable tester (BTS2004H, Hosen) from 3.5 V to 4.9 V at the rechargeable rate from 1 C to 20 C. The cycle stability of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode was also measured at  $50^\circ\text{C}$ .

### 3. Results and Discussion

**3.1. Particle Characterization of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  Powders.**  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders were continuously produced at rate of 200 g/h. Figure 2 shows an SEM image of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders obtained after 5 h. SEM image revealed that as-prepared  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  particles had spherical morphology with broad particle size distribution. The average particle size and geometrical standard deviation ( $\sigma_g$ ) of them were  $1.68\ \mu\text{m}$  and 1.34, respectively. The follow particles or spheroidal particles were also observed in Figure 2. This

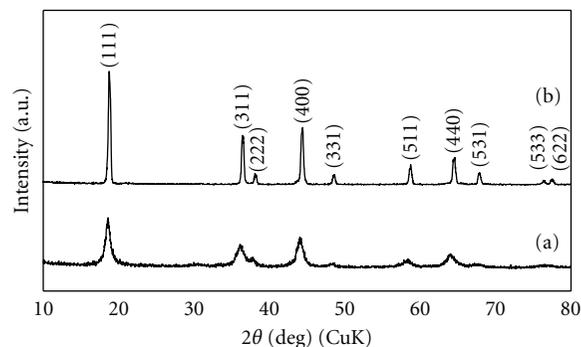


FIGURE 4: XRD patterns of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders, (a) as-prepared, (b) calcined at  $800^\circ\text{C}$ .

result suggested that the large mist derived from two-fluid nozzle atomizer with about nozzle size of  $10\ \mu\text{m}$  led to large particle size and broad size distribution of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders. Specific surface area and particle density of as-prepared  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders were  $24.6\ \text{m}^2/\text{g}$  and  $3.32\ \text{g}/\text{cm}^3$ , respectively. These results suggested that  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  particles obtained had the porous microstructure which was consisted of primary particles.

Figure 3 shows the particle morphology of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders calcined at  $900^\circ\text{C}$  for 2 h. When as-prepared  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders were calcined at  $900^\circ\text{C}$ , the primary particles were sintered to form uniform polygonal morphology. The average particle size and  $\sigma_g$  of them were  $1.01\ \mu\text{m}$  and 1.3, respectively. It was found that the particle size and size distribution became small and narrow. Specific surface area of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders decreased to  $1.03\ \text{m}^2/\text{g}$ . The particle density of them increased to  $4.17\ \text{g}/\text{m}^3$  due to the sintering of the primary particles. This led to the improvement for the dispersibility of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders in *N*-methyl-2-pyrrolidone solvent, so that the homogeneous slurry of them was obtained for the preparation of cathode.

Figure 4 shows XRD patterns of as-prepared  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders and  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders calcined at  $900^\circ\text{C}$  for 2 h. XRD revealed that the crystallinity of as-prepared powders was low, but that was well crystallized by the calcination at  $900^\circ\text{C}$ . It was seemed that these diffraction patterns of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders were identified with a cubic spinel structure with  $\text{Fd}3\text{m}$  of space group. The diffraction patterns of impurities except for spinel phase were not observed. From ICP analysis of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders calcined at  $900^\circ\text{C}$ , the molar ratio of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders was 1.0:0.49:1.49 and showed good agreement with that of starting solution. This suggested that  $\text{Li}^+$  ion,  $\text{Ni}^{2+}$  ion and  $\text{Mn}^{4+}$  ion were uniformly mixed at molecular level in each mist which was played a role as a microreactor.

**3.2. Electrochemical Properties of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  Cathode.** Figure 5 shows the charge and discharge curves of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode at discharge rate indicated. The plateau was observed at around 4.7 V in discharge curves of them. This was attributed to  $\text{Ni}^{2+}/\text{Ni}^{4+}$  redox couple [28].



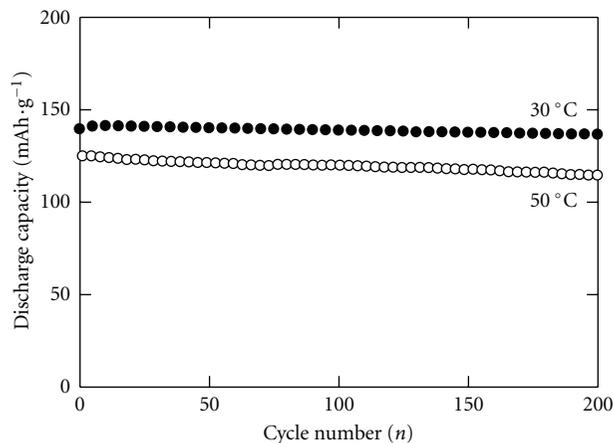


FIGURE 9: Relation between discharge capacity and cycle number at 30°C and 50°C.

and 10 C. 140 mAh/g of discharge capacity was maintained at 1 C. The discharge capacity of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode decreased to 130 mAh/g at 10 C.  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode had also good stability for the alternate cycle test at the higher and lower dischargeable rate. This suggested that the spinel structure of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  was also stable for intercalation of  $\text{Li}^+$  ion at the high and low rechargeable rate. This suggests that  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  spinel structure may lead to the high cycle stability for the volume change during the rapid intercalation of lithium ion.

Figure 9 shows the cycle performance of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode at 30°C and 50°C. The cycle test was examined at 1 C for 200 cycles. The discharge capacity of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode at 30°C was 135 mAh/g after 200 cycles. The capacity retention of it was 95%. The discharge capacity of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode at 50°C was 115 mAh/g after 200 cycles. The capacity retention of it was 91%. It was found that the cycle performance at 50°C was also stable as well as that at room temperature.

#### 4. Conclusions

$\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode materials were successfully produced by spray pyrolysis apparatus using the flame combustion. As-prepared  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders had spherical morphology with porous microstructure which had an average diameter of 1.68  $\mu\text{m}$  with broad size distribution. After the calcination,  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders with polygonal morphology and narrow particle size distribution were obtained.  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  powders were well crystallized after the calcination at 900°C and the chemical composition was in good agreement with that of starting solution. Rechargeable measurement of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode showed that the plateau was observed at 4.7V in discharge curve of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode and its discharge capacity was 146 mAh/g at 1 C. The capacity retention of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode was 95% at 1 C after 100 cycles. The discharge capacity and capacity retention of  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode was 125 mAh/g and 88% at 20 C.  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$  cathode exhibited stable cycle performance at 50°C.

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