

Microwave-assisted synthesis of LiNiO₂—a preliminary investigation

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Abstract

This paper presents the results of fundamental studies concerning the possibility of synthesizing LiNiO₂ by means of a microwave oven. Several attempts involving various precursors have been made with a view to identify suitable combinations of precursors that are microwave receptive and reactive in nature. Also, the optimization of reaction conditions, both in terms of microwave exposure time and period of post-heat-treatment required to synthesize phase-pure LiNiO₂, has been made. The optimum synthesis involves two steps, viz. exposure of precursor to microwaves for 5 min followed by a post-treatment of heating the samples in flowing oxygen or in air for about 5 h. Despite the involvement of two steps, this procedure reduces both processing time and cost. Among the various combinations of precursors chosen for the present study a microwave irradiated (5 min) LiOH–Ni(OH)₂ combination when subjected to post-heat-treatment in O₂ (5 h) results in the formation of a phase-pure LiNiO₂ sample, which exhibits good electrochemical performance (163 mAh g⁻¹) as a potential cathode material for lithium cells.

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Keywords: LiNiO₂; Microwave-assisted synthesis; Post-heat-treatment; X-ray diffraction; Electrochemical properties; Lithium cathodes

1. Introduction

Microwave energy has been in use for over 50 years in diversified fields such as communications, food processing, and textiles. The recent application of microwaves towards the synthesis of variety of organic compounds [1,2], e.g. esterification, hydrolysis, substitution, and polymerization, has opened up a new gateway in the modern era of synthetic chemistry. Further, it has been shown that microwave processing could be a viable route for the synthesis of many inorganic materials [3,4], the sintering of advanced materials and chemical analysis [2]. For instance, doped ZnS phosphor [5], nano CdSe, PbSe semiconductors [6], LaCrO₃ (an electrode/interconnector for solid oxide fuel cells) [7] are certain technologically important material that have been synthesized by microwave processing.

It is well known that, due to the high penetration depths of microwaves, its coupling with lossy materials results in rapid and uniform heating of the entire bulk of the reacting substance. This, in turn, minimizes the thermal gradients, reduces the time for particle diffusion and hence the prod-

uct can be obtained in a relatively short time. Consequently, sintering of ceramics using microwaves has been found to be advantageous in terms of reduction in particle size, sintering temperature, and firing time [8]. These parameters are reported to have significant effects on the characteristics of ceramics for commercial applications. The scope and use of microwave radiation for solid-state synthesis is restricted, however, owing to the limited availability of microwave susceptors [9].

Of late, a great deal of interest has been shown in the microwave-assisted synthesis of cathode materials such as LiMn₂O₄ [10,11], LiCoO₂ [12,13] and Li_{1.025}Mn_{1.975}O₄ [9] that are used in 4 V lithium batteries. On the other hand, no report on the microwave-assisted synthesis of the cathode material LiNiO₂ has appeared in the literature. Therefore, an attempt has been made in the present work to explore the possibility of synthesizing LiNiO₂ using microwave radiation and to optimize the irradiation time and post-heat-treatment conditions required for the formation of phase-pure LiNiO₂ with acceptable electrochemical characteristics. Also, with a view to examine the suitability of the microwave-assisted process and to have a better insight into the performance of microwave derived LiNiO₂ samples, a comparative study has been carried out of the performance of LiNiO₂ synthesized via the conventional solid-state method.

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2. Experimental

2.1. Synthesis procedure

Combinations of hydroxides, carbonates and oxides of lithium and nickel were selected to synthesize LiNiO₂ using microwaves, see Table 1. In a typical experiment, calculated amounts of selected precursors were homogenized mechanically and pelletized. The pellet was put in an alumina crucible and placed inside another larger alumina crucible, which was stuffed with glass wool. The purpose of using glass wool is mainly to avoid breaking and cracking of the glass turntable in the microwave oven by minimizing the heat transfer to the turntable. The whole assembly was kept at the centre of the turntable. The pellets were exposed to microwave radiation for various exposure times, e.g. 3, 5, 7 and 15 min. The reaction was triggered after 15–20 s with the appearance of orange colour glow, presumably due to the interaction of lithium ions with microwave radiation. The disappearance of the glow was taken as the sign of completion of the reaction for all the precursors attempted in the present study. Simultaneously, LiNiO₂ samples were synthesized by solid-state method also, using an intimate mixture of LiOH and Ni(OH)₂ (**A**) and LiOH and Ni₂O₃ (**B**). The mixtures on heating in an O₂ atmosphere at 700 °C for 21 h with intermittent grinding (to ensure phase homogeneity) resulted in the formation of fine powders of LiNiO₂.

It is very interesting to note that unlike the solid-state method, intermittent grinding of the pre-exposed reaction mixture was found to have no effective interaction with microwaves further, as indicated by the non-appearance of the receptive orange glow. Also, the expected rise in temperature was not observed, thus substantiating the non-interactive nature of the pre-exposed sample with microwaves due to intermittent grinding. In other words, the process of intermittent grinding does not appear to give any favourable result over microwave interaction with the pre-exposed samples, as far as the microwave-assisted synthesis of LiNiO₂ is concerned. Based on this, it may be concluded that ‘one-shot exposure’ (i.e. until the glow stops) to microwave radiations is ideal for optimizing the exposure time. It is considered that no such observation of the effect of intermittent grinding has been made by any research group to date for the microwave-assisted synthesis of lithium battery cathodes.

Table 1
Precursors chosen for microwave-assisted synthesis of LiNiO₂

Code	Lithium source	Nickel source	Interaction with microwaves
A	LiOH	Ni(OH) ₂	Positive
B	LiOH	Ni ₂ O ₃	Positive
C	LiOH	Basic nickel carbonate	Positive
D	Li ₂ CO ₃	Ni(OH) ₂	Positive
E	Li ₂ CO ₃	Ni ₂ O ₃	Positive
F	Li ₂ CO ₃	Basic nickel carbonate	Negative

2.2. Instruments

The synthesis of LiNiO₂ was attempted using an unmodified kitchen microwave oven (type no. WP700L17, Singer Ltd., India) rated at 700 W power output and with a frequency of 2.45 GHz. As the accurate measurement of the reaction temperature is not straightforward and requires rigorous modification of the oven, the present study focuses on the influence the time of exposure of the reactants to the microwaves.

The phase purity of the products was verified by X-ray diffraction analysis with a JEOL-JDX 8030 X-ray diffractometer. The diffractograms were recorded using nickel filtered Cu K α radiation ($\lambda = 1.5408 \text{ \AA}$) at room temperature in the 2θ range 10–80° with a scan rate set at 0.1° s⁻¹. Powder densities were measured based on the Archimedes’ principle using xylene as the liquid medium. The morphology of the powders was examined by means of a HITACHI S-3000 H scanning electron microscope, and the particle-size distribution was determined by laser diffraction with a Malvern Easy Particle Sizer. The surface area of the synthesized powders was determined by BET adsorption method using low-temperature nitrogen adsorption (Quanta Chrome Nova 1000, US).

Electrochemical performance was evaluated by assembling cathode-limited 2016 lithium coin cells. Cathodes were fabricated by slurring the cathode powders with 10 wt.% graphite and 2 wt.% PVdF as binder in *N*-methyl-2-pyrrolidone (NMP) as solvent, and coating the mixture over an Al foil current-collector. After drying at 110 °C overnight, the discs were pressed in a hydraulic press at a pressure of about 10–15 kg cm⁻² to achieve perfect adherence of the coated material over the surface of the current-collector. Discs of 1.6 cm diameter were punched out and typical cathodes were found to have an average coverage of active material of about 7 mg per disc. The electrolyte consisted of 1 M LiAsF₆ in 1:1 (v/v) EC:DMC (EC: ethylene carbonate; DMC: dimethyl carbonate), and polypropylene fabric was used as the separator. Charge–discharge studies were performed using an in-house cell-testing unit.

3. Results and discussion

The precursor **F** exhibits neither a rise in temperature nor the orange colour glow when the reactants were exposed to microwaves. Therefore, the possibility of using carbonates of lithium and nickel to synthesize LiNiO₂ was ruled out, as these compounds could not react effectively with the microwaves. By contrast, the rest of the combinations (**A–E**) are found to be microwave receptive in nature. Among the microwave receptive combinations of precursors, only combinations **A** and **B** have been chosen for further characterization in the present study. Actually, the preference for these combinations originates basically from the very objective of comparing the results of the microwave-assisted synthesis

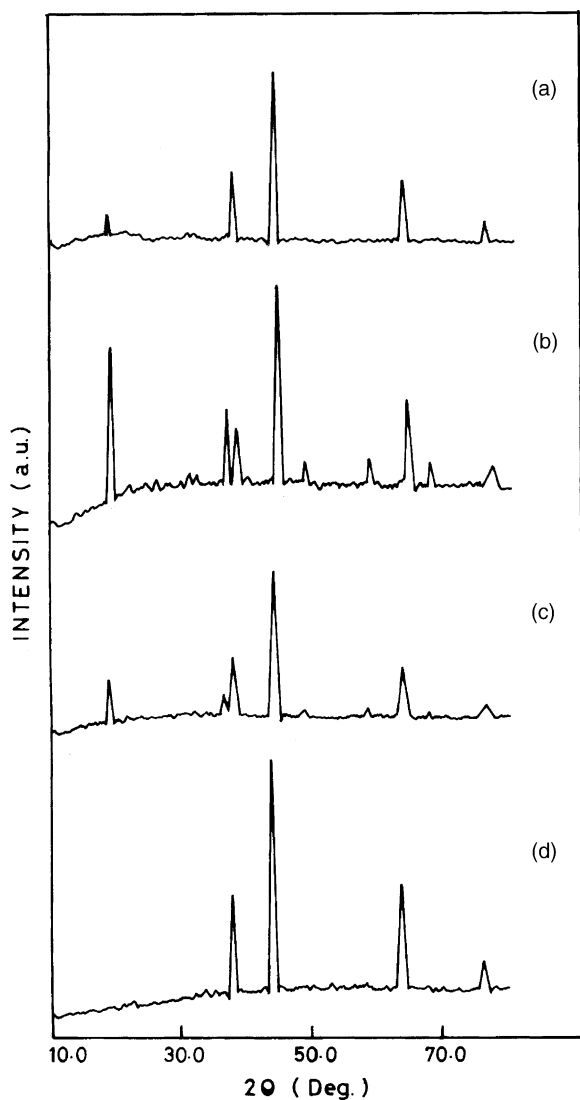


Fig. 1. XRD pattern of product obtained from microwave treatment of LiOH–Ni(OH)₂ precursor for: (a) 3 min; (b) 5 min; (c) 7 min; (d) 15 min.

and the solid-state method of synthesizing LiNiO₂ involving the same combinations of precursors.

3.1. Phase analysis by PXRD

The XRD patterns of the compounds derived from **A** and **B** using microwave irradiation for periods of 3, 5, 7 and 15 min are shown in Figs. 1 and 2, respectively. The combination **A** exposed for 3 min shows the evolution of LiNiO₂ phase, but an insufficient exposure time as evidenced from the reduced intensity of the peaks (Fig. 1a). On the other hand, the product obtained after an exposure time of 5 min (Fig. 1b) exhibits the presence of the characteristic (003) peak with a maximum intensity of approximately 90% against 100%, which is normally observed for phase-pure LiNiO₂. Therefore, in order to understand the effect of exposure time on phase purity, a systematic investigation involving longer exposure times such as 7 min (Fig. 1c) and 15 min

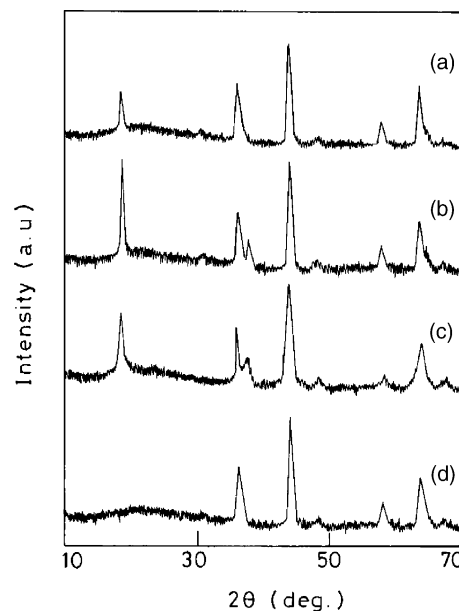


Fig. 2. XRD pattern of product obtained from microwave treatment of LiOH–Ni₂O₃ precursor for: (a) 3 min; (b) 5 min; (c) 7 min; (d) 15 min.

(Fig. 1d) were given to the same precursor individually. Surprisingly, with the increasing exposure time, the intensity of the (003) peak is found to decrease drastically and ultimately results in the mere formation of NiO or Li₂Ni₈O₁₀ phase at the end of the 7 and 15 min. From this observation, it is therefore assumed that longer exposure time could lead to a rise in temperature that may lead to the decomposition of LiNiO₂, which has already been formed from the initial microwave interaction. In other words, longer exposure time will be of little use as far as the microwave-assisted synthesis is concerned. Similarly the product obtained from precursor **B**, exposed for 5 min alone is found to exhibit a maximum (003) peak intensity of 70%, compared with that exposed for 3, 7 or 15 min (Fig. 2).

Since the product obtained from the combination **A** exhibits the (003) peak with an intensity as high as 90%, it may or may not require post-heat-treatment to improve the peak intensity further. On the other hand, for the combination **B**, it is considered that post-heat-treatment is highly essential to obtain better purity of the compound. Therefore, the products obtained from **A** and **B** were further subjected to post-heat-treatment at 700 °C in flowing O₂ and air for various times. The optimization of temperature required to synthesize phase-pure LiNiO₂ via the solid-state method has been carried out in one of our earlier studies [14] and from the satisfactory results obtained, a temperature of 700 °C has been also taken as the optimum post-heat-treating temperature for the present study. Here again, it is found that, irrespective of the type of precursor chosen, a heat-treatment for 5 h in flowing O₂ is found to be sufficient to obtain better sample purity, whereas longer heating times such as 10 and 15 h may not be required. Also, it is worth noting that a temperature of 700 °C is sufficient for the formation of

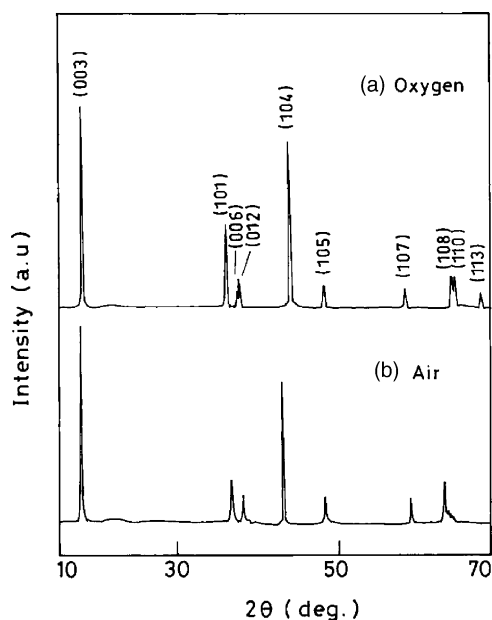


Fig. 3. Typical XRD pattern of LiNiO₂ synthesized from precursor A, post-treated in flowing (a) O₂ and (b) air for 5 h after 5 min exposure to microwaves.

phase-pure LiNiO₂ products against the reported temperature of 750–850 °C [15].

Further, it is quite interesting that the samples obtained from **A** and **B** with a post-heat-treatment in O₂ for 5 h and that derived from **A** heat treated in air for 5 h exhibited striking similarity and better crystallinity in their XRD patterns. On the other hand, the product obtained from combination **B** exhibits a slight mismatch in the XRD pattern, and thus in the related crystal constant values (when subjected to post-heat-treatment in flowing air for 5 h). From this observation, it may be inferred that a heat-treatment in flowing air 5 h would be inadequate for the combination **B** to yield phase-pure oxide. Hence, it is appropriate and advantageous to heat this precursor in flowing O₂ for just 5 h rather than in flowing air for a longer period. Typical XRD patterns of LiNiO₂ obtained from the ‘all-hydroxide’ **A** treated in O₂ and air are presented in Fig. 3. Consequently, it is understood that post-heat-treatment plays a vital role in enhancing the phase purity by way of improving the intensity of the

XRD peaks which, in turn, may lead to better electrochemical properties.

The crystal parameters calculated for various products obtained from the selected precursors of **A** and **B** treated under different conditions assuming a hexagonal setting (sp. gr. $R\text{-}3m\text{-}D_{3d}^5$) showed excellent matching (Table 2) with those of the JCPDS values [16]. From the $I_{(003)}/I_{(104)}$ ratio and c/a values (evaluated from the XRD patterns) of these post-heat-treated samples, it is confirmed that a treatment time of 5 h in the O₂ atmosphere is sufficient to obtain a product with high crystallinity and 2D structure, irrespective of the precursors employed. The sample derived from **B** in flowing air for 5 h has no distinct splitting of (108) and (110) peaks, which is an indication of a reduction in the 2D characteristics. Therefore, it is evident that the post-heat-treatment in oxygen atmosphere alone results in the formation of phase-pure LiNiO₂ when precursor **B** is used. In other words, heat-treatment in air is inefficient to synthesize pure LiNiO₂ using the microwave pre-exposed **B** type precursor. On the other hand, precursor **A** display only a slight improvement in peak intensity for both O₂ and air post-treated samples relative to the ‘as-derived’ LiNiO₂ product. Therefore, the actual choice, as well as the requirement of the post-heat-treatment can be confirmed only after subjecting these samples to systematic electrochemical characterization studies.

In an attempt to understand the effect of microwave irradiation on the selected set of precursors, two LiNiO₂ samples were synthesized from the combinations **A** and **B** by adopting the conventional solid-state method. The reacting mixtures were heated in an oxygen atmosphere for 5 and 21 h separately, with usual intermittent grinding [14]. The XRD pattern for LiNiO₂ obtained from **A** is shown in Fig. 4. Both precursors heat-treated for 5 h failed to give phase-pure LiNiO₂, whereas the XRD patterns and the crystal constants are quite similar for LiNiO₂ samples derived after 21 h of heat-treatment in flowing O₂. This observation agree with that of Ohzuku et al. [17] and confirms the requirement of at least 20 h of heating in O₂ atmosphere to obtain phase-pure LiNiO₂ by the solid-state method, irrespective of the nature of the precursor combination such as **A** and **B** chosen for the present study. A reduction in reaction time to the level of a minimum of 5 h is found, however,

Table 2

Crystal parameters, density and BET surface area of synthesized LiNiO₂ (theoretical density = 4.78 g cm⁻³)

Precursor	Post-heat-treatment	Crystal constants				Density (g cm ⁻³)	BET surface area (m ² g ⁻¹)
		<i>a</i> (Å)	<i>c</i> (Å)	<i>c/a</i>	$I_{(003)}/I_{(104)}$		
LiOH–Ni(OH) ₂ [A]	O ₂	2.873	14.193	4.940	1.29	4.111	9.8
LiOH–Ni(OH) ₂ [A]	Air	2.874	14.192	4.938	1.34	4.350	8.9
LiOH–Ni(OH) ₂ [A]	As-derived	2.870	14.182	4.941	1.23	4.158	9.4
LiOH–Ni(OH) ₂ [A]	O ₂ (21 h) (solid-state)	2.875	14.194	4.937	1.33	4.343	8.6
LiOH–Ni ₂ O ₃ [B]	O ₂	2.873	14.183	4.937	1.34	4.302	8.3
LiOH–Ni ₂ O ₃ [B]	Air	2.870	14.191	4.945	1.19	3.824	7.1
LiOH–Ni ₂ O ₃ [B]	O ₂ (21 h) (solid-state)	2.873	14.194	4.940	1.33	4.302	8.7

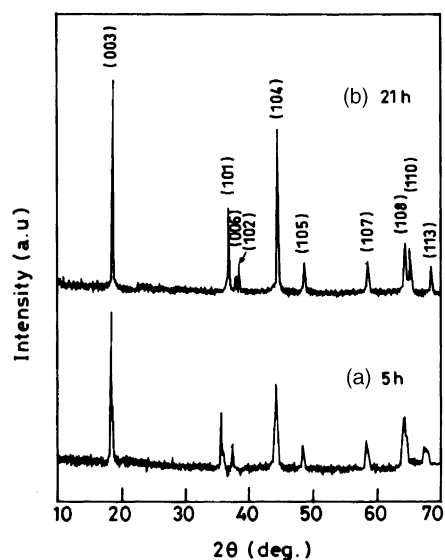


Fig. 4. Typical XRD and pattern of LiNiO_2 synthesized via solid-state route by heating precursor **A** in flowing O_2 for (a) 5 h and (b) 21 h.

to be sufficient when pre-treatment with microwaves has been adopted. Also, as the precursor **A** exposed for 5 min yields LiNiO_2 which exhibits a (003) peak with a relatively high intensity of 90%, O_2 post-heat-treatment is not mandatory. Hence, it is quite obvious that either an exposure time of 5 min under microwave radiation followed by a post-heat-treatment in a O_2 atmosphere for about 5 h, or a single step heat-treatment of the precursor in a O_2 atmosphere for at least 21 h (solid-state), is the ideal condition for the precursor **B** to synthesize phase-pure LiNiO_2 . On the other hand, the ‘all-hydroxide’ precursor **A** may require a microwave irradiation time of 5 min alone (as received) to synthesize LiNiO_2 . Consequently, a direct heat-treatment of **A** for 21 h (solid-state) or a post-heat-treatment of **A** in flowing air for 5 h yields LiNiO_2 of good purity. Evidently, post-heat-treatment in O_2 for 5 h has been found to enhance the phase purity to the optimum level, which is reflected in the electrochemical performance of LiNiO_2 (discussed below in Section 3.3) obtained from the ‘all-hydroxide’ precursor **A**. Hence, the application of microwave radiation for 5 min has been found to be significant and capable of reducing the total processing time from 21 to 5 h for both the precursors (**A** and **B**) selected for the present study.

3.2. Microstructure, particle size and distribution of post-treated LiNiO_2

A typical scanning electron micrograph of the LiNiO_2 samples obtained from precursors **A** and **B** post-treated in oxygen at 700°C for 5 h are depicted in Fig. 5a and b. The observed agglomeration is an indication of the highly sintered nature of the particles at 700°C . For all the compounds, discrete bands in the particle size range 2–5 μm and

observed from the particle-size distribution analysis. The particle-size distribution pattern for LiNiO_2 obtained from **A** is shown in Fig. 5c. For all the seven samples, the average specific surface area is found to be around $10\text{ m}^2\text{ g}^{-1}$ which is comparable with that obtained from the solid-state method reported in [18]. Similarly, the estimated powder density of all the seven samples is within 80–90% of the calculated values. This close agreement supports the fact that the product obtained from microwave method is well condensed and sintered. Physical parameters such as density, particle size and surface area, along with the crystal constant values derived from XRD data, are given in Table 2.

3.3. Electrochemical studies

Electrochemical characterization studies were conducted for a set of seven LiNiO_2 samples obtained from precursors **A** and **B** subjected to different reaction conditions. More details about the various types of LiNiO_2 samples obtained from different conditions are furnished in Table 2. The ‘as-derived’ product (without any post-heat-treatment) obtained from the microwave oven was also subjected to electrochemical characterization studies to understand the role and the necessity of post-treatment on the microwave exposed samples synthesized from precursor **A**.

To understand the electrochemical behaviour of LiNiO_2 , 2016 coin cells were assembled using the synthesized samples as cathodes against a lithium anode with 1 M LiAsF_6 in 1:1 (v/v) EC:DMC electrolyte. Charge–discharge studies were performed at a constant current of 0.1 mA cm^{-2} in the voltage range 3.3–4.3 V. The cycling performance of various $\text{Li}|\text{LiNiO}_2$ is given in Fig. 6.

The products obtained from the solid-state method using the precursors **A** and **B** show a maximum capacity of about 165 mAh g^{-1} . The microwave-exposed precursor **A** when post-heat-treated in O_2 for about 5 h exhibits a similar discharge capacity ($\sim 163\text{ mAh g}^{-1}$) during the first cycle. This result demonstrates the feasibility and the practically viable nature of synthesizing LiNiO_2 with microwaves.

Detailed studies of the performance of LiNiO_2 synthesized by the microwave-assisted method reveal considerable differences in capacity with respect to the type of precursor employed (**A** or **B**) and the nature of post-heat-treatment (O_2 or air). For example, with the post-heat-treated **A** precursor, the product obtained from O_2 heat-treatment exhibits a higher discharge capacity (163 mAh g^{-1}) than that obtained from heating in air (157 mAh g^{-1}). Further the ‘as received’ precursor **A** obtained from a ‘single-shot’ microwave exposure yields a higher (comparable with **A** heat-treated in air) capacity (155 mAh g^{-1}) than any of the samples derived from the post-treated precursor **B** ($\sim 120\text{ mAh g}^{-1}$). From this, it is concluded that microwave interactions have a specific preference for ‘all-hydroxides’ precursor **A**, presumably due to the polar nature of the reactants. On the other hand, microwave interactions are found to have least preference for ‘all carbonate’ precursors **F** (as evidenced by the

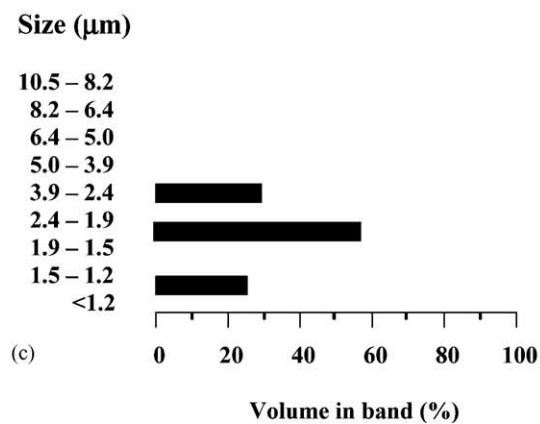
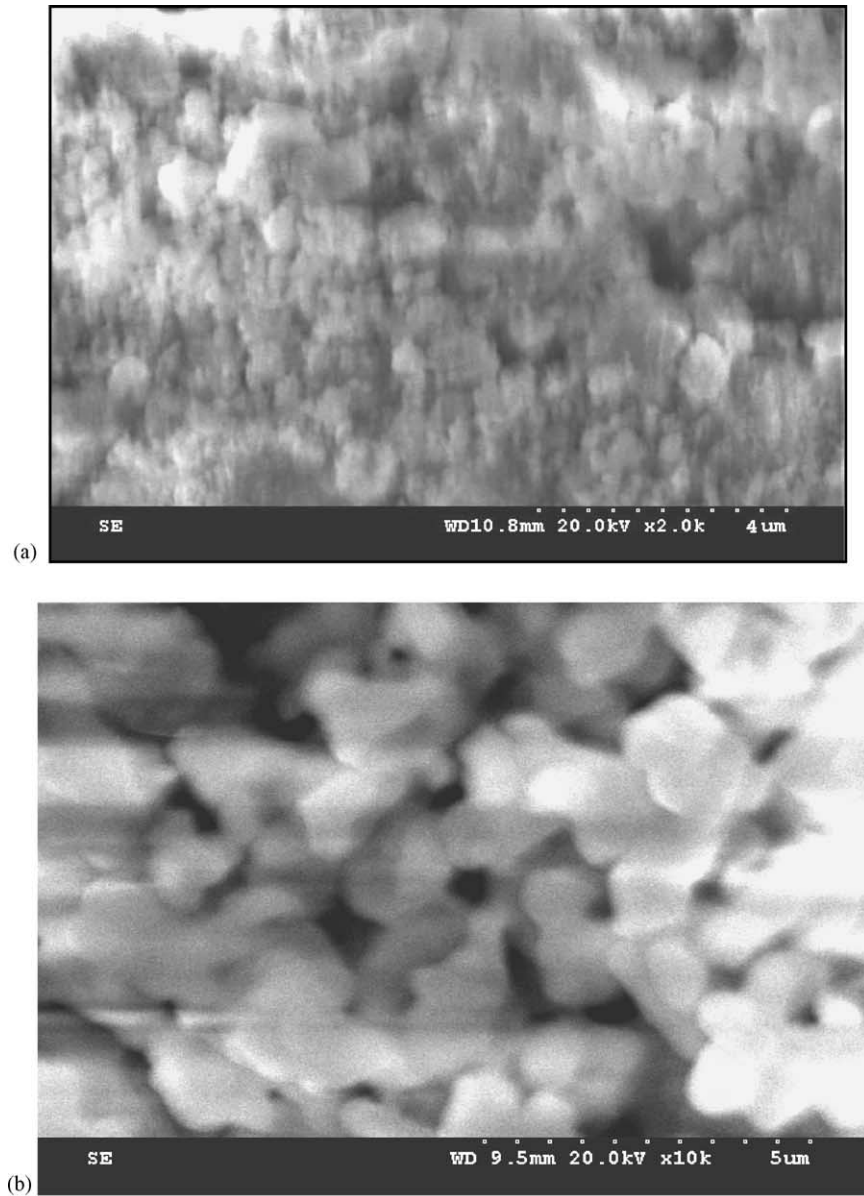


Fig. 5. Electron micrograph of (a) LiNiO_2 synthesized from O_2 post-heat-treatment of precursor A; (b) LiNiO_2 synthesized from O_2 post-heat-treatment of precursor B; (c) typical particle-size distribution pattern observed for LiNiO_2 from O_2 treated precursor A.

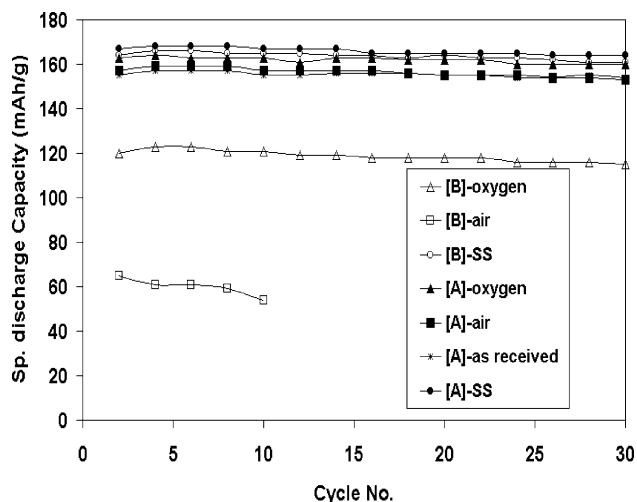


Fig. 6. Cycling behaviour of LiNiO_2 samples synthesized by various treatments (current density = 0.1 mA cm^{-2}).

non-appearance of a orange glow) based on the aforesaid polarity reasons.

For the post-heat-treated samples of precursor **B**, the O_2 post-treated product gives a higher capacity (120 mAh g^{-1}) than the air-heated counterpart (65 mAh g^{-1}). Therefore, it is clear that the products obtained from precursor **B**, even after post-heat-treatment display inferior electrochemical behaviour compared with precursor **A** with and without post-treatment. In summary, the capacities of the seven samples of LiNiO_2 are in the order:

$$\begin{aligned} \mathbf{A}_{\text{ss}} &\approx \mathbf{B}_{\text{ss}} \approx \mathbf{A}_{(\text{oxygen})} > \mathbf{A}_{(\text{air})} \\ &\approx \mathbf{A}_{(\text{as received})} > \mathbf{B}_{(\text{oxygen})} > \mathbf{B}_{(\text{air})} \end{aligned}$$

where ss denotes the solid-state method. Hence, it is concluded that the microwave-treated products derived from **A** exhibit more or less similar discharge capacity comparable with those of the compounds obtained from solid-state method, irrespective of the precursors used. This in turn substantiates the fact that the microwave-assisted synthesis is a viable process by which LiNiO_2 with improved electrochemical performance can be synthesized.

Also, based on the discharge capacity values shown by LiNiO_2 derived from different treatments, it may be concluded that the combination **A** with polar reactants is the preferred precursor as far as microwave-assisted synthesis of LiNiO_2 is concerned. Similarly, optimization of post-heat-treating conditions may also be derived from comparison of the electrochemical performance of microwave-assisted products. That is, LiNiO_2 obtained directly from microwave irradiation of **A** (for about 5 min) is capable of showing better performance (155 mAh g^{-1}), an indication that post-heat-treatment is optional only. On the other hand, the **B** precursor essentially requires post-heat-treatment wherein heating in an O_2 atmosphere results in a higher capacity (120 mAh g^{-1}) compared with the air-heated sam-

ple (65 mAh g^{-1}). Such a difference in discharge capacity values is not observed with precursor **A** when post-heated either in O_2 (163 mAh g^{-1}) in air (157 mAh g^{-1}).

The first discharge capacity of the microwave irradiated sample **A** followed by O_2 post-heat-treatment gives about 160 mAh g^{-1} , which is considerably higher than the values (135 mAh g^{-1}) reported by Chang and Kumta [19]. Interestingly, the discharge capacity values are maintained up to at least 30 cycles, without much capacity fade. That is, at the end of the 30th cycle, the discharge capacity of **A** is 164 mAh g^{-1} for the solid-state product and an average of 157 mAh g^{-1} for the post-heated, microwave irradiated samples, i.e. a capacity fade of less than 5%. Similarly, the product obtained from **B** after 30 cycles gives 164 mAh g^{-1} (solid-state method) and 115 mAh g^{-1} (microwave exposure followed by O_2 post-treatment) compared with 167 and 120 mAh g^{-1} on the first discharge capacity, respectively. The higher capacity values of both the solid-state and microwave method (using **A**) may very well be correlated with qualitative observations of (1 0 8) and (1 1 0) peak splitting and $I_{(003)}/I_{(104)}$ values of ca. 1.3 derived from XRD data [20].

By contrast, poor cycling performance has been observed for the sample synthesized from **B** heated in air, which is evident from Fig. 6. Within 10 cycles, the capacity decreases from 65 to 54 mAh g^{-1} , a value which corresponds to only 50% of those reported. This may be explained on the basis of possible cation mixing [20] and a reduction in the 2D characteristics of LiNiO_2 . The combination of these two factors is expected to hinder Li^+ diffusion and, thereby, increase unwanted polarization and cell impedance, which ultimately lowers the charge retention ability of the oxide material. Hence, the observed inferior electrochemical activity of the product obtained from **B** can be understood.

4. Conclusions

In the present study, the possibility of synthesizing LiNiO_2 using a microwave-assisted method has been examined with the selected precursor combinations $\text{LiOH-Ni}(\text{OH})_2$ (**A**) and $\text{LiOH-Ni}_2\text{O}_3$ (**B**). Generally, a two-step process which involves an initial microwave heating of the precursor for 5 min followed by an oxygen post-heat-treatment for 5 h has been found to be optimum. More specifically, the process depends upon the type of precursor employed. All the microwave-treated and the post-heated LiNiO_2 samples examined in this study are found to be phase-pure (except of $\text{LiOH-Ni}_2\text{O}_3$ heated in air) crystalline, and sintered with acceptable battery activity. It is also observed that intermittent grinding, which is part of the conventional method of preparation causes insignificant interaction of precursors with further microwave exposure. Therefore, the highlight of this method is that a combination of microwave irradiation without intermittent

grinding and a post-heat-treatment is preferable and faster than conventional furnace heating (at least 20 h) procedures. Of all the products obtained from different treatments, LiNiO₂ synthesized using precursor **A** with subsequent post-heat-treatment in O₂ for 5 h gives the best electrochemical performance. A discharge capacity value of about 160 mAh g⁻¹ (after 30 cycles) is achieved with the optimized synthesis conditions of **A**. Besides being simple in nature, the microwave method is cost-effective and energy efficient. Also, the electrochemical performance of the products are comparable with that of products made by the conventional ceramic route. Nevertheless, there is scope for improvement of the microwave method of synthesizing LiNiO₂ in terms of the precursor combination, the usage of secondary microwave susceptors, the in situ generation of an O₂ atmosphere. In addition, extensive electrochemical characterization by means of cyclic voltammetry may assist the understanding of battery activity of LiNiO₂ synthesized via microwave-assisted synthesis.

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