CHAPTER 3

EXPERIMENTAL METHODS

3.1 Introduction

Among researchers studying on the inverse spinel $LiNiVO_4$, many of them have reported their work on the cathode material as they found their interest on the compound for having such high cell employing voltage of 4.8 V. They have offered a lot of information on the structural studies, morphologies, the electrochemical studies as well as suggesting new method of synthesizing the cathode material. Almost all researchers reported on the structural, physical, chemical and electrochemical based studies using the x- ray diffraction (XRD) analyses, scanning electron microscope (SEM) analyses, transmission electron microscope (TEM) analyses, and cyclic voltammetry (CV) analyses.

3.2 Sample preparation

There are two types of synthesized method to prepare the $LiNiVO_4$ cathode material. The sol- gel method and polymer precursor technique were implemented to prepare the inverse spinel $LiNiVO_4$ cathodes. Few raw materials for preparing the cathode were used such as lithium acetate dihydrate (Fluka), nickel (II) acetate tetrahydrate (Fluka), manganese (II) acetate tetrahydrate (Aldrich), and ammonium metavanadate (Aldrich).

Table 3.1 depicts the prepared sample with the raw materials used for both methods while Figure 3.1 shows the dissolved raw materials used in this work.

Table 3.1: The prepared $LiNiVO_4$ cathode material samples with the raw materials.

Methods	Raw materials	Product
Sol- gel	1. $LiCH_3COOH.2H_2O$, Ni $(CH_3COOH)_2$. $4H_2O$, NH_4VO_3	$LiNiVO_4$
Sol- gel	2. $LiCH_3COOH.2H_2O$, Ni $(CH_3COOH)_2$. $4H_2O$, $Mn(CH_3COOH)_2$. $4H_2O$, NH_4VO_3	$LiNi_{1-x}Mn_xVO_4$
Polymer Precursor	3. $LiCH_3COOH.2H_2O$, Ni $(CH_3COOH)_2$. $4H_2O$, NH_4VO_3	$LiNiVO_4$+ PEG
Polymer Precursor	4. $LiCH_3COOH.2H_2O$, Ni $(CH_3COOH)_2$. $4H_2O$, $Mn(CH_3COOH)_2$. $4H_2O$, NH_4VO_3	$LiNi_{1-x}Mn_xVO_4$+ PEG

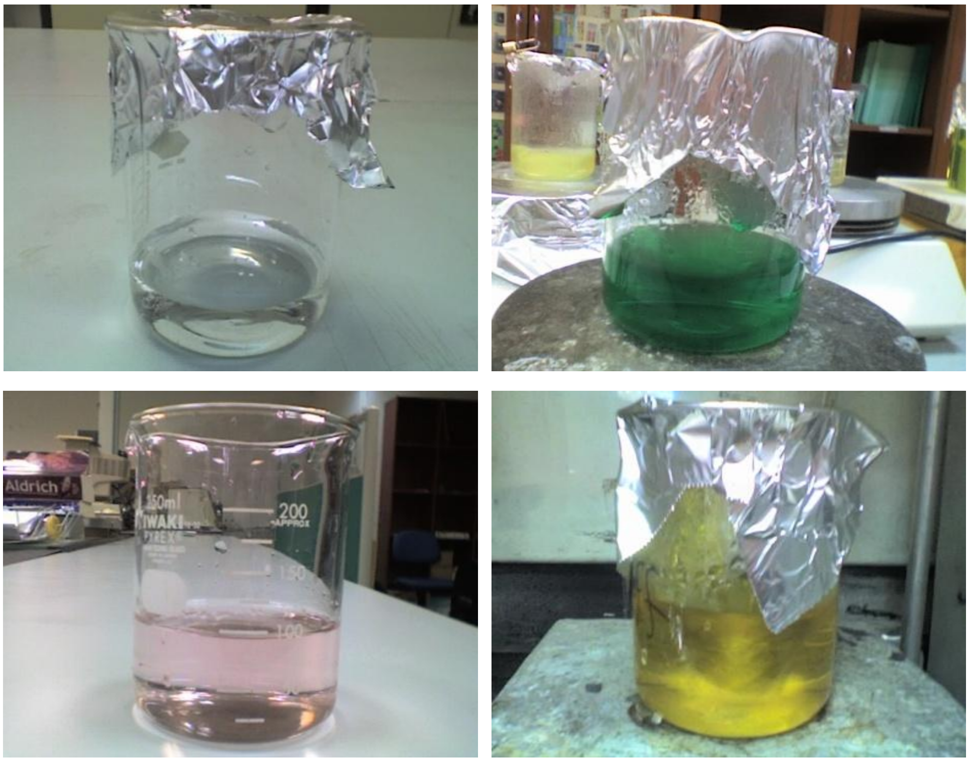


Figure 3.1 The starting materials

3.2.1 $LiNiVO_4$ Sol-gel System

The synthesized $LiNiVO_4$ precursor was prepared by the sol-gel method. The raw material used were $LiCH_3COO.2H_2O$, $Ni(CH_3COO)_2.4H_2O$ and NH_4VO_3. Figure 3.2 shows the sample preparation for $LiNiVO_4$ prepared by the sol gel method. Each mentioned material was weighed according to $LiCH_3COO.2H_2O$: Ni $(CH_3COO)_2.4H_2O$: NH_4VO_3 at (1:1:1) mole ratio and dissolved separately in distilled water. Then, those materials were mixed under homogenizer to ensure homogeneity. The mixed solution was then heated under constant stirring on a hot plate. The citric acid was added in 1:1 mole of total metal cation to the citric acid ratio. The citric acid acts as the complex agent and helps the improvement in electronic conductivity of the prepared cathode [Li *et. al*, 2007]. The ammonium hydroxide was poured into the beaker until clear solution was observed. After a while, a gas will evolve as the solution will gradually transform into jelly form. The gel was then further heated until the gel forms powder. The powder which is the intermediate compound is now a precursor for the cathode material. Then, the precursor was calcined at different temperatures ranging from 500 °C to 800 °C. Those precursors were sintered using a furnace (Naberham Program Controller). Each sample was then slowly cooled down to room temperature before removing the sample from the furnace. Table 3.2 shows the relative formula mass of each starting material used in this work.

Table 3.2: The raw materials for $LiNiVO_4$ prepared by the sol gel method.

Raw Material	Abbrv.	Relative Formula Mass (g/mole)
$LiCH_3COO.2H_2O$	(LiOAc)	102.02
$Ni(CH_3COO)_2.4H_2O$	(NiOAc)	248.86
NH_4VO_3		116.98

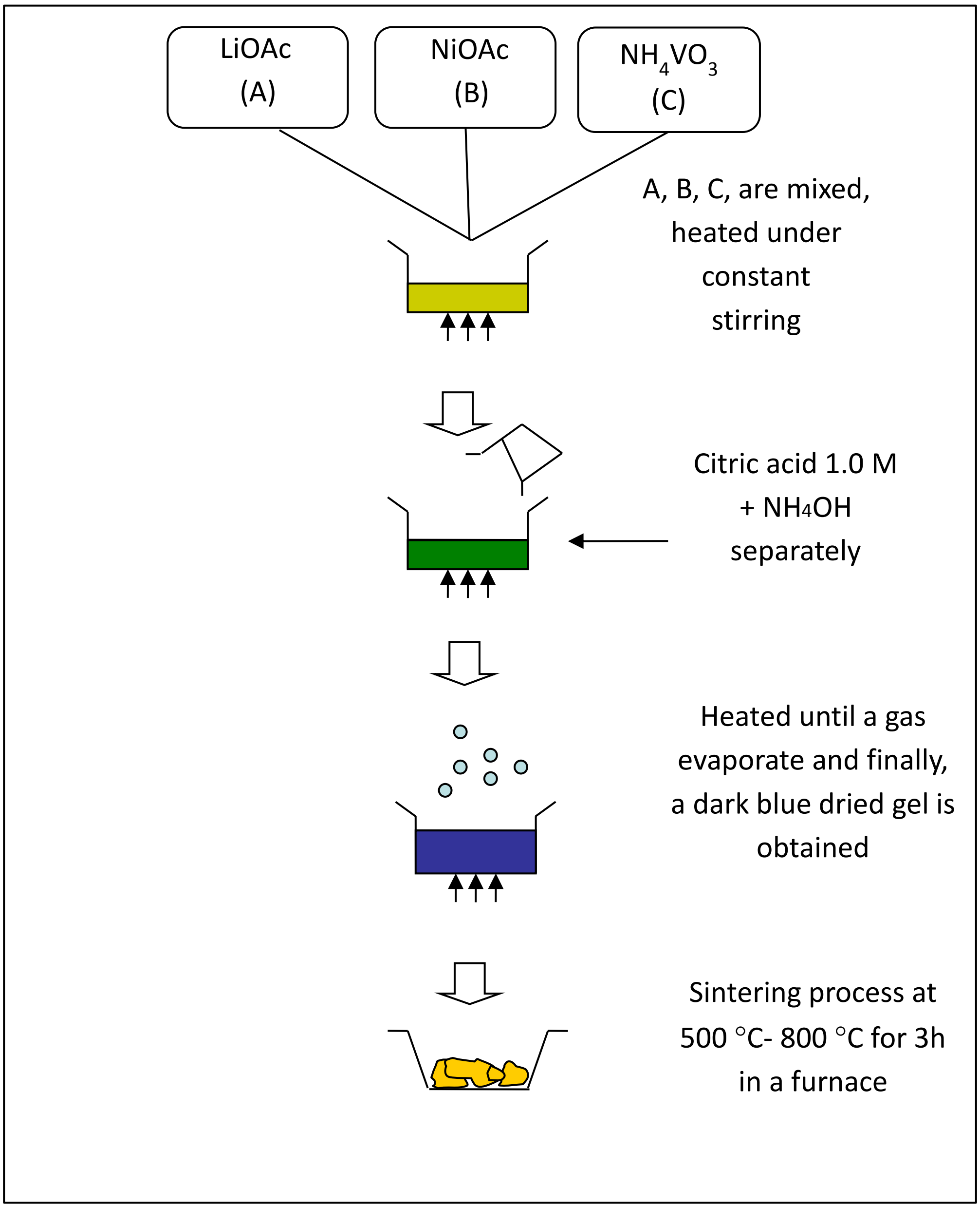


Figure 3.2 Sample Preparation for $LiNiVO_4$ prepared by the sol gel method

3.2.2 $LiNiVO_4$ Polymer Precursor System

The second system for preparing the $LiNiVO_4$ was synthesized using polymer precursor method. The polymer used in the system is poly (ethylene glycol) (Mw: 20, 000) or PEG. The PEG used was weighed and dissolved in 1wt % of acetic acid. The raw materials used were the same as the sol-gel method that are $LiCH_3COO.2H_2O$, $Ni(CH_3COO)_2.4H_2O$ and NH_4VO_3. The sample preparation for the $LiNiVO_4$ by polymer precursor method is displayed in Figure 3.3. Those starting material were dissolved separately in distilled water. Those dissolved materials were mixed using homogenizer to ensure complete mixing. The mixed solution was heated under constant stirring on a hot plate. The citric acid having 1:1 mole ratio to the total metal cations was poured into the solution. The acid was added as a carbon source and complexing agent [Hong *et. al,* 1997]. Moreover, the acid can help the better distribution of metal cation between the polymer matrixes in the solution [Fey *et. al,* 2006a; Fey *et. al,* 2006b]. Then, the ammonium hydroxide was added onto the beaker. The mixed solution was then left heated until a gas evolution occurs in the mixture. The solution will gradually turn into gelled form. The gel was further heated until the powder was obtained. The precursor was sintered in different temperature ranging from 500 °C to 800 °C in 3 hours separately in a furnace (Naberham Program Controller). Those calcined samples were left cooled to room temperature before those were removed from the furnace. Table 3.3 shows the relative formula mass of each starting material used in this work.

Table 3.3: The raw materials for $LiNiVO_4$ prepared by the polymer precursor method.

Raw Material	Abbrv.	Relative Formula Mass (g/mole)
$LiCH_3COO.2H_2O$	(LiOAc)	102.02
$Ni(CH_3COO)_2.4H_2O$	(NiOAc)	248.86
NH_4VO_3		116.98
PEG		(Mw: 20, 000)

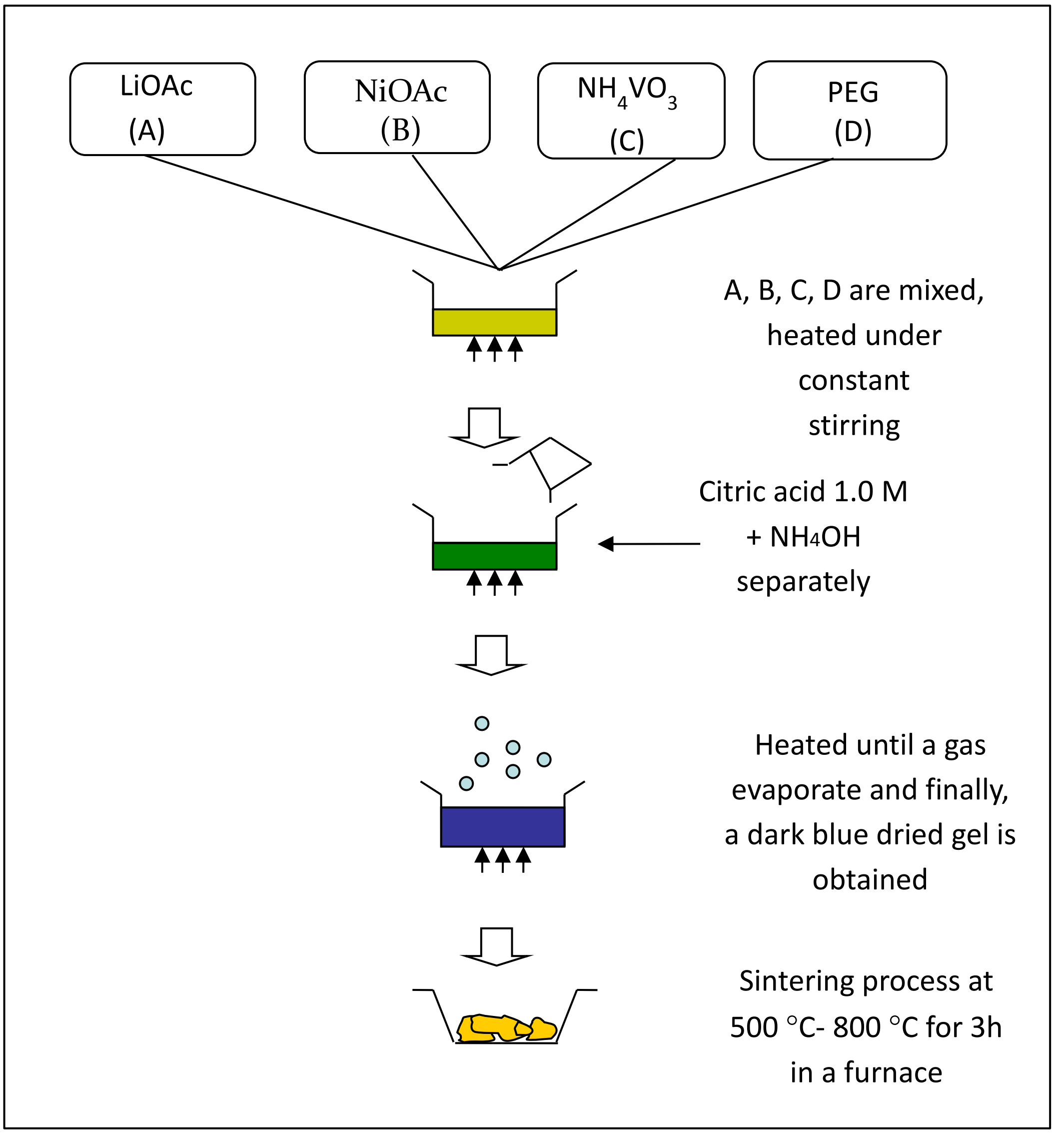


Figure 3.3 Sample preparation for $LiNiVO_4$ prepared by the polymer precursor method

3.2.3 $LiNi_{1-x}Mn_xVO_4$ Sol-gel System

The cathode materials of $LiNi_{1-x}$ Mn_xVO_4 ($0 \leq x \leq 1$) were prepared using the sol-gel method. To prepare the $LiNi_{1-x}$ Mn_xVO_4 ($0 \leq x \leq 1$), required amounts of manganese acetate tetrahydrate $Mn(CH_3COO)_2.4H_2O$ (Aldrich) were mixed with lithium acetate dihydrate, nickel acetate and ammonium metavanadate and dissolved in distilled water. Figure 3.4 shows the synthesis of the doped system $LiNi_{1-x}$ Mn_xVO_4 ($0 \leq x \leq 1$). The solutes and solvent mixture stirred with constant heating until a homogeneous solution was obtained. Citric acid and ammonium hydroxide, NH_4OH solutions were added into the homogeneous mixture until the evolution of gas was observed. The citric acid to the total metallic cation of starting material used was fixed to 1:1 mole ratio. The acid acts as the complexing agent and helps to improve the electronic conductivity of the cathode material [Li *et. al*, 2007]. In other words, the bonded complexes between the metal cations and acid addition in the mixture will be easy to obtain homogeneous and nanosized particles [Fey *et. al*, 2007]. After the occurrence of gas, a dark blue dry gel was formed. The gel obtained was further heated to form a precursor. The precursor was subjected to the thermogravimetric analysis (TGA). Results from TGA led to sintering of the powder precursor from 500 °C to 800 °C for 3 hours. The calcined sample was left cool before it was taken out from the furnace. Table 3.4 shows the relative formula mass of each starting material used in this work.

Table 3.4: The raw materials for $LiNi_{1-x}Mn_xVO_4$ (0≤ x ≤1) prepared by the sol gel method.

Raw Material	Relative Formula Mass (g/mole)
$LiCH_3COO.2H_2O$	102.02
$Ni(CH_3COO)_2.4H_2O$	248.86
$Mn (CH_3COO)_2.4H_2O$	245.09
NH_4VO_3	116.98

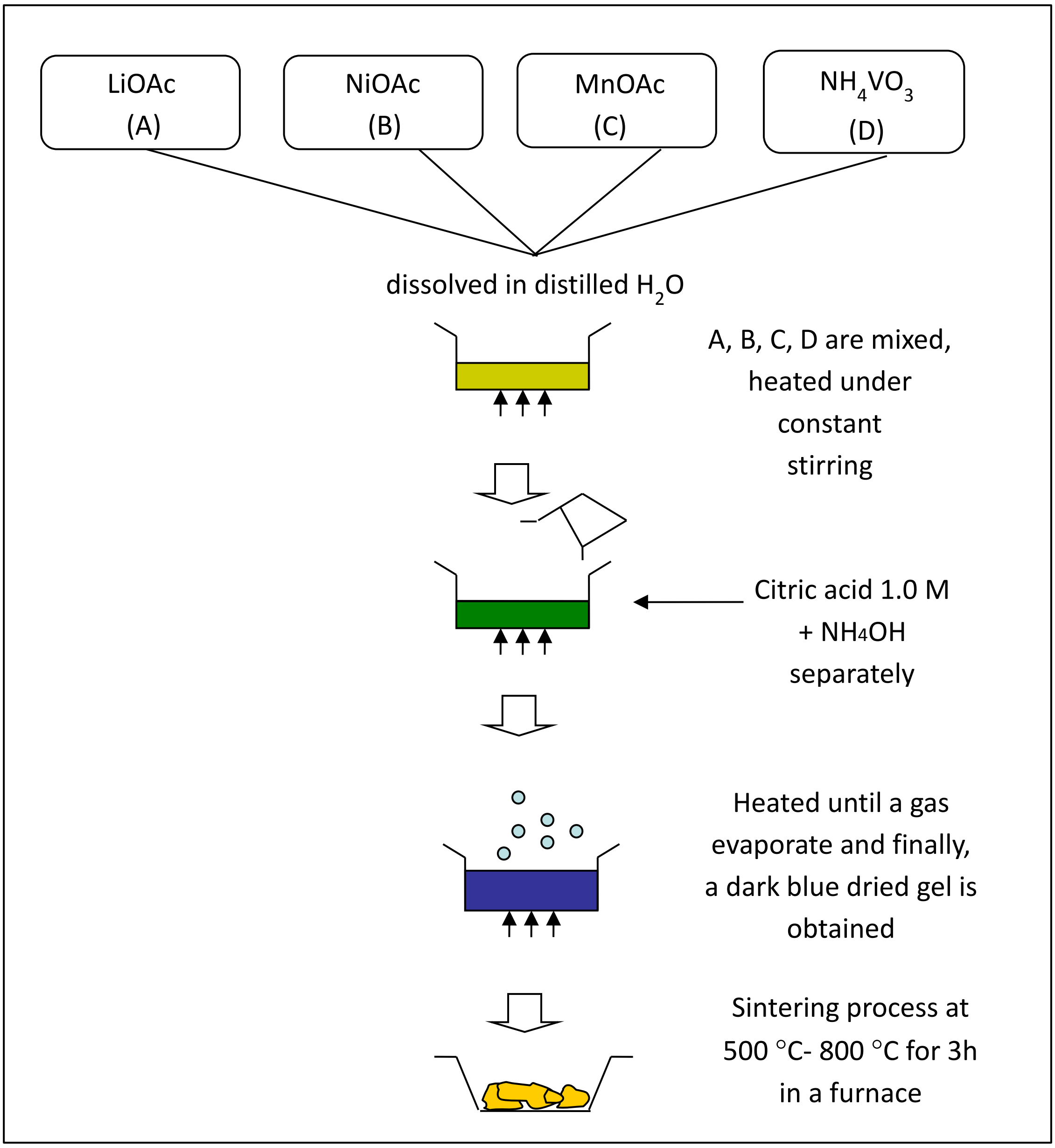


Figure 3.4 Sample preparation for $LiNi_{1-x}Mn_xVO_4$ prepared by the sol gel method

3.2.4 $LiNi_{1-x}Mn_xVO_4$ Polymer Precursor System

Lithium acetate dihydrate, $LiCH_3COO.2H_2O$ (Fluka), nickel acetate tetrahydrate Ni $(CH_3COO)_2.4H_2O$ (Ajax Chemicals), Mn $(CH_3COO)_2.4H_2O$ (Aldrich) and ammonium metavanadate NH_4VO_3 (Ajax Finechem) and PEG were used as the starting materials for polymer precursor method. The synthesis of the doped polymer system is represented in Figure 3.5. Those raw materials were weighed according to mole ratio of Li: Ni: Mn: V= 1:1:1:1 before dissolved separately in distilled water. Dissolved solutions were mixed under constant stirring. A homogenizer was used to ensure that the mixed solution blends well. The mixture was then added with citric acid followed by ammonium hydroxide. A clear greenish solution should appear after the acid was added. The citric acid acts as the chelating agent and helps the improvement in electronic conductivity of the prepared cathode [Li *et. al*, 2007]. The chelating agent has ligands where the acid can merge with positive cations in which the positive ions are distributed homogeneously in the gel precursor [Song and Lee, 2002]. The solution was left heated until a gas reaction came out until a viscous blue gel was formed. The viscous gel was further heated to form a loose powder called as a precursor. The precursor was then sintered at various temperatures ranging from 500 °C to 800 °C for 3 hours separately. Table 3.5 shows the relative formula mass of each starting material used in this work. The actual picture of the sample during initial preparation process, the gas evolution process and the final product are represented in Figure 3.6, Figure 3.7 and Figure 3.8 respectively.

Table 3.5: The raw materials for $LiNi_{1-x}Mn_xVO_4$ ($0 \leq x \leq 1$) prepared by the polymer precursor method.

Raw Material	Relative Formula Mass (g/mole)
$LiCH_3COO.2H_2O$	102.02
$Ni(CH_3COO)_2.4H_2O$	248.86
$Mn (CH_3COO)_2.4H_2O$	245.09
NH_4VO_3	116.98
PEG	(Mw: 20, 000)

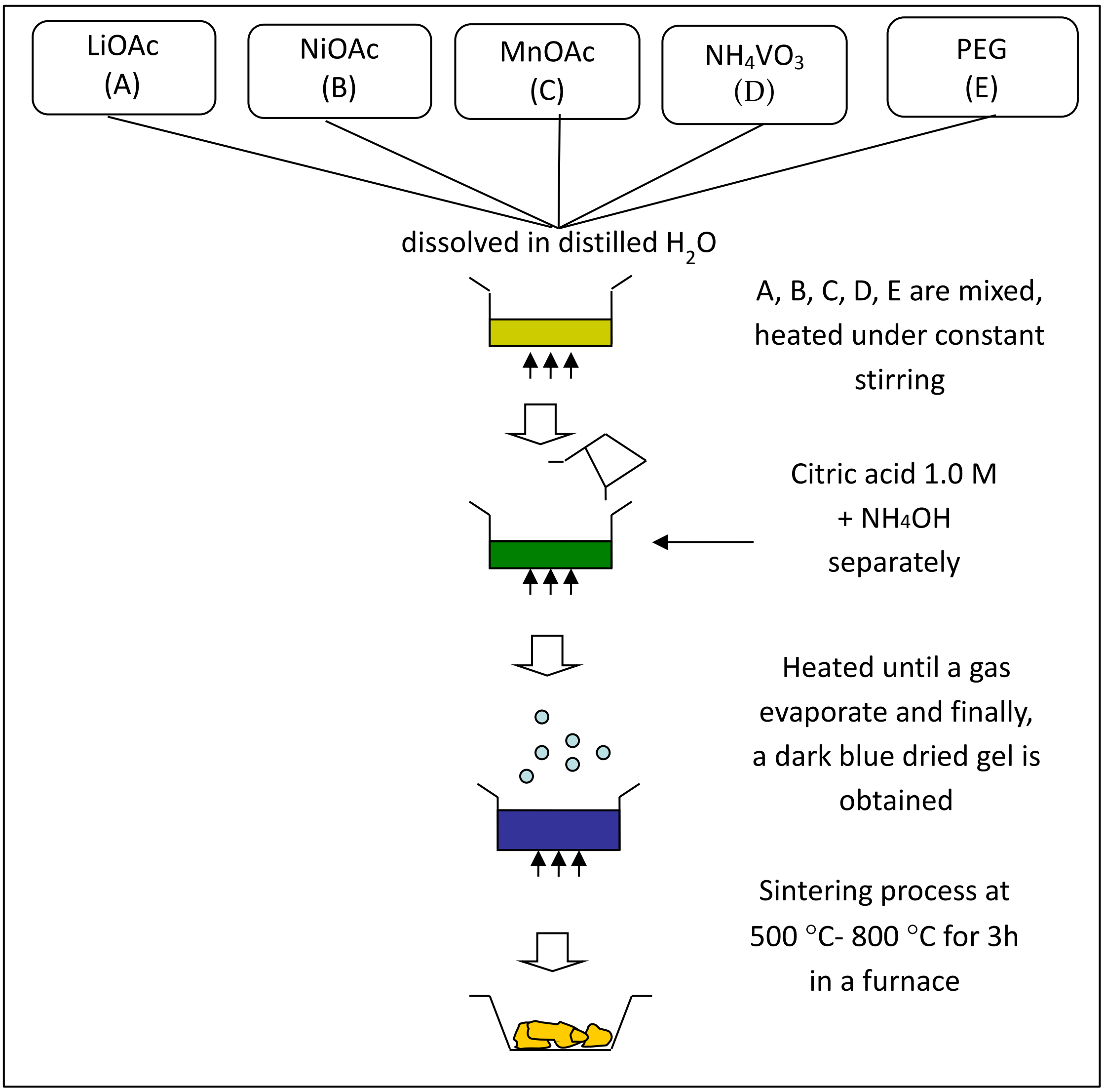


Figure 3.5 Sample preparation for $LiNi_{1-x}Mn_xVO_4$ prepared by the polymer precursor method

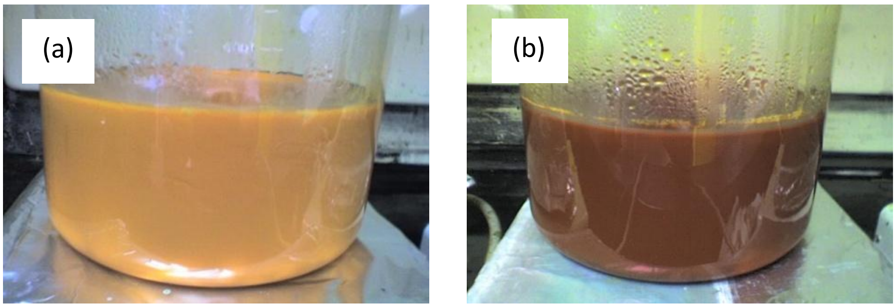


Figure 3.6 (a) The mixed solution after all raw materials are added; (b) The mixed solution under heating

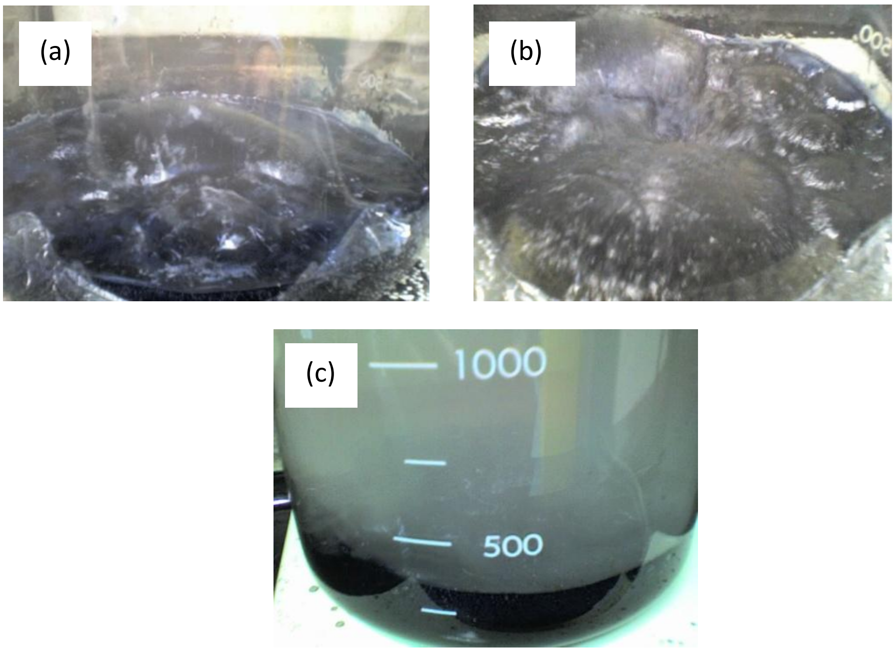


Figure 3.7 (a) The solution turns dark blue after continuous heating; (b) The mixed solution becoming gel-like solution; (c) The gas evolving process throughout the sample

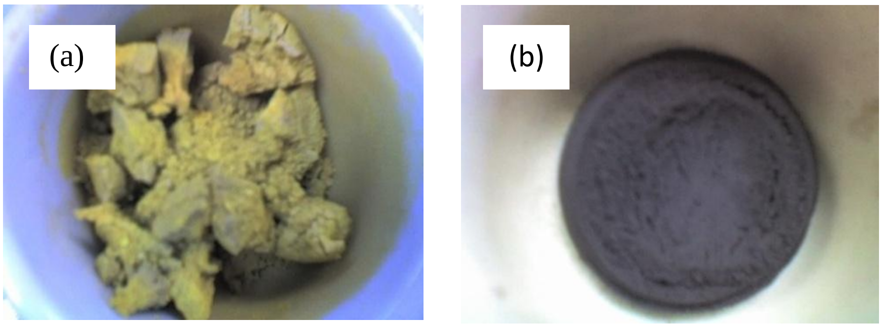


Figure 3.8 The sample after sintering process (a) $LiNiVO_4$ system; (b) $LiNi_{1-x}Mn_xVO_4$ system

3.3 X-ray Diffraction (XRD)

The X-ray diffraction (XRD) technique is the analytical measurement which gives the information about crystallographic structure, the chemical composition, and also the physical properties of materials. These non-destructive analyses are based on observing scattered intensity of an X-ray beam hitting a sample as a function of incident and scattered angle, polarization, and wavelength or energy. X-ray diffraction will give the information on the atomic structure of materials. The theta angle between the incident beam and normal to the powder will vary as the sample rotates. Bragg's Law, describes the condition for constructive interference from successive crystallographic planes (*h*,*k*,*l*), of the crystalline lattice [Myers, 2002]:

$$2d \sin \theta = \mathrm{n}\,\lambda \tag{3.1}$$

From the equation above, d is the interplanar spacing. θ is the Bragg angle, n is the order of diffraction and λ =1.54 Å is the X-ray wavelength.

The synthesized compounds were characterized for powder diffraction (XRD) analysis. The powder diffraction (XRD) technique is used to determine the crystallographic structure, crystallite size, and desirable orientation in neither powdered solid sample nor polycrystalline. Generally, the powder diffraction is used to determine the unknown compounds by comparing diffraction data over the database provided by the International Centre for Diffraction Data. The diffraction analyzer can also be used to identify the heterogeneous solid mixtures to study the relative abundance of crystalline compounds. The strains studies in crystalline materials are also commonly determined by the powder diffraction method. The phase purity and the structural type of the compound can be determined by the XRD data [Prabaharan *et. al*, 1997; Kalyani, 2002].

The broadening peaks in an X-ray diffraction can be obtained to study the effect of the finite crystallite sizes by applying the Scherrer Equation. The crystallite size plot calculated by Sherrer equation is given below:

$$d = \frac{0.9\,\lambda}{\mathrm{B}\cos\theta} \qquad (3.2)$$

where; d is the crystallite size,

B is the line broadening (FWHM in radian),

θ is Bragg’s angle.

The example of XRD pattern is shown in Figure 3.9. Lu and Liou had reported that the phase formation of $LiNiVO_4$ prepared by the solid state method can be affected by the soaking time variation at different sintering temperatures [Lu and Liou, 1999].

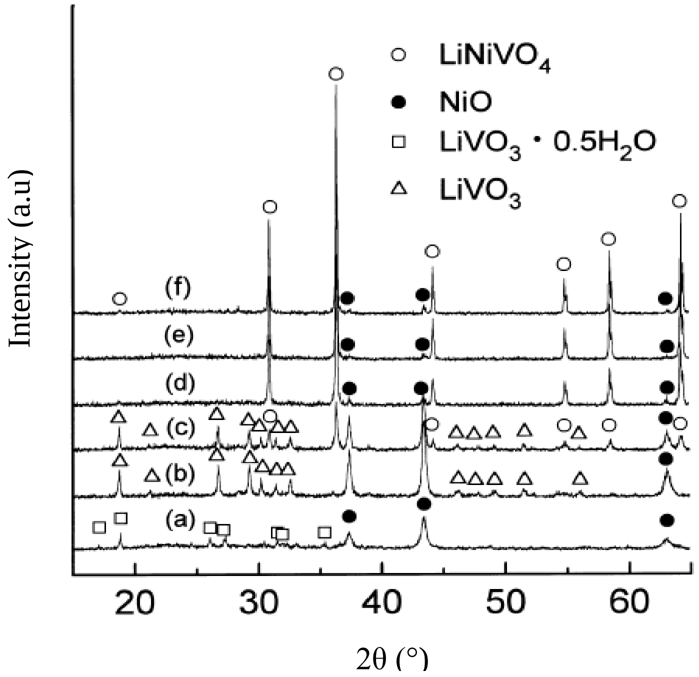
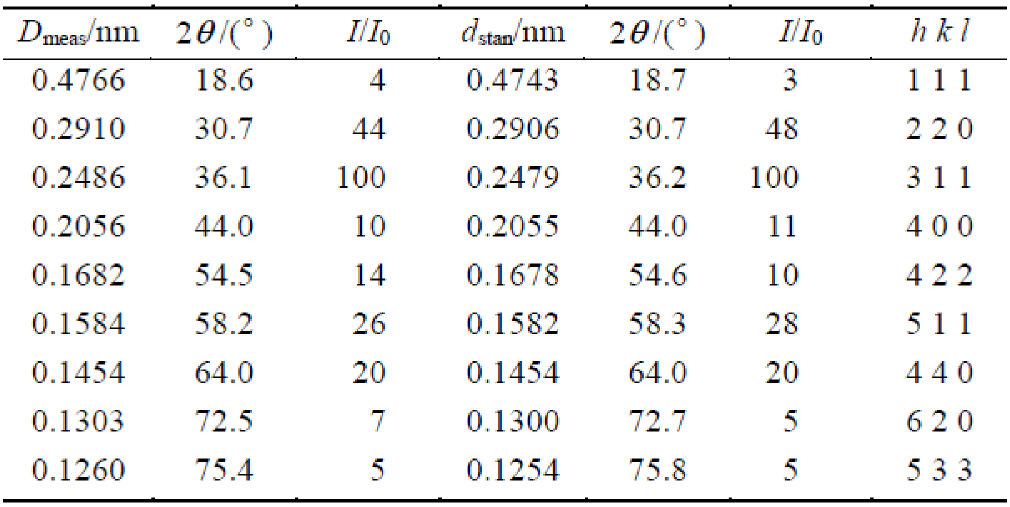


Figure 3.9 The X-ray diffraction patterns of (a) the as-prepared LiNiVO$_4$ precursor and the precursor quenched at (b) 500 °C, (c) 600 °C, (d) 700 °C, and (e) 800 °C, and (f) 1000 °C [Lu and Liou, 1999]

Lai and his co- workers have compared their LiNiVO$_4$ samples synthesized by using complexation precipitation and gelation (CPG) method [Lai *et. al,* 2001; Lai *et. al,* 2002a]. They have revealed the formation of pure LiNiVO$_4$ by providing their XRD data that agrees well with the JCPDS card *(38-1395)*. The XRD data is shown in Table 3.6.

Table 3.6 The XRD data for $LiNiVO_4$ [Lai *et. al,* 2002a]



D_{meas}/nm	$2\theta/(^\circ)$	I/I_0	d_{stan}/nm	$2\theta/(^\circ)$	I/I_0	$h\,k\,l$
0.4766	18.6	4	0.4743	18.7	3	1 1 1
0.2910	30.7	44	0.2906	30.7	48	2 2 0
0.2486	36.1	100	0.2479	36.2	100	3 1 1
0.2056	44.0	10	0.2055	44.0	11	4 0 0
0.1682	54.5	14	0.1678	54.6	10	4 2 2
0.1584	58.2	26	0.1582	58.3	28	5 1 1
0.1454	64.0	20	0.1454	64.0	20	4 4 0
0.1303	72.5	7	0.1300	72.7	5	6 2 0
0.1260	75.4	5	0.1254	75.8	5	5 3 3

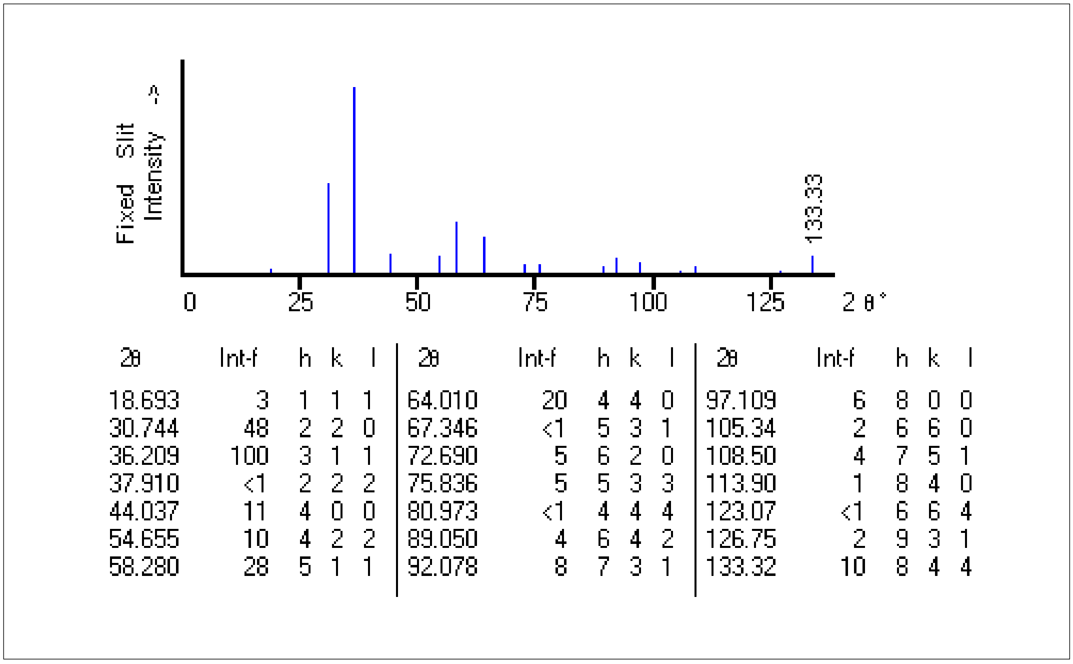
The crystallographic data also provides the information on the cubic lattice constant, *a*. subsequently the data can be calculated for the crystal volume of the compound. The cubic lattice constant, *a* can be obtained by using Bragg's formula:

$$a = d\left(h^2 + k^2 + l^2\right)^{1/2} \tag{3.3}$$

where; *d* is the distance between vicinal crystal face and

hkl is the Miller index.

The inverse spinel $LiNiVO_4$ is known to have cubic crystal in the system. Based on the literature, all the atoms in the inverse spinel structure occupy a special position in space group F*d*3*m* where both Li and Ni or Mn occupies the octahedral sites (16*d*,$\frac{1}{2}$, $\frac{1}{2}$,$\frac{1}{2}$), vanadium in the tetrahedral sites (8*a*,$\frac{1}{4}$,$\frac{1}{4}$,$\frac{1}{4}$) and oxygen (32*e, x, x, x*) [Padhi *et. al,* 1997; Gonzalez *et. al*,1994]. The structure can be expressed as $[V]_{tet}$ $[Li, M]_{oct}$ O_4 (M = Ni or Mn). The XRD database is shown in Figure 3.10.



2θ	Int-f	h	k	l	2θ	Int-f	h	k	l	2θ	Int-f	h	k	l
18.693	3	1	1	1	64.010	20	4	4	0	97.109	6	8	0	0
30.744	48	2	2	0	67.346	<1	5	3	1	105.34	2	6	6	0
36.209	100	3	1	1	72.690	5	6	2	0	108.50	4	7	5	1
37.910	<1	2	2	2	75.836	5	5	3	3	113.90	1	8	4	0
44.037	11	4	0	0	80.973	<1	4	4	4	123.07	<1	6	6	4
54.655	10	4	2	2	89.050	4	6	4	2	126.75	2	9	3	1
58.280	28	5	1	1	92.078	8	7	3	1	133.32	10	8	4	4

Figure 3.10 XRD database 38-1395 for $LiNiVO_4$

The samples were measured by using X-ray diffractometer (Siemens D5000) with operating voltage of 40 kV and current of 40 mA. The X-ray wavelength is 1.5406 Å. The X-rays measures the powdered samples at 2-theta angles ranging from 5 ° to 80 °.

3.4 Thermal Gravimetric Analysis (TGA) /Differential Thermal Analysis (DTGA)

Thermal Gravimetric Analysis (TGA) is a thermal analysis method which measures the mass changes in a compound as a function of temperature and time, which is measured in controlled surroundings. The technique used is very important to study the thermal stability of a material, or to examine its behavior in different atmospheres (e.g. inert or oxidizing). The method is desirably used with all types of solid materials, including organic or inorganic materials.

Differential thermal analysis (DTGA) is recorded simultaneously with the temperature and heat flow associated with thermal transitions in a material. The technique will allow the phase transitions to be determined (e.g. melting point, glass transition temperature, crystallization etc.). The idea of using this method is to measure both heat flow and weight changes that exhibit weight loss or gain due to decomposition, oxidation, or dehydration.

Liu et. al, (2002) have characterized the TGA and DTGA curve ranging from room temperature up to 1000 °C. They have shown that the decomposition process of the $LiNiVO_4$ precursor took place by endothermic peaks as shown in Figure 3.11.

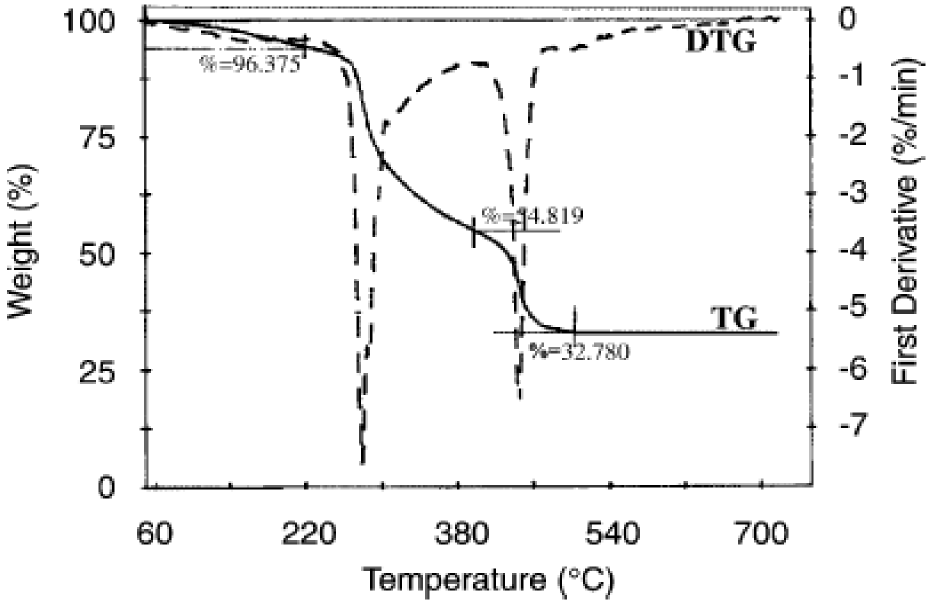


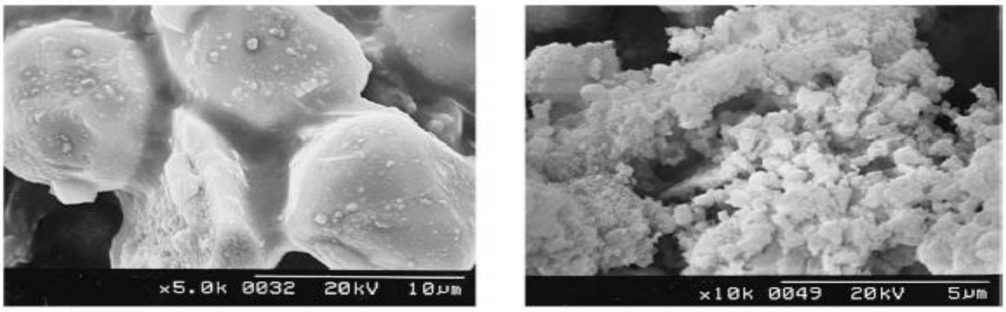
Figure 3.11 TGA/DTGA curve for $LiNiVO_4$ precursor [Liu *et. al,* 2002]

In this work, the TGA studies were carried out by using the Thermal Gravimetric Analysis (TGA). This thermal analysis was measured by using the Mettler Toledo Star System TGA analyzer. This analysis was also discussed to study the crystallization process by Prabaharan et. al, (1997), Lai et. al, (2001) and Lai et. al, (2002a) in their respective works. Those samples were put in a small alumina crucible and let the nitrogen flow through in the chamber during the analysis. The TGA analysis provides the information on the mass changes upon varying temperature from room temperature, 25 °C to 750 °C. The sample was employed at 10 °C/min. of heating rate. The firing samples were done under N_2 gas flow to avoid undesirable oxidation. The calcination process was carried out in air owing to the environmentally benign preference besides such technique rather promotes low cost synthesis.

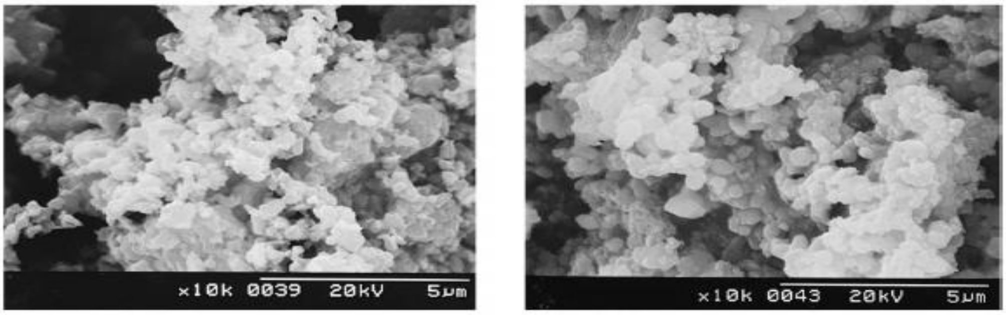
3.5 Scanning Electron Microscopy (SEM)

The SEM technique is very useful to observe the detailed information on the particle distribution generally in submicron size. This method is used by providing the high-energy beam of electrons in a raster scanning pattern. The SEM analyser uses electrons rather than light to form an image.

For scanning electron microscopy (SEM) investigation, the Leica Stereoscan S440 model scanning electron microscope was used to study the morphology of the prepared samples. Thus, the particle size and the distribution orientation can be observed by using the analyzer. Samples were vacuumed and coated with gold to prevent contamination and surface charging before the images being taken.

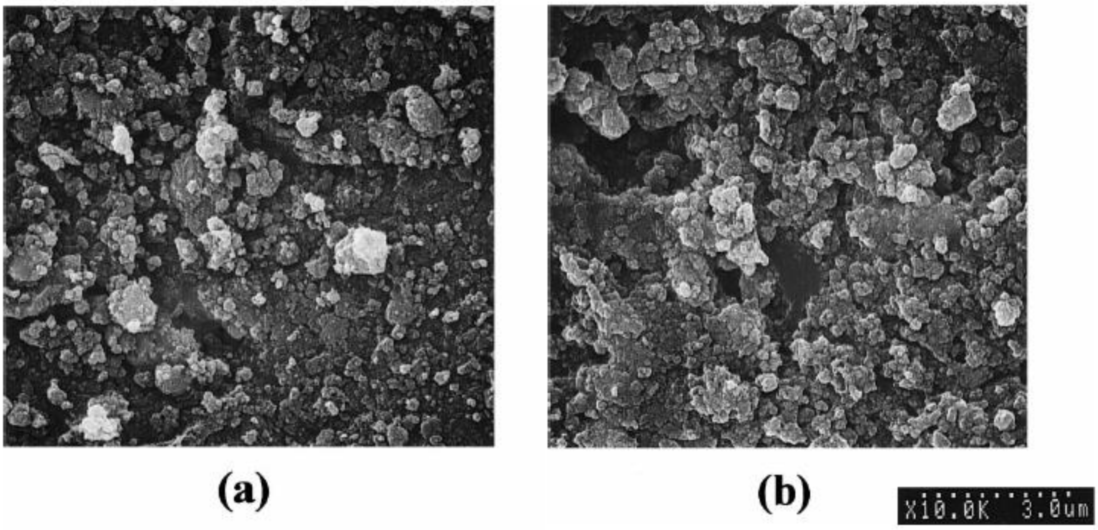


(a) HT-solid method **(b) pH=3**



(c) pH=7 **(d) pH=11**

Figure 3.12 SEM micrographs of $LiNiVO_4$ prepared by (a) HT-solid state method and the solution precipitation method: (b) pH 3; (c) pH 7; (d) pH 11 [Fey and Chen, 1999]



(a) **(b)**

Figure 3.13 Scanning electron micrographs of $LiNiVO_4$ powder prepared by the hydrothermal method at 200 °C. The concentrations of the precursor solution for (a) 0.3 M and (b) 0.6 M [Lu et. al, 1999]

Fey and Chen have reported that their varying pH conditions for $LiNiVO_4$ have affected the grain size distributions [Fey and Chen, 1999]. Lu and his co- workers have developed $LiNiVO_4$ by hydrothermal method. Their micrograph images reveal that the morphology of their sample changes as upon higher temperature treated for those samples. The images of both reported works are shown in Figure 3.12 and Figure 3.13.

3.6 Transmission electron microscopy (TEM)

The transmission electron microscopy (TEM) analyser has the same basic principle as light microscope. However, the TEM analysis uses electron instead of light. Therefore, the TEM is a microscopic technique that uses electron beam as the light sources. The electron beams are transmitted through an ultra-thin specimen thus will interact with the specimen as the beam passes through. The electron interaction of the transmitted through the specimen will form an image. The produced image can be magnified using imaging device or detected by a sensor.

Figure 3.14 The Transmission Electron Microscopy (TEM) analyser, LEO-Libra 120

The TEM analyser is designed to produce images with significantly higher resolution than light microscopes using the small de Broglie electrons wavelength. This will facilitate the user to observe even as small as a single column of atoms, which is tens of thousands times smaller than the smallest resolvable object in a light microscope. Thus, the TEM is viable upon providing thousand times better resolution than light microscope. Figure 3.14 show the TEM analyser to view the particle images in this work.

According to Subramania et. al, (2006), the prepared $LiNiVO_4$ powder produced ultrafine particles and uniformly distributed nanometer particle size and would affect the kinetics of the Li^+ ion diffusion for better performance of the cathode. Figure 3.15 depicts the nanosized particles obtained from the TEM viewing for the synthesized $LiNiVO_4$ powder.

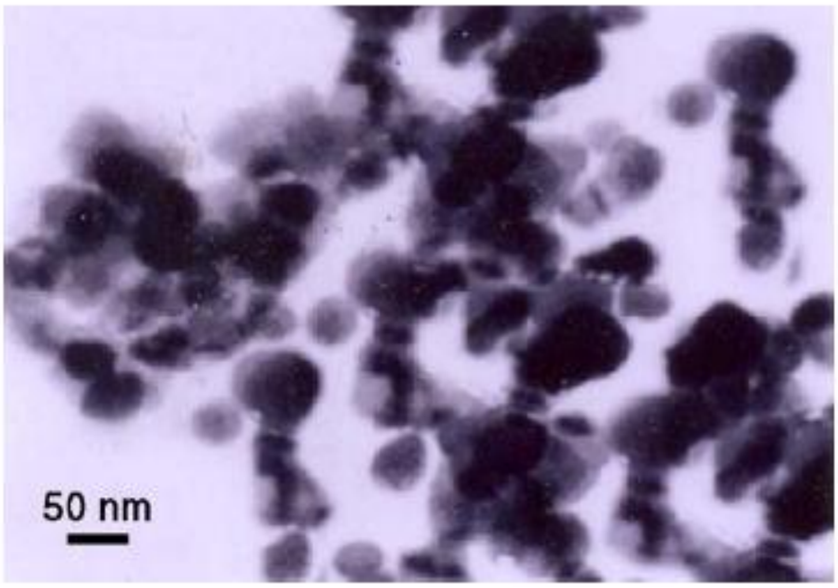


Figure 3.15 TEM photograph of $LiNiVO_4$ powder [Subramania *et. al,* 2006]

The TEM analysis was held after few days of etching sample preparation. The compound was mixed with alcohol and has it sonicate in the ultrasonic bath to agitate particles in the sample. Then, the solution was dropped onto a tiny copper formvar/ carbon mesh, a strong carbon film on copper mesh. The mounted grid was dried for few days to ensure complete drying before ready for TEM viewing. Figure 3.16 demonstrate the formvar grid used for etching process for the TEM analysis. The prepared sample was then put on the TEM chamber for viewing using transmission electron microscopy (TEM) analyser, LEO-Libra 120 at the Electron Microscopy Unit (EM Unit), Faculty of Medicine, UM.

Figure 3.16 The etching preparation for TEM analysis

3.7 Cyclic Voltammetry Analysis

The cyclic voltammetry is commonly used in the electrochemical studies. The analysis provides the qualitative information on electrochemical reaction. The technique basically offers location on redox potentials of the electrochemical species. The analysis is based on the potential curve that changed with the function of time. The scan rate is generally referred as the rate of change of potential with time.

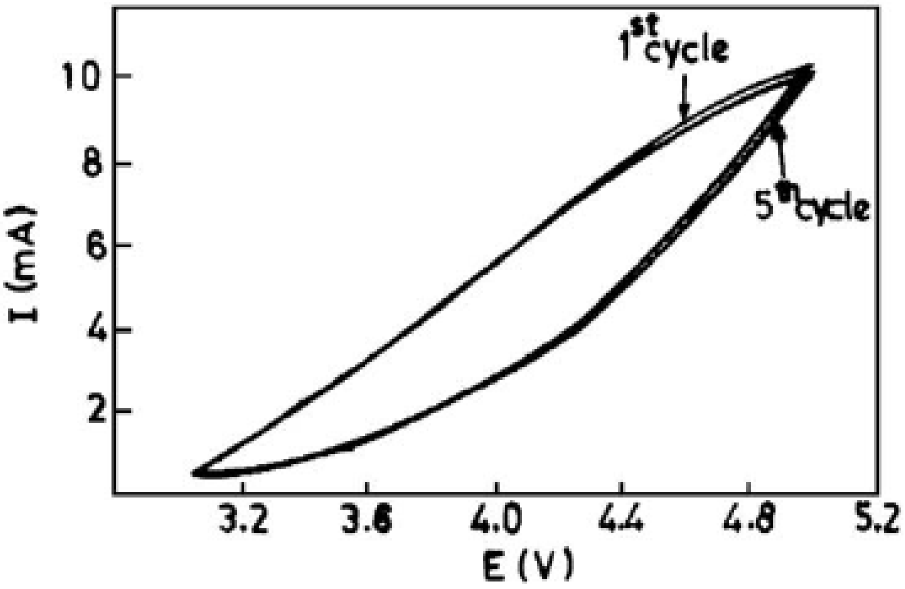


Figure 3.17 Cyclic voltammogram of C/$LiNiVO_4$ cell employing a Li^+ ion conducting PVdF-HFP based micro-porous polymer electrolyte at a scan rate of 1 mV/s [Kalyani *et. al,* 2002]

The electrochemical reaction undergoes the redox process where oxidation and reduction mechanism take place. In other words, the reaction involved two sequential electron transfer process. Cyclic voltammetry represents a pair of oxidation and reduction curve which associated with single electron loss/gain during lithium ion transfer process [Kalyani *et. al,* 2002]. According to Subramania et. al, (2006) the CV analysis reported responds well with the C/$LiNiVO_4$ cell at 3-5 V of voltage range at 1mV/s sweep rate as shown in Figure 3.17. Prabaharan et. al, (1997) has revealed the reversibility of the compound by de/ intercalating Li^+ ions cycled between 3-5 V for both Li/ $LiNiVO_4$ and C/ $LiNiVO_4$ cells.

3.7.1 Sample Fabrication

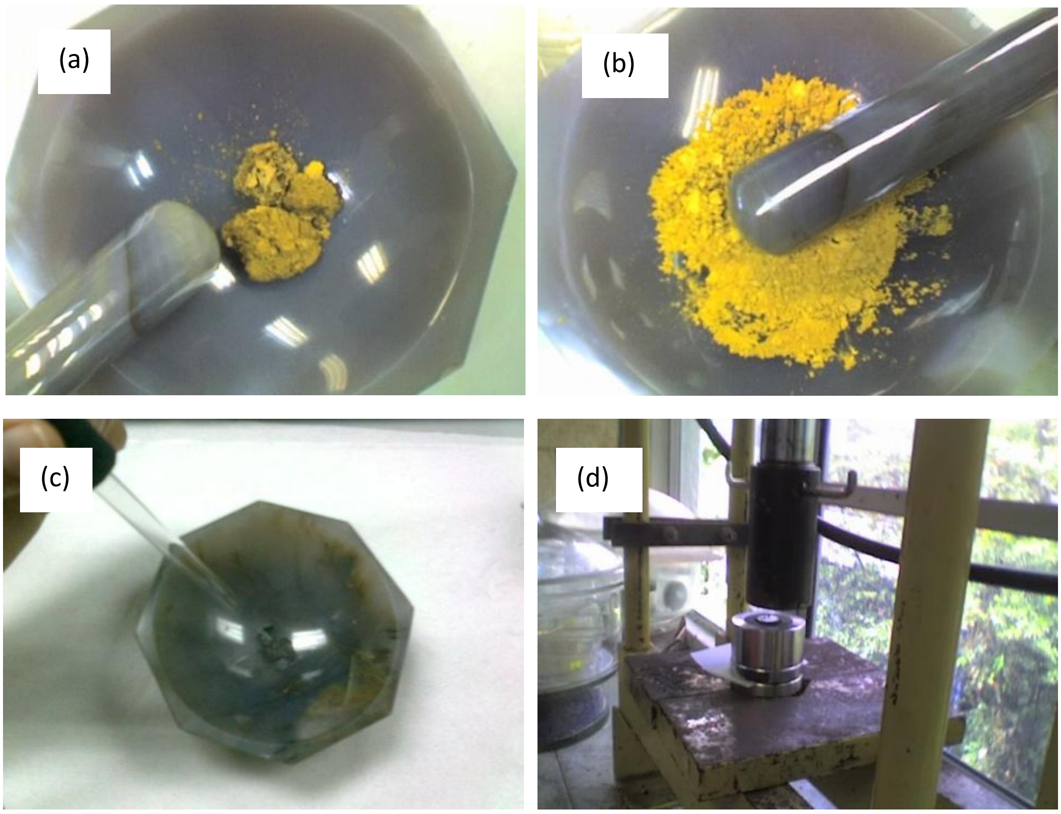


Figure 3.18 The sample before grinding; (b) The sample after grinding (c) The sample after mixing with the TAB binder; (d) The sample for pressing on the hydraulic pump

After the sample was left cooled at room temperature, the powder compound were weight for the cell fabrication process. The cathodes were prepared with 85 % active material and 15 % teflonized acetylene black (TAB). The active material compound was ground using an agate mortar. Then, the teflonized acetylene black (TAB) was added as conductive binder and the mixed materials were again ground to ensure they binds well to each other. The mixed compound was then pressed on stainless steel mesh as current collector and being pressed under hydraulic pressure of 40 kPa for perfect adherence of the compound with the mesh.

Finally, it was left dried at 70 °C, 24 h in an oven. Figure 3.18 shows the preparation process before the CV analysis was being carried out. The 1M $LiPF_6$ in 1:2 by (vol/vol %) EC/DMC was used as the electrolyte, while polypropylene membrane from Whatman GF/B microfiber was used as the separator. The half-cell was assembled in the argon-filled glove box (MBraun) using the air-tight cell holder. The CV studies were performed using AUTOLAB testing unit and recorded using the software provided. Figure 3.19 shows the schematic diagram of the CV analysis after the half-cell was being fabricated in the glove box.

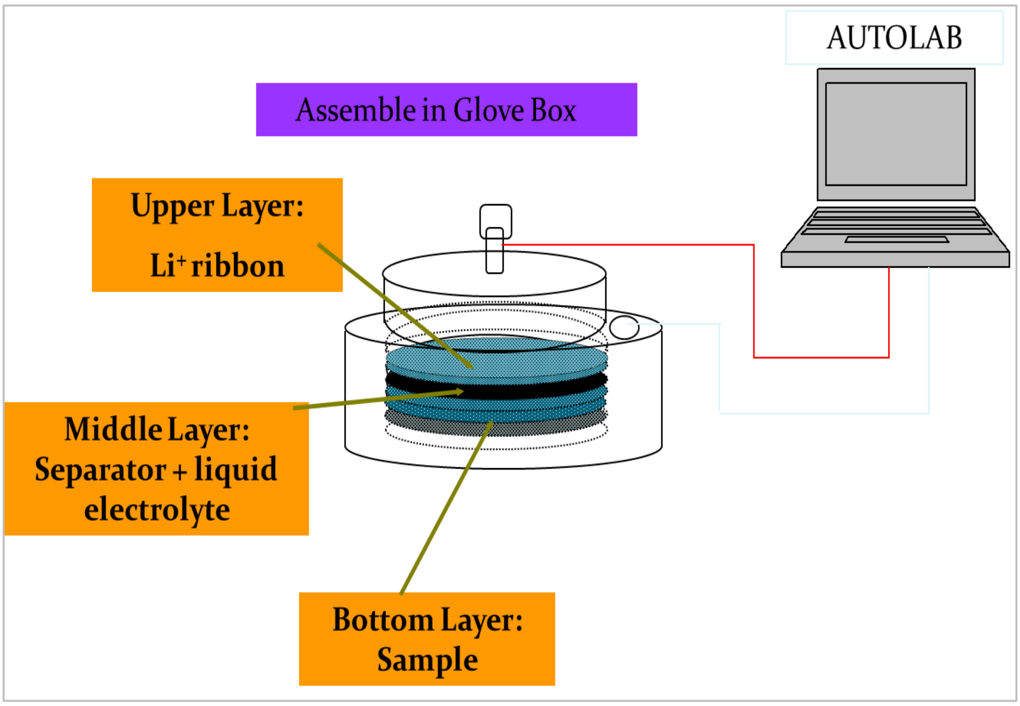


Figure 3.19 The cyclic voltammetry (CV) testing diagram

3.8 Elemental Analysis of Energy-dispersive X-ray spectroscopy (EDAX)

The EDAX technique is used to study the elemental properties for the synthesized product. EDAX provides the information on atomic percentage to investigate the presence of matter in their stoichiometric proportion. The analysis was conducted using the same scanning electron microscope (SEM) in which the EDAX signal analyser (energy dispersive X-ray spectroscopy) attaches to it. However, the EDAX outcome does not offer on lithium, the lightest metallic element information due to limitation of EDAX equipment.

The elemental analysis was employed using the scanning electron microscope (SEM) coupled with the energy dispersive X- ray spectroscopy operated at 20 kV. The analysis gives the results for amount of content of the sample which is recorded by the elemental spectrum. This work will investigate the nickel: vanadium or nickel: manganese: vanadium atomic ratios to be compared with stoichiometric composition by the predicted chemical formula of the synthesized compound.

3.9 Summary

Based on the discussed literature and experimental method, the sol-gel and polymer precursor method will be used to prepare the cathode material. The synthesized cathode will be characterized by TGA, XRD, SEM, TEM, and CV analyses.