CHAPTER-II

EXPERIMENTAL PROCEDURES
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EXPERIMENTAL PROCEDURES

2.1. INTRODUCTION

In this chapter, an overview of material preparation methods, characterization techniques, electrode fabrication process, coin cell assembling and electrochemical measurement are described. Figure 2.1 and 2.2 illustrate the flowcharts of the research strategies in this investigation.

Figure 2.1. Flowchart of the strategy of the material preparation in this investigation.
**Figure 2.2.** Flowchart of the characterization strategy in this investigation.
2.2. MATERIALS & SYNTHESIS TECHNIQUES

POLYOL PROCESS

The materials and chemicals were purchased from various companies and their details are explained in respective chapters. In this investigation, polyol technique has been adopted to prepare the bare and surface modified LiFePO$_4$ composite cathode materials. The procedures for synthesizing the LiFePO$_4$ and surface modified LiFePO$_4$ composite cathode materials are elaborated in more detail in the respective chapters of this thesis. This chapter plentifully gives description of the common steps of polyol technique. Figure 2.3 and 2.4 demonstrate the flowchart of synthesis of bare, metal oxide modified and carbonaceous materials modified LiFePO$_4$ using polyol technique. The polyol technique includes the following steps: (i) the precursor compounds of transition metal, a polyacid anionic compound and a lithium compound were thoroughly mixed and dissolved in a polyol solvent. The term "polyol" refers to a material having two or more OH groups in a molecule. The polyol solvent may be selected as any one from the group consisting of EG (Ethylene Glycol), DEG (Diethylene Glycol), TEG (Triethylene Glycol) and TTEG (Tetraethylene Glycol). The polyol serves as a solvent and a stabilizer in the electrode material synthesis procedure. Further, the polyol solvent serves to maintain the oxidation number of the transition metal, because it creates a reducing atmosphere at the boiling point thereof. (ii) dissolved mixture solution was heated nearer to the boiling point of the polyol solvent using refluxing unit with heating apparatus for a time period ranging from few hours to 72 h (iii) the resultant product was cooled down to room temperature in order to remove the remaining polyol solvent or additionally formed organic compounds after the reaction. The process of cleaning the mixed solution may be performed using water, an alcohol solution or an acetone solution.
It is preferred that the process of cleaning the mixed solution was repeated several times until the remaining polyol solvent or the additionally formed organic compound was completely removed. After the filtering and washing process, the resultant product was dried in a vacuum oven for one day. [1-2] Finally, the prepared materials were characterized for their properties.

**HYDROTHERMAL TECHNIQUE**

The raw materials of Li, Fe and PO₄ sources were dissolved in deionised water with stoichiometric molar ratio. If required, to coat on LFP, an appropriate amount of coating source has also been added to the metal oxide mixed solution. The mixed solution was poured into the air tight Teflon lined 150 ml autoclave and then reacted at 160 °C for 12 h. After the hydrothermal reaction, the reactor was cooled to room temperature. The precipitated powder was filtered and washed sequentially with deionised water, acetone and then with ethanol. The obtained powder was dried at 110 °C for 5 h under vacuum. Finally, it was calcined at 500 °C for 6 h under Ar atmosphere [3].

**SOILD STATE REACTION**

Stoichiometric molar ratio of raw materials was ball-milled for 3 h in the planetary ball mill (Retzch, Germany). The ball-to-powder weight ratio was taken as 5:1 with a rotation rate of 250 rpm. After ball milling, the powders were dried at 150 °C for 6 h in vacuum oven. Then, the dried mixture was preheated and followed by calcination under Ar atmosphere. Table 2.1 shows the details of the composition and the raw materials used in this investigation [3].
Figure 2.3 Flowchart of bare and metal oxide modified LiFePO$_4$ preparation using polyol technique.
Figure 2.4 Flowchart of carbonaceous materials modified LiFePO$_4$ preparation using polyol technique.
Table 2.1 Details of composition and raw materials used in this investigation.

<table>
<thead>
<tr>
<th>Sl.No.</th>
<th>Sample name</th>
<th>Chemical used</th>
<th>Compositions (stoichiometric molar ratio)</th>
<th>Methods/conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>LiFePO₄</td>
<td>LiH₂PO₄, FeSO₄·7H₂O, DEG</td>
<td>1.05:1 (LiPO₄:Fe)</td>
<td>Polyol Technique (245 °C for 18 h), Hydrothermal method, solid state reaction</td>
</tr>
<tr>
<td>2.</td>
<td>NiO coated LiFePO₄</td>
<td>LiH₂PO₄, FeSO₄·7H₂O, DEG Ni(CH₃COO)₂·4H₂O</td>
<td>1.05:1 (LiPO₄:Fe) 1, 2 and 3 wt. % of NiO</td>
<td>Polyol Technique 245 °C for 18 h and 500 °C for 1 h under Ar environment</td>
</tr>
<tr>
<td>3.</td>
<td>CeO₂ coated LiFePO₄</td>
<td>LiH₂PO₄, FeSO₄·7H₂O, DEG Ce (NO₃)₃·6H₂O</td>
<td>1.05:1 (LiPO₄:Fe) 1, 2 and 3 wt. % of CeO₂</td>
<td>Polyol Technique 245 °C for 18 h and 500 °C for 1 h under Ar environment</td>
</tr>
<tr>
<td>4.</td>
<td>Nb₂O₅ coated LiFePO₄</td>
<td>LiH₂PO₄, FeSO₄·7H₂O, DEG Nb₂O₅</td>
<td>1.05:1 (LiPO₄:Fe) 1, 2 and 3 wt. % of Nb₂O₅</td>
<td>Polyol Technique 245 °C for 18 h and 600 °C for 30 minutes under Ar environment</td>
</tr>
<tr>
<td>5.</td>
<td>Carbon coated LiFePO₄</td>
<td>LiH₂PO₄, FeSO₄·7H₂O, DEG Oxalic acid</td>
<td>1.05:1 (LiPO₄:Fe)</td>
<td>Polyol Technique (245 °C for 18 h), Hydrothermal method, solid state reaction</td>
</tr>
<tr>
<td>6.</td>
<td>MWCNT encrusted LiFePO₄/C</td>
<td>LiH₂PO₄, FeSO₄·7H₂O, DEG Oxalic acid MWCNT</td>
<td>1.05:1 (LiPO₄:Fe) 8 wt. % MWCNT+ 3 wt. % C</td>
<td>Polyol Technique (245 °C for 18 h)</td>
</tr>
<tr>
<td>7.</td>
<td>Graphene enfolded LiFePO₄/C</td>
<td>LiH₂PO₄, FeSO₄·7H₂O, DEG Oxalic acid and Graphite</td>
<td>1.05:1:1 (LiPO₄:Fe):C LFP/C: Graphene (99:1 wt.%)</td>
<td>Polyol Technique (245 °C for 18 h)</td>
</tr>
</tbody>
</table>
2.3. MATERIALS CHARACTERIZATION TECHNIQUES

2.3.1 Phase structure identification using powder X-ray diffraction (XRD)

X-ray diffraction (XRD) is an effective non-destructive analytical method for characterization of crystalline materials, providing the information on crystal structures, lattice parameters, orientation, and crystallite size of prepared materials [4]. The atoms and ions, which occur in regular repeating intervals in a crystalline compound, can be regarded as planes that act as semi-transparent mirrors when compounds (powders or single crystals) interact with X-rays. When the angle of incidence equals the angle of reflection for a given plane, X-ray beam can be diffracted from adjacent planes. This is seen in Figure 2.5a. Two X-ray beams with a wavelength $\lambda$ strike adjacent planes in a crystal with an angle of incidence $\theta$. Using the relations of right triangles, the path length between the two beams is related to the angle of incidence and the distance between the two planes ($d$) by two equal distances of $d\sin\theta$ (between points A and B and between points B and C in Figure 2.5). In order to have constructive overlap of the X-ray radiation, this distance must be an integer multiple of the X-ray wavelength. This relation is known as Bragg’s law:

$$n\lambda = 2d\sin\theta$$

When Bragg’s law is satisfied, the reflected beams interfere constructively and give rise to a diffraction peak. When the sample angle does not correspond to crystal plane spacing, there is a destructive interference of the X-rays. The peaks in a diffraction pattern are not a set of lines however, slight broadening occurs as a result of crystallite sizes and micro-stress within the crystallites. Figure 2.5(b) depicts a typical schematic diagram of powder diffraction experiment with the X’Pert Pro setup. A monochromatic beam of X-rays strikes the fine ground sample.
Figure 2.5. a) Representation of Bragg's law for a given set of crystal planes. b) Schematic diagram of a powder X-ray diffraction experiment.

Figure 2.6. Photograph of PAN X’ pert X-ray diffractometer (XRD)
The powder is a conglomeration of many randomly oriented tiny crystals. Thus, for each set of crystal planes, there will be a number of crystals oriented for Bragg diffraction to take place. The diffracted beams are collected with a moving detector. Plotting the angular positions and intensities of the resultant diffracted peaks of radiation produces a pattern, which is the characteristic of the sample. When a mixture of different phases is present, the resultant diffractogram is a combination of the individual patterns. All X-ray diffraction patterns presented here were obtained from a PANalytical X’Pert Pro powder X-ray diffractometer (Figure 2.6) equipped with a Cu K\(_\alpha\) radiation (\(\lambda =1.54060 \text{ Å}\)) operating at 40 kV and 30 mA in the 2\(\theta\) range of 10-80°.

### 2.3.2 Fourier transforms infrared (FT-IR) spectroscopy

Infrared spectroscopy gives the information about the bonding and molecular structure of a compound. This technique utilizes the fact that molecules absorb definite frequencies that are characteristic of their structure. i.e., the absorbed frequency of the radiation is equivalent to the frequency of the group or bond that vibrates. These frequencies take place in the IR region of the electromagnetic spectrum 4000- 400 cm\(^{-1}\).

The IR spectrum of a specimen is recorded by passing a beam of IR radiation through the specimen. Analysis of the transmitted light discloses the amount of energy absorbed at each particular wavelength. This can be executed with a monochromatic beam of radiation, which alters in wavelength over time or by means of a Fourier Transform (FT) instrument to determine all wavelengths at once. From this absorbance or transmittance spectrum, which reveal the IR wavelengths that the compound absorbs. In general, a frequency will be strongly absorbed if its photon energy coincides with the vibrational energy levels of the molecule (Figure 2.7). Therefore, IR spectroscopy is a very powerful technique, which provides fingerprint information on the chemical composition of the sample [5, 6].
Figure 2.7 Diagram and Photograph of FT-IR spectrometer.
In the present investigation, mid-infrared spectral studies in the range of 4000-400 cm\(^{-1}\) were carried out using a FT-IR Thermo-Nicolet 380 series instrument (Figure 2.7) at room temperature. Few milligrams of the compound was mixed with KBr and then it was pressed to give a translucent sample pellets.

2.3.3. Raman spectroscopy

Raman spectroscopy utilizes the Raman Effect to characterize structure and composition of materials. The Raman Effect, which describes the change in the wavelength of light that occurs when a light beam is deflected by molecules and crystalline solids, is named after Sir C.V. Raman who received the Nobel Prize in 1930 for his discovery [7]. When the incident photons collide with the atoms of the specimen, a large fraction of the photons are scattered elastically, and do not change their energy upon collision. This scattering is known as Rayleigh scattering [8]. However, a small fraction of the incident photons experiences a change in energy as a result of an inelastic scattering event (Raman scattering). In Raman scattering, the atoms in the crystal lattices gain or lose energy. Stokes Raman scattering refers to an increase in energy. The photon excites the specimen atoms to a higher vibrational or rotational energy state, and therefore loses energy upon scattering. If the specimen atoms are already in an excited vibrational state, the photon gains energy from the collision. This effect is known as Anti-Stokes Raman scattering [8]. At room temperature, Stokes Raman scattering is more likely to occur since most molecules are at a ground state. Therefore Stokes scattering gives the stronger Raman signal and is generally used for Raman analysis (Figure 2.8). The Raman frequency, or Raman shift, is the difference (in wave numbers) between the frequency of the incident light and the frequency of the scattered light. The Raman shift is thus a direct measure of the vibrational energies of a material and correspond to vibrational or rotational transitions of the scattering atoms [8].
Figure 2.8. Rayleigh and Raman scattering; frequency of incident light ($v_0$) and frequency of molecular vibration ($v_1$).

Figure 2.9. Photograph of Raman spectrometer
Raman spectroscopy relies on inelastic scattering or Raman scattering of monochromatic light from a laser source. Light from the laser interacts with phonons in the sample, resulting in a shifting of the energy of the laser photons up or down. The shift in energy gives information about the phonon modes in the system. In this work, Raman spectroscopic analysis was performed using STR Raman, SEKI Corporation, Japan system utilizing a 514.5 nm incident radiation and a 50 x aperture (N.A = 0.75), resulting in approximately a 2 \( \mu \)m diameter sampling cross section. The Raman spectrometer instrument used in this study is shown in Figure 2.9.

**2.3.4 Field-Emission Scanning electron microscopy (SEM) with EDX**

The scanning electron microscope (SEM) is a powerful tool to investigate the morphology of the samples, using a focused high-energy electron beam to generate a variety of signals at the surface of solid specimens. The signals derived from electron-sample interactions provide information on the sample such as external morphology, chemical composition, and crystalline structure. The kinetic energy from accelerated electrons in SEM is dissipated as a variety of signals produced by electron-sample interactions. These signals are generated from secondary electrons, backscattered electrons, diffracted backscattered electrons, photons, visible light and heat.

Secondary electrons and backscattered electrons are commonly used for imaging the samples (Figure 2.10a). Secondary electrons are used for showing morphology and topography on samples while backscattered electrons are explored for illustrating contrasts in composition of the multiphase samples. Characteristic X-rays produced for each element in a sample, which is excited by the electron beam can be used for compositional analysis [9].
Figure 2.10 Schematic representation of Microscopic technique and photograph of FE-SEM instrument.
In this work, the morphology of the obtained sample was investigated using two model of instruments like Field Emission Scanning Electron Microscopy (FE-SEM, Quanta 250 FEG, FEI Company) equipped with energy-dispersive spectroscopy (model: AMETEK EDS Quanta 200F) (Figure 2.10b) or field emission scanning electron microscopy (FE-SEM, LEO-1530, ZEISS, Germany) with energy dispersive spectrometer (EDS, X-MAX50) operating at 30 kV.

2.3.5 Transmission electron microscopy (TEM) with SAED

Transmission electron microscopy (TEM) is a unique and versatile characterization tool because it can provide not only a real space atomic-resolution lattice images for the nanocrystal and its surface, but also chemical information at a spatial resolution of 1 nm or better, hence allowing direct identification of the chemistry of a single nanocrystal. A simplified ray diagram of a TEM consists of an electron source, condenser lens with aperture, objective lens with aperture, specimen, projector lens and fluorescent screen.

TEM has three functions, imaging, spectroscopy and diffraction. By TEM images, we can obtain the information on the size, shape and texture of nanocrystal. For better imaging, high-resolution transmission electron microscope (HR-TEM) has to be applied, since it is capable of giving resolutions on the picometer scale due to the highly stable electron acceleration voltage and operability on the fixed current. The lattice fringes (in HR-TEM), obtained by interference between the transmitted beam and the diffracted beam, can provide information on the periodicity of the crystal. Electron diffraction (ED) is a TEM mode which allows users to determine the atomic arrangement of crystals. When combined with other functions such as EDS, it can help indentifying unknown crystals and determining the d-spacing of newly described crystals.
The ED pattern is completely dependent on the d-spacing and composition of the crystal that is being investigated. An ED of a single crystal will result in a series of diffraction spots arranged in concentric rings around the central bright spot which is comprised of transmitted electrons. While for polycrystals, some of which are oriented at the Bragg’s angle while others are not, an ED pattern with well defined concentric rings, but not spots, will appear. As to amorphous structure (i.e. no crystalline formation), only a central bright spot comprised of transmitted electrons and a single ring of randomly forward scattered electrons in ED pattern is observed.

**TEM Sample Preparation**

Sample preparation is one of the very important aspects of TEM analysis. The sample for TEM analysis can be quickly prepared by means of following steps 1) few milligrams of samples were taken in 2 ml of ethanol in a closed vial 2) Sonicated for sufficient time to disperse the sample in ethanol 3) Depositing the suspended particles on to a TEM copper grid coated with holey-carbon. Transmission electron microscopes (TEM) are capable of imaging at a high resolution of ~ 0.2 nm. In TEM, the electrons are transmitted through an ultra thin specimen, resulting in an interaction with the specimen. An image is formed from the interaction and is magnified and focused onto an imaging device, such as a fluorescent screen, photographic film, or CCD camera. In the present investigation, the nano particles and coating morphology were observed using high resolution transmission electron microscopic (HR-TEM) (Techni G2 S-TWIN, FEI, model:3000) technique (Figure 2.11).
Figure 2.11. Photograph of HR-TEM
2.3.6 X-Ray photoelectron spectroscopy (XPS)

XPS is a surface sensitive spectroscopic technique, which is widely used to
determine the electronic and chemical state of the elements present in the surface of the
material [10]. When a solid is irradiated with a flux of X-ray photons of known energy, a
number of photoelectrons escape from the top 1 to 10 nm of the solid being analyzed.
These photoelectrons originate from distinct electronic energy levels connected with
those atoms. The energy of the photoelectrons escaped from the surface is given by the
following equation

\[ E_{(K,E)} = h\gamma - E_{(B,E)} - \Phi \]

where \( h\gamma \) is the photon energy of the excitation source, \( E_{(K,E)} \) and \( E_{(B,E)} \) are the
measured kinetic and binding energy of the photoelectron respectively. \( \Phi \) is the work
function, which is the potential difference between the Fermi level and the vacuum level
of the solid. XPS is typically accomplished by exciting the sample’s surface with
monoenergetic Al k\( \alpha \) X-rays causing photoelectrons to be emitted from the sample
surface. An electron energy analyzer is used to measure the energy of the emitted
photoelectrons. From the binding energy and intensity of a photoelectron peak, the
elemental identity, chemical state, and quantity of a detected element can be determined.
Figure 2.12 shows the schematic diagram of X-ray photoelectron spectroscopy. In the
present work, powder samples were analyzed using XPS, PHI model 5802 spectrometer
with mono-energetic Al k\( \alpha \) X-ray source (\( h\nu = 1253.6 \) eV) and with the electron analyzer
set at a constant pass energy of 20 eV (Figure 2.13). The peak position of C 1s spectrum
with a binding energy of 284.6 eV, which is presented due to carbon contamination in a
very small quantity on the sample surface and it has been calibrated. The average depth of
analysis for an XPS measurement is approximately 5 nm. PHI XPS instruments provide
the ability to obtain spectra with a lateral spatial resolution as small as 7.5 \( \mu \)m.
The energy resolution of the spectrometer is 0.05 eV. The raw data spectra were fitted using XPS peak-fit software. Wherever necessary, the spectra were delineated by non-linear least square fitting with a Gauss-Lorentz (ratio 60:40) curve into multiple peaks and the corresponding binding energies were evaluated.

Figure 2.12. Schematic diagram of X-ray photoelectron spectroscopy.

Figure 2.13. Photograph of XPS.
2.3.7. Thermo gravimetric analysis (TGA)

In thermal analysis, particular physical and chemical material properties and thermal stability of prepared materials were measured as a function of the temperature. In thermal gravimetric analysis (TG), the sample’s change in weight (due to oxidation and pyrolysis effects) during a given temperature-time program at a defined atmosphere is determined. TGA is an experimental technique that is carried out on samples to determine the mass changes as a function of change in temperature or time.

Typically, it is carried out at constant heating rate under desired atmosphere (inert/air/oxidising/reducing). The choice of heat rate and atmosphere used in TGA experiments plays a crucial role on the measurement. Diverse effects can cause a sample to lose or gain weight, which produces steps in the TGA curve. These curves are known as thermograms. In this investigation, a NETZSCH STA 449 F3 Jupiter thermo gravimetric analyzer (TGA) under synthetic nitrogen or argon or air atmosphere in the range from room temperature to 1000 °C at a heating rate of 10 K/min was used for thermal analysis.

2.3.8. Atomic Force Microscopy (AFM)

In all Scanning Probe Microscope (SPM) techniques, a tip interacts with the sample surface through a physical phenomenon. Measuring a “local” physical quantity related with the interaction, allows constructing an image of the studied surface. All the data are transferred to a PC, where, with the use of the appropriate software, an image of the surface is created. The AFM measures the forces acting between a fine tip and a sample. The tip is attached to the free end of a cantilever and is brought very close to a surface. Attractive or repulsive forces resulting from interactions between the tip and the surface will cause a positive or negative bending of the cantilever. The bending is detected by means of a laser beam, which is reflected from the back side of the cantilever.
The resolution for AFM instruments is: a) 0.1 nm on sample plane (x, y) for hard and flat surfaces and 0.7-5 nm for soft materials (polymers and biological samples), b) 0.01 nm for z axis [11]. The AFM used in this experiment was the Agilent 5500 AFM (Agilent Technologies, USA), used in the non-contact Mode to obtain the high resolution images of the prepared materials. The probe used in this study was a silicon nitrate cantilever with a tip radius of less than 10 nm, nominal force constant of 48 N/m and resonance frequency at a nominal value of 190 kHz. A scan rate of 1 Hz was used. Topographic image and particle size analysis was then carried out using the picoview image processing software (Version 6).

2.3.9. Vibrating Sample Magnetometer (VSM)

Magnetic measurements were performed to get the additional information on impurities, which may be present in the prepared materials and used to analyze the magnetic nature of the samples [12]. Vibrating Sample Magnetometer (VSM) is a very sensitive magnetometer used to measure extremely weak magnetic fields. The temperature dependent magnetization measurements were carried out using Lakeshore 7300 Measurement System instrument.

2.4. ELECTRODE FABRICATION AND COIN CELL ASSEMBLY

This section is mainly focused on the preparation of electrodes and assembling of coin cell to study the electrochemical performance. In this regard, the assembling of coin cell involves several steps and these steps are described hereunder.

Fabrication of Electrode

The active material, super P/carbon black (Timcal company), and binder polyvinylidene difluoride (PVDF, Sigma-Aldrich) were taken in weight ratio of 80:10:10 and mixed in a N-methylpyrrolidone (NMP, Merck, India) solvent using a homogenizer.
The resultant slurry was coated onto a current collector (Alumina foil) whose thickness 10-20 µm using doctor blade method. The coated foil was dried at 120°C in a vacuum oven for 6-24 h to evaporate the solvent. The dried thick films (20-30 µm) of composite electrodes were then pressed between twin rollers at ~2 MPa pressure to guarantee good adherence of electrode materials on to the current collector. The prepared electrode is then punched into discs of 16 mm diameter and then dried under vacuum at 80 °C for 6 h in an antechamber prior to the insertion into glove box for assembling of coin cell [13, 14]. The active material content in the electrode was around 2~2.5 mg. Figure 2.14 shows the pictorial representation of electrode preparation method.

**Coin Cell Assembly**

The coin-cell fabrication was carried out inside an Ar-filled glove-box (MBraun, Germany) which maintains < 1 ppm of O₂ and H₂O. Standard stainless-steel cups and lids fitted with a plastic ring are used for the casing (Hohsen Corporation, Japan) of coin cell. Coin cells (size, 2032; Nominal diameter is 20 mm, nominal height is 3.2 mm and the range of weights from 2.8 g to 3.9 g) consist of composite electrode, Li metal foil as counter electrode, glass micro-fiber sheets (Whatman) or Celgard 2400 polypropylene micro-porous membrane as the separator and 1M LiPF₆ dissolved in ethylene carbonate (EC) and diethyl carbonate (DEC) or dimethyl carbonate (DMC) (1:1 by volume, Merck) as the Li⁺ ion conducting electrolyte (electronically insulated). The composite electrode is placed in the centre of the cup which forms the positive terminal of the cell and is wetted with a 100 µL of the electrolyte. This is covered with the separator that is permeable only to the Li⁺ ions but is resistant to particles of the material and is an electronic insulator. Circular Li-metal disc of the size 12 mm diameter and ~0.59 mm thick is then placed centrally on top of the separator.
Figure 2.14 Pictorial representation of electrode preparation with the appropriate instruments.
**Figure 2.15** Schematic diagram of the coin type cell configuration along with tools.
To guarantee a good electrical contact and tight sealing of the cell, a steel spring is placed inside the stainless steel lid, which then sits over a Li-metal disc and forms the negative terminal of the cell. Finally, air tight sealing is done with a mechanical hand press (Hohsen Corporation, Japan). A schematic of coin-cell (battery) is shown in Figure 2.15. The fabricated coin cells were then taken out of the glove box and are aged for 24 h at ambient atmosphere (in order to provide sufficient percolation of the electrolyte into the electrode material) before carrying out any measurements. In the present study, half cell configuration (one working electrode and one reference electrode) was employed to measure the storage performance of the synthesized materials [14].

3.4. ELECTROCHEMICAL STUDIES

The Li storage studies of prepared electrode materials were carried out under constant potential or constant current conditions. They are known as galvanostatic and potentiostatic technique, which one used to study the electrochemical storage performance. In this investigation, the fabricated coin cells were tested for charge/discharge, cyclic life, rate performance, cyclic voltammetry and impedance analyses.

**Charge/discharge analysis**

The constructed coin cells were tested for its charge/discharge and cyclic life with various current rates. In the present investigation, there are two different model battery tester were used such as Maccor 4000 Battery Tester and Neware BTS-55 computer-controlled battery test station. The consequential output of the measurement is in the form of data containing charge/discharge step time, voltage variation and overall energy output (charge/discharge capacity) in terms of mAh (coulombs) with respect to time for the weight of the active material of the electrode.
The specific charge/discharge capacity (mAh/g) (and the number of moles of Li) can then
be calculated as discussed in the previous chapter.

**Cyclic Voltammetry**

Cyclic voltammetry experiment is used to analyze the phase transitions and
oxidation/reduction potential. In this study, cyclic voltammograms were run on a
Solartron 1287 Electrochemical Interface at room temperature.

**Electrochemical impedance spectroscopy (EIS)**

Electrochemical impedance of a cell was measured by applying a small amplitude
AC potential to an electrochemical cell and measured the current through the cell. The
current measured over a broad frequency range is used to determine the resistive,
capacitive, and inductive behaviour of an electrochemical cell, which can be associated
with various polarization losses. Electrochemical impedance spectra (EIS) were
performed using CHI 660D electrochemical analyzer (CH Instruments) at room
temperature for a constant applied voltage of 5 mV in the frequency range between 100
kHz and 0.01 Hz.

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