Synthesis, Characterization and Electrochemical Analysis of Lithium Manganeze Oxide ($LiMn_2O_4$)

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Abstract

A novel combination of materials are proposed for development of the cathode material. Through the research paper we are proposing synthesis of Lithium Manganese Oxide ($LiMn_2O_4$) material using hydrothermal process. In these assumptions, the time is kept constant and temperature is varied from 120⁰C to 180⁰C. The cathode element designed using novel combination is synthesized for lithium-ion batteries. Further, different ascendency parameters like Grain Size, Morphology, Electro Chemical properties are critically observed by varying temperature by Hydrothermal Synthesis process. Additionally, the XRD, SEM and TGA techniques are preferred for characterization purpose. Finally, the electrochemical analysis is carried out to understand the performance of the prepared cathode material.

Keywords—*Rechargeable Battery, Discharge Control, Lithium-Ion, $LiMn_2O_4$, XRD, SEM, TGA*

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I. INTRODUCTION

Broadly, the battery technology can be bifurcated in to charging mechanism, charge holding techniques and discharge control mechanism. With the development in the battery technology and to fulfil the market need, concept of fast charging is introduced. Ultra-fast charging is another terminology which comes with the Electric Vehicle (EV). The ultra-fast charging creates the stress on the battery, even if the batteries are designed with the same intension. To nullify this effect, concept of step charging is introduced. In this technique, when the charging of the battery reaches to 50%, the charging current is meddled off and it is again reduced when charging reaches to 75% of the battery capacity. As far as charge holding mechanism of the battery is concerned, it is the anode and cathode terminal which are responsible for holding the charge in either charged or discharged state. Anode particularly holds the charge while charging and cathode holds the charge while discharging. These two terminals are separated by the electrolyte material to avoid contact and shorting. Here, anode is made up of graphite and the cathode can be an alloy of multiple materials like nickel, cobalt, lithium and many other materials. To fulfil need of power to be delivered to the applications and to accomplish the need of the market, already, significant researches are carried out in the development of the charging technology and charge holding techniques. But there is still large scope for the development of the discharge control mechanism.

Through this research paper, a novel combination of material is proposed for development of the cathode material which can be used for high charge density and extended life cycle. In the subsequent section, detailed literature survey is carried out. Through this, detailed analysis is done with respect to the charging speed, effects of adverse temperature, capacity loss, depth of discharge and many other significant parameters are extracted in the form of literature.

Subsequently, the methodology is proposed to design the cathode material for the lithium-ion battery. The methodology is executed in the form of Electrode Manufacturing, Cell Assembly and Cell Finishing. The research work spins around synthesis of Lithium Manganese Oxide (LiMn2O4) material. The actual manufacturing process is carried out using hydrothermal process. The cathode element is synthesized for rechargeable batteries. With these assumptions, different ascendency parameters like Grain Size, Morphology, Electro Chemical properties are critically observed by keeping time constant and varying temperature by Hydrothermal Synthesis process.

II. Literature Survey

Z. Ye, et. al. [1] In this paper, the authors have proposed security circuitry for the lithium-ion battery charger. Initially, a detailed investigation on lithium-ion battery charger security has been done, then a comprehensive system depiction was provided and lastly, experimental outputs are presented. The proposed integrated circuit has features like it gives thermal security to the battery, suitable period execution, damage cell identification, state signal, and automatic recharge, which are not available in traditional charger design. Moreover, a novel technique has been presented to realize the battery thermal control unit for the system. The proposed battery security model can set the charge current according to the ambient temperature, which provides acceleration in the charging procedure. In order to produce the system clock, a new oscillator without a comparator is designed.

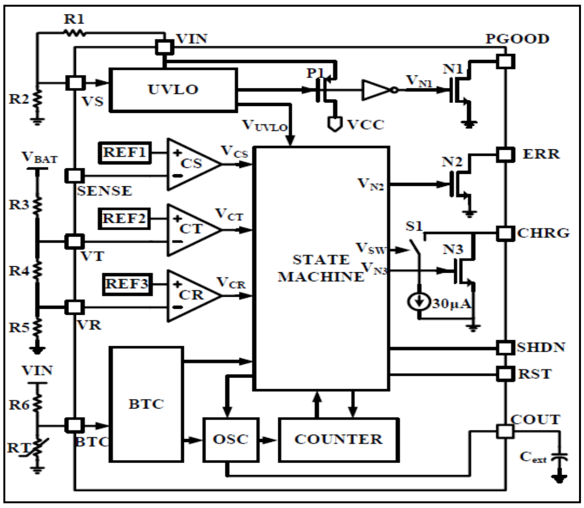

Fig. 1: Schematic of the proposed protection controller

Y. Wang, et. al. [2] For energy discharging and charging devices, bidirectional direct current to direct current converter is mostly utilized. Though, this converter shows extremely non-linear features since its inner system constraint changes depending on the function method and the interruption commitment. To deal with the non-linearity, a bidirectional direct current to direct current converter is developed which works on the basis of time delay control and is authenticated for lithium-ion battery purposes. Simulink is employed to construct a bidirectional direct current to direct current converter model. The pulse width modulation duty proportion handle method based on time delay control is developed for lithium-ion battery function regularly. To authenticate the efficiency of the proposed bidirectional direct current to direct current converter, comparative integral control is realized.

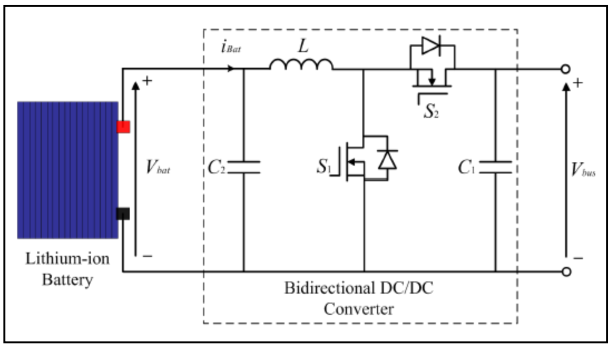

Fig. 2: Li-ion fed bidirectional DC-DC converter system

E. A. Voznesenskii [3] In the present paper, one of the most important problems in the domain of Aeronet like the inadequate period of flight operations of multi-rotor unmanned aerial vehicles due to the comparatively low precise energy utilization of advanced lithium-ion and lithium-polymer batteries has been discussed. The trouble of necessitating for the person involved in the recharging of batteries for drones, which obstructs the complete independence of flight operations is also discussed. After the relative investigation, the technique of contactless charging of the quadcopter with the assist of a particular charging position was elected for future analysis. In the experimental section, an integrated circuit of communicating elements is elected, a technique for realizing a precise drone landing is proposed, and an approach for executing the perception for future practical application is developed.

F. Dragomir, et. al. [4] Electricity as an energy component for vehicle momentum can be alternated for petrol or diesel with a broad variety of most important energy sources. This confirms the protection of energy supply and carbonless energy sources and extensive utilization of renewable in the transport domain which helps eliminate carbon-dioxide production. In a lithium-ion battery, the lithium ions travel from the negative to the positive electrode during the charging and discharging period. The electric vehicle driver is required to be familiar with how much he is going to travel before the vehicle battery needs a recharge. In this paper, the authors employed a technique for estimating the electrical corresponding device constraints utilizing pulse discharge practical information to construct lookup tables with cell temperature and system on a chip as independent variables.

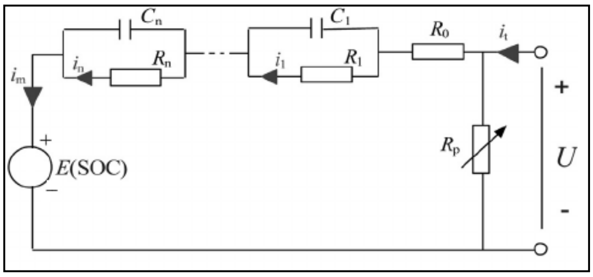

Fig. 3: Corresponding circuit modules for electrochemical cells

D. Kucevic, et. al. [5] Recharging battery electric vehicles for instance balance competitive to a standard refueling stop of an inside ignition engine vehicle creates several unanswered challenges. Traditional power frameworks face overload, in case the electrification of the transport sector picks up pace. In this paper, the authors have contrasted procedures for lattice strengthening with the utilization of lithium-ion based buffer storage under technical and economic characteristics to achieve the target of a prospect fast charge network at a power level of 500 kW and voltage levels of 900 V and above. Such tremendous fast charging potentials are not only demanded to prospect battery electric vehicles but may also facilitate recharging of electric buses and electric trucks. Two dissimilar circumstances, namely a charging station at a motorway rest region and a charging station for an electric bus depository are investigated in a further aspect.

Zhang, et. al. [6] In recent years, many types of research on cathode materials are going on to recharge lithium-ion batteries on the layered lithium nickel oxide and lithium cobalt oxide. A sol-gel technique is employed for the manufacturing of a lithium-ion secondary battery using lithium nickel manganese cobalt oxide cathode material. Discharging charging examination, cyclic voltammeter, and X-ray diffraction are used to study electrochemical and structural characterization. Manganese doping supports the diffusion of lithium in lithium nickel cobalt oxide. Furthermore, manganese doping controls the phase alteration that normally takes place in lithium nickel oxide for the duration of cycling and enhances the discharging charging reversibility of lithium nickel cobalt oxide.

H. Lin, et. al. [7] In lithium-ion batteries to evaluate the state of health, a probabilistic neural system is employed. The precise forecasting of the state of health can keep away from deadly collisions from the unexpected fault of the battery. The authors used several pieces of lithium-ion batteries for experimental purposes. The steady voltage or current recharging and steady current discharging are examined to test the life cycle of the battery. The information received from the analysis of the life cycle test of the battery and the discharging and recharging electric features are utilized to evaluate the state of health of the battery. For evaluating the state of health of the battery, the most essential features are the steady current charging period, the immediate voltage fall at the initial stage of discharging, and the open-circuit voltage.

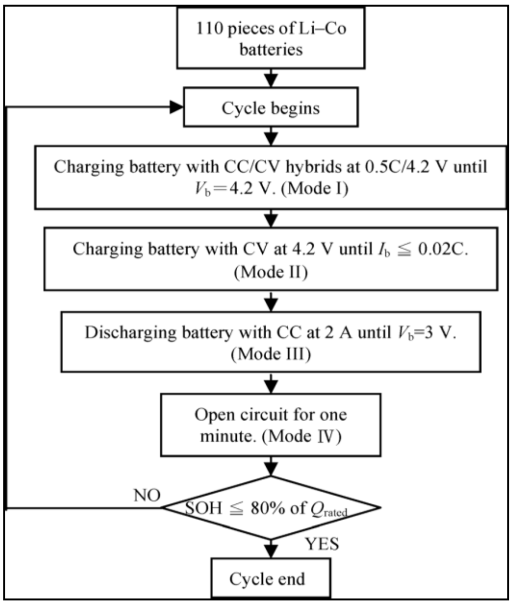

Fig. 4: Flowchart of the proposed scheme

F. Ciancetta, et. al. [8] In the domain of the automotive industry, the application of polymer electrolyte casing fuel cell stack is a most interesting topic for research. A polymer electrolyte casing fuel cell stack is incapable of providing dynamic speed range as it has non-linear characteristics in dynamic circumstances. The present paper deals with the performance characterization of the polymer electrolyte casing fuel cell stack during duplication of current step deviations. In this paper, the authors have proposed the introduction of lithium-ion battery models and a direct current to a direct current converter. The experimental outputs show that with the proposed configuration, the dimension of supercapacitors could be considerably decreased, plummeting the pressure on the battery pack without losses of performance.

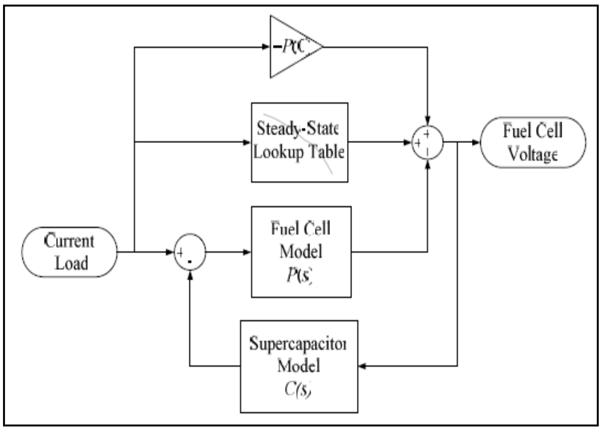

Fig. 5: Complete fuel cell module

P. Chancerel et al. [9] Batteries are considered chemical power systems in which a number of important components are required to fulfill performance and utilization necessities. Currently, batteries represent one of the most challenging waste groups when taking into account critical raw materials. In this paper, the authors have provided a summary of the information gathered and information investigation for batteries and the raw materials used. A categorization table was developed and constructed the inventory of information available on its stocks. This process comprises the number of batteries in the market and the flows of the damaged batteries gathered and treated. The outputs are shown for lithium-ion batteries and nickel-metal hybrid batteries.

H. Oman [10] The features of a gasoline-controlled vehicle are generally defined in terms of access speed, travel range, acceleration, fuel economy and life cycle price. The per kilometer price of energy for driving an electric vehicle is a fraction of the per kilometer price of energy for a gasoline drive vehicle. On the other hand, electric vehicle energy has to be stored in a battery that must be recharged. In electric vehicles, none of the batteries is capable of fully meet of these performance necessities. A lithium-ion battery, which stores the most energy is being manufactured for personal computers and laptops, where long life is not required. In a hybrid vehicle, fuel burning vehicle engine output power is supplemented with a battery. A battery is capable of providing peak power and recovering energy with dynamic braking. The best possible vehicle presentation with an available battery can be achieved if the vehicle has an on-board energy administrator which restricts the life falling pressure on the battery and frequently shows to the driver the remaining journey distance available with the energy in the battery. Nowadays, the best batteries choice for electric vehicles is the lithium-ion polymer, nickel-cadmium, lead-acid, nickel-metal hybrid and zinc-air types.

Z. Xu, et. al. [11] To indicate the strength of the power battery, the lithium batteries state of health is one of the best indicators. The aspects disturbing state of health has been complicated concerns. There are two most important research techniques, first is based on vehicle information for the online state of health assessment and analysis and another is based on the experimental situations of state of health assessment. In this paper, based on real vehicle information of electric vehicles, the authors have explained the charging characteristics and battery state of health with constraints. To indicate the charging characteristics it uses charge depth, current and charge frequency, and to indicate battery state of health it utilizes charge capability. To investigate the consequence of charge characteristics on battery state of health, the major affecting aspects of battery state of health are determined.

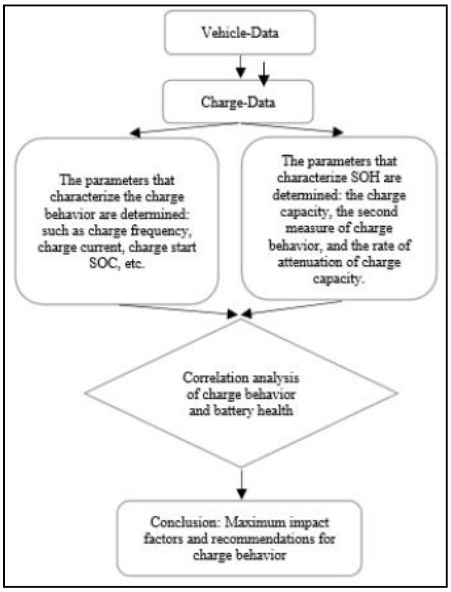

Fig. 6: The vehicle information investigation procedure

M. Nko, et. al. [12] Nowadays, solar energy is becoming one of the fastest rising renewable energy systems as it is simple and requires less maintenance. Solar energy can be employed in many applications such as home installations, street lightning, major projects associated with the grid, and installed on rooftops to power appliances directly. Such applications make solar energy the most attractive. Such energy requires storage ability to be conserved to subsequently utilize. The battery is known for its capability to store energy for subsequent utilization. The present paper projected why the lithium-ion battery is the future for renewable energy storage. With several ranges of storage batteries available in the market, lithium-ion batteries are growing as the best possible trust storage.

F. L. Mapelli, et. al. [13] Recently, many resources have been engaged on small discharge public vehicles both for public and private transportation. One of the possible solutions for public transportation like buses is a complete electric grip scheme fed by a supercapacitor that could be recharged at every stop. In reality, these kinds of vehicles are capable of covering a very short distance, generally less than a kilometer, for this cause the energy storage system required to give the low amount of energy and high peak of power. The hybrid energy storage system requires an energy administration purpose that has been constructed employing a mathematical simulation model. In this paper, the authors first proposed a mathematical simulation model and then designed a heuristic administration algorithm to deal with the onboard energy storage in the batteries as well as in a supercapacitor.

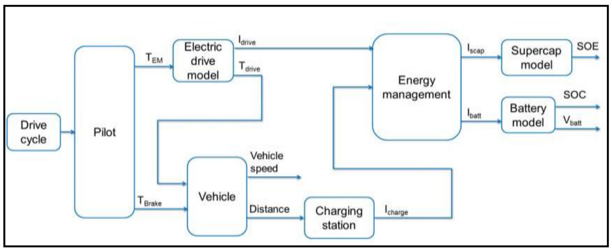

Fig. 7: Proposed simulation model

Toshio Matsushima, et. al. [14] The authors have proposed a DC power supply system designed for an outdoor setting with a maximum output current of 32 amperes and constructed employing a fixed lithium-ion battery and functionality examination was taken out in labs. The recharging features of the lithium-ion batteries are calculated by steady voltage charge and steady current charge technique and almost provides a similar discharging period that ranges from one hour to a few hours. It was observed that the cell voltage was over 3.8 volts per cell during discharge at similar current rates. By applying the lithium-ion batteries, it was found that the load of the batteries was decreased by about 25% and the volume of the system was decreased by 33% when compared with a traditional system with a valve regulated lead-acid batteries.

A. Kuperman, et. al. [15] In an electric vehicle the traction battery is the most important element as the battery features are strongly influenced by the load, price, dependability and driving range of the vehicle. In this paper, the authors have presented the performance of a fast charger for a lithium-ion electric vehicle forward motion battery.

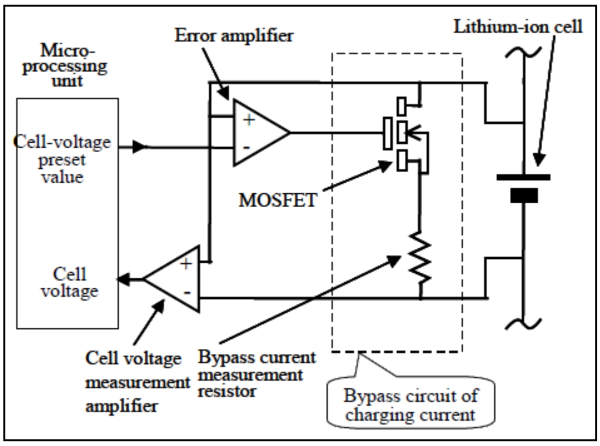

Fig. 8: Circuit of cell voltage equalizing element

The proposed charger is implemented as a dual-stage controlled direct current or alternate current converter. The initial stage contains a full-bridge diode rectifier with a push dynamic power filter. The initial step produces an unrestrained pulsating direct current bus while obeying the grid codes. The final stage contains interleaved groups of two parallel direct currents to direct current converters, fed by the unrestrained direct current bus and performing the battery charging procedure. The proposed charger has the ability to function in steady current, steady voltage and steady power charging mode.

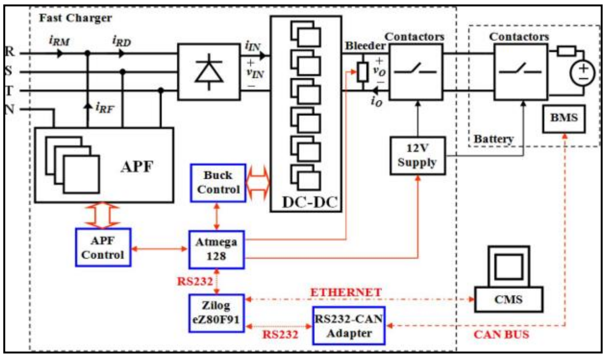

Fig. 9: Block diagram of fast charger system level

D. A. Wetz, et. al. [16] Nowadays, recharging electrochemical energy storage devices at higher speeds, such as supercapacitors, rechargeable batteries and lithium-ion capacitors is a challenging task. Some research has been done to improve the performance of charging the battery. The main objective of the research is to understand the charge rate restrictions of the devices when the eminent charge is applied in a pulsed manner. In this paper, the authors have explained the improvement in the field of a power electronics-based test stand then could be employed to transmit charge from a superior battery storage system into an individual cell utilizing pulsed currents.

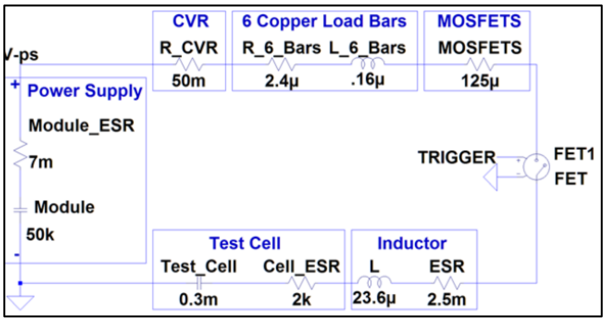

Fig. 10: Experimental recharge test stand

S. Lansburg, et. al. [17] For information technology data hub and high voltage uninterrupted power supply applications, choosing a lithium-ion polymer battery pack for critical standby power source could help consumers to advantage from a compacted installed footprint and compacted atmospheric footprint when contrasted to traditional battery solutions. High power lithium-ion batteries used as system backup have the capability to considerably decrease the installed ability while providing a similar discharge duty when a small backup period is essential. This paper shows that with a lesser amount of stored energy combined with the extremely competent recharge of the lithium cells, the full amount of energy recharge required to sustain 100% duty contributes to condensed entirety facility energy. These advantages are highlighted by the ability of the lithium-ion systems to function for a comprehensive time at the higher temperature.

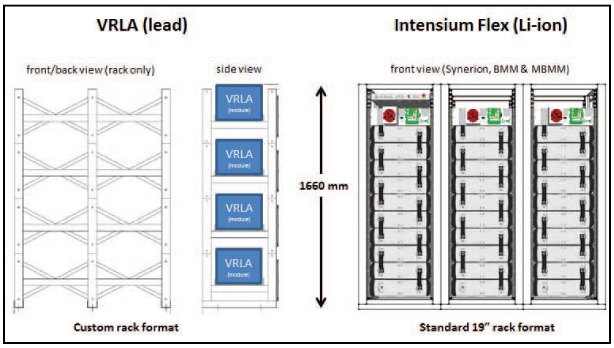

Fig. 11: Battery racking formats

F. A. LeBel, et. al. [18] Frequently collections of cells in parallel are considered to function as one individual superior element with homogeneous properties. As this might be seen as right, but it is partially wrong because it avoids the effect of the cell-to-cell current deviations and the balancing currents on pack features. Consideration of small cells into larger cells obstructs the probability of investigating the effects of inner resistance of deviations between cells on current allocation and heat production. In this paper, the authors have presented a simple electro-thermal battery module based on information of electrical and thermal characteristics of individual cells valuable for design intentions. Experimental lookup tables are employed to analyze the model constraints like the interior resistance improvement, entropic coefficient and open-circuit voltage. The experimental outputs of complete recharge, complete discharge and an interrupted discharge are shown.

III. Synthesis of Lithium Manganeze Oxide ($LiMn_2O_4$)

When the cathode is prepared, using (LiMn2O4), at higher temperature ranges, due to the fundamentals of the solid-state reactions, the final products contain, irregular particles, random distribution and high impurity. Apart from having these lacunas, it is also essential to consider the control on the crystalline growth, compositional consistency, geomorphology and microstructure features. To address such multiple issues, we are projecting the preparation of the cathode material using LiMn2O4 material and most popular Hydrothermal process. The synthesis of the LiMn2O4 material is carried out at low temperature ranges. Using hydrothermal process, it is possible to synthesize the metal oxides at low temperature ranges to obtain the same synthesis outcome as we get at 7000C to 9000C ranges. LiMn2O4 powders are hydrothermally synthesized without any additional mixtures and without having any additional chemical process. Initially, LiOH·H2O, MnO2 and Mn(NO3)2 are put into a sterilizer. The amount of MnO2 is controlled at 0.061 mol, and Mn(NO3)2 is 0.012 mol. The sterilizer is filled with the distilled water to 70% of the capacity, sealed and kept at varying temperature from 1200C, 1400C, 1600C and 1800C for fixed duration of 20 hours, and then cooled to room temperature naturally. After the hydrothermal reaction, the products were washed using distilled water and separated in a centrifuge, and then dried in air. Finally, the characterization of the prepared materials is discussed in the following section.

IV. Characterization of of Lithium Manganeze Oxide ($LiMn_2O_4$)

Characterization of the material assures how close the prepared material is come out from the expected features of the material. Out of available numerous characterization techniques, X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Thermogravimetric Analysis (TGA) techniques are adopted. Outcome of each of the characterization technique is discussed in the following section.

A. X-Ray Diffraction (XRD)

The X-ray diffraction pattern (XRD) of $LiMn_2O_4$ processed for fixed time duration of 20 hours and under different temperature range that is at 120^0C, 140^0C, 160^0C and 180^0C is disclosed in the subsequent figure.

All recognizable reflection peaks including (111), (311), (222), (400), (331), (511), (440), (531), (533), (622), (551) & (731) can be clearly indexed to the single phase of the spinel cube structure of LiMn2O4 (JCPDS file no. 88-1749) with space group Fd3m. no other impurity phases were detected. All synthesized samples were identified to have a $LiMn_2O_4$ crystal structure with the highest peak at an angle of 2θ = 18.7100 with a plane distance of d = 4.7387Å. Based on the x-ray pattern, it can be seen that the active material of the $LiMn_2O_4$ cathode has a low level of crystallinity, this is indicated by the level of peak intensity obtained at each diffraction pattern.

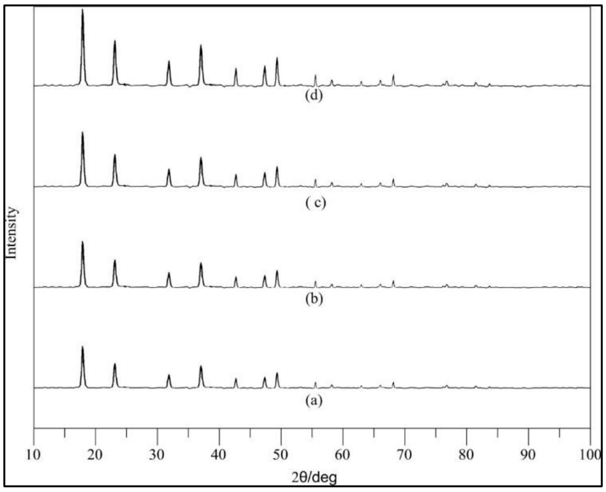

Fig. 12: XRD patterns of the products keeping time constant and at varying temperature of (a) 120^0C, (b) 140^0C (c) 160^0C and (d) 180^0C for 20 Hrs.

In all x-ray pattern one extra peak appear at angle 2θ = 48.070Å, which correspond to Li_2O impurity. From the data it is seen that as the synthesis temperature increases, the peak intensity indicates the $LiMn_2O_4$ phase is getting sharper, while the peak showing the Li_2O impurity decreases.

B. Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy is the technique that is used to create an image of the surface resulting due to the focused beam of the electron. In this technique, the electrons interact with the atoms in the sample and produces various signals which carry information about the surface topography and compositions of the material under test. The electron beam which is bombarded on the material is scanned in a raster scan pattern and the position of the beam is combined with the intensity of the detected signal to produce an image. Scanning Electron Microscopy (SEM) output of the $LiMn_2O_4$ are disclosed in the subsequent figure.

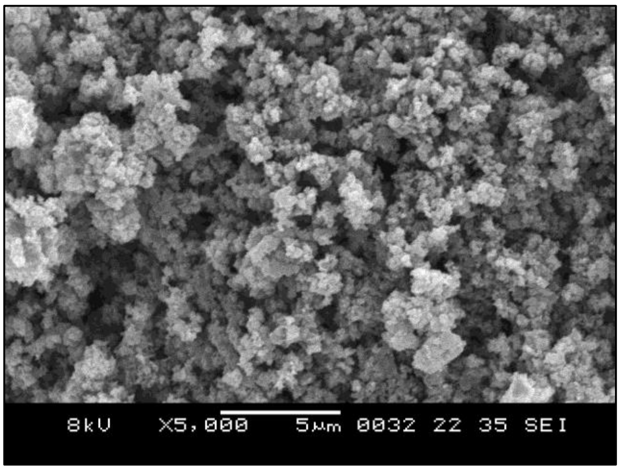

Fig. 13: SEM image of $LiMn_2O_4$ prepared by hydrothermal method at 120⁰C

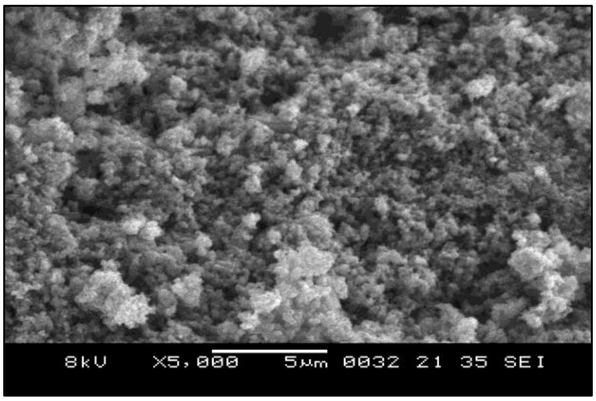

Fig. 14: SEM image of $LiMn_2O_4$ prepared by hydrothermal method at 140⁰C

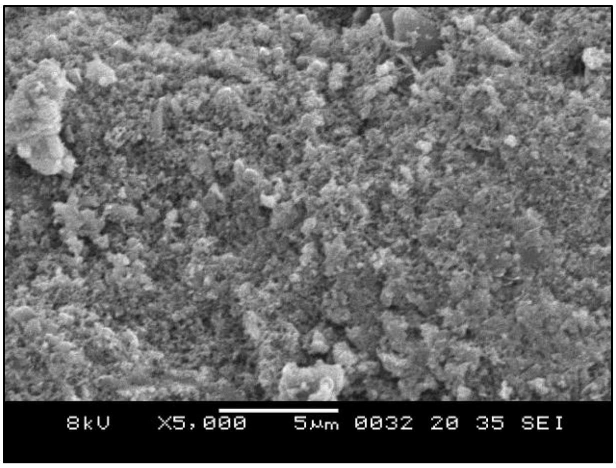

Fig. 15: SEM image of $LiMn_2O_4$ prepared by hydrothermal method at 160⁰C

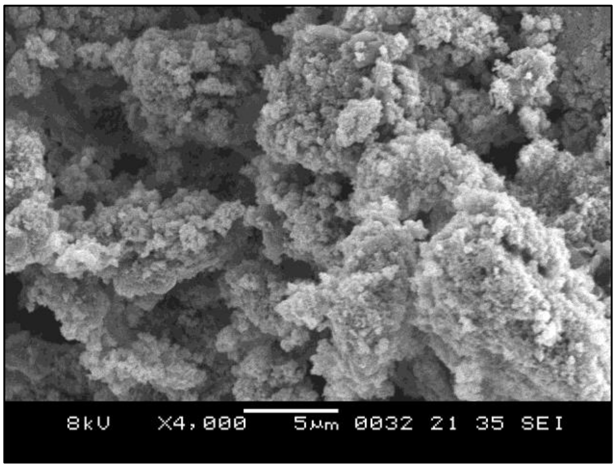

Fig. 16: SEM image of $LiMn_2O_4$ prepared by hydrothermal method at 180⁰C

Better crystallization can be seen through the above image. The distinctive well defined crystal faces exhibit single spinel crystals of octahedral shape with well-developed planes.

C. Thermogravimetric Analysis

The Thermogravimetric Analysis is carried out to evaluate the thermal stability and fraction of volatile component of the material under test. The evaluation is carried out by observing the weight change that is occurring with respect to the change in the heat applied to the material. Typical Thermogravimetric Analysis (TGA) curve of the $LiMn_2O_4$ powder prepared by the hydrothermal method is shown in the subsequent figure. Up to 130⁰C, it shows that there is only a slight weight loss of 0.5%.

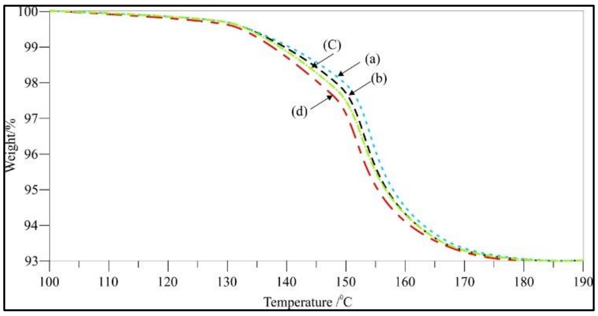

Fig. 17: TGA Curve of the $LiMn_2O_4$ Powder at (a) 120⁰C, (b) 140⁰C, (c) 160⁰C and (d) 180⁰C

Further, the most weight loss occurs above 150⁰C, which is attributed to the volatilization of lithium at such high temperature. Below 130⁰C, there is no distinct weight loss in the TGA curve. Further, $LiMn_2O_4$ powder prepared by the hydrothermal method has a steady graph after 180⁰C.

V. Electrode Preparetion

The electrode is made by dispersing a mixture of 80w% active materials, 5w% carbon black and around 2w% PVDF binder into dimethyl phthalate to obtain a slurry. The slurry is spread onto aluminum foil. Once the electrode is dried at 1200C for one and half hour, it is compressed at a rate of about 120kg/cm2. The electrodes are then dried in a vacuumed furnace for 20 hours and it is then transferred to a glove box. The electrode area is approximately 1 cm^2, and the typical thickness of the electrode is about 100μm.

VI. Electrochemical Analysis of Lithium Manganeze Oxide ($LiMn_2O_4$)

The discharge curves at various temperatures are disclosed in the subsequent figure, there are four clear discharge patterns are observed.

The $LiMn_2O_4$ cells once prepared and characterized using different characterization techniques, then finally its electrochemical performance is evaluated. For this purpose, specific discharge curve are obtain for analysis purpose. In the proposed research work, the electrochemical performance is evaluated. It is observed that the cells can be charged between 0⁰C to 60⁰C and then discharge period is observed at different operating temperatures. In our research work, as shown in the above figure, the discharge of the cells are observed at (a) 60⁰C, (b) 25⁰C, (c) 0⁰C and (d) -20⁰C.

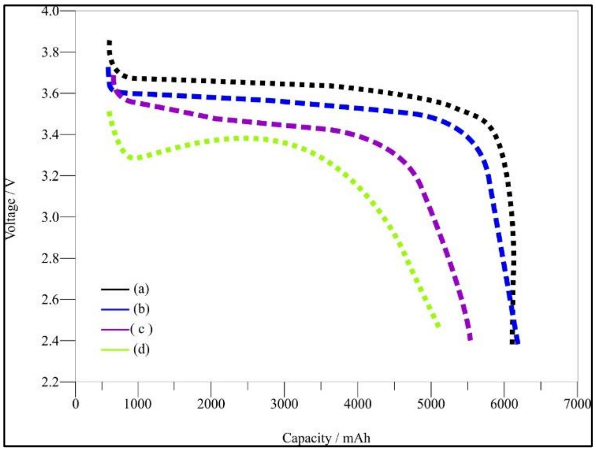

Fig. 18: Discharge Voltage at (a) 60⁰C, (b) 25⁰C, (c) 0⁰C and (d) -20⁰C

VII. Conclusion

The research work can be concluded through the following points:

- the battery cells prepared using $LiMn_2O_4$ are charged between 0⁰C to 60⁰C temperature and discharged between 20⁰C to 60⁰C.
- Through experience gained while multiple charge and discharge cycles, the optimum performance can be fetched by keeping the operating temperature to 23±2⁰C.
- It is observed that as we go on decreasing the operating temperature, cell delivers low voltage and at lower capacity.
- Also, the lowered capacity, at lower operating temperatures than standard operating temperatures results into lower energy delivery.
- On the other hand, as we go on increasing the operating temperature above the standard operating temperature, as the data is recorded for up to 60⁰C, it is observed that, cells deliver higher voltage at higher capacity.
- This is because, at higher temperature there is better flow of electrons is material.
- But, at higher operating temperatures, high voltage performance and at high capacity is at the cost of lowering the cycle life of the cells.
- In addition, keeping environment sustainability and concept of relative humidity in mind, lithium-ion cells must be operated in the range of 0% RH to 60% RH.

- Hence, performance evaluation of the discharge cycle is carried out at standard operating temperature that is in the range of 25⁰C. It is observed that, higher voltage at capacity nearly equal to the one received at the higher operating temperatures and with better cell cycle is observed at 24⁰C.

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