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Microwave Preparation, Characterizations and Thermal Study of Nano sized $LiMn_2O_4$

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ABSTRACT

Microwave synthesis method for the synthesis of nanomaterials is the energy efficient method, less time consuming method and also known for its simplicity. Metal oxide nanomaterial preparation using microwave route finds much importance in the field of synthetic technology. Here is an attempt to prepare lithium manganate ($LiMn_2O_4$) nanomaterials by microwave route using polymer as a fuel. As prepared lithium manganate sample was characterized for its structure by employing powder X-ray diffraction (XRD) tool. The morphology of the prepared oxide material was studied by Scanning Electron Micrograph (SEM) tool. Fourier Transform infrared (FTIR) spectral study was undertaken to know the bonding in the prepared oxide sample. Thermal behavior of the sample was well studied.

Keywords: Microwave, nanomaterials, Bonding, Structure, Morphology.

INTRODUCTION

The recent research diverted towards metal oxide nanomaterials due to its specific properties and potential applications. Metal oxides ceramics are given much importance because of its high conduction and high dielectric properties [1-3]. Synthesis of metal oxide materials at nano dimension were attracted to many researchers because of its great technological importance [4-5]. Interest has increased following the observation that the properties are strongly dependent on the size of particles with dramatic changes when nanometric sizes are acquired [6-7]. Microwave route of the preparation of metal oxide materials attracted many researchers for the preparation of nano structured metal oxide ceramics and is under constant investigation due its much importance for its simplicity [8-9].

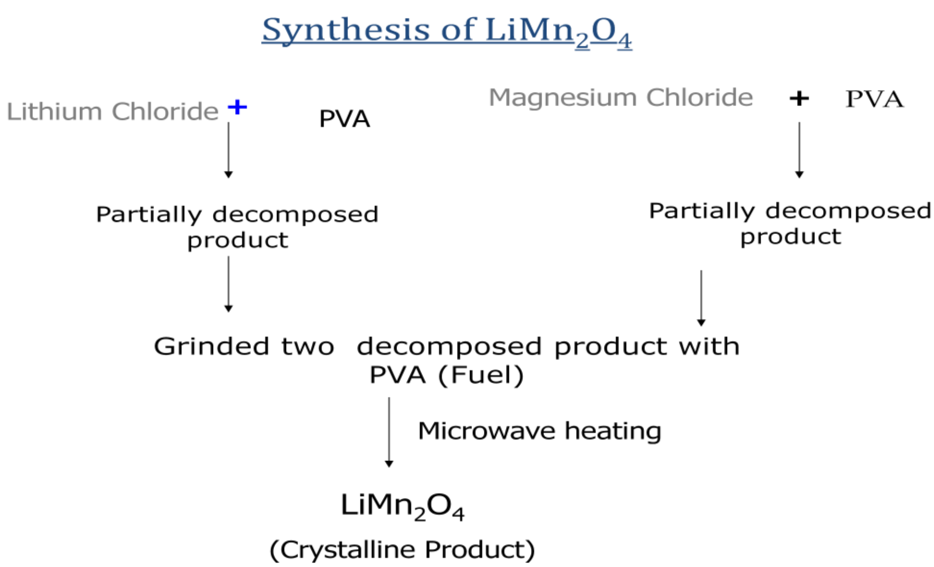
Lithium manganate is electrode materials for lithium ion batteries because of its high reduction potential and acceptable environmental impact[10]. Combustion of reaction mixture converts the required solid crystalline product at moderate rate and gives the application oriented crystalline product. Metal oxide ceramics obtained by combustion route shows crystalline nature and also good morphology [11]. Among the various metal oxides, $LiMn_2O_4$ is an important ceramic oxide used for various applications, especially in battery material. Hence, much attention is given for the preparation, characterization and properties of $LiMn_2O_4$ material.

Present investigation reports the synthesis of lithium manganate nanomaterials employing microwave route. Polyvinyl alcohol is used as a fuel for the preparation of $LiMn_2O_4$ nanomaterials. Combustion treatment is given for the complete conversion of salt in to oxide nanomaterials. As prepared lithium manganate sample was well characterized for its structure by X-ray diffraction (XRD), morphology by Scanning Electron Microscope (SEM) and bonding by Fourier Transform Infrared study (FT-IR) techniques. Thermal stability of the sample was studied by thermal method.

MATERIALS AND METHODS

Materials: Lithium Chloride, Manganese chloride, polyvinyl alcohol chemicals are used in the present study was of AR (Analytical Reagent) grade. Polyvinyl alcohol is used as fuel for the combustion process and Microwave method is adopted for the synthesis of lithium manganate nanomaterials.

Preparation of Lithium manganate nanomaterials: One gram of lithium chloride and manganese chloride ignited with polyvinyl alcohol fuel in a separate container on electrical oven to get partially decomposed product. After complete evolution of the fumes, both samples were again grinded with polyvinyl alcohol in weight ration 1:1:4. The partially decomposed product formed may be may be attributed to the low temperature of the reaction giving rise to insufficient energy needed for complete conversion. Hence, the sample was under microwave irradiation in domestic microwave woven having frequency 2.45GHz for about ten minutes at highest power level. The solid burns by producing different coloured light and leaving behind lithium manganate nano product. The preparation scheme is given in the scheme 1 [12].



Scheme-1

Characterization: The structures of as prepared zinc oxide were studied by X – ray diffraction using Phillips X – ray diffractometer (PW3710) with Cu Kα as source of radiation. Morphology and bonding of the above oxide was studied by Phillips XL 30 ESEM and Perkin–Elmer 1600 spectrophotometer in KBr medium tools respectively. The thermal studies were undertaken using Mettler Toledo Star instrument.

RESULTS AND DISCUSSION

X-ray diffraction: Figure-1 shows XRD pattern of prepared $LiMn_2O_4$ sample. The pattern shows the presence some peaks confirm the formation of crystalline product. The d-spacing values of the sample matches well with standard 35-782 JCPDS file. Unit cell parameters ware obtained by least –square refinement of the powder XRD data. This study reveals that the sample is mono phasic lithium manganate with cubic spinal structure having nano sized particles.

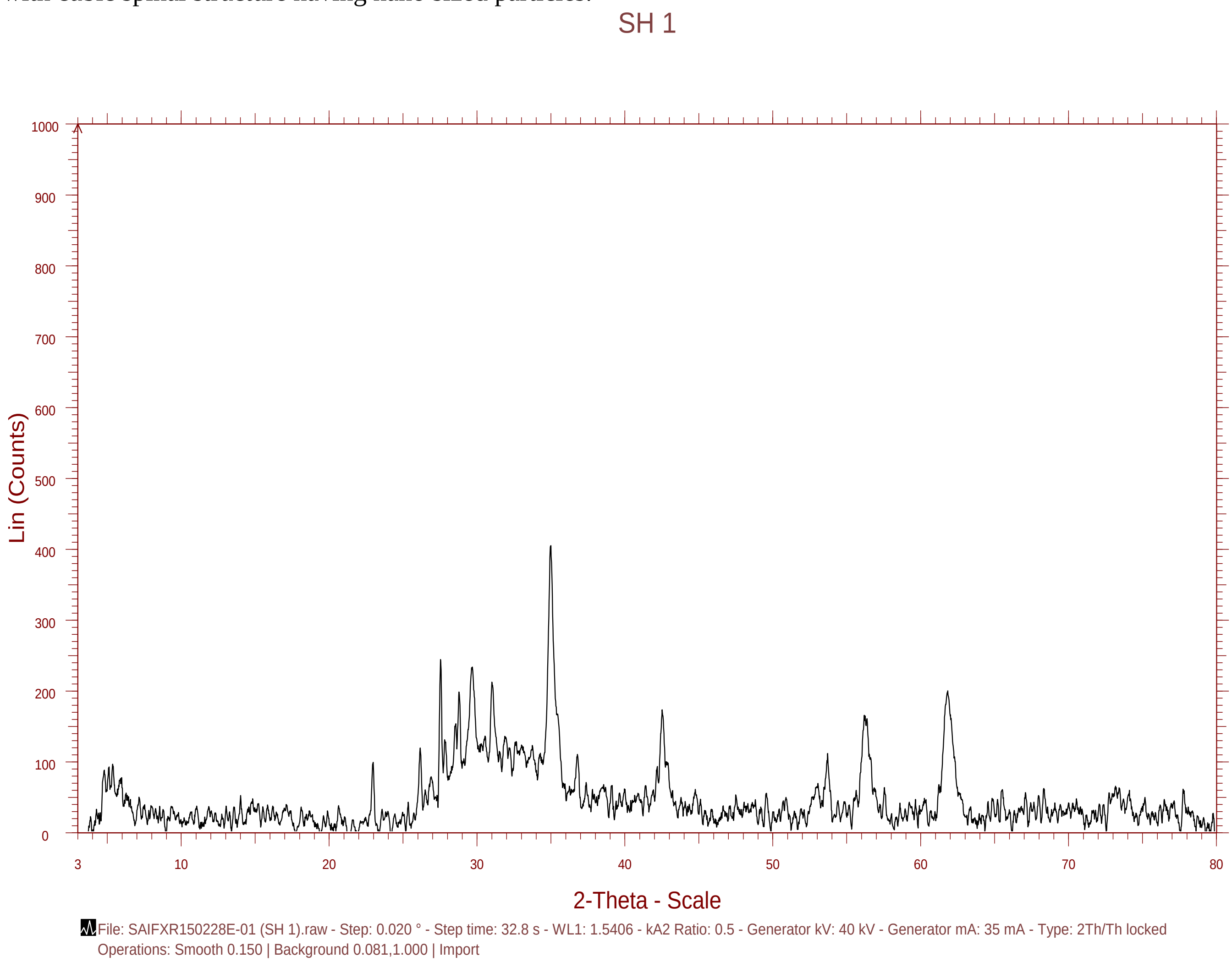


Figure-1: XRD pattern of $LiMn_2O_4$ sample

Scanning Electron Microscopy (SEM): Figure-2 shows SEM image of prepared lithium manganate sample. This image shows the most of the particle agglomeration and shows irregular shape particles. In addition to this a close compact arrangement forms due to crystalline behavior is also observed.

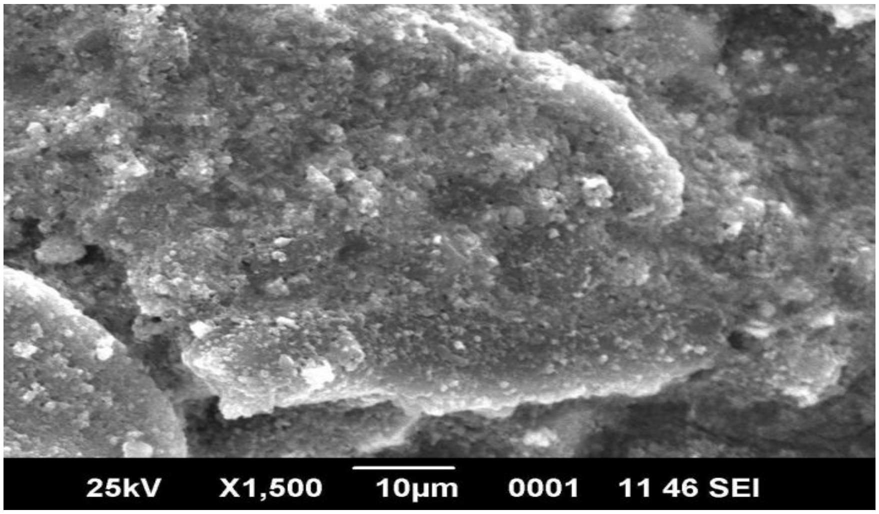


Figure-2: SEM image of $LiMn_2O_4$ sample

Infrared Study: The metal- oxygen bonding and nature of the synthesized lithium manganate sample was carried out by infrared tool. Metal oxides generally give absorption bands below 1000cm^{-1} arising from inter-atomic vibrations [13]. Observed vibrational frequencies of the said sample are given in the table-1. The sample shows the absorption in the region 3435, 2161, 1666, 1456, 1245, 1130, 1000, 928, 672, and 541cm^{-1}. The peak 3400 cm-^{1} corresponds to water of absorption and the peak at 1245 and 1130 cm^{-1} may be due to the presence of some overtones. The peaks below 1000 cm^{-1} corresponds to Metal-oxygen (M-O) vibrational modes of the sample. This conform the formation of lithium manganate sample.

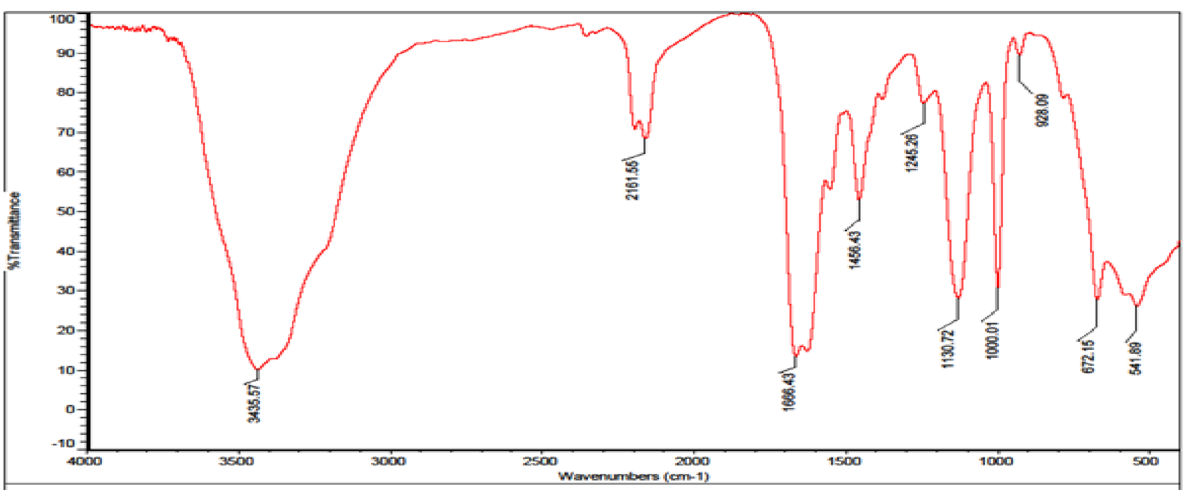


Figure-3: FT-IR spectra of $LiMn_2O_4$ sample

Table-1. Vibrational Frequencies if $LiMn_2O_4$

Sample	$LiMn_2O_4$
Vibrational frequency(cm^{-1})	3435, 2161, 1666, 1456, 1245, 1130, 1000, 928, 672, 541

Thermal Study: Figure-4 shows TGA trace of $LiMn_2O_4$ sample. The trace shows two step weight loss, the first weight loss is due to the loss of adsorbed moisture present in the sample. A second loss from 150 to 300 ^{o} C indicates the weight loss due to due to decomposition of the sample. The weight loss is slow process and is multistep one.

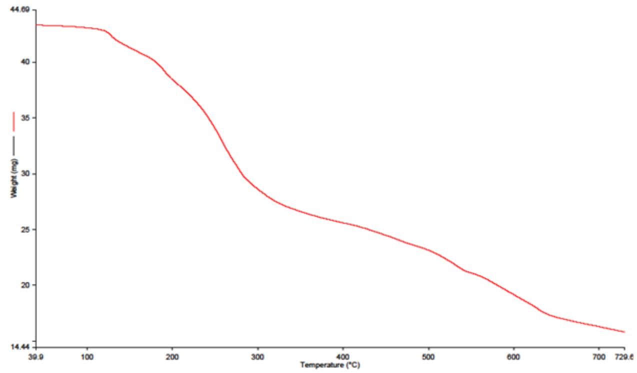


Figure-4: TGA trace of $LiMn_2O_4$ sample

APPLICATIONS

This method can also be useful for the synthesis of other metal oxide materials at nano dimensions

CONCLUSIONS

Microwave assisted route is used for the synthesis lithium manganate sample using polyvinyl alcohol as fuel. This method finds its importance because of its simplicity and less time consuming. Hence, this method can also adopt for the synthesis of other metal oxide materials at nano dimensions. Crystalline materials formed with simple way with applicable morphology.

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