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Synthesis of $Li_{1.25}Mn_{0.125}Ni_{0.125}Co_{0.5}O_2$ nano powders, via sol-gel method, as a candidate for cathode material of lithium - ion batteries and investigating the effect of gelatin amount on their physical and structural Properties

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Abstract
In this research, $Li_{1.25}Mn_{0.125}Ni_{0.125}Co_{0.5}O_2$ Nano powders, were synthesized using sol-gel method at different temperatures, lithium nitrate, manganese nitrate, nickel nitrate, cobalt nitrate as raw materials. Thermal and thermal gravimetric of TGA/DTA, characterization of structure properties of samples prepared by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) were performed. Also, with X-ray diffraction scheme, at different temperatures, it was concluded that the calcination temperature is suitable for synthesis. The size of Nano crystals was determined using Scherer and Williamson Hall method. The FTIR results showed that the metal bands in the 500 region increased sharply with increasing temperature to 850C, which is consistent with the XRD results.

Keywords: Cathode, lithium ion battery, sol-gel method

Introductions

Human dependence on energy in everyday life is constantly increasing, even with the advancement of various technologies, which makes the need to use energy resources and its optimal use more and more obvious. Basically, one of the signs of economic growth is the increase in energy consumption. Economic calculations made in recent years have shown that the rate of increase in world energy consumption is constantly increasing and is expected to increase again [1]. In 1990, it was the most significant development in environmental technology. Until then, portable devices were traditionally powered by gasoline. Economic and environmental problems related to the use of fossil fuels are improving. Considerable efforts have been made in the energy industry to replace fossil fuel sources with electrochemical energy conversion components such as fuel cells and electrochemical energy storage components such as super capacitors and batteries [2]. By using a combination of super capacitors, fuel cells and batteries, it is possible to find an alternative to combustion engines that do not require fossil fuels, fuels that produce greenhouses, which has caused concern in modern society [3]. By the limitation of fossil fuels and a long period of rehabilitation, human beings seek to use new and renewable sources of energy such as solar, wind, waves, waters and so on. But since these types of energy are not available in times of need, it is imperative that most of them be stored in the time frame of energy and consumed in times of need. The battery is a source of electrochemical energy that converts the energy released from a chemical reaction directly into electricity [4]. The desperate need for portable electronics that are both lightweight and compact has led to a major international effort to develop rechargeable batteries. Therefore, the commercialization of li-ion batteries has attracted the attention of the world market [5, 6]. Li-ion batteries at this time have the highest energy density among the rechargeable battery industry, and the use of these batteries in electronic devices has been evolving in recent decades. However, the use of li-ion batteries in portable vehicles is necessary to improve various factors such as energy density, durability, safety and cost [7, 8]. Batteries are programmed and adjusted differently with different voltage, current, power and discharge time levels. Proper use of batteries requires the provision of an electrical model that mimics their electrochemical behavior. Batteries directly contribute to the advancement and expansion of portable electronic technology for the necessary and appropriate energy for vehicles [9]. The electrochemical efficiency of a

battery depends on its cathode. Many cathodes materials are used in li-ion batteries, among which metal-dependent oxides such as $LiCoO_2$, $LiMn_2O_4$, $LiNiO_2$, $LiMnO_2$ can be named [10]. The structure of $LiCoO_2$ has problems such as the high cost and toxicity of cobalt. To solve this problem, $LiMO_2$ structures (M = Co, Ni, Mn) have been considered due to the high capacity in the range (250 mAh/g2) and low costs as well as the reduction of toxicity compared to today's commercial $LiCoO_2$ batteries [16].

Conclusion and Discussion

In order to study the performance of the sample heat more precisely and to determine the temperature required for calcination in the sol-gel method, the TGA, DTA curves for the dried gel sample $Li_{1.25}Mn_{0.125}Ni_{0.125}Co_{0.5}O_2$ are shown in Fig 1. An exothermic minimum is observed around 88.02C in the DTA diagram, corresponding to the weight loss in the TGA diagram, which is due to the water absorbed during the mixing of the precursors and the decomposition of the gelatin. In the 320C to 460C range, the exothermic peak appears in the DTA diagram, which, with a sharp mass peak in the TGA diagram, may be related to molecular decomposition. Its intensity is in 441.77C, which is related to the decomposition of precursors and then the formation of raw materials [15]. From then until the temperature of 320C to 800C is not observed. Which probably indicates the crystal structure of the substance by the way, by adding some gelatin, the heating process increases, according to Fig (1b) it is 190C, Fig (1a) is 215C and Fig (1c) is 240C. The total mass lost in this temperature range in a sample (1:1 is about 48.71%, 1:2 is about 62.01% and 1:4 is about 81.35%).

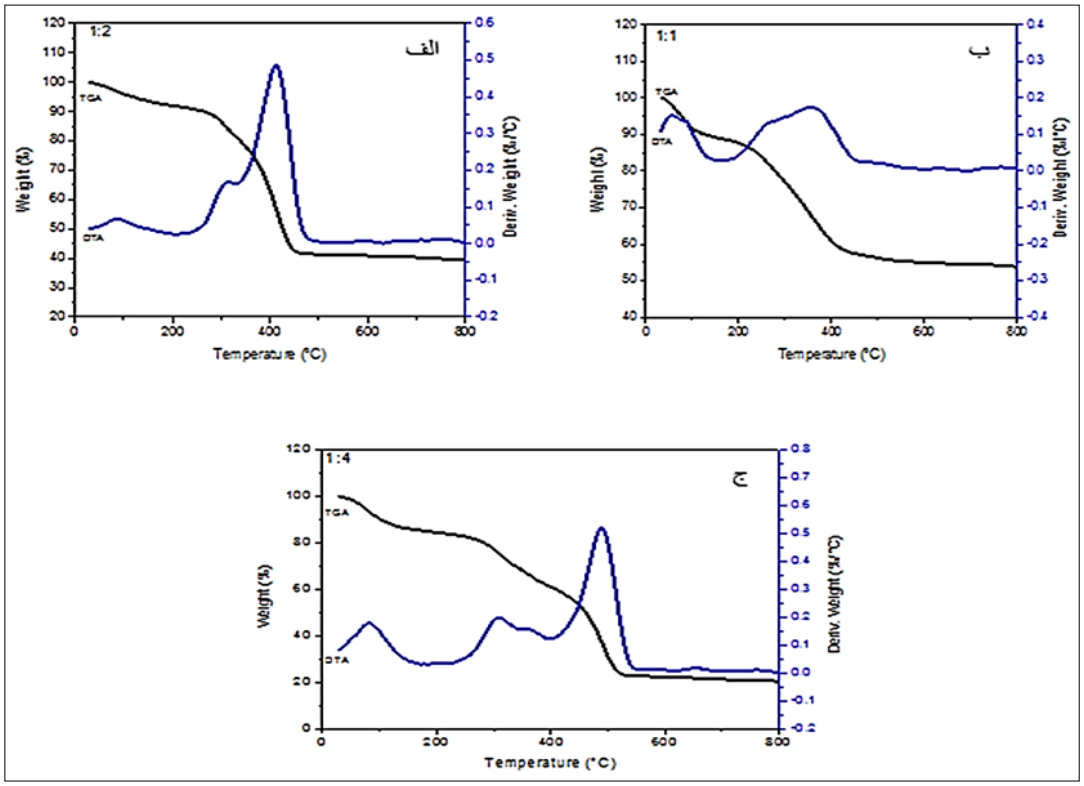

Fig 1: DTA/TGA curve for gel dried in $Li_{1.25}Mn_{0.125}Ni_{0.125}Co_{0.5}O_2$ powders, a) 1:2, b) 1:1 and c) 1:4

The X-ray diffraction pattern of the sample at different temperatures is shown in Fig 2. Most of the main spectral lines can be identified according to the hexagonal structure with the spatial group R3m, which indicates the presence of $LiMO_2$ phase [7].

The presence of weak peaks in the area of 20-25 indicates the presence of Li_2MnO_3 monoclinic phase with spatial group (C2/m) which is due to the movement of lithium ions to the metal layer mediating the Li_2MnO_3 component and the formation of a super grid in it [8]. Several features indicate the high crystallinity of the specimens: (1) Bragg's narrow lines, (2) separation of dual reflection lines (206)/(060) and (133)/(200). (3) Higher ratio of 1.2 intensity of the two main peaks of the XRD diagram, I (002)/I (202) [12].

Peaks obtained at lower temperatures are less intense. As the temperature rises to 850C, the intensity of the peaks increases and the pairs of peaks (060), (206) and (200), (133) are clearly separated, which improves the layered structure and crystallization. Shows well at this temperature [14].

The size of nanocrystals was determined by Scherer and Williamson-Hall methods. In Debbie Scherer method using the following relation:

$$D = \kappa\lambda/\beta\cos\Theta \qquad (1)$$

In Equation 1, λ the mean size of the crystals is, which may be less than or equal to the grain size. Bragg angles are [13]. According to Table 1, the crystal size of the sample is 1:4 at different temperatures.

Table 1: Average size of crystals for sample 1:4 at different temperatures

Temperature in celsius	2Θ		D (nm)
700	18/16	19/8	18/48
800	18/28	19/64	25/2
850	18/36	19/64	23/1

On the other hand, we know that the widening of Bragg peaks is due to factors such as the share of flattening (due to machine errors), strain and the size of the crystals. Both the strain and magnitude effects can be examined using the Williamson-Hall method.

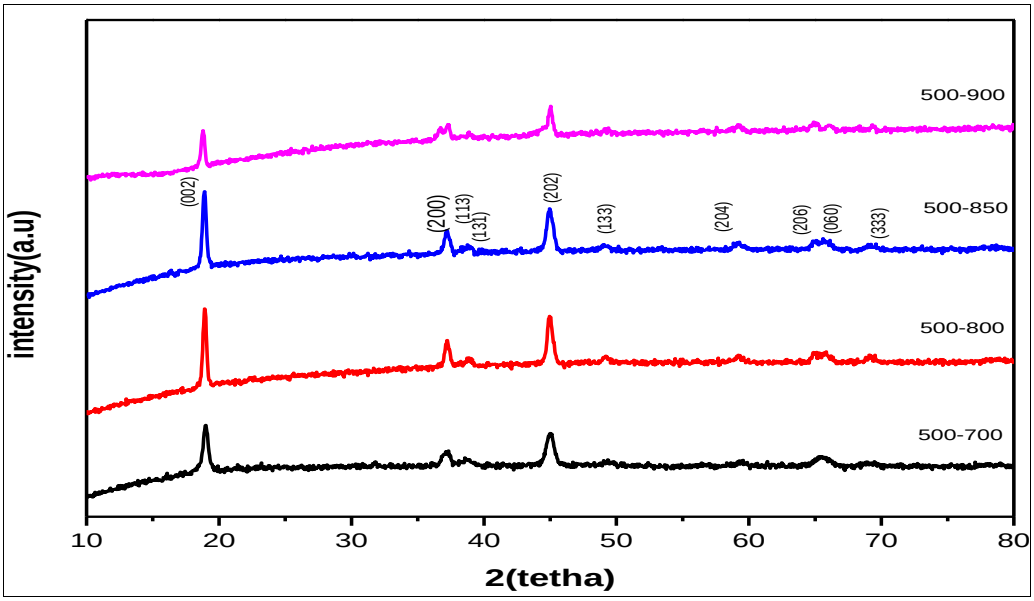

Fig 2: X-ray diffraction of $Li_{1.25}Mn_{0.125}Ni_{0.125}Co_{0.5}O_2$ nanoparticles at temperatures of 700C, 800C, 850C and 900C

Infrared spectroscopy based on radiation absorption has been used to investigate the vibrational mutations of molecules and polyatomic ions. FTIR spectroscopic results for $Li_{1.25}Mn_{0.125}Ni_{0.125}Co_{0.5}O_2$ nano powders, between is drawn in Fig (3). By increasing the temperature, as seen in the XRD results, the organic matter is removed and the metal peaks below become stronger and the structure becomes more crystalline. In this example, the valleys in the wave number, less than the wave number, are related to fluctuations in the octahedral [9]. Valleys are in the range of wave number and are related to different bonds and bending fluctuations of the water group and symmetric and asymmetric group bonds. [9, 10].

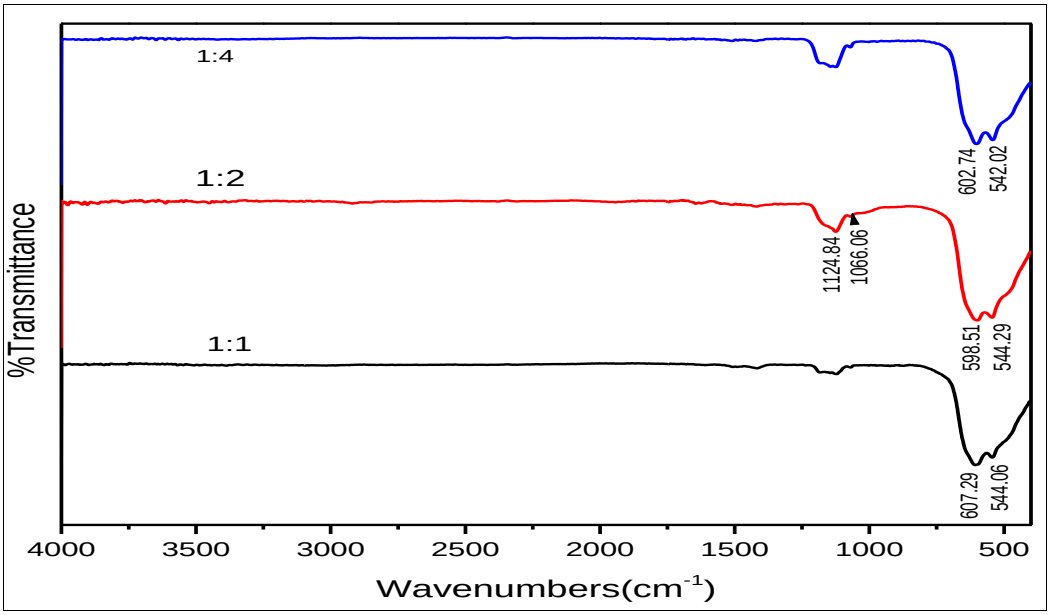

Fig 3: Spectrum of FTIR nano powder $Li_{1.25}Mn_{0.125}Ni_{0.125}Co_{0.5}O_2$, 1:1, 1:2 and 1:4 sample

Morphology (FESEM)

Field emission scanning electron microscopy (FESEM) was used to examine the morphology of the magnified images of $Li_{1.25}Mn_{0.125}Ni_{0.125}Co_{0.5}O_2$ powder. Scanning electron microscope is one of the branches of electron microscopes. By performing a series of scanning operations by electron beam, high quality images are prepared from the sample surface. Also shown in Figure 4, the particles are well crystallized. All sample ratios have approximately the same hexagonal (hexagonal) morphology and have sizes between 100-150 nm. The particle size has been slightly reduced by changing the gelatin to a ratio of 1:2. The distinctive edges and smooth sides of the dense bamboo are a characteristic feature of these particles.

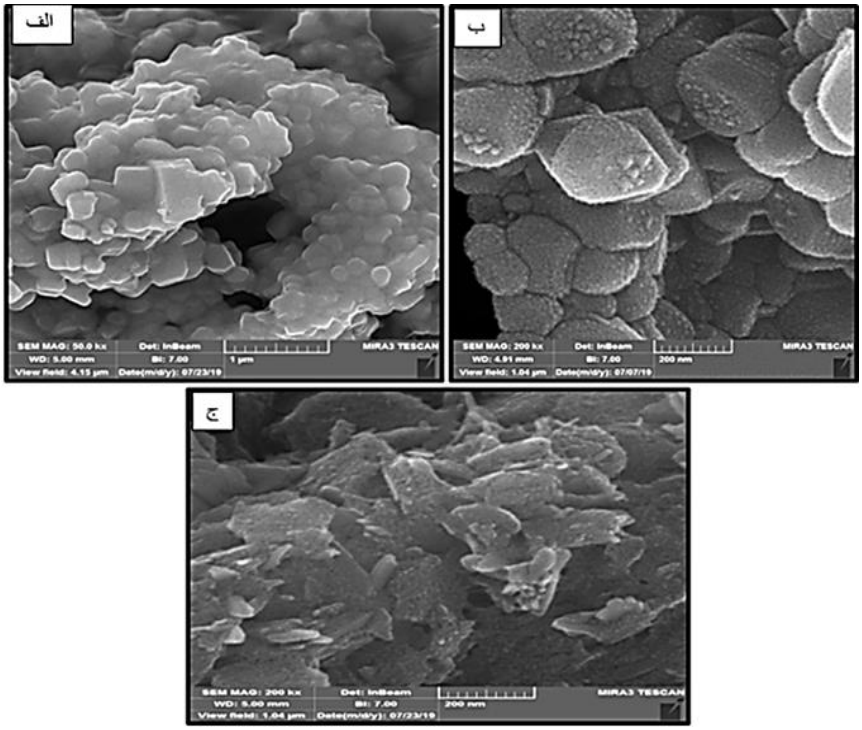

Fig 4: FESEM images for gelatin ratios, a) 1:1, b) 1:2 and c) 1:4

Conclusion

In this paper, the effect of temperature on the structural properties of $Li_{1.25}Mn_{0.125}Ni_{0.125}Co_{0.5}O_2$ powders synthesized by sol-gel method was investigated. Thermal and thermal gravimetry of TGA/DTA, characterization of samples synthesized by X-ray diffraction (XRD), infrared spectroscopy (FTIR), field emission scanning microscope (FESEM), was performed. Examination of XRD results shows that with increasing temperature, the structure of the material becomes more regular and crystalline and the intensity of the peaks increases. FTIR spectral analysis showed that with increasing temperature, the metal bands in the region increase sharply, which is consistent with the results of the X-ray diffraction spectrum.

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