Citric Acid-Assisted Inexpensive Semi-Wet Combustion Synthesis and Characterization of Ultrafine $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$ and LiFePO_4 Polycrystalline Materials

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Abstract

A solution based economically low cost, eco-friendly and energetically advantageous alternative route for the fabrication of ultrafine $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$ and LiFePO_4 , polycrystalline material by semi-wet combustion and sol-gel synthesis is presented. The thermal analysis, crystal structure, morphology and composition of the materials were investigated using different physio-chemical characterizations. TG/DTA, X-ray diffraction, TEM, SEM, EDX, and XPS. The route required auto-combustion of an aqueous metal nitrate solutions in air with the aid of citric acid and is efficient for the fabrication of high quality, $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$ and LiFePO_4 at low temperature, in the combustion residue itself. On a micro-structural studies the grain size was seen in submicron-size that was acquired in a long-established sol-gel combustion and succeeding calcination method. Consequently, the present method reported can yield the ultrafine LiFePO_4 and $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$ at moderate temperature and is look forward to be applicable for other iso-structural polycrystalline materials.

1. Introduction:

LiFePO_4 is of practical interest because of its low toxicity, economically cheap and high theoretical capacity of 170 mAhg^{-1} with a smooth charge-discharge potential at 3.4 V versus Li/Li^+ owing to the $\text{Fe}^{2+}/\text{Fe}^{3+}$ redox couple reaction [1]. LiFePO_4 possess the good thermal stability of which permits the safety issue of the battery mainly for large scale applications in electric vehicles [2]. Since olivine structured based cathode materials, especially the LiFePO_4 which is simply synthesized at large scale and environmentally -friendly in contrast with LiCoO_2 , LiMn_2O_4 and LiNiO_2 [3]. The current research in the area of materials chemistry for the enhancement of the electrochemical performance of cathode materials for Li-ion batteries for improving the synthesis technology of these materials. The fabrication of LiFePO_4 polycrystalline material via conventional solid-state reaction routes is very difficult because it requires multistep in processing such as calcination and sintering at high temperature and long duration with intermediate grinding using different organic solvent, but this cause poor stoichiometry control, large particle size, non-homogeneity [4, 5]. In recent years, the wet chemical routes at low temperature for synthesizing LiFePO_4 polycrystalline materials have boosted the importance due to the production of small particles with homogeneous distribution [6]. There are various wet chemical routes such as hydrothermal [7], polymeric precursor [8], sol–gel [9], spray pyrolysis [10], co-precipitation etc. [11] are used to produce high quality LiFePO_4 polycrystalline materials; although, these techniques have not received much industrial importance due to the use of costly reagent materials and various multi steps of processing taking the long time for the fabrication of these material. Moreover, the merits of the sol-gel method is the feasibility to fabricate ultrafine polycrystalline materials. As per the literature the large numbers of wet chemical methods have been established through the last decade to fabricate the polycrystalline LiFePO_4 cathode material. Sol–gel based auto-combustion synthesis is a peculiar method, with a solitary consolidation of the combustion and sol–gel procedure giving a fluffy and voluminous as final product [12]. Since there is no report on the characteristics of LiFePO_4 and $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$

polycrystalline material, taking in to the consideration of the viability of the semi-wet auto combustion synthesis. In the present research work, a modified sol-gel method called semi-wet combustion synthesis has been developed for synthesizing $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$. We have fabricated ultrafine $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$ by semi-wet combustion synthesis for the first time. In the presented route, the metal ions were taken as nitrate solutions and put together with TiO_2 in solid state form which is low cost. The semi-wet combustion synthesis possess the several merits of over other traditional methods. Firstly, $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$ powder simply fabricated by a very easy semi-wet combustion. Secondly, There are several chemical methods were used for the synthesis of Ti substituted LiFePO_4 where Ti source is utilized as $\text{Ti}(\text{OR})_4$, which is expensive and not be easy handle in lab. Here in, the simplistic fabrication and systematic analysis of $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$ by a semi wet combustion, applying low cost and solid TiO_2 powder as the Ti source and citric acid as the oxidant fuel.

2. Experimental Procedure:

2.1. Material Synthesis

Ultrafine LiFePO_4 (LFP) and $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$ (LFTP) polycrystalline material were synthesized by the sol-gel and semi wet combustion synthesis using citric acid as a chelating agent. The analytical grade chemicals with high purity lithium nitrate, $\text{Li}(\text{NO}_3)$ (Junsei, $\geq 99\%$), iron oxalate di-hydrate, $\text{FeC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ (Junsei, $\geq 99\%$), ammonium di-hydrogen phosphate, $\text{NH}_4\text{H}_2\text{PO}_4$ (Sigma Aldrich, $\geq 99\%$) and solid TiO_2 powder (Sigma Aldrich, $\geq 99\%$) as starting materials. $\text{Li}(\text{NO}_3)$ and $\text{NH}_4\text{H}_2\text{PO}_4$ were mixed in distilled water to get aqueous solutions and $\text{FeC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ was mixed in distilled water with the addition of 50 ml dilute nitric acid. Stoichiometric amounts of the standard aqueous solutions of Li^+ , Fe^{2+} and PO_4^{4-} were mixed in beaker for the synthesis of LiFePO_4 and Li^+ , Fe^{2+} and PO_4^{4-} were mixed in separate beaker along with stoichiometric proportion of solid TiO_2 for the synthesis of $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$. Then, the calculated amount of citric acid equivalents to metal ions was added to the both solutions. The resulting solution for LFP and dispersion for LFTP were put on hot plates at temperature 80–90 °C for the evaporation of the excess water. After the evaporation of the water then it's dried and starts to expand and given the large volume of foamy mass. Within the few minutes, the foamy mass starts to self-combustion without any flame. The whole process of ignition was occurred in air and burnt under self-combustion and obtained the fluffy mass of LFP and LFTP powders. The fine powder LFP and LFTP were obtained by grounding the fluffy mass using pestle and mortar. The precursor powder of LFP and LFTP were calcined at a rate of 5 °C min⁻¹ to 700 °C for 5 h under argon atmosphere. A simplified flow chart of the fabrication procedure of LFP and LFTP is shown in Fig. 1.

2.2. Material characterization

The calcined powders of LFP and LFTP were subjected to various physiochemical characterizations. Simultaneous thermo-gravimetric (TG) and differential thermo-analytic (DTA) measurements in flowing synthetic nitrogen (100 ml/min) carried out using a SDT Q600-0772 system. The X-ray diffraction (XRD)

patterns of the fabricated samples were analyzed by using a Ultima IV X-ray diffractometer (Rigaku, Japan) with Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$). It operated at 40 kV and 30 mA over a 2θ range of between 10° and 70° , in continuous scan mode at 2° min^{-1} . The microstructural features of the prepared materials was investigated using a JSM6500F (Jeol, Japan) field emission scanning electron microscopy (SEM) and the particle size was evaluated using a high resolution transmission electron microscope (TEM, FEI Tecnai F30, Japan). The energy dispersive X-ray analyser (EDX, model Kevex, Sigma KS3) attached with SEM was employed for elemental analysis. The specimen for transmission electron microscopy analysis was developed by dispersing the calcined powders of LFP and LFTP in acetone by ultra-sonication. The acquired suspensions were deposited onto carbon-coated copper grids. X-ray photoelectron spectroscopy (XPS) analyses were conducted using a K-Alpha (Thermo Scientific, USA) in wide scan survey mode, and high-energy resolution with Al K α (1486.6 eV).

3. Result And Discussion:

The thermo gravimetric analysis (TGA) and differential thermal analysis (DTA) were carried out to determine the ideal temperature for the decomposition of precursor gel of LFP and LFTP. Simultaneous TG/DTA characterizations, performed on LiFePO_4 and $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$ of dried precursor powder at a heating rate of $10^\circ \text{C min}^{-1}$ in nitrogen from room temperature to 900°C , are shown in Fig. 2 (a & b). As shown in Fig. 2 (a & b) the LFP and LFTP show nearly the similar type of thermal behavior. The thermograms of the samples reveals the two-step weight losses. The initial one displayed the very slight weight loss within the temperature range room temperature to 185°C . Secondly the major weight loss is found in the temperature range, 180 to 440°C while in the LFTP is 208 to 490°C . There is no further weight loss was noticed beyond 600°C . The initial weight loss is owing to evaporation of residual water and the dehydration of hydrated water present in the sample. The second major weight loss is associated to the combustion of gel and excess citric acid [13]. On the DTA curve, there were two exothermic DTA signals observed on decomposition processes. The initial exothermic process has been rise at temperature of 275°C and the other signal appears between 340 and 470°C . These exothermic signals demonstrate the auto-combustion process where the nitrate ions of reagents act as an oxidizing agent and citric acid as fuel. The lack of signal in the DTA curve after 600°C also validate the findings of TG analysis and established the formation of LFP and LFTP at around 700°C .

X-ray diffraction (XRD) patterns of LFP and LFTP calcined at 700°C for 5 h are shown in Fig. 3. The XRD patterns are agree very well with that of phospho-olivine standard phase and indexed to an orthorhombic crystal structure (space group $Pnma$) [14]. The samples displayed the narrow diffraction peaks demonstrating the good crystallinity degree and there is no Ti compound phase or other impurity phase was observed. In inset in Fig. 3 showed the magnified patterns in the 2θ range of $35.4\text{--}36^\circ$ which displayed the shifting of the XRD peak towards lower 2θ which suggest the enhancement of lattice parameter as listed in Table 1. It is noted from the table that the remarkable increases in parameters a, b and c are observed in LFTP and confirms the Ti^{4+} ions successfully incorporated at Fe^{2+} sites in LiFePO_4 .

Table 1

The lattice parameters and unit cell volume of the samples are calculated using cell software and values are shown in Table 1.

Specimen	Lattice Parameter (Å)			Lattice Volume (Å ³)
	a	b	c	
LiFePO ₄	10.262	5.997	4.678	287.964
LiFe _{0.95} Ti _{0.05} PO ₄	10.310	6.014	4.689	290.827

Scanning electron microscopy characterization was made to determine the morphology and grain size using scanning electron microscope (SEM). Figure 4 show the SEM images of LFP and LFTP polycrystalline material. As seen from the SEM images the morphology of the grains are irregular in shape in both materials. The LFP displayed the separate grains while in the case of LFTP showing the large grain with some agglomeration. The grain size of LFP and LFTP polycrystalline material found to be in the range of 120 nm-0.4 µm and 200 nm- 0.45 µm, respectively.

Figure 5 (a, b) displayed the energy dispersive X-ray (EDX) images of the LFP and LFTP polycrystalline material to ascertain the composition of the grains during the SEM measurement. As seen from the Fig. 5 (a, b) all the metal elements were detected in the grain region except Li because it has very low energy of characteristic radiation, not easy to detect in EDX analysis. It is confirmed that the existence of Fe, P and O in LFP and Fe, P, Ti and O metal ions in LFTP which confirms that Ti ion successfully enter into the crystal lattice of LiFePO₄.

The LFP and LFTP polycrystalline material were further analyzed with High resolution transmission electron microscopic (HR-TEM) analysis as shown in Fig. 6. As displayed in Fig. 6a, the LFP crystallites have irregular shape in the size range 200–300 nm while LFTP has larger crystallites size in the size range 200 nm. It is also evidently observed from the TEM images that each crystallite of LFP and LFTP were coated by a carbon layer to form a carbon–shell structure generated by carbonization of the citric acid. As distinguished from Fig. 6 (b, d) the thickness of the carbon layer is about 2.4 nm and 7.5 nm for LFP and LFTP polycrystalline material, respectively.

Furthermore, the EDX spectra of LFP and LFTP polycrystalline material were observed with HR-TEM (Fig. 7a-d) showing the existence of Fe, P and O in LFP and Fe, P, Ti and O metal ions for LFP and LFTP polycrystalline material, respectively which are well matched with the EDX analysis (Fig. 5).

The distribution of elements is shown in the EDS mapping images (Fig. 8 (a) and (b)) for LFP and LFTP polycrystalline material. As can be seen, the Ti dopant ions is distributed homogeneously in the LiFePO₄

crystal structure without the presence of any trace amounts of the impurities. A very homogeneous uniform distribution of the Fe, P, O and Fe, Ti, P, O elements is also detected in LFP and LFTP polycrystalline material because during the synthesis of LFP and LFTP polycrystalline material, the constituents Li^+ , Fe^{2+} and PO_4^{3-} ions were dissolved in the water and mixed on the atomic scale. This investigation confirms the advantages of the citric acid-assisted semi-wet combustion synthesis.

X-ray photoelectron (XPS) spectra of LFP and LFTP polycrystalline material was carried out to ascertain the oxidation state of the metal ions and elemental composition as shown in Fig. 9 and Fig. 10.

The wide spectrum in Fig. 9 (a) and 10 (a) evidently shows the existence of Li 1 s, Fe 2p, P 2p, O 1 s and Li 1 s, Fe 2p, P 2p, Ti 2p, and O 1 s XPS signals of these elements, which verified the EDX observation and immaculacy of LFP and LFTP polycrystalline material with additional peak of carbon C 1 s located at ≈ 284.8 eV [Figs. 9 (f) and 10 (f)] was used as a reference for binding energy calibration. From the Figs. 9 (b) and 10 (b) LFP and LFTP displayed the Li 1 s XPS spectrum located at 55.78 eV and 55.27 eV corresponding to the lithium in LFP and LFTP polycrystalline material [15]. The positions of the Fe 2p region peaks [Figs. 9 (c) and 10 (c)] in the XPS spectra of the both material were corresponding to the Fe $2p_{3/2}$ main peak situated at 710.5 eV & 710.9 and Fe $2p_{1/2}$ main peak located at 724.3 eV & 724.4 for LFP and LFTP, respectively. These binding energy positions of Fe correspond to the characteristic peaks of the Fe^{2+} , and the observations are consistent with earlier [16]. XPS spectra, shown in [Figs. 9 (d) and 10 (d)], demonstrate that the only one P 2p doublet peaks with the binding energy values of 133.5 eV and 133.3 eV for LFP and LFTP, respectively. The binding energy positions of the P 2p affirm the existence of $(\text{PO}_4)^{3-}$ phosphate group [17]. The O 1 s [Figs. 9 (e) and 10 (e)] of the LFP and LFTP materials were similar at 531.4 eV with respect to the corresponding spectra for oxygen in LiFePO_4 [18]. For the dopant as Ti in LiFePO_4 in Fig. 10e, the XPS peaks of Ti 2P are located at 458.4 and 464.3 eV for Ti $2p_{3/2}$ and Ti $2p_{1/2}$, respectively, which affirm the oxidation state of Ti ions is + 4 in good consistency with oxidation state of Ti [19, 20].

4. Conclusions:

Ultrafine LiFePO_4 and $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$ were successfully synthesized by sol-gel and semi-wet combustion synthesis. The concept of the using semi-wet combustion synthesis for Ti doping in LiFePO_4 is very efficient to synthesize the isomorphs of LiFePO_4 . The fabricated LiFePO_4 and $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$ materials are ultrafine in nature with particle size range $\approx 100\text{--}300$ nm. The synthesized LiFePO_4 and $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$ materials were very high quality having homogeneous distribution of elements confirmed by using the different physiochemical characterizations of EDX, EDS mapping and XPS.

Declarations

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Figures

Figure 1

Systematic representation for the synthesis of ultrafine LiFePO_4 (LFP) and $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$ (LFTP) polycrystalline material.

Figure 2

TG-DTA curves for the precursor powders of (a) $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$ (LFTP), (b) LiFePO_4 (LFP).

Figure 3

X-ray powder diffraction patterns of (a) LiFe_{0.95}Ti_{0.05}PO₄ (LFTP), (b) LiFePO₄ (LFP) calcined at 700 °C for 5 h.

Figure 4

SEM images of (a) LiFePO₄ (LFP) (b) LiFe_{0.95}Ti_{0.05}PO₄ (LFTP) calcined at 700 °C for 5 h.

Figure 5

Energy dispersive X-ray spectra of the (a) LiFePO₄ (LFP) (b) LiFe_{0.95}Ti_{0.05}PO₄ (LFTP) calcined at 700 °C for 5 h during SEM analysis.

Figure 6

(a) TEM image of LiFePO_4 (LFP) (b) corresponding HR-TEM image (c) $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$ (LFTP) (d) HR-TEM image.

Figure 7

Energy dispersive X-ray spectra of the (a) LiFePO_4 (LFP) (b) corresponding EDX spectra of specific area of carbon matrix of LiFePO_4 (LFP) (c) $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$ (LFTP) (d) corresponding EDX spectra of specific area of carbon matrix of $\text{LiFe}_{0.95}\text{Ti}_{0.05}\text{PO}_4$ (LFTP) calcined at 700°C for 5 h during TEM analysis.

Figure 8

EDS mapping image of (a) LiFePO₄ (LFP) of the elements Fe, P, O and C (b) LiFe_{0.95}Ti_{0.05}PO₄ (LFTP) of the elements Fe, Ti, P, O and C.

Figure 9

X-ray photoelectron spectra of the LiFePO₄ (LFP) calcined at 700 °C for 5 h.

Figure 10

X-ray photoelectron spectra of the LiFe_{0.95}Ti_{0.05}PO₄ (LFTP) calcined at 700 °C for 5 h.

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