

# Synthesis and Characterization of $\text{Li}_x\text{FePO}_4/\text{C}$ -PPy Composite Cathode Material with Excess of $\text{Li}^+$ Doping



Rajeev Sehrawat, Rashmi Mittal, Deepak Kr Tyagi, Milan Kr Bera, Anil Kr Sharma

**Abstract:** *In-situ Polypyrrole (PPy) coating was performed on the surface of  $\text{Li}_x\text{FePO}_4/\text{C}$  ( $x=0.95$  to  $1.20$ ) particles using iron (III) tosylate as oxidizer. The composite material  $\text{Li}_x\text{FePO}_4/\text{C}$  ( $x=0.95$  to  $1.20$ ) was synthesized by two step method.  $\text{FePO}_4$ /polyaniline particles were first synthesized by chemical precipitation and were further heat treated with lithium acetate and sucrose under reduced atmosphere. XRD pattern confirms that  $\text{Li}^+$  addition to  $\text{LiFePO}_4$  has increased interplanar spacing and of the unit cell size. Impurity phase appears with  $x=1.15$  and  $1.20$  which further disappears after polymer coating. After polymerization process the XRD pattern shows  $\text{Li}_{0.05}\text{FePO}_4$  and  $\text{LiFePO}_4$  phases and both the phases have same electrochemical behavior. Morphology of the  $\text{Li}_x\text{FePO}_4/\text{C}$  and  $\text{Li}_x\text{FePO}_4/\text{C}$ -PPy was studied by using FE-SEM and it was found that particles are spherical with size range below  $200\text{nm}$ . Transmission Electron Microscope (TEM) also confirms that  $\text{Li}_x\text{FePO}_4/\text{C}$  isolated particles were well encapsulated within the polymer matrix*

**Keywords:**  $\text{LiFePO}_4/\text{C}$  composite, coating, polymer, in-situ polymerization

## I. INTRODUCTION

The olivine structured  $\text{LiFePO}_4$  has developed much interest as a promising cathode material for lithium ion battery due to its high theoretical capacity ( $170\text{mAh/g}$ ), high structural stability excellent safety and low cost since it was first proposed by Goodenough [1, 2, 3].

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\* Correspondence Author

**Dr Rajeev Sehrawat\***, Department of Physics, Maharishi Markandeshwar (Deemed to be University), Mullana, Ambala, Haryana 133207, India. Email: [rajeev.sehrawat@mmumullana.org](mailto:rajeev.sehrawat@mmumullana.org), [rajeev.sehrawat@gmail.com](mailto:rajeev.sehrawat@gmail.com)

**Dr Rashmi Mittal**, Department of Physics, Maharishi Markandeshwar (Deemed to be University), Mullana, Ambala, Haryana 133207, India. Email: [rashmimittal3@gmail.com](mailto:rashmimittal3@gmail.com)

**Dr Deepak Kr Tyagi**, Department of Physics, Maharishi Markandeshwar (Deemed to be University), Mullana, Ambala, Haryana 133207, India. Email: [deepak.tyagi76@gmail.com](mailto:deepak.tyagi76@gmail.com)

**Dr Milan Kr Bera** Department of Physics, Maharishi Markandeshwar (Deemed to be University), Mullana, Ambala, Haryana 133207, India. Email: [m.k.bera.iitkgp@gmail.com](mailto:m.k.bera.iitkgp@gmail.com)

**Dr Anil Kr Sharma** Department of Physics, Maharishi Markandeshwar (Deemed to be University), Mullana, Ambala, Haryana 133207, India. Email: [anil67042@gmail.com](mailto:anil67042@gmail.com)

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However, the low electronic conductivity and slow 1-dimensional insertion/extraction of lithium ion through the structure have restricted the commercialization of this material [4, 5, 6].

The electrical conductivity of this material can be increased by metal doping in the structure and conductive surface coating [7, 8]. Further lithium ion diffusion can be increased by reducing the particle size [9, 10]. The conducting form of polyaniline (PANI) coated  $\text{LiFePO}_4$  has increased the cyclability and rate capability [11].

Polypyrrole can be used as successful coating material to increase the cyclic stability of  $\text{LiFePO}_4/\text{C}$  cathode material [12]. Since the polypyrrole also have higher cyclability (500cycle), good theoretical capacity ( $70\text{mAh/g}$ ) to store  $\text{Li}^+$  and is electrochemically active so, it can increase the cyclability without much affecting the overall energy density of cathode [13].

During the processing such as synthesis and electrochemical activity of cathode material, the partial loss of  $\text{Li}^+$  occurs. The first loss occurs during first charge of battery and was due to the formation of passivating solid electrolyte interphase (SEI) layer [12]. The second loss occurs due to long time charging/discharging process in the cathode material, this leads to the adherence of some Li-ion on surface of anode. And the final loss occurs if there is conductive surface coating of polymer, because during polymerization reaction loss of lithium occurs and polymer coated  $\text{LiFePO}_4/\text{C}$  material was observed as lithium deficient [13]. So, excess of lithium doping would be required in the structure of  $\text{LiFePO}_4/\text{C}$  material to improve the cyclability and capability of composite material. Till now the properties of polypyrrole coated  $\text{LiFePO}_4/\text{C}$  with higher lithium doping has not studied. In the present work initially we have synthesized  $\text{Li}_x\text{FePO}_4/\text{C}$  ( $x = 0.95$  to  $1.20$ ) particles in the nano-range to decrease the diffusion length and finally these particles were in-situ coated by polypyrrole to improved the cyclability and rate capability.

## II. SYNTHESIS

### A. Synthesis of $\text{Li}_x\text{FePO}_4/\text{C}$

The materials  $\text{Li}_x\text{FePO}_4/\text{C}$  ( $x=0.95$  to  $1.20$ ) was synthesized in two step. In the first step  $\text{FePO}_4/\text{PANI}$  was synthesized by chemical precipitation method. To synthesize  $\text{FePO}_4/\text{PANI}$ , aqueous solution of  $0.0278\text{mol}$  of  $(\text{NH}_4)_2\text{H}_2\text{PO}_4$  and  $\text{FeCl}_3$  was added with  $0.5\text{ml}$  of aniline monomer units with continuous stirring for 8hrs at  $65^\circ\text{C}$ .

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On addition of  $\text{FeCl}_3$  to  $(\text{NH}_4)_2\text{H}_2\text{PO}_4$  white precipitation of  $\text{FePO}_4$  starts growing and turns dark green due to formation of Polyaniline after 3hr. The precipitate was filtered out and washed with di-ionized water and acetone.

In the second step different molar ratio of  $\text{CH}_3\text{COOLi}$  (0.95, 1.00, 1.05, 1.10, 1.15 and 1.20) with respect to  $\text{FePO}_4$  were homogeneously mixed using mortar-pestle for 1hr. The resulting mixture was further calcined at  $700^\circ\text{C}$  under reduced atmosphere ( $\text{Ar}(90\%)+\text{H}_2(10\%)$ ) for 15hrs and  $\text{Li}_x\text{FePO}_4/\text{C}$  material was obtained.

## B. Synthesis of $\text{Li}_x\text{FePO}_4/\text{C}$ -PPy

For the synthesis of  $\text{Li}_x\text{FePO}_4/\text{C}$ -PPy ( $x=0.95$  to  $1.20$ ) composite material a known quantity of  $\text{Li}_x\text{FePO}_4$  (1g) was added to 20 ml ethanol containing 20wt.% Iron(III)tosylate. The mixture was stirred in the temperature range of  $1-5^\circ\text{C}$  for 30min followed by addition of  $252\mu\text{l}$  liquid pyrrole monomers. The suspension was kept stirred for another 30min in the temperature range of  $2-5^\circ\text{C}$ . The solution was filtered and washed with ethanol. The resulting composite  $\text{Li}_x\text{FePO}_4/\text{C}$ -PPy was kept in a vacuum oven for 5 hrs at  $55^\circ\text{C}$ .

## III. CHARACTERIZATION

The morphology was observed with field emission scanning electron microscopy (FE-SEM, FEI, Quanta 200F) operating at 20kV. The X-ray diffraction pattern was obtained, on Bruker, D8 ADVANCE diffractometer with  $\text{CuK}_\alpha$  radiation. The phase and structure analysis was performed using XRD data on PANalytical X'Pert HighScore Plus software. Images of samples were observed using transmission electron microscopy (TEM, with FEI-TECHNAI-20) operating at 200kV

## IV. RESULT AND DISCUSSION

### A. XRD Analysis

The fig. 1 shows the X-ray diffraction pattern of  $\text{Li}_x\text{FePO}_4/\text{C}$  ( $x=0.95$  to  $1.20$ ). All the peaks of patterns for the samples having composition  $x = 0.95$  to  $1.10$  represent pure single phase and can be indexed on the basis of olivine type orthorhombic structure with space group  $\text{pnma}$  of  $\text{LiFePO}_4$  (with JCPDS file no. 98-005-9133). For  $x=1.15$  and  $1.20$  compositions the impurity phase  $\text{Li}_3\text{PO}_4$  appears along with  $\text{LiFePO}_4$ .

From the fig. 1 it was observed that there is continuous shift of peak towards lower Bragg angle for  $\text{Li}_x\text{FePO}_4/\text{C}$  ( $x=0.95$  to  $1.15$ ). The peak shift was due to the excess of lithium along 1-dimensional (010) direction [14]. Peak shift relative to  $\text{LiFePO}_4$  can be easily observed from fig 2 and it was found that maximum peak shift occurs for the sample composition  $x = 1.15$ . But for the same material impurity phase  $\text{Li}_3\text{PO}_4$  appears at  $2\theta=22.32^\circ$  and  $23.16^\circ$  and grows for further addition of lithium (for  $x=1.20$ ). The peak shift towards lower Bragg angle has increased the interplaner spacing, this results to generate uniform strain in the crystal lattice [14]. Due to this uniform strain the crystal structure becomes unrelaxed. From the diffraction pattern it was found that crystallites for  $x=1.10$  have maximum strain, without  $\text{Li}_3\text{PO}_4$  phase impurity. The  $\text{Li}_3\text{PO}_4$  considered as impurity phase, because the phase

does not have electrochemical properties [15, 16]. For  $x=1.20$  peaks tends to come at their respective positions and there was a slight increase in the peak intensity of  $\text{Li}_3\text{PO}_4$  impurity phase, showing that addition of more lithium goes to the crystallites of  $\text{Li}_3\text{PO}_4$  instead to provide strain to crystallite of  $\text{LiFePO}_4$ . The absence of carbon peak in the diffraction pattern may be due to amorphous phase of carbon or/and low content.

The lattice parameter of  $\text{LiFePO}_4$  was worked out using the Bragg law for the orthogonal system:

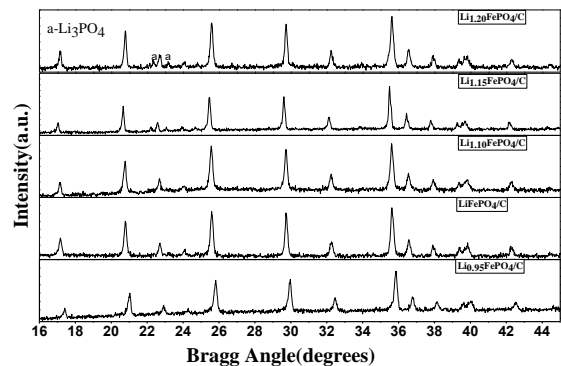
$$d = \left( \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \right)^{-1/2} \quad (1)$$

The calculations were performed using three maximum intensity peaks and obtained values of lattice parameters were listed in table 1.

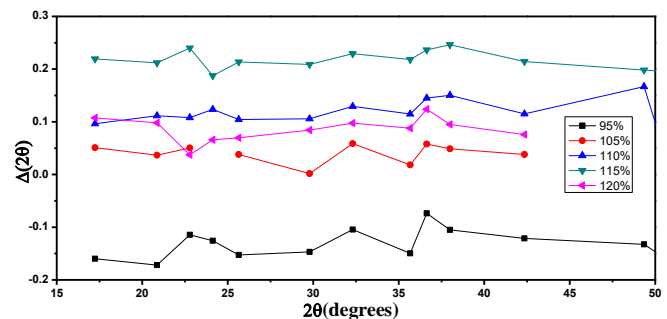
**Table 1. Variation of lattice parameter with  $\text{Li}^+$  doping.**

$\text{Li}_x/\text{FePO}_4$	a (Å)	b (Å)	c (Å)	V (Å <sup>3</sup> )
x = 0.95	10.2994	5.9935	4.6263	285.58
x = 1.00	10.3154	5.9949	4.6707	288.84
x = 1.05	10.3212	6.0041	4.6794	289.99
x = 1.10	10.3219	5.9515	4.7268	290.38
x = 1.15	10.3561	6.0284	4.7236	294.90
x = 1.20	10.3421	5.9937	4.6902	290.97

So, excess lithium content has increased the crystallite volume from 285.58 to 294.90 Å<sup>3</sup> for  $\text{Li}_x\text{FePO}_4/\text{C}$  and hence interplaner spacing, this originates the shift of peak intensity towards lower Bragg angle.



**Fig.1 XRD pattern of  $\text{Li}_x\text{FePO}_4/\text{C}$  ( $x=0.95$  to  $1.20$ )**

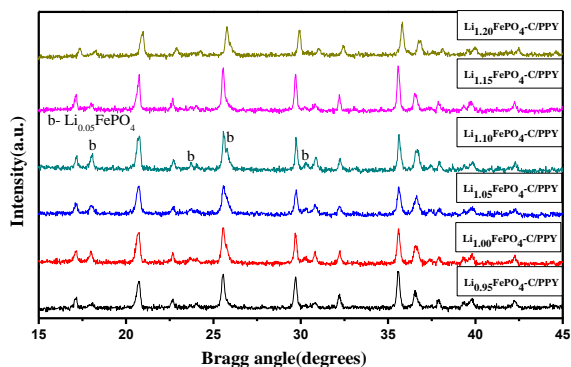


**Fig. 2 Relative shift in  $2\theta$  values with respect to  $\text{Li}_{1.00}\text{FePO}_4$**

**B. Explanation of XRD after PPy coating**

Fig.3 shows the XRD pattern of  $\text{Li}_x\text{FePO}_4/\text{C}$  ( $x=0.95$  to  $1.20$ ) coated with Polypyrrole composite material ( $\text{Li}_x\text{FePO}_4\text{-C/PPy}$  ( $x=0.95$  to  $1.20$ )). All the major peaks were matched with olivine type pnma structure (with JCPDS file no. 01-083-2092).

From the diffraction pattern it was found that all the peaks have nearly same position and there was no significant shift in the peak positions, showing that  $\text{LiFePO}_4$  crystallites were strain free. Diffraction pattern of composite material  $\text{Li}_{0.95}\text{FePO}_4\text{-C/PPy}$  shows a weak intensity peak corresponding of  $\text{Li}_{0.05}\text{FePO}_4$  (with JCPDS No. 98-10-8284) at  $17.95^\circ$ .  $\text{Li}_{0.05}\text{FePO}_4$  and  $\text{LiFePO}_4$  both have same electrochemical behavior and lithium storage capacity [17]. Other peaks of  $\text{Li}_{0.05}\text{FePO}_4$  phase become significant gradually for higher lithium content (for  $x=0.95$  to  $1.10$ ) and  $\text{Li}_{1.10}\text{FePO}_4\text{-C/PPy}$  composite material shows highest no/intensity of peaks for  $\text{Li}_{0.05}\text{FePO}_4$  phase. Peak intensity for this phase further decrease for  $x=1.15$  and  $1.20$ . The increase in the peak intensity of  $\text{Li}_{0.05}\text{FePO}_4$  phase for  $x=1.10$  shows that this material donate maximum  $\text{Li}^+$  to PPy.



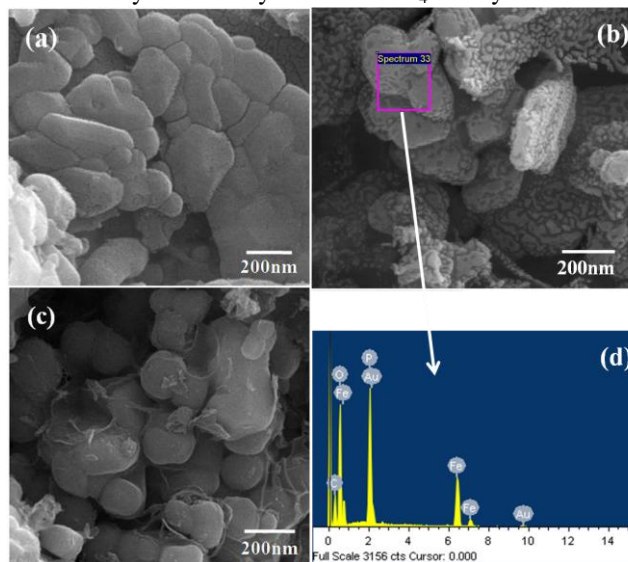
**Fig.3 XRD pattern of  $\text{Li}_x\text{FePO}_4/\text{C-PPy}$  ( $x=0.95$  to  $1.20$ )**

The highest strain for  $x=1.10$  pure phase structure results maximum  $\text{Li}_{0.05}\text{FePO}_4$  phase. It may happen because, due to strain, the crystallites donate more  $\text{Li}^+$  to polymers to come in relax stage. After coating no peaks were observed in the XRD patterns of  $\text{Li}_x\text{FePO}_4\text{-C/PPy}$  (for  $x=0.95$  to  $1.20$ ) for  $\text{Li}_3\text{PO}_4$  impurity phase. It is concluded that this phase dilute during polymerization reactions and lithium of the phase may goes to the polymer chains as polymer chains also have the ability to store  $\text{Li}^+$  [18]. For  $\text{Li}_x\text{FePO}_4\text{-C/PPy}$  (for  $x=1.15$  and  $1.20$ ) composites both the phases  $\text{Li}_3\text{PO}_4$  and  $\text{LiFePO}_4$  contributes to the saturation of polymer chains for  $\text{Li}^+$ . For  $\text{Li}_x\text{FePO}_4\text{-C/PPy}$  (for  $x=1.15$  to  $1.20$ ) composite materials the observed intensity peak for  $\text{Li}_{0.05}\text{FePO}_4$  is reduced because, conversion of  $\text{LiFePO}_4$  to  $\text{Li}_{0.05}\text{FePO}_4$  is suppressed due to presence of  $\text{Li}_3\text{PO}_4$  phase.

**C. Powder morphology**

The morphology of as synthesized  $\text{Li}_x\text{FePO}_4/\text{C}$  and  $\text{Li}_x\text{FePO}_4/\text{C-PPy}$  powders is as shown in fig. 4(a, c) and (b) respectively. The SEM image of  $\text{Li}_x\text{FePO}_4/\text{C}$  shows spherical particles are agglomerated through carbon shell having size nearly 200nm this was further confirmed by Dynamic light scattering (DLS) studies. The carbon coating on  $\text{Li}_x\text{FePO}_4/\text{C}$  was confirmed by Energy Dispersive Spectroscopy (EDS).

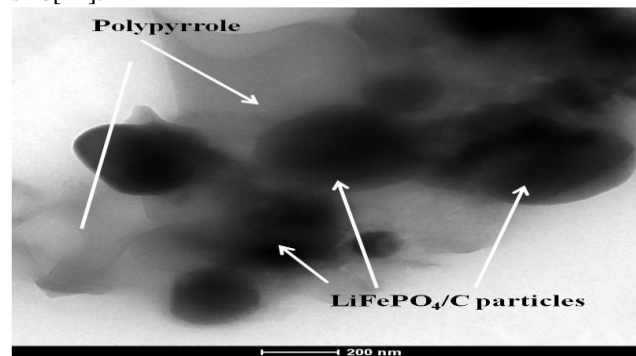
The EDS also confirms the ratio of Fe:P:O is 1:1:4. Fig.4(b) shows the SEM image of  $\text{Li}_x\text{FePO}_4/\text{C-PPy}$ . The two phases  $\text{Li}_x\text{FePO}_4/\text{C}$  and PPy are clearly distinguishable. The EDS shows that the ratio of Fe:P:O = 1:1:4 for  $\text{LiFePO}_4$  (for  $x=1$ ) is still maintained which clearly shows that stictometry of elements is same in the PPy matrix showing  $\text{Li}_x\text{FePO}_4/\text{C}$  is attached to PPy by physical bonding. The particle size of  $\text{LiFePO}_4/\text{C}$  in the pyrrole composite is still same. From the constant particle size and EDS it is confirmed that there was no dilution of  $\text{Li}_x\text{FePO}_4$  particle during polymerization of pyrrole. This confirms that  $\text{LiFePO}_4$  particles were chemically stable during polymerization; the fact was already confirmed by XRD analysis of  $\text{LiFePO}_4/\text{C-PPy}$ .



**Fig.4 FE-SEM micrograph of (a)  $\text{Li}_{1.00}\text{FePO}_4/\text{C}$  and (b)  $\text{Li}_{1.15}\text{FePO}_4/\text{C-PPy}$  and (c)  $\text{Li}_{1.15}\text{FePO}_4/\text{C}$  (d) EDS spectra of  $\text{Li}_{1.15}\text{FePO}_4/\text{C-PPy}$**

**D. TEM Analysis**

The typical TEM image of  $\text{Li}_x\text{FePO}_4/\text{C-PPy}$  is shown in fig 5. For the  $\text{LiFePO}_4/\text{C-PPy}$  composite, TEM image confirms that particles are embedded in the continuous polypyrrole matrix. The TEM image also confirms that particles are mono-dispersed and does not stick together due to their magnetic behavior as it can be seen in case of  $\text{LiFePO}_4/\text{C}$  from fig 5. The polypyrrole matrix behaves as fast conducting bridge and for such a system of mono disperse particles diffusion length of  $\text{Li}^+$  completely depends upon particles size[22].



**Fig. 5 TEM image of  $\text{LiFePO}_4/\text{C-PPy}$**

## V. CONCLUSION

Polypyrrole coated  $\text{Li}_x\text{FePO}_4/\text{C}$  ( $x=0.95$  to  $1.20$ ) composite was successfully synthesized. XRD pattern of  $\text{Li}_x\text{FePO}_4/\text{C}$  ( $x=0.95$  to  $1.20$ ) confirms that  $\text{Li}^+$  addition to  $\text{LiFePO}_4$  has increased the cell volume and d-spacing due to which a uniform stress induced in the crystal. Impurity phase  $\text{Li}_3\text{PO}_4$  has appeared for  $x=1.15$  and  $1.20$ . During polymerization the impurity phase diluted and was not appeared in XRD pattern. FE-SEM confirms the spherical morphology of  $\text{Li}_x\text{FePO}_4/\text{C}$  and  $\text{Li}_x\text{FePO}_4/\text{C}$ -PPy particles having size  $200\text{nm}$ . The carbon shell and polymer coating on the particles were clearly visible in FE-SEM micrograph. TEM has further confirmed the  $\text{Li}_x\text{FePO}_4/\text{C}$  particles are encapsulated in the polypyrrole matrix.

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## AUTHORS PROFILE



**Dr. Rajeev Sehrawat** is currently an Assistant Prof. at MMDU. My field of research interest is on “Cathode materials of Lithium ion batteries”.



**Dr. Rashmi Mittal** is currently an Assistant Prof. at MMDU. His field of research interest is on “Modelling and Simulation of AI alloys”



**Dr. Deepak Kumar Tyagi** is currently an Assistant Prof. at MMDU. His field of research interest is on “Modeling and Inversion of Electromagnetic Data”.



**Dr. Milam Kumar Bera** is currently an Assistant Prof. at MMDU. His field of research interest is on “Experimental and Theoretical Modelling of Electronics Devices”.



**Dr. Anil Kr. Sharma** is currently an Assistant Prof. at MMDU. His field of research interest is on Computational Study of Biocative Molecules”.