



METHOD FOR SURFACTANT-ASSISTED HYDROTHERMAL SYNTHESIS OF NANO-SIZED LiFePO_4 /CARBON COMPOSITE
IITM Technology Available for Licensing

PROBLEM STATEMENT

- Conventional methods for the preparation of the LiFePO_4 /Carbon composites **do not allow homogeneous carbon coating** which is essential for a long-life cycle & high-rate capability of the cathode material for Li-ion battery application.
- Further, said prior art techniques **do not offer a reduced particle size to nanoscale size (<50nm)** which can tremendously improve the rate capability of LiFePO_4 .
- As a result, said procedures often **do not provide optimal performance of LiFePO_4** .
- Hence, there is a need to address the issues.

INTELLECTUAL PROPERTY

IITM IDF Ref. 1767; IN Patent No: 399209

TECHNOLOGY CATEGORY/ MARKET

Technology: Synthesis of nano-sized LiFePO_4 /C composite;

Industry & Application: Energy, Raw Material Electric Vehicle, Automotive, Power Industry;

Market: The global LiFePO_4 Batteries market is projected to grow at a **CAGR of 37.3%** during **2024-2029**.

TRL (TECHNOLOGY READINESS LEVEL)

TRL-3/4, Proof of Concept ready, tested in lab.

TECHNOLOGY

- Present invention describes an improved method for synthesis of nano-sized **LiFePO_4 /Carbon Composite** using a **tri-blocked copolymer-based surfactant assisted-hydrothermal process**.
- **STEP 1.** The LiFePO_4 /C composite is **synthesized** by surfactant-assisted hydrothermal method from the stoichiometric mixture of $\text{LiOH}\cdot\text{H}_2\text{O}$, $\text{FeSO}_4\cdot 7\text{H}_2\text{O}$ & H_3PO_4 (**3:1:1 molar ratio**) using a tri-block copolymer Pluronic® 31R1 (average Mn ~3,300).

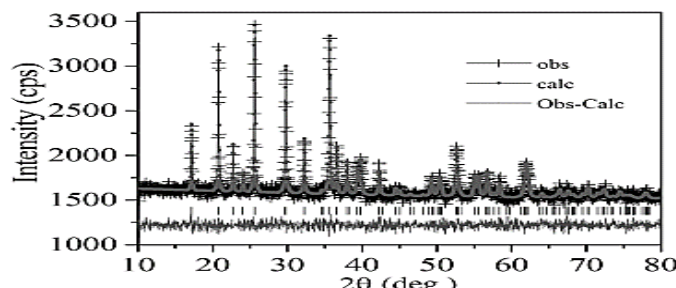


Fig. 1: Illustrates a graphical representation of the Rietveld refinement of LiFePO_4 /carbon composite, calcined at 923 K for 6 h.

- The method comprising a few steps including synthesizing process:

2nd Step

- **Dissolving 3 mL of the tri-block Pluronic® 31R1 copolymer in 15 mL of distilled water at 35°C & 30 mmol of $\text{LiOH}\cdot\text{H}_2\text{O}$ was added to the surfactant solution.**

3rd Step

- **Adding 10 mmol H_3PO_4 under stirring to obtain a milky white suspension and a 3 mL ethylene glycol was subsequently added into the resulting white suspension to disperse the inorganic salts;**

4th Step

- **Dissolving the requisite amount of ferrous sulphate ($\text{FeSO}_4\cdot 7\text{H}_2\text{O}$; 10 mmol) in 15 mL deionized water & adding with ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$; 2.5 mmol)**
- **Finally, transfer the resulting suspension into a 150 mL Teflon-lined stainless-steel autoclave & heat at 180°C for 16h followed by washing the precipitate with water & ethanol & drying at 80°C after the hydrothermal reaction.**

RESEARCH LAB

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KEY FEATURES / VALUE PROPOSITION

❖ Technical Perspective

- Present invention provides mesoporous LiFePO_4/C composite in **spindle shape & particle size ($< 50 \text{ nm}$)** controlled by Pluronic®31R1. (Refer Fig.2)
- It is noted that **Ascorbic acid** acts as a reducing agent in autoclave-based hydrothermal synthesis.
- Further, **20 wt% sucrose** uniformly decorated over LiFePO_4 and H_2/Ar gas act as a second reducing agent.

❖ Industry Perspective

- The main objective of the present invention is to **provide a Nontoxic Pluronic®31R1** assisted route for synthesis of the superior cathode in lithium-ion-batteries (LIB) with **excellent reversibility & long-term cyclability**.

Images

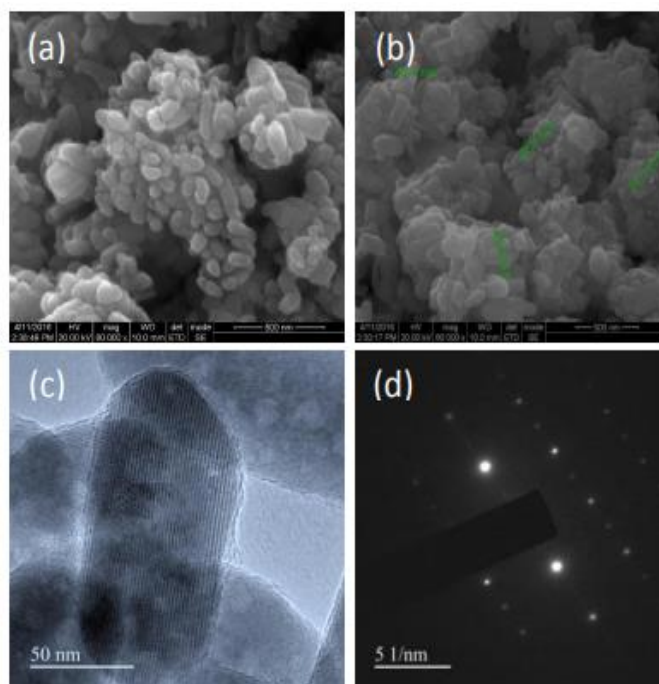


Fig. 2: Illustrates the graphical representation of SEM (2a-2b), TEM (2c) & corresponding SAED pattern (2d) of mesoporous $\text{LiFePO}_4/\text{Carbon}$ composite.

Results

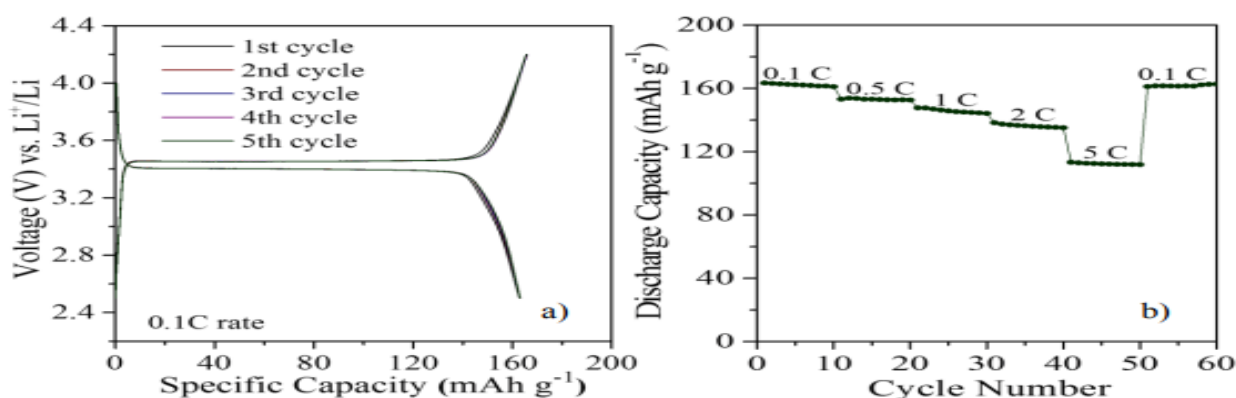


Fig. 3: Illustrates graphical representation of galvanostatic charge-discharge profiles (3a) recorded at 0.1C for LiFePO_4/C sample and rate performance (3b) of LiFePO_4/C composite electrode at different current rates.

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