SYNTHESIS AND CHARACTERIZATION OF LITHIUM IRON PHOSPHATE NANOWIRES FROM SAGO PITH WASTE CELLULOSE NANOFIBRIL AEROGEL

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To my beloved father and mother,

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ABSTRACT

This project aimed to produce highly crystalline lithium iron phosphate coated aerogel ($LiFePO_4$ nanowires) via coating and sintering $LiFePO_4$ on the surface of an aerogel template prepared from cellulose nanofibril (SPCNF) extracted from sago pith waste (SPW). The effects of SPCNF aerogel quantity (X_q), sintering duration (X_t) and sintering temperature (X_T) on the quality of $LiFePO_4$ were studied. Specifically, SPCNF were first extracted from SPW through combined chemical and mechanical treatments, followed by a freeze-drying step to produce an aerogel. The resulting white and spongy SPCNF aerogel was characterized to elucidate its morphology, crystallinity, and thermal resistance using field-emission scanning electron microscopy, powder x-ray diffraction (PXRD) and thermogravimetric analysis, respectively. Results showed that the morphology of SPCNF aerogel resembled that of a web-liked structure with the diameters of each SPCNF measured to be within 15-30 nm. The degree of crystallinity of the aerogel was approximately 88.38% and its thermal degradation temperature was in the range of 260-350 °C. The aerogel was then coated with $LiFePO_4$ through direct coating on aerogel by applying a uniform solution containing Li^+, Fe^{2+} and PO_4^{3-} in 1:1:1 molar ratio and followed by calcination and sintering. The final product, $LiFePO_4$ nanowires had wire-liked structure with the diameters between 15-30 nm, and the PXRD and transmission electron microscopy verified the nanowires were covered with $LiFePO_4$. Results from design of experiment of full-factorial design showed that the three parameters are significant and the coating quality, Y can be correlated with the following equation: $Y = 5.662 + 7.23875X_q + 7.23875X_t + 5.60125X_T + 7.23875X_qX_t + 5.60125X_qX_T + 5.60125\ X_tX_T + 5.60125\ X_qX_tX_T$.

ABSTRAK

Projek ini bertujuan untuk menghasilkan kristal litium besi fosfat bersalut aerogel (nanodawai $LiFePO_4$) melalui penyalutan ke permukaan templat aerogel nanofibril selulosa (SPCNF) yang diekstrak daripada hampas sagu (SPW). Kesan kuantiti aerogel SPCNF (X_q), tempoh pensinteran (X_t) dan suhu pensinteran (X_T) ke atas kualiti $LiFePO_4$ telah dikaji. Secara khusus, SPCNF diekstrak daripada SPW melalui gabungan rawatan kimia dan mekanikal, diikuti oleh pengeringan secara pembekuan untuk menghasilkan aerogel. Aerogel SPCNF yang bertekstur lembut dan berwarna putih dicirikan dari segi morfologi, darjah penghabluran dan rintangan haba, masing-masing melalui kaedah bidang pelepasan mengimbas mikroskopi elektron, pembelauan sinar-x serbuk (PXRD) dan analisis termogravimetri. Hasil ujian menunjukkan bahawa morfologi aerogel SPCNF adalah dalam struktur bentuk web dengan diameter SPCNF dalam lingkungan 15-30 nm. Darjah penghabluran aerogel SPCNF adalah sebanyak 88.38% dan degradasi termal berlaku pada suhu antara 260-350 °C. Aerogel tersebut kemudian disaluti dengan $LiFePO_4$ melalui penyalutan langsung ke atas aerogel dengan menggunakan satu larutan homogen yang mengandungi ion Li^+, Fe^{2+} dan PO_4^{3-} dalam nisbah molar 1:1:1 dan diikuti oleh proses pengkalsinan dan pensinteran. Produk terakhir, nanodawai $LiFePO_4$ mempunyai struktur bentuk dawai dengan diameter dalam lingkungan 15-30 nm, dan analisis PXRD dan transmisi elektron mikroskopi mengesahkan nanodawai telah diliputi dengan $LiFePO_4$. Keputusan daripada rekabentuk eksperimen faktorial penuh menunjukkan bahawa ketiga-tiga parameter tersebut adalah penting dan boleh dihubungkaitkan dengan kualiti salutan, Y melalui persamaan berikut: $Y = 5.662 + 7.23875X_q + 7.23875X_t + 5.60125X_T + 7.23875X_qX_t + 5.60125X_qX_T + 5.60125\ X_tX_T + 5.60125\ X_qX_tX_T$.

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LIST OF ABBREVIATIONS AND SYMBOLS

1D	-	1-Dimensional
2D	-	2-Dimensional
3D	-	3-Dimensional
AGU	-	Anhydroglucose Unit
BET	-	Brunauer–Emmett–Teller
BOD	-	Biological Oxygen Demand
C	-	Carbon atom
-C=O	-	Carbonyl group
C_2H_5OH	-	Ethanol
C_7H_8	-	Toluene
-C-C	-	Carbon-carbon
CFs	-	Carbon Fibers
-CH	-	Methyl group
CH_3COOLi	-	Lithium Acetate
CMC	-	Cellulose Microcrystal
CMF	-	Cellulose Microfibril
CNC	-	Cellulose Nanocrystal
CNF	-	Cellulose Nanofibril
CNTs	-	Carbon Nanotubes
Co	-	Cobalt
-C–O	-	Carbon-Oxygen Bond
CO_2	-	Carbon Dioxide
COD	-	Chemical Oxygen Demand
CVD	-	Chemical Vapour Deposition
DoE	-	Design of experiment
DP	-	Degree of Polymerization
DSC	-	Differential Scanning Calorimetry

EDX	-	Energy Dispersive X-ray Analysis
EG	-	Ethylene Glycol
EVs	-	Electric Vehicles
Fe	-	Iron
$Fe(CH_3COO_2)_2$	-	Iron (II) Acetate
Fe^{2+}	-	Iron (II) ion
Fe_2P	-	Iron Phosphide
$FeC_2O_4.2H_2O$	-	Iron (II) Oxalate Dihydrate
$FeC_2O_4H_2O$	-	Iron (II) oxalate monohydrate
$FeCl_2.4H_2O$	-	Iron (II) chloride tetrahydrate
FeO_6	-	Iron Oxide
$FePO_4$	-	Iron (III) Phosphate
$FePO_4(H_2O)_2$	-	Iron (III) Phosphate Dihydrate
FE-SEM	-	Field Emission Scanning Electron Microscopy
FTIR	-	Fourier Transform Infrared Spectroscopy
H_3PO_4	-	Phosphoric acid
HCl	-	Hydrochloric Acid
HEVs	-	Hybrid Electric Vehicles
HIUS	-	High Intensity Ultrasonication
HSO	-	Sulfuric Acid
Li^+	-	Lithium ion
Li_2CO_3	-	Lithium Carbonate
$Li_4P_2O_7$	-	Lithium Pyrophoshate
LIBs	-	Lithium Ion Batteries
$LiCoO_2$	-	Lithium Cobalt Oxide
LiF	-	Lithium Fluoride
$LiFePO_4$	-	Lithium Iron Phosphate
$LiFePO_4/C$	-	Lithium Iron Phosphate Coated Carbon
$LiMn_2O_4$	-	Mn-based spinels lithiated transition metal oxides
$LiMO_2$	-	lithiated transition metal oxides
LiO_6	-	Lithium Oxide
$LiOH.H_2O$	-	Lithium hydroxide monohydrate
$Li_xM_y(XO_4)_z$	-	polyanion-type lithiated transition metal oxides

Mn	-	Manganese
N_2	-	Nitrogen Gas
$NaClO_2$	-	Sodium Chlorite
NaOH	-	Sodium hydroxide
$NH_4H_2PO_4$	-	Ammonium dihydrogen phosphate
$(NH_4)_2HPO_4$	-	Diammonium phosphate
NH_4OH	-	Ammonium hydroxide
Ni	-	Nickel
NMMO	-	N-benzyl-morpholine-N-oxide
O	-	Oxygen atom
-OH	-	Hydroxyl group
P	-	Phosphorus
PO_4^{3-}	-	Phosphate ion
S	-	Sulfur
Si	-	Silicon
SnO_2	-	Tin Oxide
SPCNF	-	Sago Pith Cellulose Nanofibril
SPW	-	Sago Pith Waste
STA	-	Simultaneous Thermal Analysis
TEM	-	Transmission Electron Microscopy
TEMPO	-	2,2,6,6- Tetramethyl-piperidin-1-yl)oxyl
TGA	-	Thermogravimetric Analysis
TMP	-	TEMPO-Mediated Oxidation
wt %	-	Weight percentage
XRD	-	X-ray powder diffraction

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Malaysia is the world's third largest exporter of sago starch with a total sago palm plantation area of 68,000 ha, after Indonesia (1,843,278 ha) and Papua New Guinea (1,020,000 ha) (Ahmad, 2014). Each year, a large amount of agro-residue termed sago pith waste (SPW) or *hampas* by the locals, is generated during the processing of the rasped pith of the sago palm trees. Improper handling of this agro-waste can eventually cause environmental problems (Toh *et al*., 2011). To date, different works have been done in order to turn the SPW into value-added products. For example, as an alternative substrate for fermentable sugars production (Linggang *et al.*, 2012), bioethanaol production (Saravana *et al.*, 2014) and etc.

Aerogel is a highly porous material with high specific surface area, low density and low thermal conductivity. It can be used as thermal insulators, super adsorbents, batteries and more. Kitsler (1932) published the first report on aerogel prepared from inorganic gels. The main drawback of these inorganic aerogel is their poor mechanical properties (brittle and fragile) and thus, organic aerogels were introduced (Kim *et al*., 2011). Cellulose nanofibril (CNF) aerogel is a strong organic aerogel. The fibrillar morphology of and strong mutual hydrogen bonds in CNF facilitate the mechanical ductility and flexibility of the aerogel (Chen *et al*., 2011 and Pääkkö *et al*., 2008). Due to their mechanical robustness, CNF aerogels can be used in various applications such

as templates for synthesizing inorganic hollow nanotubes (Korhonen *et al*., 2011) and cobalt-ferrite nanoparticles (Olsson *et al*., 2010).

Lithium iron phosphate ($LiFePO_4$) has been identified as the most promising cathode material for making lithium ion batteries because of its low cost, high theoretical capacity (170 mAh g^{-1}) and long cycle life (Zhang, 2010). $LiFePO_4$ in the form of nano-sized particles has a higher surface electrochemical reactivity, shorter diffusion length for electrons and Li-ions and thus, better electrochemical performance (Yamada *et al*., 2001, Jamnik *et al*., 2003, Dominko *et al*., 2007 and Saravanan *et al*., 2009). Therefore, different synthetic methods were introduced for the preparation of nano-sized $LiFePO_4$ (Gong *et al*., 2011). For instance, hydrothermal method was used by Yang *et al*., (2001) to produce high purity, single crystalline $LiFePO_4$ nano-particles. This method, however, requires long time i.e. three days to complete the synthesis process. After that, solvothermal processes were utilized by Muraliganth *et al*. (2008), Murugan *et al*. (2008) and Saravanan *et al*. (2009) to produce nanostructured $LiFePO_4$. Some drawbacks of this method include, low precursor solubility, high cost and high reaction temperature.

Numerous studies have also been done to synthesize nano-sized $LiFePO_4$ with different morphologies such as nanoparticles (Delacourt *et al.*, 2006), nanoplates (Saravanan *et al.*, 2009) and nanowires (Lin *et al*., 2008 and Zhang, *et al*., 2013). Among the wide range of morphologies, nanowires are identified as the most promising morphology because they offer a better electrical percolation behaviour which means a higher conductivity comparing to that of other morphologies (Bruce *et al.*, 2008).

In this project, $LiFePO_4$ nanowires were synthesized by using a new approach in which, the precursors of $LiFePO_4$ are first coated onto sago pith cellulose nanofibrils (SPCNF) aerogel. The coated SPCNF aerogel is then sintered at high temperature in a conventional furnace under N_2 atmosphere to produce highly crystalline $LiFePO_4$ nanowires.

1.2 Problem Statement

As the world's third largest exporter of sago starch, Malaysia produces up to 47,000 metric tons of sago starch annually (Uthumporn *et al*., 2014). It is estimated that for every kilogram of sago starch extracted, a kilogram of SPW is produced (Lai *et al*., 2013). These wastes are normally dumped into the rivers together with the waste water. In other words, each year, 52,000 tons of SPW ends up polluting the rivers by increasing the biochemical oxygen demand (BOD) of the water. Microbiological degradation of the waste consumes oxygen dissolved in the water, leaving the water with insufficient oxygen to support higher forms of life.

One of the ways to minimize the impact of SPW is to utilize it and convert it into some value-added products such as biofuels, biomaterial, template etc. Kumaran and co-workers (1997) utilized SPW as a substrate for the production of enzyme via solid substrate fermentation. Besides that, SPW was also used as an additional carbon source in anaerobic digesters for the production of biogas (Abd-Aziz, 2002), an alternative substrate for fermentable sugars production (Linggang et al., 2012) and bioethanaol production (Saravana et al., 2014). To the best of the author's knowledge, to date, no report on the extraction of SPCNF from SPW is available.

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In average, SPW contains about 23% by weight of cellulose (Linggang *et al*., 2012). It is believed that SPCNF could be extracted from SPW via chemical, mechanical or a combination of both methods, like what other researchers obtained from different types of lignocellulosic agricultural wastes (Jiang *et al*., 2013 and Chen *et al*., 2014). The extracted SPCNF adopt a shape similar to that of nanowires and their surface contain plenty of hydroxyl functional groups which could interact with different types of chemical compounds including metal ions via electrostatic attraction or complexation. Therefore, SPCNF has the potential to serve as a template for the synthesis of inorganic nanowires, for example $LiFePO_4$.

$LiFePO_4$ is an important nano-sized material for the manufacturing of rechargeable battery. It is used as cathode because of its high specific capacity (170 $mAhg^{-1}$), a relatively high redox potential (3.5V), long cycle life, and high stability (Wang *et al*., 2008, Wu *et al*, 2011). Researchers reported that, $LiFePO_4$ nanowires are more efficient as compared to that of in the form of spherical nanoparticles in conducting electricity, due to its longer mean free path (Zhu *et al*., 2006). Unfortunately, to date, $LiFePO_4$ only available commercially in spherical nanoparticle form because its synthesis method is easier to be industrialized for mass production (Park *et al*., 2009, Ban *et al*., 2010 and Carbana *et al*., 2010). Therefore, the need for the works on developing a simple and scalable method for synthesizing $LiFePO_4$ nanowires has certainly been recognized in order to solve this problem.

Hence, it is hypothesized that $LiFePO_4$ nanowires can be synthesized via a two-stage, facile synthetic method which involves the coating and calcination of $LiFePO_4$ on a SPCNF template. SPCNF surface contains plenty of hydroxyl functional groups which are able to form complex with iron (II). Lithium ion, Li^+ and phosphate, PO_4^{3-} precursors can then be added to form $LiFePO_4$ upon calcinations.

Although the direct use of SPCNF is hypothesized to be able to assist in the formation of $LiFePO_4$ nanowires, it is highly likely that $LiFePO_4$ particles could formed as a by-product, along with the formation of the nanowires. If this happened, these particles would be challenging to be separated from the nanowires and will exist as a contaminant (Lele *et al*., 2014). To circumvent this particle contamination issue, it is hypothesised that converting the free-standing SPCNF into a SPCNF aerogel with large surface area would help. In aerogel form, the precursor solutions of $LiFePO_4$ could be better absorbed via capillary force on the surface. After drying, a thin layer of amorphous $LiFePO_4$ could be annealed into crystalline $LiFePO_4$ nanowires formed on the SPCNF surface and following the wire-like contour of individual SPCNF within the aerogel (Melone *et al*., 2013).

Therefore, this aim of this project is focused on producing $LiFePO_4$ nanowires from the precursor coated SPCNF aerogel. The precursors are expected to be coated onto the aerogel uniformly after solvent removal and be transformed into crystals at elevated temperature. Individual SPCNF in the aerogel are expected to serve as a template/platform for $LiFePO_4$ nanowires to grow upon.

1.3 Objectives

1. To synthesize and characterize SPCNF aerogel interconnected structure with web-like appearance.

2. To synthesize and characterize $LiFePO_4$ nanowires and study the effects of SPCNF aerogel's quantity, sintering temperature and sintering duration on the quality of the $LiFePO_4$ nanowires.

1.4 Significance of the Study

The most significant contribution of this study is that $LiFePO_4$ nanowires, a very important cathode material can be obtained via a relatively safe and simple method namely, coating of $LiFePO_4$ onto the SPCNF aerogel followed by calcinations. The proposed use of aerogel template is expected to reduce the formation of particulate impurities. Additionally, this project might lead to a new application for SPCNF, thus converting this low value, environmental-polluting agricultural waste into value-added consumer products.

1.5 Scope of the Study

In this project, SPCNF was extracted from SPW using a combination of chemical and mechanical methods. The extracted SPCNF was transformed into aerogel via freeze drying. Meanwhile, $LiFePO_4$ precursors were synthesized from Iron (II) chloride, $FeCl_2$, lithium hydroxide monohydrate, $LiOH.H_2O$ and phosphoric acid, H_3PO_4 using a mixture of ethylene glycol and water.

Coating of the $LiFePO_4$ precursors onto the SPCNF aerogel to produce $LiFePO_4$ nanowires were studied by varying three parameters namely, quantity of the SPCNF aerogel (0.02-0.06 g), sintering temperature (300-500 °C) and sintering duration (60-180 min). Effects of the three parameters toward the quality of the $LiFePO_4$ nanowires were evaluated using design of experiment (DOE) with the aid of a software namely, JMP 13.

Characterizations of the SPCNF aerogel and $LiFePO_4$ nanowires were done through the following techniques: lattice structure and elemental composition were analysed by Transmission Electron Microscopy (TEM) and Energy Dispersive Spectroscopy (EDS), surface morphology was characterized by Field-Emission Scanning Electron Microscopy, (FE-SEM). Chemical analysis was accomplished via Fourier Transform Infrared Spectroscopy (FTIR) and Powder X-ray diffraction (PXRD). Lastly, thermal stability was evaluated by using Thermo-gravimetric Analysis (TGA) and Simultaneous Thermal Analysis (STA).

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