

CHAPTER 1:

**INTRODUCTION TO
DISSERTATION**

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Rechargeable lithium ion batteries were first commercialized by Sony in 1990. Batteries based on lithium provide high energy density. In fact lithium is the most electronegative (-3.04 V) versus standard hydrogen electrode (SHE). It is also the lightest metal with equivalent molecular weight of 6.94 g mol^{-1} and specific gravity 0.53 g cm^{-3} [Tarascon and Armand, 2001]. The performance of secondary batteries is measured in terms of the energy density, power density (the ability of high current discharge), working voltage, flatness of the discharge curve, rapid recharging, cycleability, safety and discharge capability at low temperatures [Takamura, 2002]. Performances of these batteries are dependent on electrode and electrolyte materials. In this dissertation emphasis is on an anode material

$\text{Li}_4\text{Ti}_5\text{O}_{12}$ is a candidate for anode materials in lithium-ion battery. It has zero-strain properties and thermally stable at high temperatures. There are many techniques to prepare $\text{Li}_4\text{Ti}_5\text{O}_{12}$ such as solid state reaction, ball-milling, hybrid microwave and sol-gel methods. Sol-gel method was employed in this work to synthesize $\text{Li}_4\text{Ti}_5\text{O}_{12}$. This is because sol-gel reactions occur at low temperature through hydrolysis and condensation reactions and form high homogeneities products.

This dissertation will also include ceramic-polymer composites. Miniaturization of electrical devices requires an integration of passive circuit elements such as resistors, capacitors and inductors. For the integration of these passive components, high dielectric constant thin films are essential. Although ferroelectric lead zirconate titanate

and barium titanate (BaTiO_3) are high dielectric constant materials and candidates for capacitors, high temperature processing is required to embed capacitors using these materials in resin substrates. Several methods have been adopted in order to realize low temperature processing. These include application of a nano-crystalline seeding technique (Kobayashi *et al.*, 2008) which enables temperatures as low as $350\text{ }^\circ\text{C}$ to be applied.

Another method that enables low temperature processing is the use of ceramic-polymer composites. This technique combines the low temperature processing of polymers and the high dielectric constant of ceramics. These composites can be prepared by mixing a dielectric polymer solution and submicron- or micron-sized ceramic particles, and evaporate the solvent of the polymer solution.

In this work, poly (vinyl alcohol) is chosen as the host polymer due to its ability to form film, non-toxic, water-soluble and biocompatible. It is a biodegradable polymer with excellent mechanical properties. However, most organic polymers have relatively low dielectric constant and in order to increase the dielectric constant of the material, ceramic powders with high dielectric constant were added [Popielarz and Chiang, 2007]. Hence PVA was used in the preparation of composites doped with inorganic particles, like perlite [Tian and Tagaya, 2008], montmorillonite [Kaczmarek and Podogórski, 2007a] and graphite [Kaczmarek and Podogórski, 2007b]. In this work $\text{Li}_4\text{Ti}_5\text{O}_{12}$ is the ceramic material used to produce the polymer composite.

Based on the gathered facts, the aims of the present dissertation are:

1. To synthesize the $\text{Li}_4\text{Ti}_5\text{O}_{12}$ anode material for lithium ion batteries using the sol-gel technique.

2. To characterize the anode materials using thermo gravimetric (TG) and x-ray diffraction (XRD) in order to understand the formation of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ from the raw materials used and for purpose of identification respectively.
3. To fabricate the electrochemical cell and study the performance of the cell using the $\text{Li}_4\text{Ti}_5\text{O}_{12}$ anode material prepared.
4. To study the effect of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ doping in poly (vinyl alcohol). The prepared films will also be characterized using x-ray diffraction (XRD), fourier transform infrared (FTIR) spectroscopy, electrochemical impedance spectroscopy (EIS) and UV-Vis absorption spectroscopy.

Chapter 2 present an inexhaustive overview of $\text{Li}_4\text{Ti}_5\text{O}_{12}$. Chapter 3 presents the method of sample preparation and sample characterization techniques such as x-ray diffraction (XRD), fourier transform infrared (FTIR) and UV-Vis spectroscopies. The precursor of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ will be analyzed using thermo gravimetry to determine the range of formation temperature and stability of the $\text{Li}_4\text{Ti}_5\text{O}_{12}$ compound. Results on the characterization of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ and performance of the half-cell are presented in Chapter 4. The effects of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ doping in PVA are presented in Chapter 5. Dielectric properties and type of relaxation of PVA doped with $\text{Li}_4\text{Ti}_5\text{O}_{12}$ will be determined in this chapter. The results obtained from Chapter 4 and 5 will be discussed in Chapter 6. The conclusions with suggestions for future work are dealt in Chapter 7.