

**UNIVERSITI TEKNOLOGI MARA**

**ELECTROCHEMICAL DEPOSITION AND  
CHARACTERIZATION OF TUNGSTEN OXIDE AS  
ELECTROCHROMIC FILMS**

**WAN DANIAL SHAHIZUAN BIN WAN GHAZALI**

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## ABSTRACT

Tungsten oxide ( $\text{WO}_3$ ) has been extensively studied material for preparation of electrochromic film for smart windows applications. This work reports the preparation of  $\text{WO}_3$  films by electrodeposition technique. The effects of electrodeposition parameters such as deposition solution and deposition conditions on the structural, morphological and electrochromic properties of electrodeposited  $\text{WO}_3$  films were investigated. The deposited  $\text{WO}_3$  films were characterized using FESEM for surface morphology, EDAX for elemental analysis, XRD for structural properties, Cyclic Voltammetry for electrochromic performance and UV-Vis Spectroscopy for optical properties. The stability of bath solution containing tungsten ions from two different precursors (W salt and W powder) played an important factor in the formation of  $\text{WO}_3$  films on ITO-glass substrate. It was found that smooth, well adhered and transparent  $\text{WO}_3$  films with good electrochromic performances (switching colouration and cycling stability during intercalation and deintercalation of  $\text{H}^+$  ions) can be prepared from 0.05M tungstate ions (using W powder) + 10mL  $\text{H}_2\text{O}_2$  in the presence of 30 mL 2-propanol by applying constant potential of  $-0.45\text{V}$  vs Ag/AgCl for 300s at  $40^\circ\text{C}$ . Post-treatment by annealing has significantly changed the surface morphology and structural property of  $\text{WO}_3$  films from amorphous to crystalline after annealed at  $T > 300^\circ\text{C}$ . The crystalline  $\text{WO}_3$  films have poor electrochromic performance than the amorphous films due to the entrapment of  $\text{H}^+$  ions into the crystalline structure of  $\text{WO}_3$  films. The coloured  $\text{WO}_3$  film has reflected about 70% the transmission of near-IR light (ie: solar heat). The prepared  $\text{WO}_3$  film is a great potential to be used as an electrochromic film in vehicles and building industries.

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# CHAPTER ONE

## INTRODUCTION

### 1.1 BACKGROUND OF STUDY

Over the last two decades, there has been progressive interest in the fabrication of thin solid film materials for many advanced applications. Thin films of metal oxides such as Zinc Oxide (ZnO), Nickel Oxide (NiO), Lead Oxide (PbO<sub>2</sub>) and Tungsten Oxide (WO<sub>3</sub>) have been the materials of quickly growing attention due to their interesting physicochemical and optical properties. Properties of WO<sub>3</sub> films such as electrochromism, electrical and optical have been exploited for many technological applications such as for the development of sensors (Sadex, Woldarski, & Shin, 2008), smart windows (Lampert, 1984), electrochromic devices (Badilescu & Ashrit, 2003), catalytic material (Xiang, et al., 1995) and electrosynthesis of organic compounds (Guglielmi, 1972).

WO<sub>3</sub> films can be deposited onto substrates with the use of various methods, such as vacuum techniques sputtering (Nanba et al., 1994), thermal evaporation (Bohnke, Bohnke, Robert, & Carquille, 1982) and chemical vapor deposition (Bange, 1999); and also chemical methods such as spin coating, spray pyrolysis, sol-gel deposition and electrochemical deposition (Lee, 1997; Leftheriotis & Yianoulis, 2008; Yu, Jia, Du, & Zhang, 2000). Electrochemical deposition is a versatile technique for the synthesis of materials with a variety of morphologies and structures. The control of current (i.e.: galvanostatic) or voltage (i.e.: potentiostatic) permits to vary the amount or thickness of deposited materials using electrochemical deposition technique.

The fabrication of WO<sub>3</sub> films with nanostructure and highly porous opens up the way to tailor the physical (e.g.: effective surface area, thermal stability, energy gap) and chemical (e.g.: surface reactivity, stoichiometry, chemical stability) properties in view of electrochromic devices development. The electrochemical and optical properties of WO<sub>3</sub> films are strongly related to their morphology, porosity and the size of the WO<sub>3</sub> particles (Yang, Li, Blackford, & Luca, 2006). The electrochromic activities of WO<sub>3</sub> film can be improved by reducing its particle size