

**UNIVERSITI TEKNOLOGI MARA**

**SYNTHESIS OF TIN OXIDE FILM  
FROM TIN OCTOATE USING SOL-  
GEL DIP-COATING METHOD ON  
GLASS SUBSTRATES**

**RAFIZAH BINTI ZAITON**

Thesis submitted in fulfillment  
of the requirements for the degree of  
**Master of Science**

**Faculty of Chemical Engineering**

**October 2019**

## ABSTRACT

The work presented in this thesis deals with experimental and theoretical studies related to tin oxide, SnO<sub>2</sub> thin films. Tin oxide widely used in optical and electronic applications. The main purpose of this research is to synthesis and to characterize the SnO<sub>2</sub> film for coating on a glass substrate by using a sol-gel dip-coating method. The crucial part of this study was the formulation of the coating solution. Therefore, the effect of solvent, acid catalyst, aging time and binder on viscosity also was investigated in this study. Tin octoate was used as the main material that acts as a precursor, Ethylene Glycol (EG) as a solvent and Nitric acid (HNO<sub>3</sub>) as a catalyst. In this research, SnO<sub>2</sub> thin film was synthesized through the sol-gel method with the addition of an organic binder which is Polyethylene glycol (PEG). The obtained gel and coated substrates were then dried at 200 °C. Chemical properties of SnO<sub>2</sub>, were characterized using FTIR, XRD and SEM-EDX to study the structural/bonding determination, phase of the coated samples and also the surface morphology of sol coatings on the glass substrate and sol powder after drying. Besides that, mechanical properties also studied using adhesion tests on the coated layer. The best formulation for coating solution to obtain SnO<sub>2</sub> film was arbitrarily selected at 1:10:1 represent Sn(Oct)<sub>2</sub>, E.G and HNO<sub>3</sub> respectively at 24 h .and concentration of PEG binder used was 8g. This formulation gives a viscosity range of 40cp which was suitable for coating on glass substrates. Based on the FTIR result, Sn-O and Sn-O-Sn bonding were obtained on the absorption peak between 500-700nm. XRD result also shows the phase of SnO<sub>2</sub> obtained by referring XRD pattern from (JCPDS file No.86-2265), it shows peaks of both the tetragonal structure of Sn and SnO (JCPDS file No. 06-0395). However, the result indicates that this temperature (200°C) tin not fully oxidized yet. In addition, it was clearly observed that tin, Sn dominated the composition of the element on the SnO<sub>2</sub> coating film by 68.30 %, followed by oxygen, O (28.32 %) and, carbon, C (3.38 %) when characterized using EDX. In this research, the average thickness of the thin film was 256 μm. Thus, it shows the thickness of the coating was proved by the illustrations made in by the SEM micrograph. Last but not least, an adhesion test showed 66.7% was peeled off from the surface of the glass when no PEG binder was added compared with only 26.7% was peeled off from the surface for a coating solution with a binder. This can be concluded that an addition of binder can help to produce a better adhesion result which leads to producing a stronger film.

## ACKNOWLEDGEMENT

### *In the name of Allah, the Most Gracious and the Most Merciful*

Alhamdulillah, all praises to Allah S.W.T for His guidance and blessing for giving me strength and ability for making this research project accomplishment. First and foremost, my special appreciation goes to my supervisor, Dr. Norliza Ibrahim for her support and very useful advice with a continuous stream of suggestions, comments, and encouragement. What I had learned throughout the whole period of my research project had been a meaningful and worthwhile experience.

I would like to express my appreciation to the Dean and the Deputy Deans, Faculty of Chemical Engineering for their support and help towards my postgraduate affairs. My acknowledgement is also goes Dr Putri Nadzrul Faizura Megat Khamaruddin and Mrs Adeebah Md Zin for their cooperation and support during my journey in this study.

Many thanks to all technicians especially Mrs Azizan Din, Mr Mohd Rizuan Razlan and Mr Mohd Yazid Yusof in Faculty of Chemical Engineering for providing me their guidance and assistance throughout the experimentation period.

I would like to extend my sincere thanks to all my friends especially Izni Mariah, Sxureha Yunus, Fitriah Rabani, Fatin Alia, Zatul Iranati, Faridatul Akmal, Ezan Suhaila, Noorani Kamarolzaman, Nur Shafarina Zainon and all members of postgraduate for their moral support and helps during my study. Thank you very much for great memories.

Finally, my sincere warm-hearted gratefulness goes to my parents, Mr Zaiton bin Othman and [REDACTED] and my siblings for their everlasting love, understanding, patience, du'a and support. Without them, none of my accomplishment would have been possible

***RAFIZAH ZAITON, 2019***

# TABLE OF CONTENTS

	<b>Page</b>
<b>CONFIRMATION BY PANEL OF EXAMINERS</b>	<b>ii</b>
<b>AUTHOR'S DECLARATION</b>	<b>iii</b>
<b>ABSTRACT</b>	<b>iv</b>
<b>ACKNOWLEDGEMENT</b>	<b>v</b>
<b>TABLE OF CONTENTS</b>	<b>vi</b>
<b>LIST OF TABLES</b>	<b>ix</b>
<b>LIST OF FIGURES</b>	<b>x</b>
<b>LIST OF SYMBOLS</b>	<b>xiii</b>
<b>LIST OF ABBREVIATIONS</b>	<b>xiii</b>
<b>CHAPTER ONE: INTRODUCTION</b>	<b>1</b>
1.1 Research Background	1
1.2 Problem Statement	2
1.3 Objectives	3
1.4 Scope of Research	3
1.5 Thesis Outline	4
<b>CHAPTER TWO: LITERATURE REVIEW</b>	<b>5</b>
2.1 Overview	5
2.2 Thin Film Deposition Technique	8
2.3 Sol-Gel Process	11
2.3.1 Sol-gel Processing Parameter	15
2.3.2 Effect of Solvent	15
2.3.2.1 Ethylene Glycol as Solvent	16
2.3.3 Effect of Sol Concentration	18
2.3.4 Effect of Acidity	19
2.3.5 Effect of Ageing Time	23
2.3.6 Effect of Binder	24

2.3.6.1	<i>Poly Ethylene Glycol (PEG)</i>	25
2.3.7	Effect of Viscosity	27
2.3.8	Effect of Drying	29
2.3.9	Thin Film Fabrication	29
2.3.10	Glass Substrates as a Support	31
2.3.11	Characterization Technique of Sol-Gel and the Thin Film of SnO <sub>2</sub>	32
2.4	Application of Tin Oxide in Industrial Sector	35
 <b>CHAPTER THREE: RESEARCH METHODOLOGY</b>		<b>37</b>
3.1	Overview of Methodology	37
3.2	Materials	39
3.3	Film Forming Formulation	39
3.3.1	Material Formulation of a Coating Solution	39
3.3.2	Effect of Concentration of Solvent on SnO <sub>2</sub> Sol	40
3.3.3	Effect of HNO <sub>3</sub> Concentration on Prepared SnO <sub>2</sub> Sol	40
3.3.4	Effect of Ageing Time on SnO <sub>2</sub> Sol	41
3.3.5	Effect of PEG Binder Concentration on SnO <sub>2</sub> Properties and Structures	41
3.3.6	Sol-gel Dip-coating to Produce SnO <sub>2</sub> Thin Films	42
3.4	Characterization Studies of the Coating Solution and Thin Film	44
3.4.1	pH Measurement	44
3.4.2	Chemical Bond and Functional Group Analysis	44
3.4.3	Thickness Measurement	45
3.4.4	Crystallinity and Phase Analysis	45
3.4.5	Morphology Analysis	45
3.4.6	Elemental Composition Analysis	46
3.4.7	Durability of the Coated Film	46
 <b>CHAPTER FOUR: RESULTS AND DISCUSSION</b>		<b>47</b>
4.1	Introduction	47
4.2	Preliminary Study on Formulation of a Coating Solution	47
4.3	Parameter Effect on Viscosity of Coating Solution	50
4.3.1	Dependency of the Viscosity of SnO <sub>2</sub> Sol Depending on Ethylene Glycol	