

R

**THE EFFECT OF WEAK ACIDS HYDROLYSIS
ON THE SOL-GEL FABRICATION OF SILICA
THIN FILM**

ABDUL HAMID

**UNIVERSITY OF MALAYA
2002**

Perpustakaan Universiti Malaya



A511032938

**THE EFFECT OF WEAK ACIDS HYDROLYSIS
ON THE SOL-GEL FABRICATION OF SILICA
THIN FILM**

BY

ABDUL HAMID

**Research Project Report Submitted in Fulfillment of
MSc in Analytical Chemistry & Instrumental
Analysis by course work**

**DEPARTMENT OF CHEMISTRY,
UNIVERSITY OF MALAYA,
Kuala Lumpur, 50603 Malaysia**

2002

Acknowledgement

Alhamdulillah, all praises be to Allah. I am very thankful to ALLAH for giving me the opportunity to complete this project report within the stipulated time. Certainly without His mercy and blessing nothing will ever be within my reach.

I am also indebted to my supervisor, Dr. Misni Misran for all the guidance and advices given me throughout this project and also his persistent effort in helping me to complete the project.

And I am also grateful to all my friends for their help. A heartfelt appreciation is to Rohingya Student Development Movement (RSDM), and its members for their persistent encouragement and assistance.

Table of Contents

Acknowledgement-----	i
Table of contents-----	ii
Lists of illustration -----	vi
List of tables-----	viii
Chapter one: Introduction-----	1
 Chapter two: Literature Review	
2.1 The sol-gel technology -----	4
2.2 Sol-gel process -----	5
2.3 Sol-gel research -----	6
2.4 Hydrolysis reaction mechanism -----	7
2.5 Silane chemistry -----	10
2.6 Sol-gel applications -----	11
 Chapter three: Experimental procedure	
3.1 Surface pre-treatment -----	15
3.2 Chemical reagents -----	15
3.3 Instruments -----	16
3.3.1 Spin coater -----	16
3.3.2 Atomic Force Microscope -----	17
3.3.3 Scanning Electron Microscope -----	18
3.3.4 Thermogravimetric Analyzer -----	19

3.4 Preparation of sol-gel solution -----	22
3.4.1 Methodology for sol preparation -----	24
3.5 Spin coating -----	25
3.5.1 Developing thin film -----	25
3.5.2 Drying and sintering -----	26
3.5.3 The effect of temperature-----	27
3.6 Summarization of gel formation route -----	32
3.7 Heat treatment schedule -----	33
Chapter four: Results and Discussion -----	34
4.1 Acetic acid catalyzed sol coated thin films -----	36
4.2 Propionic acid catalyzed sol coated thin films-----	43
4.3 Malonic & Maleic acid catalyzed sol coated thin films -----	51
Chapter five: Conclusion -----	61
References -----	63

List of illustration

Fig 1. Process flow for the sol-gel silica thin film fabrication-----	3
Fig 2. Thermogram of sample A (TEOS + EtOH + Acetic acid)-----	28
Fig 3. TG curve showing mass reduce of sample A (TEOS + EtOH + acetic acid) due to heat -----	29
Fig 4. Thermo gram of sample B (TEOS + EtOH + Malonic acid)-----	30
Fig 5. TG curve showing mass reduce of sample B (TEOS + EtOH + malonic acid) due to heat-----	31
Fig 6. Schematic diagram of organic and inorganic catalyzed sol-gel method---	32
Fig 7. Schematic diagram showing heat treatment of silica thin film-----	33
Fig 8. SEM micrograph of 12 mol dm^{-3} acetic acid catalyzed thin film after sintering at $300 \text{ }^\circ\text{C}$ -----	38
Fig 9. AFM micrograph image of 12 mol dm^{-3} acetic acid catalyzed thin film after sintering at $300 \text{ }^\circ\text{C}$ -----	38
Fig 10. SEM micrograph of 12 mol dm^{-3} acetic acid catalyzed thin film after sintering at 250°C -----	39
Fig 11. AFM micrograph image of 12 mol dm^{-3} acetic acid catalyzed thin film after sintering at $250 \text{ }^\circ\text{C}$ -----	39
Fig 12. SEM micrograph of 10 mol dm^{-3} acetic acid catalyzed thin film after sintering at $250 \text{ }^\circ\text{C}$ -----	40
Fig 13. AFM micrograph image of 10 mol dm^{-3} acetic acid catalyzed thin film after sintering at $250 \text{ }^\circ\text{C}$ -----	40

Fig 14. SEM micrograph of 8 mol dm ⁻³ acetic acid catalyzed thin film after sintering at 250 °C -----	41
Fig 15. AFM micrograph image of 8 mol dm ⁻³ acetic acid catalyzed thin film after sintering at 250 °C -----	41
Fig 16. SEM micrograph of 6 mol dm ⁻³ acetic acid catalyzed thin film after sintering at 250 °C -----	42
Fig 17. AFM micrograph image of 6 mol dm ⁻³ acetic acid catalyzed thin film after sintering at 250 °C -----	42
Fig 18. SEM micrograph of 2.0 ml propionic acid catalyzed thin film after sintering at 300 °C -----	45
Fig 19. AFM micrograph image of 2.0 ml propionic acid catalyzed thin film after sintering at 300 °C -----	45
Fig 20. SEM micrograph of 2.0 ml propionic acid catalyzed thin film after sintering at 250 °C -----	46
Fig 21. AFM micrograph image of 2.0 ml propionic acid catalyzed thin film after sintering at 250 °C -----	46
Fig 22. SEM micrograph of 1.00 ml propionic acid catalyzed thin film after sintering at 250 °C -----	47
Fig 23. AFM micrograph image of 1.0 ml propionic acid catalyzed thin film after sintering at 250 °C -----	47
Fig 24. SEM micrograph of 2.0 ml propionic acid catalyzed thin film after sintering at 120 °C -----	48

Fig 25. AFM micrograph image of 2.0 ml propionic acid catalyzed thin film after sintering at 120 °C	48
Fig 26. SEM micrograph of 1.5 ml propionic acid catalyzed thin film after sintering at 120 °C	49
Fig 27. AFM micrograph image of 1.5 ml propionic acid catalyzed thin film after sintering at 120 °C	49
Fig 28. SEM micrograph of 1.0 ml propionic acid catalyzed thin film after sintering at 120 °C	50
Fig 29. AFM micrograph image of 1.0 ml propionic acid catalyzed thin film after sintering at 120 °C	50
Fig 30. SEM micrograph of 0.70 mol dm ⁻³ malonic acid catalyzed thin film after sintering at 75 °C	53
Fig 31. AFM micrograph image of 0.70 mol dm ⁻³ malonic acid catalyzed thin film after sintering at 75 °C	53
Fig 32. SEM micrograph of 1.00 mol dm ⁻³ malonic acid catalyzed thin film after sintering at 75 °C	54
Fig 33. AFM micrograph image of 1.00 mol dm ⁻³ malonic acid catalyzed thin film after sintering at 75 °C	54
Fig 34. SEM micrograph of 1.72 mol dm ⁻³ malonic acid catalyzed thin film after sintering at 75 °C	55
Fig 35. AFM micrograph image of 1.72 mol dm ⁻³ malonic acid catalyzed thin film after sintering at 75 °C	55

Fig 36. SEM micrograph of 2.58 mol dm ⁻³ malonic acid catalyzed thin film tafter sintering at 75 °C -----	56
Fig 37. AFM micrograph image of 2.58 mol dm ⁻³ malonic acid catalyzed thin film after sintering at 75 °C -----	56
Fig 38. SEM micrograph of 0.70 mol dm ⁻³ maleic acid catalyzed thin film after sintering at 75 °C -----	57
Fig 39. AFM micrograph image of 0.70 mol dm ⁻³ maleic acid catalyzed thin film after sintering at 75 °C -----	57
Fig 40. SEM micrograph of 1.00 mol dm ⁻³ maleic acid catalyzed thin film after sintering at 75 °C -----	58
Fig 41. AFM micrograph image of 1.00 mol dm ⁻³ maleic acid catalyzed thin film after sintering at 75 °C -----	58
Fig 42. SEM micrograph of 1.72 mol dm ⁻³ maleic acid catalyzed thin film after sintering at 75 °C -----	59
Fig 43. AFM micrograph image of 1.72 mol dm ⁻³ maleic acid catalyzed thin film after sintering at 75 °C -----	59
Fig 44. SEM micrograph of 2.58 mol dm ⁻³ maleic acid catalyzed thin film after sintering at 75 °C -----	60
Fig 45. AFM micrograph image of 2.58 mol dm ⁻³ maleic acid catalyzed thin film after sintering at 75 °C -----	60

List of tables

Table 1: The components ratio of various acetic acids sol-gel solution -----	23
Table 2: The components ratio of various malonic and maleic acid sol-gel solution -----	23
Table 3: The components ratio of various propionic acid sol-gel solutions -----	23
Table 4: The thickness and roughness of acetic acid catalyzed thin films -----	27
Table 5: The thickness and roughness of propionic acid catalyzed thin films ----	37
Table 6: The thickness and roughness of malonic acid catalyzed thin films -----	43
Table 7: The thickness and roughness of maleic acid catalyzed thin films -----	52
Table 8: the temperature effect on thin film thickness -----	52