UNIVERSITI PUTRA MALAYSIA

FREQUENCY BEHAVIOUR OF QUARTZ CRYSTAL MICROBALANCE (QCM) IN CONTACT WITH SELECTED SOLUTIONS

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By

ZURAIDAH BABA

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia in Fulfilment of the Requirements for the Degree of Master of Science

May 2007

Devoted to my family and my beloved hubby

Zainab Bt Yasin

Zanariah Bt Baba & Azahar Alias

Rosli Bin Baba

Zakaria Bin Baba & Merlyna Zulfa

Anuar Bin Abdul Aziz

Thanks for the love, support, prayers, understanding and encouragement through the duration of this research project.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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By

ZURAIDAH BABA

May 2007

Chairman : Associate Professor Zainal Abidin Talib, PhD

Faculty : Science

This thesis investigated the interfacial behavior of some selected solutions in contact with one surface of Quartz Crystal Microbalance (QCM). A device was constructed to monitor viscosity of solutions using fundamental frequency of 9 MHz and 10 MHz quartz crystal. Piezoelectric quartz crystals with gold electrodes were mounted by O-ring in between liquid flow cell. Only one side of the crystal was exposed to the solutions which were pumped through silicon tube by a peristaltic pump. The measured frequency shift was observed in order to investigate the interfacial behavior of some selected solution in contact with one surface of Quartz Crystal Microbalance (QCM). An analysis of the interaction between an AT-cut quartz crystal microbalance and various liquid system of analytical interest is presented. The analysis which included piezoelectric effects and other influences; liquid properties, experimental conditions and the characteristic of the solution are reported. Oscillation in distilled water was taken as a reference. The frequency change caused by the density (ρ, gcm^{-3}) and viscosity (η,

$gcm^{-1}s^{-1}$) were found to be proportional to the square root of the product, ($\rho\ \eta$). The result suggested that analysis of small frequency shifts during EQCM studies needs to account for changes in ρ and η of the solution.

In this work the frequency responses of the QCM in contact with the saccharide (sucrose, maltose and glucose), alliphatic alcohols groups (methanol, ethanol and 1-propanol), polyethylene glycol (degree of polymerization of 400, 4000, 10000 and 20000), urea, dimethyl sulfoxide, glycerol and aromatic hydrocarbon (hexane, benzene and toluene) were measured.

Generally, all the liquid tested showed an increment of the frequency shift with increasing content of the solutes. For each solution, the frequency was recorded as the concentration increases from distilled water to a very concentrated solution. The frequency measurements carried out for saccharide solution produces the maximum changes of frequency shift compared with other solutions.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

PERLAKUAN FREKUENSI KUARZA KRISTAL MIKROJISIM BERHUBUNG DENGAN LARUTAN TERPILIH

Oleh

ZURAIDAH BABA

Mei 2007

Pengerusi : Profesor Madya Zainal Abidin Talib, PhD

Fakulti : Sains

Tesis ini mengkaji perlakuan antara permukaan beberapa larutan terpilih apabila bersentuhan dengan satu permukaan Kuarza Kristal Mikrojisim (QCM). Satu alat telah direkabentuk untuk menguji kelikatan larutan menggunakan frekuensi asas kuarza crystal 9 MHz dan 10 MHz. Kuarza kristal piezoelektrik dengan elektrod emas dikepit menggunakan O-ring di antara sel aliran cecair. Hanya satu permukaan kristal didedahkan kepada larutan yang dipam melaui tiub silicon menggunakan pam peristaltik. Ukuran perubahan frekuensi diperhatikan ketika perlakuan antara permukaan terhasil di antara larutan terpilih yang bersentuhan dengan satu permukaan Kuarza Kristal Mikrojisim (QCM). Analisis ke atas tindak balas di antara potongan-AT kuarza kristal mikrojisim dan sistem pelbagai cecair diterangkan secara terperinci. Laporan analisa termasuk kesan piezoelektrik dan kesan lain, antaranya ciri-ciri cecair, keadaan eksperimen dan sifat-sifat larutan. Ayunan di dalam air suling diambil sebagai rujukan. Perubahan frekuensi disebabkan oleh kepekatan (ρ, gcm^{-3}) dan kelikatan (η, $gcm^{-1}s^{-1}$)

telah dikenal pasti berkadar terus dengan punca kuasa dua produk, ($\rho\ \eta$). Keputusan mencadangkan bahawa analisis perubahan kecil pada frekuensi semasa proses EQCM perlu mengambil kira perubahan pada kepekatan, ρ dan kelikatan η larutan.

Dalam kajian ini, tindakbalas frekuensi QCM apabila bersentuhan dengan larutan sakarida (sukrosa, maltosa dan glukosa), kumpulan aliphatik alcohol (metanol, etanol dan 1-propanol), polietilena glikol dengan darjah pempolimeran adalah 400, 4000, 10000 dan 20000, urea, dimetil sulfoksida, gliserol dan aromatik hidrokarbon (heksana, benzena dan toluena) telah diukur.

Secara amnya, semua larutan yang diuji menunjukkan penambahan pada perubahan frekuensi apabila kandungan bahan terlarut juga bertambah. Bagi setiap larutan, perubahan frekuensi direkod untuk setiap kenaikan kepekatan larutan dari air suling kepada larutan pekat. Bacaan frekuensi yang diperolehi menunjukkan larutan sakarida menghasilkan perubahan frekuensi yang maksimum berbanding dengan larutan lain.

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Alhamdulillah, I grateful thank God, Almighty ALLAH S. W. T. , for giving me health and strength for successfully completing my project and writing this thesis. I also extend greet to Prophet, Muhammad S. A. W.

This dissertion ends a very fruitful and fascinating period of Master research. I especially appreciate the enthusiastic and cooperative efforts to my Supervisor, Assoc. Prof. Dr. Zainal Abidin Talib for his kind advice, proper guidance, attention and support throughout the project. Our long scientific discussion helped planting the seeds of my research, crystallizing ideas and experimental results to a whole and harmonized picture. Without his interest and continuous support, this academic research project will not be possible to finish.

I would also like to express my deep gratitude and appreciation of the assistance rendered to me by my co-supervisors, Assoc. Prof. Dr. Ionel Valeriu Grozescu and Prof. Dr. Anuar Kassim on the valuable advice and guidance in bringing this research study to completion and in editing the text for publication.

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I hope that all the experience, knowledge and findings of this research would be useful for others in the future.

Finally, I am grateful to those who have assisted me in preparing this thesis. The financial support provided by the Malaysian Government through the IRPA (Vote Number: 09-02-04-0458-EA001) program.

Thank you very much.

May Allah Ta'ala bless you all.

I certify that an Examination Committee met on 4th May 2007 to conduct the final examination of Zuraidah Baba on her Master of Science thesis entitle "The Frequency Behaviour of Quartz Crystal Microbalance (QCM) in contact with selected solutions." In accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulation 1981. The committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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This thesis submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee are as follows:

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Professor/ Dean
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Date: 9 August 2007

DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any degree at UPM or other institutions.

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ZURAIDAH BABA

Date: 1 August 2007

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS/NOTATION/GLOSARRY OF TERM

AT-cut	A slice through a quartz rod at an angle of approximately 35^{o} with respect to crystallographic *x* axis. The most common used for QCM applications.
A_o	maximum vibration amplitude
$\overset{0}{A}$	Armstrong ($\overset{0}{A}$ =3 x 10^{-8} m)
δ	penetration depth
∂z	acceleration of the slab
F	stress (Nm^{-1})
f_q	quartz resonator frequency (MHz)
ΔF	resonant frequency shift (MHz)
Δf_W	frequency different in water (MHz)
f_o	fundamental frequency of quartz crystal microbalance (MHz)
k	propagation constant of the instantaneous shear wave velocity decay/$k = \omega(\rho_q \mu_q)^{0.5}$
K	parameter ($cm^2 g^{-1} s^{-1/2}$)
ΔM	mass (g)
η_1	liquid viscosity (Poise)
η_L	absolute viscosity (Poise)
η_W	viscosity of water (Poise)
ρ	density of quartz (ρ =2.648 gcm^{-3})
ρ_1	liquid density (gcm^{-3})

ρ_L	fluid density (gcm^{-3})
ρ_W	density of water (gcm^{-3})
Quality factor, Q	The ratio of peak energy stored to energy lost per cycle during oscillation
QCM	Quartz Crystal Microbalance
t	efflux time (s)
μ	shear modulus of the quartz crystal (dyne cm^{-2})
u_x	elastic displacement along x
U	Amplitude
v_x	fluid velocity in the x direction – parallel to the resonator – liquid interface.
ω	angular frequency (MHz)