



UNIVERSITI PUTRA MALAYSIA

**PREPARATION AND CHARACTERIZATION OF GLASS-CERAMICS
SYNTHESIZED FROM SODA LIME GLASS AND WASTEWATER
SLUDGE**

SYAHARUDIN BIN ZAIBON

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By

SYAHARUDIN BIN ZAIBON

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

January 2011



DEDICATION

*To my beloved parents Zaibon Mohd Yatim and Ainun Hj Jalal
for their boundless love and repeated encouragement ..*

*To my family members
for their wonderful support and concern...*

*To my supervisors:
Assoc. Prof. Dr. Zaidan Abdul Wahab and Dr. Khamirul Amin Matori
for their guidance and advice...*

*To all my friends
for their assistance and supports...*



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

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Chairman: Associate Professor Zaidan Abdul Wahab, PhD

Faculty: Science

This project reports the utilization and vitrification of wastewater sludge (WS) and soda lime silica (SLS) glass via converting them into glass-ceramic materials. The vitrification method has been used and also exploited as a solution for the disposal of WS and SLS glass and to minimize any environmental hazards.

The glass ceramics were prepared from a mixture of wastewater sludge and SLS glasses and melted at 1375 °C for 3 hours and quenched by pouring it into water to obtain a coarse frit. The frit glass was then crushed and sieved to 106µm and pressed into a pellet. The sintering process was performed at various temperatures between 700-1000 °C for 2 hours at 50 °C intervals. The combination of WS and SLS provided a suitable chemical composition for the production of glass-ceramic. The resulting samples were then characterized using energy dispersive X-ray fluorescence spectrometer (EDXFS), differential thermal analysis (DTA), X-ray diffraction (XRD), scanning electron microscopy (SEM) and laser flash apparatus (LFA).



In this work, a thermal diffusivity measuring apparatus was used for measurement of thermal diffusivity (α) of glasses and glass-ceramics. The measurements of α were carried out at room temperature up to 300 °C with the intervals of 50 °C. Experimental results showed that α value for samples A (25%WS 75%SLS) are in the range of 0.386 - 0.767 mm²s⁻¹, samples B (20%WS 80%SLS) are in the range 0.322 - 0.726 mm²s⁻¹, samples C (15%WS 85%SLS) are in the range 0.3 - 0.66 mm²s⁻¹, samples D (10%WS 90%SLS) are in the range 0.283 - 0.623 mm²s⁻¹, and for samples E (5%WS 95%SLS) are in the range 0.24 - 0.615 mm²s⁻¹. The changes of the nature of the crystallinity of the samples, heat treatment temperature and changes of the density value have been suggested to be responsible for the variation in the thermal diffusivity behavior.

DTA study indicated that there were only inflection points of the endothermic peaks in the DTA curves of the glass samples. XRD analysis showed the amorphous state of the glass samples and also the presence of the diopside sodian, augite, cristobalite low and calcium silicate phases in the heat-treated samples. SEM investigations revealed that small amount of crystallites occurred in the microstructure of the heat treated samples such as flower shape, hemispherical shape, leaf shape, flaky shape and also residual glassy phase in contrast to the amorphous structure of the samples.

It is recommended that future researches can be carried out to develop a good quality of glass-ceramic and would make them attractive to industrial application such as study in hardness, bend strength, erosive wear resistance, fracture strength and toughness.

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

PENYEDIAAN DAN PENCIRIAN SERAMIK KACA YANG DISINTESIS DARI KACA KAPUR SODA DAN ENAPAN CEMAR AIR BUANGAN

Oleh

SYAHARUDIN BIN ZAIBON

Januari 2011

Pengerusi: Profesor Madya Zaidan Abdul Wahab, PhD

Fakulti: Sains

Projek ini melaporkan penggunaan dan pengcaaan enapan cemar air buangan dan kaca silika kapur soda dengan menukarkannya ke bahan seramik-kaca. Kaedah pengcaaan ini telah diguna dan dieksploitasikan sebagai satu penyelesaian untuk pelupusan enapan cemar air buangan dan kaca silika kapur soda dan meminimumkan bahan-bahan yang berbahaya kepada alam sekitar.

Seramik-kaca disediakan daripada campuran enapan cemar air buangan dan kaca silika kapur soda dan dileburkannya pada suhu 1375 °C selama 3 jam dan dilindap secara pantas dengan menuangkannya ke dalam air untuk mendapatkan butiran kaca yang kasar. Kemudian butiran kaca kasar ini dihancurkan dan ditapis sehingga 106 µm dan dibentuk menjadi pelet. Proses rawatan haba dilakukan pada suhu diantara 700-1000 °C selama 2 jam pada sela suhu 50 °C. Gabungan WS dan SLS menyediakan komposisi kimia yang sesuai untuk pengeluaran seramik-kaca. Kemudiannya sampel-sampel dicirikan menggunakan spektrometer pendarfluor sinar-X penyebaran tenaga (EDXRF), analisis perbezaan terma (DTA), belauan sinar-X (XRD), mikroskop pengimbas elektron (SEM), dan alat kilauan laser (LFA).



Dalam kerja ini, satu alat pengukuran koresapan terma digunakan untuk mengukur koresapan terma (α) kaca dan seramik-kaca. Resapan terma, α diukur pada suhu bilik sehingga 300 °C dengan sela suhu 50 °C. Hasil eksperimen menunjukkan nilai α untuk sampel A (25%WS 75%SLS) adalah dalam julat 0.386-0.767 mm²s⁻¹, sampel B (20%WS 80%SLS) adalah dalam julat 0.322-0.726 mm²s⁻¹, sampel C (15%WS 85%SLS) adalah dalam julat 0.3-0.66 mm²s⁻¹, sampel D (10%WS 90%SLS) adalah dalam julat 0.283-0.623 mm²s⁻¹, dan sampel E (5%WS 95%SLS) adalah dalam julat 0.24-0.615 mm²s⁻¹. Perubahan keadaan penghabluran pada sampel, suhu rawatan haba, dan perubahan nilai ketumpatan telah dicadangkan sebagai penyebab kepada perubahan ciri koresapan terma.

Kajian DTA menunjukkan bahawa hanya terdapat titik infleksi puncak endoterma dalam lengkungan DTA untuk sampel-sampel kaca. Analisis XRD menunjukkan keadaan amorfus pada sampel-sampel kaca dan juga kehadiran fasa-fasa seperti diopsid sodian, augit, kristobalit rendah dan kalsium silikat dalam sampel-sampel yang dirawat haba. Analisis SEM mendedahkan bahawa sejumlah kecil sahaja kristalit terhasil di dalam struktur mikro sampel yang dirawat haba seperti yang berbentuk bunga, bundar, daun, berkelopak dan juga lebih fasa kaca.

Penyelidikan yang akan dilaksanakan pada masa akan datang, dicadangkan agar menghasilkan seramik kaca yang berkualiti tinggi dan mempunyai tarikan untuk kegunaan industri dengan mempelajari dan mendalami sifat-sifat seperti kekerasan, kekuatan lenturan, rintangan haus hakis, kekuatan retakan, dan ketegapan.

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I certify that an Examination Committee has met on 27 January 2011 to conduct the final examination of Syaharudin bin Zaibon on his Master of Science thesis entitled “Preparation and Characterization Of Glass-Ceramics Synthesized From Soda Lime Glass and Wastewater Sludge” in accordance with the Universities and University College Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The committee recommends that the student be awarded the Master of Science.

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This thesis was 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 were follows:

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DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

SYAHARUDIN BIN ZAIBON

Date: 27 January 2011



TABLE OF CONTENTS

		Page
	DEDICATION	ii
	ABSTRACT	iii
	ABSTRAK	v
	ACKNOWLEDGEMENTS	vii
	APPROVAL	viii
	DECLARATION	x
	LIST OF TABLES	xiii
	LIST OF FIGURES	xiv
	LIST OF ABBREVIATIONS	xvi
	LIST OF SYMBOLS	xi
	CHAPTER	
1	INTRODUCTION	1
	1.1 Research Problems	2
	1.2 Importance of the Study	6
	1.3 Objectives of the Study	11
	1.4 Scope of Study	12
2	LITERATURE REVIEW	13
	2.1 Introduction	13
	2.2 Review of Glass –ceramic	13
	2.2.1 Glass-ceramic	13
	2.3 The production of glass-ceramic by using waste materials	15
	2.4 Basics of heat transfer	17
	2.4.1 Thermal Diffusivity Measurement Using Laser Flash Technique	18
	2.4.2 Thermal Diffusivity of Solid Material	24
3	METHODOLOGY	30
	3.1 Introduction	30
	3.2 Sample preparation	30
	3.2.1 Selection of Raw Materials	30
	3.2.2 Batch composition	31
	3.2.3 Powder Preparation	31
	3.2.4 Mixing Process	32
	3.2.5 Glass Fabrication	33
	3.2.6 Molding Process	33
	3.2.7 Thermal Treatment Process	34
	3.3 Characterization of Samples	36
	3.3.1 Thermal Diffusivity Measurement using Laser	36

	Flash Method	
	3.3.2 X-ray Diffraction Analysis	39
	3.3.3 Chemical analysis	39
	3.3.4 Differential Thermal Analysis	40
	3.3.5 Microstructure analysis	42
	3.3.6 Density measurement	44
4	RESULTS AND DISCUSSION	45
	4.0 Introduction	45
	4.1 Energy Dispersive X-ray Analysis	45
	4.2 X-ray Diffraction Analysis	47
	4.3 Differential Thermal Analysis (DTA)	59
	4.4 Scanning Electron Microscopy Analysis	60
	4.4.1 Further Discussion in SEM Analysis	66
	4.5 Density of samples	75
	4.6 Laser Flash Technique Analysis	78
5	CONCLUSION AND RECOMMENDATIONS	90
	5.1 Conclusion	90
	5.2 Preparation of Sample Improvement	92
	5.3 Further Studies	92
	REFERENCES	R1
	APPENDICES	A1
	BIODATA OF THE STUDENT	B1
	PAPER PUBLISHED AND PRESENTED	P1

