UNIVERSITI PUTRA MALAYSIA

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

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

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 ºC 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 mm^2s^{-1}, samples B (20%WS 80%SLS) are in the range 0.322 - 0.726 mm^2s^{-1}, samples C (15%WS 85%SLS) are in the range 0.3 - 0.66 mm^2s^{-1}, samples D (10%WS 90%SLS) are in the range 0.283 - 0.623 mm^2s^{-1}, and for samples E (5%WS 95%SLS) are in the range 0.24 - 0.615 mm^2s^{-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 pengacaan enapan cemar air buangan dan kaca silika kapur soda dengan menukarkannya ke bahan seramik-kaca. Kaedah pengacaan ini telah diguna dan dieksploitasikan sebagai satu penyelesaian untuk pelupusan enapan cemar air buangan dan kaca silica 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 ºC. 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 keresapan terma digunakan untuk mengukur keresapan 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 mm^2s^{-1}, sampel B (20%WS 80%SLS) adalah dalam julat 0.322-0.726 mm^2s^{-1}, sampel C (15%WS 85%SLS) adalah dalam julat 0.3-0.66 mm^2s^{-1}, sampel D (10%WS 90%SLS) adalah dalam julat 0.283-0.623 mm^2s^{-1}, dan sampel E (5%WS 95%SLS) adalah dalam julat 0.24-0.615 mm^2s. Perubahan keadaan penghabluran pada sampel, suhu rawatan haba, dan perubahan nilai ketumpatan telah dicadangkan sebagai penyebab kepada perubahan ciri keresapan 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 dirawati haba. Analisis SEM mendedahkan bahawa sejumlah kecil sahaja kristalit terhasil di dalam struktur mikro sampel yang dirawati haba seperti yang berbentuk bunga, bundar, daun, berkelopak dan juga lebihan 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.

ACKNOWLEDGEMENT

In the name of Allah, the most gracious and most merciful, I would like to express my biggest and sincere gratitude towards my beloved parents. Your unwavering love, supports and understanding are highly appreciated. It goes without saying.

My great pleasure also goes to my project supervisor, Associate Professor Dr. Zaidan Abdul Wahab for his expertise and continuous guidance, constructive suggestions, untiring supports, invaluable guidance and encouragement which help me to complete this project. He is willing to spend his previous time in discussing the project. Plus, he ensured that my project will always be on a right track.

To Dr. Khamirul Amin Matori, thanks for being such a helpful and dedicated advisor towards the end of my study.

I would also like to dedicate my appreciations towards my lab-mates Mr. Mohd Zul Hilmi Mayzan, Miss. Norfarezah Hanim Edros and Mr. Thai Ming Yeow. Your guidance, supports and cooperations are highly appreciated. Thanks for being a good companion of mine.

My special dedication also goes to my beloved one, Siti Ayisah Abdullah. Thanks for all your kindness, love and supports.

To all my friends, Mohd Norizam Md Daud, Mohd Sabri Mohd Ghazali, Ahmad Warid Suhat, Norfarhana Ayuni Joha, Aina Suhaiza Nazir and Ahmad Mustaza Ahmad Rusli, thanks for your encouragement and moral support. May Allah bless you with His Rahmat. A very special thanked also goes to the technical staffs of Physics Department, Faculty of Science, Universiti Putra Malaysia.

In truth, Only Allah can reciprocate all the kindness. May Allah bless you…

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.

Members of the Examination Committee were as follows:

Jumiah Hassan, PhD
Associate Professor
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(Chairman)

Azmi Zakaria, PhD
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Date: 19 April 2011

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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Khamirul Amin Matori, PhD
Lecturer
Faculty of Science
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(Member)

HASANAH MOHD GHAZALI, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

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

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