



UNIVERSITI PUTRA MALAYSIA

**EFFECTS OF HEAT TREATMENT ON STRUCTURE AND THERMAL
DIFFUSIVITIES OF $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-Na}_2\text{O-CaO-CaF}_2$ GLASS-CERAMICS
FROM WASTE MATERIALS**

LOY CHEE WAH

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**MASTER OF SCIENCE
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FROM WASTE MATERIALS**



**By
LOY CHEE WAH**

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

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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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ON STRUCTURE AND THERMAL DIFFUSIVITIES
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FROM WASTE MATERIALS**

By

LOY CHEE WAH

December 2012

Chairman: Khamirul Amin Matori, PhD

Faculty: Science

Recycling waste materials into dental application is a developing field of study. Soda-lime-silica (SLS) glass and clamshell (CS) were used in this research to make dental glass-ceramics. The manufacture of glass-ceramics by using waste materials for dental industries can reduce a large amount of solid wastes produced daily. This research was focused on the phase transformations and thermal diffusivities of $\text{SiO}_2\text{-Na}_2\text{O-Al}_2\text{O}_3\text{-CaO-CaF}_2$ glass-ceramics which made by different weight ratio of CS and CaF_2 powders. This research is important to obtain an optimum composition and heat treatment temperature to improve the properties of glass-ceramic for dental applications. Two series of $\text{SiO}_2\text{-Na}_2\text{O-Al}_2\text{O}_3\text{-CaO-CaF}_2$ mixture samples (SM1-SM4) and glass samples (SG1-SG4) were produced. The raw materials used in this research were SLS glasses, α -alumina (Al_2O_3) powder, CS and calcium fluoride (CaF_2) powder. Both SLS glasses and CS were collected from waste products in food industries. The weight formula of the samples were $(\text{SLS})_{0.4} (\text{Al}_2\text{O}_3)_{0.3} (\text{CS})_{0.3-x} (\text{CaF}_2)_x$, where $x = 0, 0.1, 0.2$ and 0.3 . The SM1-SM4 were produced by mixing raw

materials according to their weight ratio and followed by pressing them into pellet form. Heat treatments at 500-1200 °C for 3 hours were carried out on these pellets to obtain crystalline phases. XRD, FTIR, TGA, DTA, FESEM, and thermal diffusivity tests were carried out on SM1-SM4. The SG1-SG4 were produced by rapid cooling of molten mixture powders which based on their weight ratio. The glasses obtained were ground into powder form and followed by pressed them into pellet form. Heat treatments at 500-1300 °C for 3 hours were carried out on these pellets to obtain crystalline phases. The crystallized SG1-SG4 glasses after being heat treated were called as glass-ceramics. XRD, FTIR, DTA, FESEM, and thermal diffusivity tests were also carried out on SG1-SG4. The result shows that all the SM1-SM4 and SG1-SG4 consists of nepheline and gehlenite phases after heat treatment at high temperature. The nepheline phase is an important phase in various dental materials, especially for dental crown. The nepheline phase at lowest heat treatment temperature was found in SG3 at 800 °C. The glass transition and crystallization temperatures of SG3 were 680 and 872 °C respectively.

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

**KESAN RAWATAN HABA TERHADAP
STRUKTUR DAN KERESAPAN HABA
BAGI $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-Na}_2\text{O-CaO-CaF}_2$ SERAMIK-KACA
DARIPADA BAHAN-BAHAN BUANGAN**

Oleh

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Kitar semula bahan buangan untuk aplikasi pergigian adalah bidang kajian yang sedang berkembang. Kaca soda-kapur-silika (SLS) dan kulit kerang (CS) telah digunakan di dalam kajian ini untuk menghasilkan seramik-kaca pergigian. Pengeluaran seramik-kaca dengan menggunakan bahan-bahan buangan untuk industri-industri pergigian dapat mengurangkan jumlah besar bahan-bahan buangan pejal seharian. Kajian ini menumpu kepada transformasi fasa dan keresapan terma bagi seramik-kaca $\text{SiO}_2\text{-Na}_2\text{O-Al}_2\text{O}_3\text{-CaO-CaF}_2$ yang diperbuat daripada serbuk-serbuk CS dan CaF_2 dengan nisbah berat yang berlainan. Kajian ini adalah penting untuk memperoleh komposisi dan suhu rawatan haba yang optimum bagi memperbaiki ciri-ciri seramik-kaca untuk aplikasi-aplikasi pergigian. Dua siri sampel-sampel campuran (SM1-SM4) dan sampel-sampel kaca (SG1-SG4) $\text{SiO}_2\text{-Na}_2\text{O-Al}_2\text{O}_3\text{-CaO-CaF}_2$ telah dihasilkan. Bahan-bahan mentah yang digunakan dalam kajian ini adalah kaca-kaca SLS serbuk α -alumina (Al_2O_3), CS and serbuk kalsium fluorida (CaF_2). Kedua-dua kaca-kaca SLS dan CS diperoleh daripada produk-produk buangan dalam industri-industri makanan. Formula berdasarkan berat

bagi sampel-sampel adalah $(SLS)_{0.4} (Al_2O_3)_{0.3} (CS)_{0.3-x} (CaF_2)_x$, di mana $x = 0, 0.1, 0.2$ and 0.3 . SM1-SM4 dihasilkan melalui pencampuran bahan-bahan mental mengikut nisbah beratnya dan diikuti dengan penekanan supaya membentuk *pellet*. Rawatan haba telah dijalankan ke atas *pellet-pellet* tersebut pada suhu 500-1200 °C selama 3 jam demi memperoleh fasa-fasa hablur. Ujian-ujian seperti XRD, FTIR, TGA, DTA, FESEM, and keresapan terma dikendalikan ke atas SM1-SM4. SG1-SG4 telah dihasilkan melalui penyejukan kejut leburan yang mengandungi campuran serbuk-serbuk berdasarkan nisbah beratnya. Kaca yang diperoleh telah dihancurkan kepada bentuk serbuk dan diikuti dengan penekanan supaya membentuk *pellet*. Rawatan haba telah dijalankan ke atas *pellet-pellet* tersebut pada suhu 500-1300 °C selama 3 jam demi memperoleh fasa-fasa hablur. SG1-SG4 yang telah dihablurkan selepas dirawat dengan haba dipanggil sebagai seramik-kaca. Ujian-ujian seperti XRD, FTIR, DTA, FESEM, and keresapan terma turut dijalankan ke atas SG1-SG4. Keputusan menunjukkan kesemua SM1-SM4 dan SG1-SG4 mengandungi fasa *nepheline* dan *gehlenite* selepas rawatan haba dijalankan pada suhu yang tinggi. Fasa *nepheline* adalah fasa yang penting di dalam pelbagai bahan-bahan pergigian, terutamanya untuk korona gigi. Fasa *nepheline* pada suhu rawatan haba terendah didapati di dalam SG3 pada 800 °C. Suhu transformasi kaca and penghabluran bagi SG3 adalah 680 dan 872 °C masing-masing.

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I certify that a Thesis Examination Committee has met on 26 December 2012 to conduct the final examination of Loy Chee Wah on his thesis entitled "**Effects of Heat Treatment on Structure and Thermal Diffusivities of SIO₂-NA₂O-AL₂O₃-CAO-CAF₂ Glass-Ceramics from Waste Materials**" in accordance with the Universities and University Colleges 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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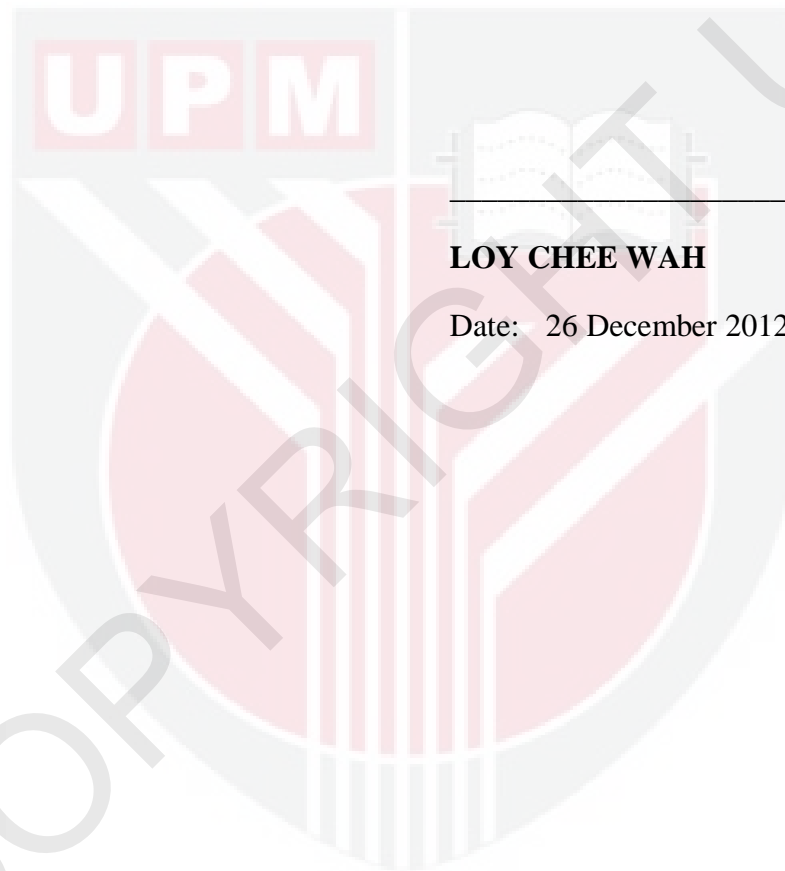
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DECLARATION

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.



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Date: 26 December 2012

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