UNIVERSITI PUTRA MALAYSIA

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MASTER OF SCIENCE

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By

HASLINAWATI MOHD MUSTAPHA

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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Chair : Khamirul Amin Matori, PhD

Faculty : Faculty of Science.

In this study, the chemical contents of rice husk (RH) were determined using Inductive Couple Plasma (ICP). Various types of acid were used in the leaching process of the RH samples. ICP data shows that, samples that have undergone acid leaching contained a lower percentage of metallic elements compared to untreated sample. Hydrochloric acid (HCl) leaching of husk is superior to Sulfuric acid (H_2SO_4) and Nitric acid (HNO_3) for removing the metallic elements. It also contained more percentage of silica (SiO_2) which is 94.61%, treated with HNO_3 (93.71%), treated with H_2SO_4 (92.56%) and for untreated (85.48%).

For heat treatment, the required combustion temperature is 500 °C and above to produce white ash within a reasonable time (~1 hour in this study). By heat treating rice husk ash (RHA) at 800 °C for 2 hours, all the carbon contained in RHA (black ash) was removed

and an amorphous white rice husk ash (WRHA) is produced. The weight loss of RHA sample treated at 500 °C was found to be about 80.71% and it was increased to 88.64% after treatment at 800 °C.

Ceramics produced from WRHA and Erbium Oxide (Er_2O_3) was made by mixing, milling, grinding, pressing and sintering procedure. They were well analyzed by XRD, scanning electron microscopy (SEM) and energy dispersive x-ray (EDX), fourier transform infrared (FTIR) and laser flash apparatus (LFA). The XRD revealed the changes in crystal phase due to sintering temperature. Cristobalite and trydimite phase were observed from all compositions. While for sample with addition of 10% and 20% of Er_2O_3, Er_2O_3 and $Er_2O_7Si_2$ phase were observed as well as cristobalite and trydimite phase. SEM is useful tool to analyze structural changes that occur at the surface of ceramics. The morphology analysis of samples showed that the microstructures of samples are related to the phase of the crystal. On the other hand, EDX analysis confirm the composition of elements contain in the samples. FTIR analysis showed the chemical group presents in the samples. The main band is observed as Si-O-Si stretching band in all samples due to its silica composition. Laser flash system was used to measure thermal diffusivity in order to compare the structural of samples with their thermal features. It was shown that thermal diffusivity is increased as sintering temperature increased. Changes in crystal phase as proved by XRD (from cristobalite to tridymite and Er_2O_3 to cristobalite and tridymite) are closely followed by thermal diffusivity. Thermal diffusivity is also dominated by their microstructure, density and temperature.

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

TERMOFIZIKAL PENCIRIAN SERAMIK SINTESIS DARIPADA SEKAM PADI DAN ERBIUM OXIDE

Oleh

HASLINAWATI MOHD MUSTAPHA

Disember 2010

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Dalam kajian ini, kandungan kimia di dalam sekam padi (RH) di kira menggunakan ICP. Berbagai jenis asid digunakan dalam proses penyingkiran sampel sekam padi. Keputusan ICP menunjukkan sampel yang telah melalui proses penyingkiran mengandungi kandungan bahan logam yang sedikit berbanding sampel yang tidak malalui proses tersebut. Asid hidroklorik (HCl) lebih baik daripada asid sulfuric (H_2SO_4) dan asid nitric (HNO_3) untuk menyingkirkan bahan logam. Ia juga mengandungi lebih banyak peratus silika (SiO_2) iaitu 94.61%, rawatan dengan HNO_3 (93.71%), rawatan dengan H_2SO_4 (92.56%) and tanpa rawatan (85.48%).

Bagi perawatan haba, suhu pembakaran yang diperlukan ialah 500 ˚C dan keatas untuk menghasilkan abu putih di dalam masa yang sesuai (~1 jam bagi kajian ini). Dengan

merawat RHA pada suhu 800 ˚C untuk 2 jam, semua kandungan karbon didalam RHA (abu hitam) telah disingkirkan dan abu putih (WRHA) amorfos dihasilkan. Peratus kehilangan berat sampel RHA yang di bakar pada suhu 500 °C adalah sebanyak 80.71% dan nilai tersebut meningkat kepada 88.64% selepas di bakar pada suhu 800 °C.

Seramik yang dihasilkan daripada WRHA dan Erbium Oxide (Er_2O_3) di buat melalui proses pencampuran, pengisaran, pemampatan, dan pembakaran. Sampel tersebut telah dianalis menggunakan XRD, FTIR, DTA, SEM and EDX. XRD mendedahkan perubahan didalam fasa hablur berhubung dengan suhu pembakaran. Fasa cristobalite dan trydimte telah di dapati dalam semua komposisi sampel. Manakala penambahan 10 dan 20 peratus komposisi Er_2O_3, fasa yang terlibat ialah Er_2O_3 dan $Er_2O_7Si_2$ serta cristobalite dan trydimite. SEM merupakan alat yang berguna untuk menganalisa perubahan struktur yang berlaku pada permukaan seramik. Analisis morfologi sampel menunjukkan bahawa struktur mikro sampel berkait dengan fasa kristal yang berlaku. Analisis EDX mengesahkan komposisi unsur-unsur di dalam sampel. Analisis FTIR menunjukkan kumpulan kimia yang wujud dalam sampel. Kumpulan utama diperhatikan sebagai kumpulan Si-O-Si dalam semua sampel adalah disebabkan oleh komposisi silikanya. Sistem laser digunakan untuk mengukur penyerakan haba untuk membandingkan struktur sampel dengan ciri-ciri habanya. Ia telah menunjukkan bahawa penyerakan haba meningkat dengan suhu pembakaran. Perubahan fasa kristal sampel sebagaimana dibuktikan melalui XRD (daripada cristobalite kepada trydimite dan Er_2O_3 kepada cristobalite kepada trydimite) diikuti rapat oleh penyerakan haba. Penyerakan haba juga didominasi oleh struktur mikro, ketumpatan dan suhu

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I certify that an Examination Committee has met on 30 December 2010 to conduct the final examination of Haslinawati binti Mohd Mustapha on his Master of Science thesis entitled "Thermophysical Characterization of Ceramic Synthesized from Rice Husk Ash and Erbium Oxide" in accordance with University Putra Malaysia (Higher Degree) Act 1980 and University Putra Malaysia (Higher Degree) Regulation 1981. The Committee recommends that the student be awarded the degree of Master of Science.

Members of Examination Committee were 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 degree of Master of Science. The members of the Supervisory Committee are as follows:

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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 institutions.

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HASLINAWATI MOHD MUSTAPHA

Date: 30 December 2010

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