UNIVERSITI PUTRA MALAYSIA

PHYSICAL AND ELECTRICAL PROPERTIES OF CERAMIC OBTAINED FROM WHITE RICE HUSK ASH AND SODA LIME SILICA GLASS

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DEDICATION

Dedicated to

My Loves Family And Husband

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

PHYSICAL AND ELECTRICAL PROPERTIES OF CERAMIC OBTAINED FROM WHITE RICE HUSK ASH AND SODA LIME SILICA GLASS

By

NASIM HEIDARI BATENI

September 2014

Chairman: Associate Professor Mohd Nizar B Hamidon, PhD

Faculty : Engineering

Rice Husk (RH) is a biodegradable cheap waste product, which is available in more than 75 rice producing countries. RHs are treated as waste, causing pollution and disposal problems. Due to the environmental concern and the need to conserve energy and resources, a number of studies and researches have been addressed and confronted for the conversion of RH biomass into a high value-added and useful income-generating products. It is found that RH can be used as an excellent source to produce silicon-based materials, since it is known to have high silica ash in the range of 20-25%. Thermal combustion of RH at moderate temperature and heated in an air atmosphere produces the white rice husk ash (WRHA), which contains >90% of amorphous silica. Meanwhile, soda lime silica (SLS) glass is the most common type of glass. The commercially made glass bottle was used in this study as an urban waste. Mixing the SLS glass with WRHA may improve the physical and electrical properties. Therefore, in this present study, three different mixtures of WRHA and SLS glass consists of 97.5 wt.%, 95 wt.% and 92.5 wt.% of WRHA and 2.5 wt.%, 5 wt.% and 7.5 wt.% of SLS glass, respectively. These samples were sintered at difference temperatures of 900 °C, 1000 °C and 1200 °C and been labeled as S_1, S_2 and S_3. Chemical, physical and electrical characteristics were analyzed using X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), Archimedes' method, ASTM C373, Scanning Electron Microscope (SEM), LCR meter and Sawyer-Tower circuit. XRF result shows that the main constituent of the mixture specimens is silica (91-94 wt.%) and impurities are such as CaO, K_2O, SO_3, Fe_2O_3. XRD result illustrates the crystalline structure of S_1, S_2 and S_3. The physical analysis reveals that S_3 shows better physical properties than other samples because it has the highest density (2.17 g/cm^3), lowest porosity (22.4 %) and lowest LS (1.8 %). Electrical analysis shows that S_1 (at 1200 °C) has the best electrical properties ($\varepsilon' = 11.1$ and $\tan\delta = 0.2$ at 100 kHz) comparing with S_2 and S_3. The reason is that S_1 has the lowest dielectric loss (0.26)

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and its P-E loop evaluation shows capacitor response. As a result, S_1 is the best dielectric material among other ceramics.

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

SIFAT-SIFAT FIZIKAL DAN ELEKTRIKAL BAGI SERAMIK YANG DIPEROLEHI DARI ABU PUTIH SEKAM PADI DAN KACA SILICA SODA KAPUR

Oleh

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Sekam Padi (RH) adalah sisa produk terbiodegradasikan yang murah serta boleh diperolehi dari 75 buah negara pengeluar padi. RH dianggap sebagai sisa yang boleh menyebabkan pencemaran dan masalah pelupusan. Oleh kerana kesedaran alam sekitar serta keperluan untuk memelihara tenaga dan sumber, beberapa kajian dan penyelidikan telah dijalankan untuk penukaran biomass RH kepada produk yang dapat menjana pendapatan dan mempunyai nilai tambah. Didapati bahawa RH boleh digunakan sebagai sumber yang baik untuk menghasilkan bahan-bahan berasaskan silikon, kerana ia diketahui mempunyai abu silika yang tinggi dalam lingkungan 20-25%. RH dibakar pada suhu sederhana dan dipanaskan dalam suasana udara, abu sekam padi putih (WRHA) terhasil dan mengandungi > 90 % daripada amorfus silika. Soda kapur silika (SLS) kaca adalah jenis kaca yang paling biasa. Botol kaca dibuat secara komersial telah digunakan dalam kajian ini sebagai sisa buangan. Campuran WRHA dan SLS berkemungkinan dapat menambah-baik sifat fizikal dan eletrikalnya. Oleh itu, kajian ini memberi tumpuan pada sintesis seramik berasaskan campuran WRHA dan SLS yang terdiri daripada 97.5, 95 dan 92.5 wt.% daripada WRHA dan 2.5, 5 dan 7.5 wt.% kaca SLS. Mereka disinter dengansuhu yang beza pada 900, 1000 dan 1200 °C serta dilabel dengan S_1, S_2 dan S_3. Sifat kimia, fizikal dan elektrikal kimia mekara dianalisa dengan menggunakan X-ray pendarfluor (XRF), pembelauan sinar-X (XRD), kaedah Archimedes, ASTM C373, mikroskop imbasan elektron (SEM), LCR meter dan litar Sawyer–Tower. Keputusan XRF menunjukkan bahawa juzuk utama daripada specimen adalah silika (91-94 wt.%) dan bendasing adalah seperti CaO, K_2O, SO_3, Fe_2O_3. Keputusan XRD memaparkan struktur amorfus di dalam serbuk bertukar kepada struktur Kristal apabila seramik telah terbentuk bagi S_1, S_2 dan S_3. Keputusan analisa fizikal mendedahkan sampel S_3 menunjukkan sifat fizikal yang terbaik berbanding sampel lain kerean ia mempunyai ketumpatan yang tertinggi (2.17 g/cm^3), keliangam rendah (22.4 %) dan LS yang rendah (1.8 %). Sifat elektrikal pula menunjukkan sampel S_1 (pada suhu 1200 ^{o}C) mempunyai sifat elektrikal terbaik ($\varepsilon' = 11.1$ dan tan $\delta = 0.2$ pada 100 kHz) berbanding sampel S_2 dan S_3. Ini kerana sampel S_1 mempunyai kekurangan dielektrik terendah (0.26) dan penilaian

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gelung P-E menunjukkan tindakbalas kapasitor. Maka Kesimpulannya, S_1 adalah bahan dielektrik yang terbaik dibandingkan denagn sampel-sample yang lain.

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APPROVAL

I certify that a Thesis Examination Committee has met on …. 2014 to conduct the final examination of NasimHeidariBateni on his thesis entitled "**PHYSICAL AND ELECTRICAL PROPERTIESOF CERAMIC OBTAINED FROM WHITE RICE HUSK ASH AND SODA LIME SILICA GLASS**." 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 relevant degree of Master.

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

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LIST OF ABBREVIATIONS

A	Area of electrodes
ASTM C373	American standard test method C373
Biocon	Biological Control company
C	Capacitance
CMOS	Complementary Metal Oxide Semiconductor
d	Thickness of sample
DRAM	Dynamic Random Access Memory
E	Electric Field
FEUP	Faculty of Engineering of University of Porto
f	Frequency
IC	Integrated circuit
IMD	Inter-metal dielectric
LS	Linear shrinkage
LTCC	Low Temperature Cofired Ceramic
P	Polarisation
PVA	Polyvinyl alcohol
RF	Radio frequency
RH	Rice husk
RHA	Rice husk ash
SEM	Scanning Electron Microscopy
SLS	Soda-lime-silica
tan δ	Loss tangent
U.S. EPA	US Environmental Protection Agency
WRHA	White rice husk ash
XRF	X-ray Fluorescence
XRD	X-ray Diffraction
W_d	Dry weight of the sample
W_S	Weight of the suspended sample

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W_M	Saturated weight of the sample
Wt.%	Weight percentage
ρ_a	Density of acetone
d_a	Average pellet diameter after sintering (mm)
d_o	Original pellet diameter (mm)
λ	Lambda, wavelength
θ	Theta, Position of XRD peaks
ρ	Density
ε'	Dielectric constant
ε_o	Permittivity of free space

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Generally, the annual rice paddy production is almost 600 million tons in the world wide (Omatola & Onojah, 2012) as the 20% of the rice paddy is husk. In Malaysia, more than two million tons of RH is produced annually (Matori, Haslinawati, Wahab, Sidek, Ban, & Ghani, 2009). Considerable amount of the husk is burnt as the cheapest and easiest way to banish and decompose it into fertilizer, in the majority of rice producing countries. Many researchers proposed methodologies of treating and recycling RH in order to reduce the pollution. These studies not only provide methods to decrease the amount of potential pollution, but also help to save more landfill area and care for human health. RH is known to have high silica ash content (20-25%) (Real, Alcala, & Criado, 1996; Chouhan, Kujur, Amritphale, & Chandra, 2000; Patel, Karera, & Prasanna, 1987; Liou, 2004; Siqueira, Yoshida, Pardini, & Schiaavon, 2009). Thermal combustion of RH at moderate temperatures in atmosphere, yield an amorphous silica content >90% (Liou, 2004; Chandrasekhar, Satyanarayana, Pramada, Raghavan, & Gupta, 2003; Xiong, Sekiya, Sujaridworakun, Wada, & Saito, 2009; Xiong, Saito, Wada, & Sekiya, 2009; Mishra, Chakraverty, & Banerjee, 1985). Further, it was reported in 1974 that such a high percentage of silica is very unusual within nature and that no other plant waste even approaches the amount of silica found in RH (Beagle, 1974). In fact, RH is a form of waste from the rice milling which is the most economical and abundant source of silica (Kaupp, 1984). Nowadays, efforts are being made to produce low cost silicon and the sources of silica and silicon in biomass resources such as RH for industrial applications (Shinahara & Kohyama, 2004). Extracted silica from RH mainly uses as a reactive SiO_2 and has been extensively applied in ceramic industries (Haslinawati, Matori, Wahab, Sidak, & Zainal, 2009; Khan, Jabbar, Ahmad, Khan, Naeem Khan, & Mirza, 2012). RH is largely used as fuel and in large-scale for electrical power generation (Sun, Zhao, Ling, & Fengming, 2009; Wu, Yin, Ma, Zhou, & Chen, 2009; Oliveira, Neto, Inocencio, Ando Junior, Bretas, & Perrone, 2012; Lin, Wang, Lin, & Juch, 1998). Researches have exhibited that incorporation of RHA produces high strength and durable concrete (Chindaprasirt, Rukzon, & Sirivivatnanon, 2008; Dong, Huu, & Lan, 2008; Satish, Vikrant, & Kavita, 2013). RHA as an amorphous silica source is utilised to supply different silicon compounds such as silicon nitride (Rahman, 1994), silicon carbide (Romero & Reinoso, 1996; Gorthy & Pudukottah, 1999), silica aerogel (Li & Wang, 2008; Dorcheh & abbasi, 2008) and silica gel (Della, Kuhu, & Hotza, 2002). The great field of applications for silica aerogel is thermal insulating (Pajonk, 2003) and electrical insulating (Gurav, Jung, Park, Kang, & Nadargi, 2010). The silica obtained from RH is useful in the semiconductors manufacturing, solar cells for photovoltaic power generation (Amick, 1982; Genieva, Turmanova, Dimitrova, & Vlaev, 2008; Hunt, Dismuked, Amick, Schei, & Larsen, 1984; Sun & Gong, 2001) and other electronic applications. Furthermore, studies have illustrated crystalline RHA can cause silicosis and pneumoconiosis (Liu, Liu, & Li, 1996).

As the most prevalent category of glass, SLS glass (Prado, Fredericci, & Zanotto, 2003) utilised for container wares and windowpanes. Based on U. S. EPA report in 2011, over 41% of soft drink and beer bottles, 34% of wine and liquor bottles, and 15% of food and other glass jars were recovered for recycling. Researchers provide methods on using these

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waste glasses for the commercial applications to decrease the amount of them. Most commercially made glasses are composed primarily of silica (70.9-80 wt.%) (Poutos, Alani, Walden, & Sangha, 2008). In addition to silica, glasses also contain other oxides such as CaO, Na_2O, K_2O and Al_2O_3, which influence their properties (Zanotto, 1991). The use of the recycled glass has become popular in modern times, as aggregate in concrete, with large-scale research being carried out. Findings of the recent research proved that concrete containing recycled glass aggregates tend to show better thermal insulation and better long term strength because of its better thermal properties of the glass aggregates (Poutos et al., 2008). In this work, commercial SLS glass was added to WRHA to take advantage of SLS glass's chemical stability (Zanotto, 1991) to improve the resistance of the pellets to cracking.

This dissertation presents an attempt to prepare crystalline silica-based ceramic from WRHA and SLS glass, waste materials, to be utilised for electronic applications. Generally, ceramic is a kind of inorganic or non-metallic materials prepared by passing through heat treatment and subsequent cooling process according to Non-destructive Testing Education Resources (2001-2012) (NDT Educational Resource Center, 2001-2012). Ceramic materials may have a crystalline or partly crystalline structure, or may be amorphous. Ceramics materials are commonly hard and brittle. In addition, they are typically electrical and thermal insulator (Hayashi, 1984; Nagata, Katsui, Hoshi, Tsuchiya, Toh, Zhao, Shikama, & Hodgson, 2013; Farid, 2013). To this reason, electrical properties of the WRHA-SLS glass ceramic were considered for electronic applications, in this work.

The electrical properties, in this work, consisting of dielectric properties (dielectric constant and dissipation factor) and piezoelectric-electric field (P-E) hysteresis loop evaluation. Dielectric properties exhibit insulating capabilities of a material which could be made to show an electric dipole structure and thus are used within electrical circuits to separate conductive elements. A dielectric material also exhibits the ability of a capacitor to store a charge. Nagata et al. (2013) reported that silica and alumina are popular dielectric materials and will be extensively used as insulator in heating and current-drive systems. A major use of dielectrics is in fabricating capacitors (Ramesh, Shutzberg, Huang, & Gao, 2003). Another major application of dielectric materials is in semiconductor chips to insulate transistors from each other (Sugimura, Imai, Kawasaki, Kamata, Fujii, Fujito, Yonehara, Teramoto, Sugawa, & Ohmi, 2008). Dielectric materials are also utilised for dielectric resonator antenna applications (Huitema & Monediere, 2012).

The dielectric constant of a material is attributed to its capacity to store energy in an electric field, whereas the loss tangent value is also indicative of a material's energy dissipation characteristics (Nelson & Stetson, 1976). Dielectric constant is an important parameter in sophisticated electronic equipments such as amplifiers, semiconductors, transducers and in material processing, electronics and biomedical engineering.

P-E hysteresis loop for a device is a plot (a hysteretic curve) showing the variation of polarisation (P) with the electric field (E) applied to that device at a given frequency. The poling process of applying an electric field to polycrystalline materials is necessary to develop a better understanding of its conductive or dielectric behaviours in order to improve engineered material and device properties.

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In this work, the solid-state reaction between WRHA and SLS glass powders was considered. In order to determine the chemical compositions and phase changes during reaction in the batches, XRF and XRD analysis were performed, respectively. The physical characteristics of the sintered pellets were determined by bulk density, apparent porosity, and LS measurements. Besides, SEM micrographs of the pellets were considered to evaluate the porosity of the surface. The dielectric properties of the sintered pellets were measured using LCR meter and their P-E hysteresis curve was detected by using Sawyer-Tower circuit.

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1.2 Problem Statement

- From a global viewpoint, environmental protection concern has aroused over the recent years. Disposal of bulky RH is a global concern. Open burning leads to environmental pollution. Therefore, if it is not disposed in time, it will become a serious environmental problem (Foo & Hameed, 2009; Pijarn, Jaroenworaluck, Sunsaneeyametha, & Stevens, 2010).

- Glass is generally accepted internationally as urban waste. The waste used glass is usually crashed and re-melted. It can be used as a raw material for the production of other glass-based products. Not all-waste glasses can be recycled into new glasses, and so it is attributed to disposal problem (Steering Committee on Vitrification of Radioactive Wastes, Environment and Resources Commission on Geosciences, Division on Earth and Life Studies, & National Research Council, 1997).

- At the moment, extracting pure silica from natural deposits of quartzite rock or quartz sand results in high production costs that is subsequently reflected in the silica dielectric material’s high market price (Tanner, Yan, & Zhang, 2000; Tomozawa, Kim, & Lou, 2001). Thus, a new study needs to be done by looking at more sustainable technology such as waste materials.

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1.3 Motivation of Research

Motivation of this research work to select WRHA for evaluation of physical and electrical properties has been mentioned bellow:

- conversion of a waste material (RH) into the useful income-generating product
- Producing low cost dielectric material (dielectric silica-based ceramic) due to high silica content of WRHA (because silica is a good dielectric material)

There were some motivations for addition of SLS glass to WRHA, which including:

- conversion of urban waste (SLS glass bottles) into the useful income-generating products
- High silica content

1.4 Objectives

This project was carried out based on vital goal of producing silica-based ceramics from WRHA and SLS glass precursors and evaluating their physical and electrical properties for electronic applications. Hence, the objectives of this study are listed below:

- To synthesize silica-based ceramic from WRHA and SLS glass mixture

- To evaluate the physical and electrical characteristics of the ceramics

1.5 Scope of Study

In order to achieve the objectives of the study, its scopes are based on listed categories. The ceramic-based samples were prepared using conventional powder pressing technique. For the powder pressing technique, a pellet dies in the size of 13 mm has been used because it was the only available dies in the laboratory. Because of time limitation, addition of the SLS glass to the WRHA was done only in three weight- ratio. Each pellet consists of 0.5 g of the mixture powder because the pellet with 0.3 g of the powder was broken frequently. Due to the consolidation of the ceramic powder particles, the pellets were sintered at 900 °C, 1000 °C and 1200 °C. There was not significant physical and chemical properties for WRHA at sintering temperature of 1100 °C, so this sintering temperature was ignored. Sintering temperature must not reach the melting point of a material, so the samples were not sintered at temperature upper than 1200 °C. Any crystallization or densification of the composite at temperature lower than 900 °C is undesirable as this can prevent evaporation of the organics and binder. The chemical constituents of the SLS glass, the WRHA, and mixture ceramic will be analysed using XRF. Then, the physical characteristics, e.g., bulk density and linear shrinkage of WRHA-SLS glass ceramic will be measured using Archimedes' principle in acetone and direct geometrical measurement, respectively. Porosity of the specimens was measured using ASTM C373 and the SEM micrographs of the ceramic samples were considered to support the results of ASTM. Electrical characteristics of the specimens were appraised in this project with measurement of dielectric properties and P-E hysteresis loop. Due to the lack of the facilities in the Universiti Putra Malaysia, the electrical properties of the samples were measured at Suranaree University of Technology in Thailand. Dielectric

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constant and dissipation factor were measured using LCR meter at 1 kHz, 10 kHz, and 100 kHz at room temperature. The test frequency cannot go up to MHz and GHz because the LCR meter in the laboratory has frequency limitation in the range of 12 Hz-200 KHz.

Lastly, P-E hysteresis loop of samples was measured using Sawyer-Tower circuit with applying the electric field (500 V/mm) to specimen at 100 kHz. At the higher electric field, the ceramic samples would break down because the samples have high conductivity (high loss).

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