UNIVERSITI PUTRA MALAYSIA

PHYSICAL, STRUCTURAL, OPTICAL AND ELASTIC PROPERTIES OF ZINC SILICATE GLASS AND GLASS CERAMIC DERIVED FROM RICE HUSK

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By

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

July 2017

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

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July 2017

Chairman : Associate Professor Khamirul Amin Matori, PhD
Faculty : Science

In this study, fabrications of zinc silicate ($ZnO$$SiO_2$) system were fabricated using zinc oxide (ZnO) and white rice husk ash (WRHA). Rice husk was burned to form WRHA and was used as a substitution for silica. Compositions of $(ZnO)_x(WRHA)_{1-x}$ (x = 0.55, 0.60, 0.65 and 0.70 wt.%) was labeled S1, S2, S3 and S4 using the melt-quench method. First of all, the chemical composition of the samples fabricated has been analyzed using Energy Dispersive X-ray Fluorescence (EDXRF) technique. Its physical properties have been measured with density through Archimedes principles and molar volume. Density of the samples increases from 2.94 to 3.66 g cm^{-3} while the molar volume decreases from 23.97 to 20.18 cm^3 mol^{-1} when the amount of ZnO in the samples increases. Next, structural properties of the samples have been analyzed using X-Ray Diffraction (XRD) and Fourier Transform Infrared (FTIR) Spectroscopy. XRD shows that not all samples produced are in amorphous state and only sample S1 is in amorphous while samples S2, S3 and S4 is in crystalline as the amount of ZnO increases. FTIR results shows that non-bridging oxygens (NBOs) are formed as ZnO increase in the samples. Optical properties of the samples have been analyzed using UV-Visible Spectroscopy (UV-Vis). As the ZnO in the $ZnO$$SiO_2$ samples increase, the absorption increases with the increase of crystallization and shifts to higher wavelength. Optical band gap of the samples decreases with the increase in ZnO as NBOs increases. Optical band gap of the samples arises with E_{opt} values for n = 3/2 after comparing the value from the exitation coefficient and differential curve with various number of n. The optical band gap E_{opt} values for n = 3/2 decreases from 4.50 to 4.41 eV as ZnO increases. Ultrasonic velocity of the samples was used to calculate the elastic properties of the samples. Decrease of longitudinal and shear velocity was 3857.03 to 2482.31 ms^{-1} and 1951.95 to 966.27 ms^{-1} respectively. Elastic moduli such as longitudinal modulus, shear modulus, Young modulus, bulk modulus, Poisson's ratio, fractal bond connectivity and Micro-hardness of the samples have been calculated. All the elastic moduli decrease except for Poisson's ratio. Elastic moduli are an important tool to measures sample's resistance to be deformed elastically or

permanently. Elastic moduli such as longitudinal modulus, shear modulus, Young modulus, bulk modulus, Poisson's ratio was compared with the theoretical elastic moduli from Rocherulle's model. Comparison of both of these elastic moduli shows a similar trend in longitudinal modulus, shear modulus, Young modulus and bulk modulus except for the Poisson's ratio.

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

FIZIKAL, STRUKTUR, OPTIK DAN SIFAT KEKENYALAN KACA DAN KACA SERAMIK ZINK SILIKA DARIPADA SEKAM PADI

Oleh

LEE CHEE SUN

Julai 2017

Pengerusi : Profesor Madya Khamirul Amin Matori, PhD
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Dalam kajian ini, fabrikasi sistem zink silikat ($ZnO-SiO_2$) telah menggunakan zink oksida (ZnO) dan abu putih sekam padi (WRHA). Sekam padi dibakar untuk membentuk WRHA dan digunakan sebagai pengganti silika. Komposisi $(ZnO)_x(WRHA)_{1-x}$ (x = 0.55, 0.60, 0.65 dan 0.70 wt.%) melambangkan S1, S2, S3 dan S4 masing-masing telah dengan menggunakan kaedah teknik lebur dan lindap kejut. Pertama sekali, komposisi kimia sampel telah dianalisis menggunakan teknik (EDXRF) manakala sifat-sifat fizikal sudah dianalisis dengan ketumpatan sampel melalui prinsip Archimedes dan isipadu molar. Ketumpatan sampel meningkat daripada 2.94 to 3.66 g cm^{-3} manakala isipadu molar menurun dari 23.97 daripada 20.18 cm^3 mol^{-1} apabila jumlah ZnO dalam sampel meningkat. Seterusnya, sifat-sifat struktur sampel telah dianalisis menggunakan (XRD) dan (FTIR). XRD menunjukkan bahawa tidak semua sampel yang dihasilkan berada dalam keadaan amorfus dan hanya sampel S1 berada dalam keadaan amorfus manakala sampel S2, S3 dan S4 berada keadaan kristal apabila ZnO bertambah. Data FTIR menunjukkan penambahan (NBOs) terbentuk disebabkan oleh penambahan ZnO. Sifat-sifat optikal sampel telah dianalisis menggunakan UV-Vis. Peningkatan ZnO dalam sampel $ZnO-SiO_2$ menyebabkan peningkatan penyerapan disebabkan dengan peningkatan penghabluran dan kemudiannya beralih kepada gelombang yang lebih panjang. Jurang band optik sampel berkurangan dengan peningkatan ZnO apabila NBOs meningkat. Jurang band optik sampel muncul dengan nilai E_{opt} untuk n = 3/2 selepas membandingkan nilai dari pekali pengasingan dan lengkung berbeza dengan pelbagai bilangan n. Nilai jurang band optik E_{opt} untuk n = 3/2 menurun daripada 4.50 hingga 4.41 eV apabila ZnO meningkat. Halaju ultrasonik sampel digunakan untuk mengira sifat elastik sampel. Pengurangan kelajuan memanjang dan ricih ialah dariapda 3857.03 hingga 2482.31 ms^{-1} dan 1951.95 kepada 966.27 ms^{-1} untuk kelajuan masing-masing. Modulus elastik seperti modulus memanjang, modulus ricih, modulus Young, modulus pukal, nisbah Poisson, penyambungan ikatan fraktal dan kekerasan Micro sampel telah dikira. Semua modulus elastik mengalami penurunan kecuali nisbah Poisson. Modul elastik adalah ujian yang penting untuk mengukur rintangan sampel sama ada untuk diubah

secara anjal atau secara kekal. Modulus elastik seperti modulus memanjang, modulus ricih, modulus Young, modulus pukal, nisbah Poisson dibandingkan dengan modulus teori dari model Rocherulle. Perbandingan kedua-dua modulus ini menunjukkan trend yang sama dalam modulus memanjang, modulus ricih, Modulus Young dan modulus pukal kecuali nisbah Poisson.

ACKNOWLEDGEMENTS

First and foremost, I would like to express my deepest appreciation to my supervisor, Associate Professor Dr. Khamirul Amin Matori for his continues guidance and advice regarding my project throughout the year. Not only that, he is a very experience person in my field of study and I thoroughly appreciate his advice, encouragement, support and guidance for me throughout this project.

Apart from that, I would like to thank my co-supervisors Prof. Dr. Sidek Hj. Abdul Aziz and Associate Professor Dr. Halimah Mohamed Kamari for giving me a lot of support, guidelines and guidance during my research in various aspects. Next, I would like to thank my great mentor Dr. Mohd Hafiz Mohd Zaid for his continuous support for my project and he often offers me a lot of guidance and support for me throughout my project. Moreover, he also shares his experience and ideas about his projects and this surely acts as a boost for me to complete this project. Not only have them, staff of the Faculty of Science and Institute of Advanced Technology of Universiti Putra Malaysia also provides me with a lot with guidance when I send them my samples for analysis

In a nutshell, I would also like to thank my family members who support me throughout the experiment and after completing this master project, I have gained a lot of knowledge and experiences on after completing this master's degree.

I certify that a Thesis Examination Committee has met on 20 July 2017 to conduct the final examination of Lee Chee Sun on his thesis entitled "Physical, Structural, Optical and Elastic Properties of Zinc Silicate Glass and Glass Ceramic Derived from Rice Husk" 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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LIST OF ABBREVIATIONS

Al_2O_3	Alumina
CaO	Calcium oxide
CuO	Cupric oxide
Fe_2O_3	Ferric oxide
K_2O	Potassium oxide
MgO	Magnesium oxide
Na_2O	Sodium oxide
SiO_2	Silica
ZnO	Zinc oxide
BOs	bridging oxygens
NBOs	non-bridging oxygens
EDXRF	Energy Dispersive X-ray Fluorescence
ICP	Inductive Couple Plasma
XRD	X-ray Diffraction
FTIR	Fourier Transform Infrared Spectroscopy
UV-Vis	Ultraviolet-Visible Spectroscopy
V_L	longitudinal velocity
V_S	shear velocity

CHAPTER 1

INTRODUCTION

1.1 Research Background

Glass is an amorphous (non-crystalline) solid that has no long range, atomic arrangement, periodic and exhibits time dependent glass transformation behavior which consists mainly of silicon dioxide, (SiO_2). Glass making dates back to 3500 BC in Egypt, where glass serves as jewelry for the royals and the upper class (Zabiegaj et al., 2014). Next, one thing special about glass is that it forms hard, but brittle with no visible crystals formed as it cools down rapidly making it easy to be molded into a shape and stays unchanged. Nowadays, glass is cheap and easy to be manufactured compared to the old days as new technology emerges such as precision glass molding technique which allows the fabrication of high precision optical glass components from glass without grinding and polishing (Prater et al., 2016). Glass plays a major role in our community nowadays as there is more emerging application of glass ever since the Pilkington process is invented and flat glass can be produced when the glass is floated on a layer of tin.

Furthermore, glass is an inert material which makes it ideal for heat and chemical applied techniques even in the kitchen or laboratory. Examples of glass which includes soda-lime silica glass which is normally made up of 60-75% SiO_2, 12-18% soda (Na_2O), and 5-12% lime (CaO) and the major use of it includes bottles, jars, glassware and window glasses (Dervos et al., 2007). Lindbrathen (2005) also states that lead glass is composed of 54-65% SiO_2, 18-38% lead oxide (PbO), 13-15% Na_2O and it is used mainly as radiation shielding glass because lead absorbs gamma rays and other forms of harmful radiation in hazardous conditions such as the nuclear industry.

Moreover, borosilicate glass is mainly composed of 70-80% SiO_2, 7-13% boric oxide (B_2O_3) and smaller amounts of the alkali such as 4-8% of Na_2O and potassium oxide (K_2O), and also 2-7% aluminium oxide (Al_2O_3) which is mainly used in laboratories or the pharmaceutical industry for its great resistance to thermal shock which provides greater accuracy (Stevenson, 2012).

Glass-ceramics in the other hand are polycrystalline materials of fine microstructure that are produced by the control or uncontrolled crystallization of a glass. Control crystallization is usually generated by nucleating additives while uncontrolled crystallization is crystallization which happens because of variation of compositions during the fabrication of glass (Rawlings et al., 2006). There is only few specifics kind of glass which is easy to be crystallized and some glass is very stable and is not easy to crystallize such as ordinary window glass. Glass ceramics generally is not fully crystalline and there is one or more glassy phase which exists in the crystalline phase of glass ceramic and percentage of crystallinity in glass ceramic varies from 0.5% to 99.5% but most of them occur at 30% to 70% (Zanotto, 2010).

Furthermore, the properties of glass ceramics includes high strength, toughness, low thermal expansion, high temperature stability, fluorescence, high chemical inertness and breakdown voltage which are magnificent for kitchen hot plates with relatively low production costs, photolithographic processes since the low thermal expansion coefficient which are compatible and for substrates for telescopes (Rawlings et al., 2006). Next, properties of glass ceramics that is fabricated can be structured and varied according to their degree of crystallinity and microstructure by heat treatment or chemical compositions (Martin et al., 2014). Glass ceramic is generally produced by two methods which involve melting and quenching the glass in glass manufacturing production and heat treatment of glass above its glass transition temperature (Soman et al., 2012; Staff et al., 2016).

Nowadays, utilization of rice husk (RH) to substitute conventional silica has been a major breakthrough as RH is an industrial waste and it's often considered worthless and by using RH to form glass and glass ceramic is an alternative ways to deal with these waste. RH has to be burned up to form white rice husk ash (WRHA) which has high percentage of silica (Azmi et al., 2016). Next, based on two research papers by Lee et al. (2013) and Leenakul et al. (2016) respectively, burning RH for 1000 °C for 2 hours and 4 hours contains 96-99% and 94.96% of SiO_2 respectively. Utilizing RH to substitute silica is a brilliant idea because Malaysia has a vast variety of agricultural production and one of them is rice and this will contribute to a vast amount of RH waste (Hadipramana et al., 2016).

Next, these WRHA can be used as an oil spill absorbent, waterproofing chemicals, flame retardants, and as a carrier for pesticides and insecticides because of its magnificent absorbent and insulating properties (Ahmad et al., 2014). Furthermore, it also can act as an additive for cement and concrete fabrication thermal, insulation and water purification (Kumar et al., 2012; Krishna et al., 2016). Not only that, due to its high silicon content, these WRHA has become a silica source for preparation of elementary silicon and a number of silicon compounds, such as silicon carbide and silicon nitride (Patil et al., 2014; Rahim et al., 2015).

Nowadays there are various types studies which involves the research on glass and glass ceramic and silicate glass and glass ceramic got most of the attention from researchers as its capability as a network former with enormous capabilities. However, there are just a small amount of studies which explains the effect of a glass modifier on silicate glass with RH as silica source and thus in this study, effect of zinc oxide (ZnO) as a glass modifier on silicate glass and glass ceramic from RH will be explored by producing a series of $(ZnO)_x(WRHA)_{1-x}$ glass and glass ceramic. Main objective of this research is to the study the effect of ZnO on the physical, structural, optical and elastic properties of zinc silicate ($ZnO{-}SiO_2$) glass and glass ceramic with RH as silica source.

First of all samples produced were analyzed using the Energy Dispersive X-ray Fluorescence (EDXRF) Spectroscopy to obtain its chemical composition as this study acquires WRHA as substitution for conventional silica. Next, physical used in this study is the density and molar volume analysis while structural analysis acquire in this study consist of X-Ray Diffraction (XRD) and Fourier Transform Infrared (FTIR) Spectroscopy which gives the phase and functional group of the samples. Furthermore optical analysis of this study makes use of Ultraviolet-Visible (UV-Vis) Spectroscopy technique as the absorption band obtained from the samples can be converted to optical band gap thus obtaining band gap energy.

Finally, the most attractive part of this study is to obtain elastic moduli from ultrasonic velocities which is obtained using the Ritec, Ram-5000 Snap System. After obtaining both the wave velocities the longitudinal modulus, shear modulus, Young's modulus, bulk modulus, Poisson's ratio, fractal bond connectivity and micro-hardness of the samples can be deduced and these elastic moduli clearly illustrates the strength of the samples and Rocherulle's model prediction for elastic moduli using chemical compositions has also be used to compare the experimental elastic moduli with the theoretical elastic moduli. Understanding especially the elastic moduli $ZnO–SiO_2$ glass and glass ceramic from RH enables researchers to manipulate its composition to revolutionize the glass and glass ceramic industry.

1.2 Problem Statement

Malaysia is one of the major producers of rice in Asia and this will surely produce a lot of RH as the unwanted waste products (Piramli et al., 2016). RH poses a series of threat to our environment and this has seriously increased the amount of rubbish. In this past 20 years, many efforts have been made to recycle these waste products to a profitable by byproduct such as WRHA (Cui et al., 2016; Saad et al., 2016). Based on the statistics provided by the Food and Agriculture Organization of the United Nations (2016), 2.6 million metric tons of rice is mass produced in Malaysia annually on 2016 and the amount of rice will keep on increasing as newer techniques and technology such as precision farming insect-proof net cultivation (IPN), rice-duck farming (RD) is being implemented in the cultivation process and this clearly implies the RH produced will increase as the years went on (Liu et al., 2017).

Generally, farmers and rice developers often burn the RH openly as wastes, and this release CO_2 into the atmosphere and this is a well-known as the greenhouse gas which endanger our ecosystem (Arai et al., 2015; Marchal et al., 2015). Numerous health related problem arises from direct combustion of RH in open air and this has cause numerous problems to the government as it will create a black fog and increases the temperature of earth (Hadipramana et al., 2016). Hence, by conducting experiments on ways to manipulate and reuse RH, optimization of agricultural waste and create an alternative economical source for the farmers instead of just burning it in the open air which causes severe pollutions.

Furthermore, there are not much research has been done on $ZnO-SiO_2$ glass and glass ceramic from RH and by conducting this study, a new approach can be done on acquiring RH as silica source rather than conventional silica which will definitely safe cost and benefit humanity.

1.3 Objective of the research

This study was carried out based on some clear and precise objectives and the purpose of this research are:

1. To extract high purity silica from waste RH.
2. To determine the effect of zinc oxide on the physical, structural, optical, ultrasonic velocity and elastic moduli of $ZnO-SiO_2$ glass and glass ceramic samples.
3. To compare the experimental elastic moduli with the theoretical elastic moduli which is obtained through Rocherulle's model of the $ZnO-SiO_2$ glass and glass ceramic samples.

1.4 Scope of the study

In order to achieve the objectives of the study, the scopes of the study as follow in this research are:

1. The glass samples of $ZnO-SiO_2$ glass has been produced based on the stiochiometric equation of $(ZnO)_x(WRHA)_{1-x}$ which x = 0.55, 0.60, 0.65 and 0.70 using WRHA and ZnO powder by conventional melting and quenching technique
2. In order to identify the chemical composition of the samples, energy dispersive EDXRF has been used in this study
3. The structure of $(ZnO)_x(WRHA)_{1-x}$ samples will be measured using X-ray diffraction technique to confirm the amorphous structure of the sample
4. The optical properties (the band gap and type of the band gap) of the samples have been measured using UV-Vis technique
5. The ultrasonic wave velocity of the $(ZnO)_x(WRHA)_{1-x}$ samples have been measured using the Ritec, Ram-5000 Snap System which will be used to obtain the elastic properties of the samples

1.5 Important of the study

WRHA is a promising substitution containing high amount of silica and it is relatively cheaper compared to conventional silica. Melting silica from conventional silica to form glass requires high temperature and it is not easy to perform compared to silica from RH which melts at lower temperature.

Malaysia which has vast amount of paddy fields will supply numerous amount of RH waste in its production and most of it ended in landfills or burned in an open air and if nothing is being done upon it, it ended up devastating our daily life. Malaysia has currently 230 landfill sights and 80% of them will reach maximum capacity within the next two years, and with insufficient landfill at disposal, it is going to a major problem for the younger generation in the future.

In order to curb the harmful environmental effect caused by the landfill and open burning disposal solid waste, many researches is working round the clock to discover new techniques to divert these wastes to produce applicable new products or applications in industries.

In this research, ZnO had been choose as an oxide to be added into the WRHA samples to form glass and g $ZnO-SiO_2$ glass ceramic because of its good properties with improvement in its mechanical properties and chemical inertness (Arjmandi et al., 2015; Gupta et al., 2016).

1.6 Thesis Outline

The thesis is structured as follows. Chapter 1 gives the research background, objectives, scopes and also the importance of this study. Next, Chapter 2 comprises of the literature review which study the past studies related to this current study. Chapter 3 in the other hand is reserved for the experimental technique characterization used in this study. Results of this study is reported in chapter 4 which includes the physical, structural, optical and elastic properties of glass and glass ceramic produced from RH and Chapter 5 provides the conclusion and suggestion for future works.

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