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การศึกษาผลของตะกั่วออกไซด์ต่อสมบัติโครงสร้างและสมบัติยืดหยุ่นของแก้วที่รีไซเคิลจากซิลิกาเจลโดยใช้เทคนิคอัลตราโซนิกและเอฟทีไออาร์

A Study of the Effect of Lead Oxide on Structural and Elastic Properties of Recycled Silica Gel Glass by Ultrasonic and FTIR Technique

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บทคัดย่อ

งานวิจัยนี้มีจุดมุ่งหมายเพื่อทำการศึกษาสมบัติทางโครงสร้างและสมบัติยืดหยุ่นของแก้วรีไซเคิลซิลิกาเจล (RSG) โดยใช้เทคนิคการวัดด้วยคลื่นเสียงความถี่สูงอัลตราโซนิก(Ultrasonic technique) และการวัดการดูดกลืนรังสีอินฟาเรด(FTIR technique) แก้วตัวอย่างอยู่ในระบบ 10CaO – xPbO – (90–x)RSG เมื่อ x มีค่าเท่ากับ 20 25 30 35 40 และ 45 เปอร์เซ็นต์โดยโมล ซึ่งเตรียมขึ้นโดยใช้กระบวนการหลอมแล้วทำให้เย็นตัวลงอย่างรวดเร็ว การวัดความหนาแน่นของแก้วตัวอย่างจะวัดโดยใช้หลักการของอาร์คิมิดีส โดยใช้เอ็นเฮกเซนเป็นของเหลวและวัดความเร็วคลื่นเสียงอัลตราโซนิกตามยาวและตามเฉือนของแก้วตัวอย่างโดยใช้วิธีพัลส์เอคโค ความถี่ 4 MHz ที่อุณหภูมิห้อง ผลที่ได้แสดงให้เห็นว่าความเข้มข้นของ PbO ทำให้สมบัติยืดหยุ่นของแก้วตัวอย่างในระบบนี้เปลี่ยนแปลงไป ดังนั้น PbO ทำให้พันธะในโครงข่ายแก้วถูกทำลาย และส่งผลให้เกิด non-bridging oxygen (NBO) และผลที่ได้จากการวิเคราะห์แก้วตัวอย่างโดยใช้การวัดการดูดกลืนรังสีอินฟาเรดช่วยสนับสนุนผลที่ได้จากเทคนิคการวัดความเร็วคลื่นเสียงอัลตราโซนิก

ศัพท์สำคัญ : แก้วเลดซิลิเกต ซิลิกาเจล เอฟทีไออาร์สเปกโทรสโกปี เทคนิคอัลตร้าโซนิก

Abstract

The purpose of this work is to study structural and elastic properties of recycled silica gel glass (RSG) by ultrasonic and FTIR techniques. The 10CaO – xPbO – (90–x) RSG glass systems (where x = 20, 25, 30, 35, 40 and 45 mol%) were prepared by using melt-quenching method. Densities of the glass samples were measured using Archimedes' principle with n-hexane as the immersion liquid. The longitudinal and shear ultrasonic velocities in the samples were measured at room temperature and at 4 MHz frequency using a pulse echo technique. The obtain results showed that changes in elastic properties of the glass samples depended on the concentration of PbO. Therefore the presence of PbO resuled in the destruction of the glass network and subsequent formation of non-briding oxygen (NBO). Moreover, these results can also be supported by FTIR technique.

Keyword: lead silicate glasses, silica gel, FTIR spectroscopy, ultrasonic technique

Introduction

Today, the glass materials are developed for being used as radiation protection. Due to its diverse applications, glass has been used from time immemorial. Glass material is comparatively cheap, relatively easy to produce and synthesize in a wide range of composition [1]. It was observed that many types of glasses can be used as shielding materials which are suitable for each of the radiation sources. Recently, M.H. Kharita and et al. (2012) observed radiation shielding properties of phosphate glasses [2]. R. Laopaiboon and et al. (2011) have used the ultrasonic technique to study the elastic properties of borosilicate glass before and after irradiation [3] and K.J. Singh and et al. (2008) investigated gamma-ray shielding and structural properties of lead silicate glass [4].

More than 15 years ago, silicate glass had a potential application in radiation shielding. The major element of silicate glass shielding ismade from silicon dioxide (SiO_2). The properties of silicate glass can be modified by chemical composition and preparation techniques [2], [4], [5]. Addition of some non-conventional glass former materials such as lead oxide (PbO) into the glass matrix may lead to drastic change in properties such as high density, high nonlinear optical susceptibility, high thermal conductivity and chemical durability [6]. The silica gel is made synthetically from sodium silicate where SiO_2 is the major element [7]. It is used as a desiccant to control humidity to avoid spoilage or degradation of some commodity. When silica gel was used for more time, its performance would be deteriorated and then be discarded as wastes.

Elastic properties are of great interest to investigate the anomalous variation as a function of glass composition. The radioactive wastes are immobilized in the glasses, and they are left in a different environment such as temperature, humidity and pressure. Among these studies [3], [4], [7], [8], the characterization of the glasses through ultrasonic non-destructive testing is an effective way to examine their structural and elastic properties [8]. Therefore, ultrasonic technique is a promising tool to investigate the microstructure change with different composition of glasses.

The main objective of this present work is to investigate the role of PbO content in the calcium–lead–silicate glass systems. The recycled silica gel based glass system is selected in order to seek for the possible use as a new radiation shielding materials. The structural and elastic properties of recycled silica gel glass is studied by ultrasonic technique. More ever, the glass samples were investigated using FTIR spectroscopy to confirm its ultrasonic velocity.

Experimental

1. Silica gel preparation and characterization

The recycled silica gel (RSG) samples for this research were obtained from Department of Physics, Faculty of Science, Ubon Ratchathani University. It was silica gel degradation. The chemical composition of RSG sample was determined by EDS technique as shown in Table 1. It was found that there is high content of Si in RSG sample. Therefore, Si in silica gel is used as represented network former glass system in this study.

Table 1 Chemical composition of RGS by EDS technique

Element	mol%
O	64.42
Na	0.51
Al	0.48
Si	34.60
Total	100.00

2. Glass preparation

Glass samples of compositions 10CaO – xPbO – (90–x)RSG (where x = 20, 25, 30, 35, 40 and 45 mol%) were prepared by the melt-quenching technique. Appropriate amounts of CaO, PbO and RSG were weighted using an electronic balance with an accuracy of 0.0001 g. The homogeneity of the chemical mixture was achieved by repeated grinding in agate mortar. The mixture was melted in ceramic crucibles at around 1250 ^{o}C in an electric furnace under normal atmospheric condition. The molten glass was then poured into warmed stainless steel molds, annealed at around 400^{o}C for 2 h and allowed to cool to room temperature. After that, the two opposite faces of the glass samples were grounded and polished using different grades of silicon carbide paper respectively. Opposite faces were finished optically flat and parallel with a mirror like surface. Chemical composition of the prepared glass samples is provided in Table 2.

3. Density and molar volume

The density of glass samples at room temperature were measured using Archimedes' principle with n-hexane as immersion fluid and applying the relation [9]

$$\rho = \rho_b\left(\frac{W_a}{W_a - W_b}\right) \tag{1}$$

Where ρ_b is the density of buoyant, W_a and W_b are the sample weights in air and the buoyant, respectively.

The molar volume V_g, have been determined as, [10]

$$V_g = \frac{M}{\rho} \tag{2}$$

Where M is the molar weight of the glass

3.1 Ultrasonic velocity measurement and elastic moduli calculation

The ultrasonic velocities were obtained by applying the pulse-echo technique. The elapsed time is measured between the initiate and the receiver of the pulse appearing on the screen of an equipment (Sonatest Sitescan 230). All velocity measurements in this study were carried out at 4 MHz frequency, and at room temperature. The ultrasonic velocity can be calculated as give in the following relation [11], [12].

$$v = \frac{2x}{\Delta t} \tag{3}$$

Where x is the sample thickness (cm) and t is the time interval (s^{-1}). The measurements were repeated three times to check the reproducibility of the data.

Both longitudinal and shear velocities (v_l and v_s) and density (ρ) of the glass samples were used to calculate the elastic moduli as the following the standard relation [3], [13].

Longitudinal modulus: $L = \rho v_l^2$ (4)

Shear modulus: $G = \rho v_s^2$ (5)

Bulk modulus: $K = L - \left(\frac{4}{3}\right)G$ (6)

Young's modulus: $E = 2(1+\sigma)G$ (7)

Poisson's ratio: $\sigma = \frac{L-2G}{2(L-G)}$ (8)

Micro-hardness: $H = \frac{(1-2\sigma)E}{6(1+\sigma)}$ (9)

Table 2 Chemical compositions, densities (ρ) and molar volumes (V_g) of RSG with different PbO contents

Sample	Composition (mol%)			ρ (g/cm^3) ±0.001	V_g (cm^3) ±0.001
	CaO	PbO	RSG		
RSG1	10	20	70	5.3646	19.1252
RSG2	10	25	65	5.4733	20.1012
RSG3	10	30	60	5.5457	21.1770
RSG4	10	35	55	5.5980	22.3046
RSG5	10	40	50	5.6610	23.3671
RSG6	10	45	45	5.7227	24.4223

3.2 FTIR spectroscopy

The FTIR absorption spectra were measured at room temperature in the wavenumber range 400–1900 cm^{-1} using FTIR spectrometer (Spectrum RXI, Perkin-Elmer) with a spectral resolution of 4 cm^{-1}. A KBr pellets technique was used by mixing 2 mg of the fine glass powder with 200 mg of KBr powder in an agate mortar. Then the mixture was subjected to press in an evocable die to produce homogenous transparent disks. After that, the FTIR measurements were carried out immediately.

Results and discussion

The recycled silica gel glasses with different PbO concentration from 20 to 45 mol% has been accomplished. Fig. 1 illustrates variation of density and molar volume as a function of PbO mol%. It can be seen that the densities of glass samples are significantly increasing with PbO content, due to replacement of SiO_2 (molecular weight is 60.0843 g/mol), in silica gel by PbO (molecular weight is 223.1994 g/mol). This behavior is normally observed when increasing the content of a heavy metal oxide in glass system [3], [14], [15]. The density values were used to calculate the molar volume which is defined as the volume occupied by unit mass of the glass samples and the exact values are shown in Table 2. Shelby [16] reported that the molar volume depend on the ionic radius of the modifier. If the ionic radius of the modifier ions (ionic radius of Pb^{2+} is 1.19 Å) is larger than the interstices of the network structure (ionic radius of Si^{4+} is 0.40 Å), their attraction to the oxygen ions can lead to an argument in the size of the interstices and an enhance of the molar volume. The molar volume can be used as a parameter to identify an open structure [17], [18]. It can be seen from Table 2 that a composition of 10CaO-45PbO-45RSG glass corresponds to the maximum open structure.

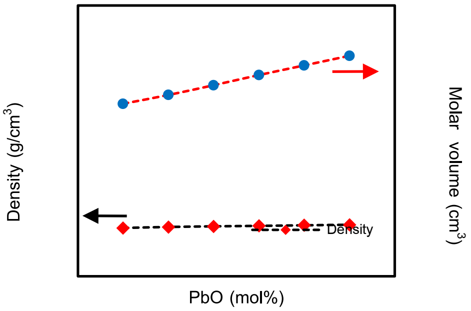

Fig.1. Variation of density and molar volume as a function of PbO mol%

The ultrasonic velocities (v_l and v_s) in glass samples with different mol% of PbO are shown in Fig. 2. Both longitudinal and shear velocities change with increasing PbO concentration. It was mentioned that the change in Geometrical configuration, cross-link density, bridging oxygen an dimensions of interstitial space of glass adjudicate the ultrasonic velocity and therefore, ultrasonic velocity can be used as a tool for indicating the degree of change in structure with composition of glasses [3], [11], [19]. Therefore it can be concluded that the decrease of ultrasonic velocity is implicated to the increase in the number of non-bridging oxygens (NBOs) and the increase of ultrasonic velocity is related to the increase of the bonding of the glass network [9].

In this work, both densities and the ultrasonic velocities value were used to calculate the elastic moduli (longitudinal modulus: L, shear modulus: G, bulk modulus: K and Young's modulus: E) as shown in Table 3.

Table 3 The longitudinal and shear velocities (v_l and v_s), elastic moduli (L, G, K, E) and micro-hardness (H) of RSG with different PbO contents.

Sample	v_l (m/s)	v_s (m/s)	*L* (GPa)	*G* (GPa)	*K (GPa)*	*E (GPa)*	*H(GPa)*
	±8	±7	±0.31	±0.16	±0.45	±0.21	±0.13
RSG1	3653	2405	71.6267	31.0291	30.2546	69.3709	7.9054
RSG2	3555	2292	69.1070	28.7276	30.8035	65.7435	6.8131
RSG3	3498	2213	67.7927	27.2086	31.5145	63.3815	6.0814
RSG4	3532	2275	69.8089	28.9732	31.1779	66.3623	6.8525
RSG5	3506	2228	69.4664	28.1264	31.9646	65.2421	6.3790
RSG6	3532	2294	71.5257	30.1505	31.3251	68.4796	7.324

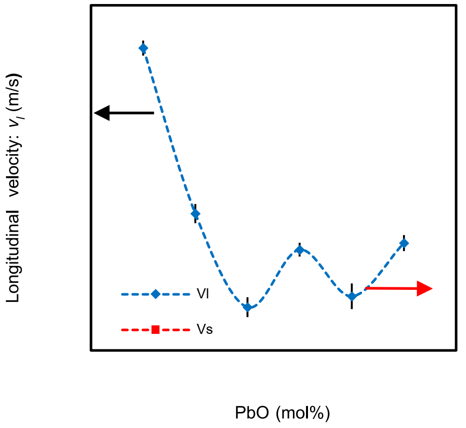

Fig. 2 Variation of ultrasonic velocities (both v_l and v_s) in glass samples with different mol% of PbO

In general, the elastic moduli increase when the density or ultrasonic velocity increase. In Fig. 3 and 4, the continuous increase of PbO content from 20 to 30 mol% results in the decrease of longitudinal, shear and Young's moduli. However, further increase of PbO content (30–45 mol%) lead to the flotation in value of these moduli. Bulk modulus are opposite to the trend of other moduli mentioned above. From the results of the elastic moduli, it can be said that the increase of these moduli is caused by the increase in formation of non-bridging oxygen atoms that cause the formation of compactness structure [20], [21].

Micro-hardness (H) expresses the stress required to eliminate the free volume of the glass. Table 3 shows that an increase in PbO content from 20 to 30 mol% results in a decreased in micro-hardness. On the other hand, the PbO content (30–45 mol%) increase further the value of micro-hardness increases. The change in micro-hardness with an addition of PbO implies a change of rigidity [22], which further confirms the compactness in structural network of the glass samples.

The infrared spectra of CaO–PbO–RSG glass system show some distinct frequency regions (Fig. 5). The mid-region extending from 400 to 1200 cm^{-1} is related to the vibrations of silicate networks [23]. This region consist of very broad bands indicating the overlapping of the bands due to major silicate groups with other bands due to partner lead groups whenever they are vibrating close in wave numbers. The band from 478 to 485 cm^{-1} is assigned to the vibrations of Si–O bonds overlapped by a band attributed to vibrations of Ca–O [24], [26].

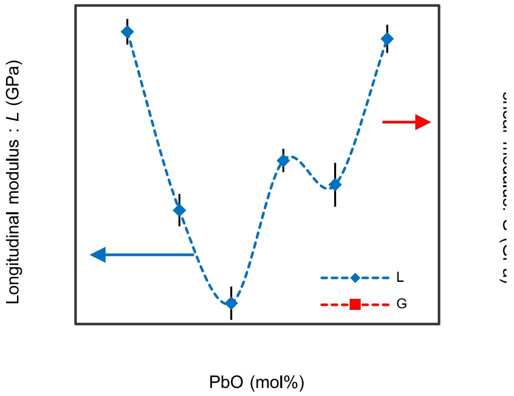

Fig. 3 Variation of elastic moduli (both L and G) in glass samples with different mol% of PbO

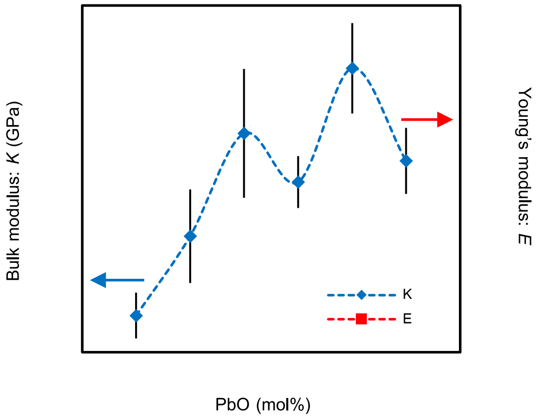

Fig. 4 Variation of elastic moduli (both K and E) in glass samples with different mol% of PbO

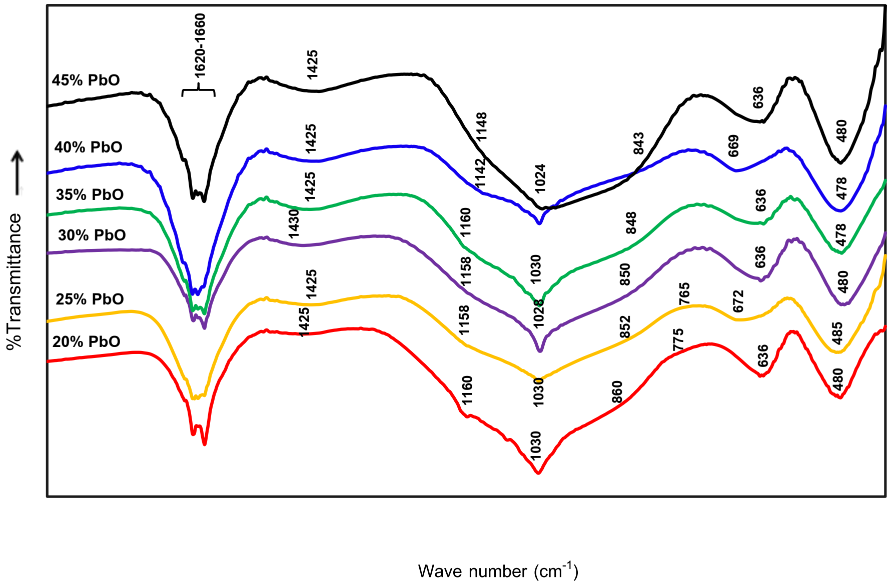

Fig.5 Infrared transmission spectra of calcium-lead-silicate glass systems

The band from 630–680 and 760–780 cm^{-1} is assigned to vibrations modes of SiO_3, SiO_4 and PO_4 groups [6], [27], [28]. As the content of PbO increases, the band becomes narrow and the peak position shifts to 630–680 cm^{-1}. This can be attributed to a change in the bond angles and/or bond lengths of the main structural units. Similarly, the obvious decrease in intensities of the IR bands also implies changes in the bond angles and/or bond lengths as well as number of non-bridging oxygen in the main structural units. Such postulation have been introduced by Primak et al. [29], Hobbs et al. [30] and Piao et al. [31]. The band from 850 to 860 cm^{-1} is assigned to the vibrations of Pb–O bond with non-bridging oxygens [6], [32]. The position and intensity of the main broad prominent band from 1024 to 1030 cm^{-1} is assigned to combined stretching vibrations of Si–O–Si network of tetrahedral structural units consisting of silicate groups overlapped by a band attributed to stretching vibrations of Si–O–Pb and Pb–O bonds in structural units of PbO_3 [6], [32], [33]. Moreover, an increased intensity with increasing PbO content in this region can also be assigned to stretching vibration of SiO_4 and PbO_3 with one or two non-bridging oxygen atoms. The peaks at 1620 to 1660 cm^{-1} relate to water, H-O-H, Si-OH vibrations [23], [32], [34].

FT infrared absorption spectra indicate the presence of silicate groups vibrations which are clearly identified as the major modes due to the presence of SiO_2 in higher percent (45–70 mol.%) than that of its partner PbO (20–45 mol%) and the rest is modifier CaO (10 mol%) [23]. Assignment of the various vibration modes is summarized in Table 4. From infrared spectra in the 1024–1030 cm^{-1} range, it is concluded that increased PbO content affects the structure of the RSG glasses by breaking Si–O and Pb-O bonds within tetrahedral unit. This conclusion from infrared spectra compliments our conclusions from molar volume and ultrasonic velocities measurements.

Table 4 Assignment of absorption bands in the infrared spectra of the glass system

Wave number (cm^{-1})	**Assignment**
1620–1660	Water, H–O–H, Si–OH vibrations.
1425–1430	Ca–O bonds
1024–1030	Si–O–Si of [SiO_4] structural unit, stretching vibrations of Si–O–Pb, Pb–O bonds in [PbO_3] structural units
850–860	Pb–O bonds in [PbO_4] structural units
760–780	stretching vibrations of Si–O–Si Pb–O bonds in [PbO_4] structural units
630–680	bending vibrations of O–Si–O
478–485	bending vibrations of Si–O–Si, Si–O in the framework of the glass, Ca–O bonds, Covalent Pb–O
400–1200	vibrations of silicate networks [SiO_3,SiO_4] vibrations of Pb–O networks [PbO_3,PbO_4]

Conclusions

The density and molar volume of glass system change with increasing the amount of PbO content. This can be attributed to breaking of the glass network or the increase in non-bridging oxygens. Moreover the value of elastic property depends also on the concentration of PbO, since PbO has an effect on rigidity of the glass network structure. Micro-hardness also shows the same trend as the ultrasonic velocities since this value also relates to the rigidity of the glass. The infrared spectra shows that with increasing PbO content, there is the change in the bond angles and/or bond lengths of main structure unit. This is related to the breaking of Si–O and Pb–O bonds within structural unit of SiO_4. Therefore FTIR spectral data confirm the formation of non-bridging oxygens and support the results of ultrasonic velocity. This paper shows that the silica gel can substitute for the pure silica, in the terms of elastic properties and micro-hardness. For future studies, the radiation properties of this glass system should be included for study.

Acknowledgments

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