JSSE JOURNAL OF SCIENCE & SCIENCE EDUCATION
Faculty of Science, Ubon Ratchathani University
Vol. 6 No. 2 (Jul.– Dec. 2023): 171 - 180
http://doi.org/10.14456/jsse.2023.15

Research Article

Influence of manganese ions concentration on structural and elastic properties in recycled window glasses by using ultrasonic technique and FTIR spectroscopy

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Received <11 November 2022>; Revised <31 March 2023>; Accepted <3 April 2023>

Abstract

This study aimed to examine recycled window glasses' structural and elastic properties (RWG). The recycled window glasses were prepared in the 90RWG – $10Na_2O$ – $xMnO_2$ system (x = 0.001, 0.01, 0.1, and 1.0 mol %) by the conventional melt quenching method at 1250 °C. The densities of the glass samples were determined using Archimedes' principle. Elastic moduli, such as longitudinal modulus, shear modulus, Young's modulus, bulk modulus, Poisson's ratio, and microhardness, were calculated using ultrasonic velocity data from the ultrasonic technique. The ultrasonic velocity measured the velocities of the glass samples at a frequency of 4 MHz and room temperature. FTIR spectra were measured in a wave number range of 400 – 2000 cm^{-1} to study the structure of the glass samples by FTIR spectroscopy. The results show that the glass structure was changed and could be improved by increasing the concentration of MnO_2 in the glass system. Adding MnO_2 to the glass network resulted in the formation of SiO_4 bridging oxygen and cross-link density in the glass network. These results lead to changes in the elastic moduli of the glass and an increase in the rigidity of the glass structure. The Si-O-Si linkages of the bridging oxygen were verified by FTIR spectroscopy.

Keywords: Glass, Recycled window glasses, Ultrasonic technique, FTIR spectroscopy

บทความวิจัย

อิทธิพลของแมงกานีสไอออนต่อสมบัติทางโครงสร้างและความยืดหยุ่นของกระจกหน้าต่างรีไซเคิลโดยใช้เทคนิคอัลตร้าโซนิกและอินฟราเรดสเปกโทรสโกปี

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บทคัดย่อ

งานวิจัยนี้มีวัตถุประสงค์เพื่อตรวจสอบสมบัติทางโครงสร้างและสมบัติความยืดหยุ่นของกระจกหน้าต่างรีไซเคิลที่ถูกเตรียมให้อยู่ในระบบ 90RWG – 10Na_2O – xMnO_2 โดยวิธีการหลอมที่อุณหภูมิสูง 1250°C เมื่อ x = 0.001 0.01 0.1 และ 1.0 mol% ความหนาแน่นของตัวอย่างแก้วสามารถหาได้จากหลักการของอาร์คิมิดีส สมบัติความยืดหยุ่น เช่น มอดูลัสตามยาว มอดูลัสเฉือน มอดูลัสของยัง บัลค์มอดูลัส อัตราส่วนปัวซอง และความแข็งระดับไมโคร สามารถคำนวณได้จากความเร็วคลื่นเสียงอัลตร้าโซนิคโดยใช้เทคนิคคลื่นเสียงความถี่สูง การวัดความเร็วคลื่นเสียงของตัวอย่างแก้วถูกวัดที่ความถี่ 4 MHz และที่อุณหภูมิห้อง จากนั้นศึกษาโครงสร้างด้วยเทคนิคอินฟราเรดสเปกโตรสโคปีในช่วงของเลขคลื่น 400 – 2000 cm^{-1} จากผลการศึกษาพบว่าโครงสร้างของระบบแก้วมีการเปลี่ยนแปลงและสามารถปรับปรุงโครงสร้างโดยการเติมความเข้มข้นของแมงกานีสออกไซด์ในระบบแก้ว เนื่องจากการเติมแมงกานีสออกไซด์ในระบบแก้วสร้างการก่อตัวของโครงสร้าง SiO_4 และความหนาแน่นของ cross-link ในโครงข่ายแก้ว ผลลัพธ์นี้นำไปสู่การเปลี่ยนแปลงของสมบัติความยืดหยุ่นและความแข็งแรงทางโครงสร้างเพิ่มขึ้น ซึ่งโครงสร้างของ Si-O-Si สามารถถูกตรวจสอบได้โดยใช้เทคนิค FTIR

คำสำคัญ: แก้ว กระจกรีไซเคิล เทคนิคอัลตร้าโซนิค อินฟราเรดสเปกโตรสโคปี

Introduction

Glass materials, such as window glass for decorations, glassware in the laboratory, and optical lenses, have been used extensively. Glass materials are interesting because they are insulators and are transparent to visible light. In addition, the corrosion resistance to chemicals, high strength, and impurities gives the glass its color, and glass can be blown into an infinite number of shapes. Silicate glass (soda-lime Glass) is one of the most widely used glass types because of its ability to be modified and remelted numerous times. This is one reason that leads to increasing glass waste in the environment. Silicate glass composition is 70-75 wt% silica, 12-16 wt% Na_2O, and 10-15 wt% CaO. This system contains a large amount of silica (SiO_2), which leads to a high melting point of the glass. In terms of glass formation, SiO_2 forms the basis of glass and is called the glass former. However, its high melting point and viscosity make it difficult to melt. Na_2O can modify the form of glass, which is known as a glass modifier. Its effect on decreasing the melting point and viscosity (Srisittipokakun *et al*., 2011). However, the decomposition of glass waste takes time and effort. Reusing and recycling glass waste is an interesting solution to environmental problems (Khazaalah *et al*., 2022). One of the glass waste management methods is melting at a high temperature. In addition, the adding of transition metals oxide (TMOs) into the recycled glass can improve and develop the properties of glass. Moreover, transition metals (TM) are used as coloring agents for glasses. Glass doped with transition metals has attracted much attention because of its interesting optical, electrical, and mechanical properties. Modifier oxide added into the glass network can break the inter-tetrahedral bonds, which is the change in some properties of glass and colors (Abdeldaym *et al*., 2021). In addition, many researchers have reported that metal ions can create several non-bridging oxygens (NBOs) and present a higher mechanical strength. Manganese is a transition metal with a color that improves the microstructure of glass when added to glass systems. Many forms of Mn^{2+}, Mn^{3+}, Mn^{4+}, and MnO^{4-} ions exist. It is well known that manganese oxide transforms into Mn orthomanganate when heated to 1000°C (Gaddam *et al*., 2014). Mn^{2+} acts as a modifying cation and has a coordination number of six in silicate glass. Mn^{4+} forms polyhedral coordinates $[MnO_4]^{4-}$ and may participate in the formation of a glass network together with Si^{4+} (Gaddam *et al*., 2014 and Wahab *et al*., 2021).

In this work, the recycling of window glass waste is interesting and suitable for this work. Commercial window glass was recycled (RWG) using the conventional melt-quench method and was prepared in a 90RWG – $10Na_2O$ – $xMnO_2$ system (where x = 0.001, 0.01, 0.1, and 1.0 mol%). Recycled window glass (RWG) is the base glass system and then added MnO_2 into the glass base for studies of the structure and elastic properties. The structural properties of the glass were studied using ultrasonic techniques and FTIR spectroscopy. Elastic moduli, such as longitudinal modulus, shear modulus, Poisson's ratio, bulk modulus, Young's modulus, and microhardness, were calculated using the ultrasonic velocity data and density data from Archimedes' principle.

Material and Methods

Sample preparation

Commercial window glass waste was recycled (Recycled Window Glass = RWG). There are included a careful cleaning and are crushed into a powder. Crushed glass was prepared in 90RWG – $10Na_2O$ – $xMnO_2$ (where x = 0.001, 0.01, 0.1 and 1.0 mol%) and mixed until homogeneous. Homogeneous mixtures were melted in an electric furnace at 1250°C for 5 h. The melted glass was poured into preheated stainless-steel molds and annealed at 500°C for 2 h. The samples were then naturally cooled to room temperature. Glass samples were cut and polished using different silicon carbide grades and aluminum powder for ultrasonic velocity measurements. Chemical composition analyses of the glass system were carried out using WDXRF. Commercial window glass composition is 73.390% of silica (SiO_2), Na_2O = 15.170%, CaO = 7.339%, MgO = 3.408%, Al_2O_3 = 0.586%, Fe_2O_3 = 0.063%, TiO_2 = 0.026%, and K_2O = 0.023% (Sopapan *et al*., 2019).

Density measurements

Density of all glass samples was determined employing Archimedes' principle using n-hexane and applying the relation (Gaafar, 2007).

$$\rho = \rho_l \left(\frac{W_a}{W_a - W_b}\right) \quad (1)$$

where ρ_l is density of immersion liquid, W_a and W_b are the glass sample weights in air and weights in the immersion liquid, respectively. The molar volume (V_m) of the glass samples are given by

$$V_m = \frac{M}{\rho} \quad (2)$$

where ρ is the density of the glass and M is the molecular weight of the glass system (Singh *et al*., 2008).

Ultrasonic velocity measurements

The ultrasonic velocities of the glass samples were measured using a pulse-echo technique. The ultrasonic wave was generated from a ceramic transducer with a resonant frequency of 4 MHz and simultaneously acted as a transmitter-receiver. This study used an ultrasonic flaw detector, SONATEST Sitescan 230, for ultrasonic velocity measurement. The velocity measurements were repeated three times to check the velocity data. The elastic moduli were calculated using the following standard equation (Afifi *et al*., 2003).

Longitudinal modulus (L):

$$L = \rho V_l^2 \quad (3)$$

Shear modulus (G):

$$G = \rho V_S^2 \quad (4)$$

Bulk modulus (K):

$$K = L - \frac{4}{3}G \quad (5)$$

Young's modulus (E):

$$\mathrm{E} = (1 + \sigma)2\mathrm{G} \quad (6)$$

Poisson's ratio (σ):

$$\sigma = \frac{L-2G}{2(L-G)} \quad (7)$$

Micro-hardness (H):

$$H = \frac{(1-2\sigma)E}{6(1+\sigma)} \quad (8)$$

Where v_l is longitudinal velocity and v_s is shear velocity of the glass samples.

Infrared absorption measurements

The glass samples were measured at room temperature in the wavenumber range 400–2000 cm^{-1} by a Fourier transform infrared spectrometer. The powdered glass samples were mixed with KBr at a ratio of 1:100 mg of glass powder: KBr.

Results and discussion

Density and Molar volume

Density is an essential parameter for studying the physical properties of a material. The density and molar volume of all glass samples are shown in Figure 1. It was observed that the density increased from 2.552 g/cm3 to 2.581 g/cm^3 as the concentration of MnO_2 in the glass system increased. The increase in the density of the glass is due to the higher molecular weight of MnO_2 in the glass network. The molecular weight of MnO_2 is 86.93 g/mol, and 60.09 g/mol of silica. Thus, adding MnO_2 to the glass system increased the density of the glass samples. Basically, the molar volume represents the volume occupied by one mole of material and it depends on the ionic radius of the modifier. Shelby reported that if the ionic radius of the modifier ions is smaller than the interstices of the glass network their attraction to the oxygen ions can lead to a decrease in consequently decreases the molar volume. The Mn^{2+} ions in the glass network may be inserted to replace Na^{+} ions, decreasing the molar volume at 0.001 mol% MnO_2. This led to shrinkage of the glass structure. The ionic radius of Mn^{2+} ions (0.83 Å) is smaller than that of Na^{+} ions (1.02 Å) (Laopaiboon *et al*., 2014). Then, the increase in molar volume at 0.01 to 1.0 mol% MnO_2 due to Mn^{2+} ions increase in the glass network. Due to the ionic radius of Mn^{2+} is larger than Si^{4+} (the ionic radius of Si^{4+} is 0.400 Å) which is an increase in the bond length or inter-atomic spacing between the atoms, whereby the glass network will expand. This result indicates a decrease in the compactness of the glass by increasing the number of non-bridging oxygens and expands the glass structure. Furthermore, the size of manganese ions can depend on their oxidation state. The ionic radius of manganese (II) ion (Mn^{2+}) is about 0.83 Å, manganese (III) ion (Mn^{3+}) is about 0.67 Å, manganese (IV) ion (Mn^{4+}) is about 0.53 Å. FTIR spectra can check the creation of NBOs at approximately 960 cm^{-1} and as shown in Figure 7.

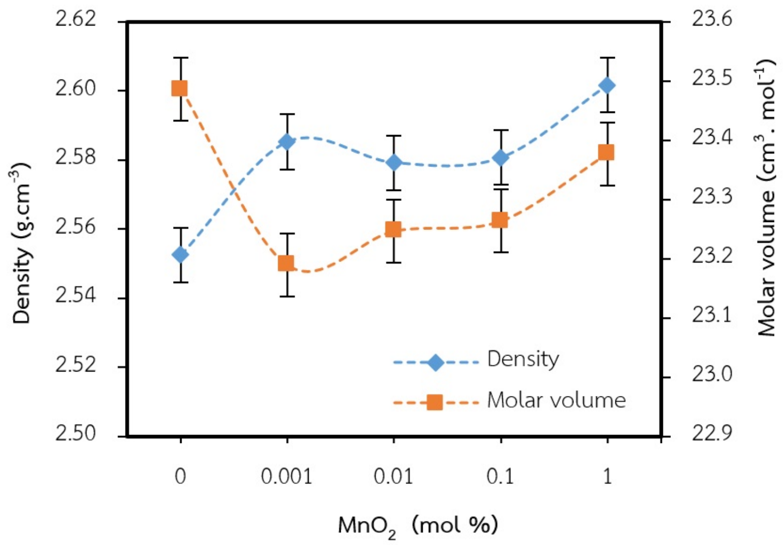

Figure 1. Variation of density and molar volume with the concentration of MnO_2

Ultrasonic velocity and elastic moduli

The ultrasonic velocity, such as the longitudinal and shear velocities, was measured using the pulse-echo technique at a frequency of 4 MHz and room temperature. Ultrasonic velocity measurement is one of the techniques used to study the glass structure. The longitudinal and shear velocities are shown in Figure 2. The longitudinal velocities increased from 5822 m/s to 5891 m/s, and the shear velocities increased from 3402 m/s to 3588 m/s. Adding MnO_2 can create bridging oxygen (BOs) in the glass network. This leads to higher connectivity of the glass network bonds and an increase in the ultrasonic velocities of the glass (Marzouk, 2009). The longitudinal and shear moduli are shown in Figure 3. The elastic moduli, such as longitudinal modulus, shear modulus, bulk modulus, Young's modulus, Poisson's ratio, and microhardness, were calculated using the following equation (3-8). It is well known that the longitudinal and shear moduli directly correlate with glass density. The increase in density leads to an increase in the longitudinal and shear moduli (Laopaiboon *et al*., 2016). From Figure 3., the value of longitudinal modulus increases from 86.5 to 90.2 GPa and increases from 29.5 to 33.5 GPa of the shear modulus.

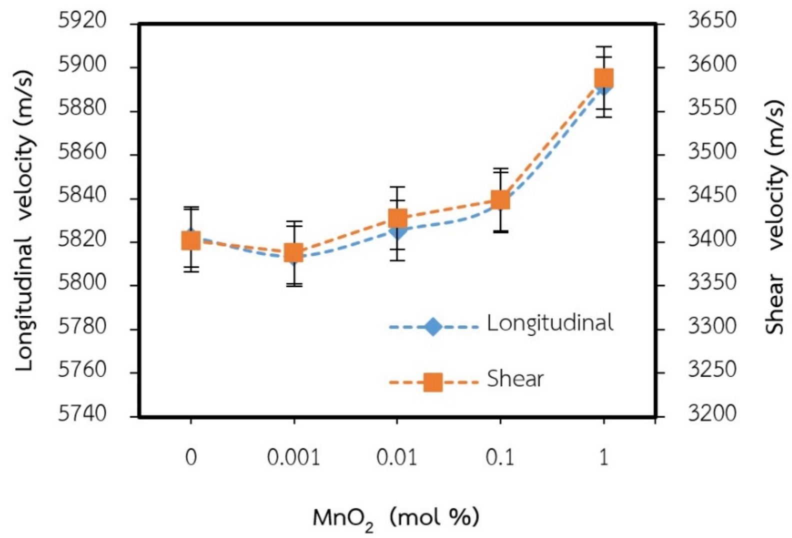

Figure 2. Variation of longitudinal and shear velocities with the concentration of MnO_2

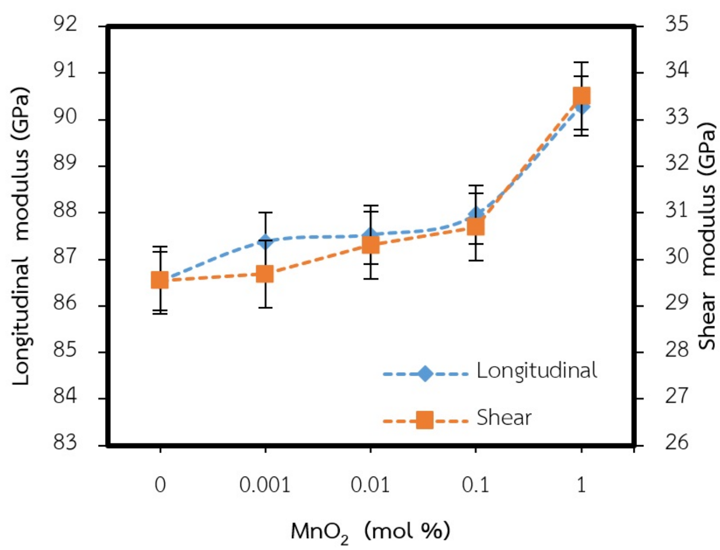

Figure 3. Variation of longitudinal and shear modulus with the concentration of MnO_2

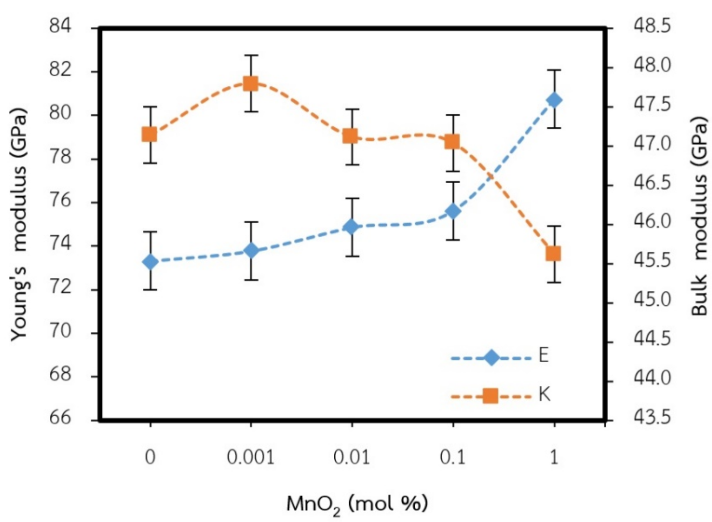

Figure 4. Variation of Young's modulus and Bulk modulus with the concentration of MnO_2

The values of Young's modulus and bulk modulus, as shown in Figure 4. Young's modulus increased with an increase in the concentration of MnO_2 in the glass system. The value of Young's modulus increased from 73.3 to 80.7 GPa with an increase in MnO_2 mol% content. The high value of Young's modulus is related to the increase in the rigidity of the glass structure and is associated with the change in the cross-linking of the glass network. The elastic moduli are related to the average strength of the bond and depend on the value of the cation-anion forces (Singh, 2014). However, adding MnO_2 to the glass system decreased the bulk modulus from 47.1 to 45.6 GPa. Poisson's ratio is shown in Figure 5. It can be observed in Figure 5. that the Poisson's ratio decreases from 0.24 to 0.20 with the increase in MnO_2 content. Poisson's ratio reveals a cross-link density in the glass structure. The range of Poisson's ratio 0.1 to 0.2 presents a high cross-link density, while 0.3 to 0.5 is a low cross-link density. In this study, the value of Poisson's ratio remained in the range of a high cross-link density. Adding Mn^{2+} ions to the glass system can create bridging oxygens (BOs) in the glass network. This leads to the formation of bridging oxygen of Mn-O-Si bonds, and the results reveal increasing rigidity of the glass structure (Saddeek *et al*., 2004). In addition, the decrease in Poisson's ratio revealed covalent bonds in the glass structure and a correlation between the number of bridging bonds per cation in the glass system (Zaid, 2011).

The microhardness value, as shown in Figure 6. The micro-hardness of glass samples increases from 5.1 GPa to 6.5 GPa. These results show that the rigidity of the glass sample increased with the addition of MnO_2 to the glass system. Mn^{2+} ions create cross-link density in the glass network.

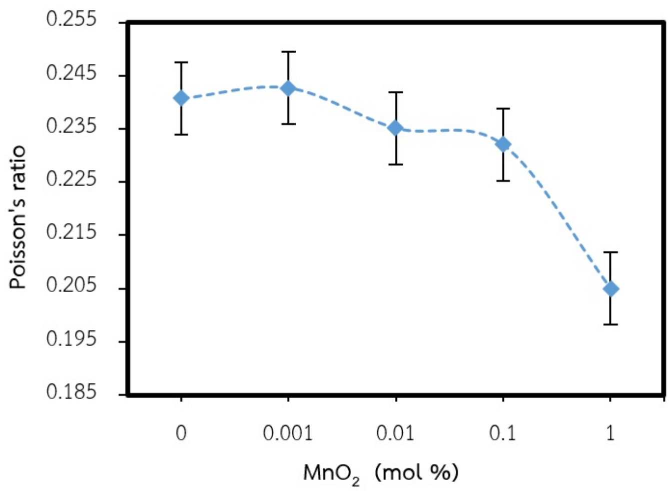

Figure 5. Variation of Poisson's ratio with the concentration of MnO_2

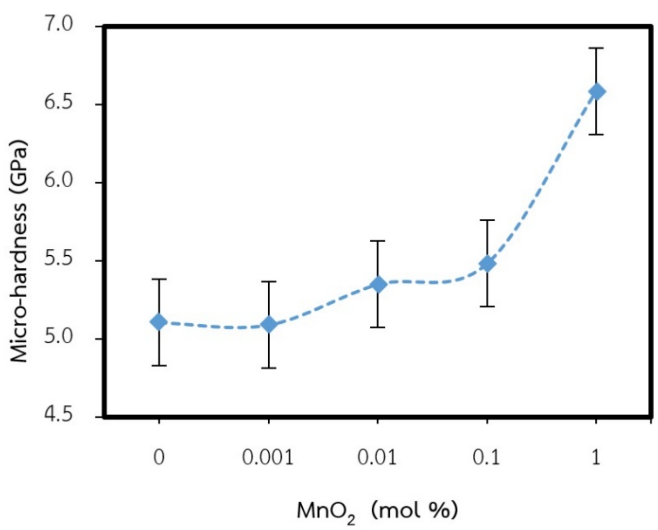

Figure 6. Variation of micro-hardness with the concentration of MnO_2

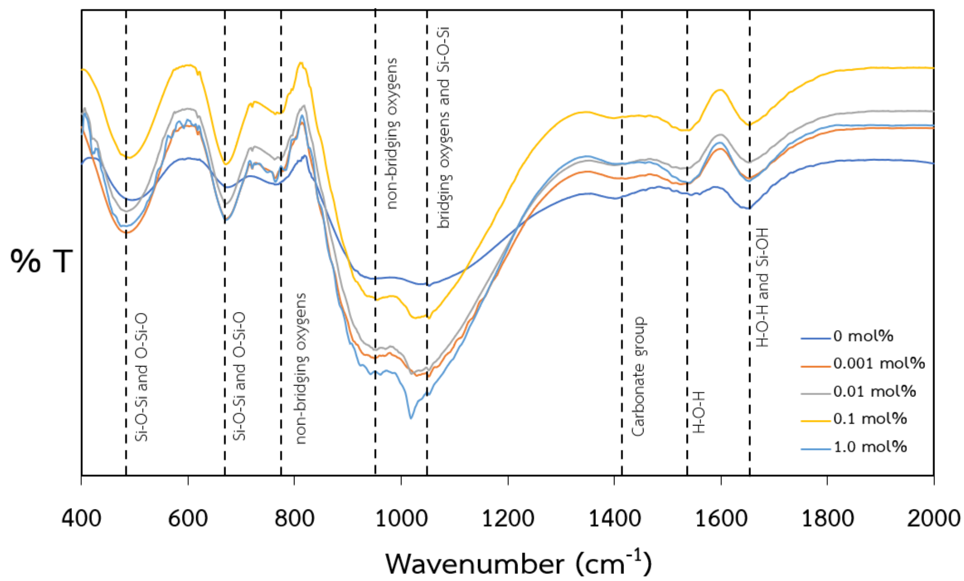

Figure 7. The FTIR spectra of all glass samples

FTIR spectroscopy

The glass samples in this work were measured at room temperature in the wavenumber range of 400 - 2000 cm^{-1}. The FTIR spectra of the glass samples are shown in Figure 7, and the infrared absorption peaks are presented in Table 1. It can be seen that the first region of the IR band in the frequency around 470 cm^{-1} is due to the Si-O-Si bending modes of bridging oxygen. The second peak at approximately 640-680 cm^{-1} corresponds to the vibration of the Si-O-Si and O-Si-O bending modes. The weak IR band located at approximately 775-800 cm^{-1} is assigned to the vibrations of the O-Si-O bonds. The strong peak of the IR spectra at approximately 960 cm^{-1} is the vibrations of nonbridging oxygen (NBO). The absorption at 960 cm^{-1} of the glass sample non-doped with MnO_2 into the glass system is a strong peak. The glass network builds Si-O-Si of nonbridging oxygen (SiO_4) in the network. In addition, MnO_2 doped glass sample (1.0 mol%) had a more substantial peak than the glass sample (non-doped MnO_2). These results show that Mn^{2+} ions can create nonbridging oxygen in the glass network, leading to an open structure of the glass networks. The peak at approximately 1050-1120 cm^{-1} was assigned to the Si–O–Si antisymmetric stretching of Si-O-Si of bridging oxygen (SiO_4). It can be seen that bridging oxygen can be created and increased when adding the MnO_2 into the glass system, owing to the structure of the nonbridging oxygen of SiO_4 and SiO_2, the bridging oxygen of SiO_4 (Abdeldaym, 2021 and ElBatal, 2009). This leads to an increase in the ultrasonic velocity of the glass and elastic moduli. The bands in the region of ~1600 cm^{-1} were assigned to molecular water- or hydroxyl-related bands.

Table1. Depicts the detailed assignments of IR bands in sodium silicate glasses (Abdelghany *et al*., 2014).

Peak position (cm^{-1})	Assignment
460-480	Bending vibration of Si-O-Si linkages
640-680	Si-O-Si and O-Si-O bending
775-800	Symmetric stretching vibrations of O-Si-O bonds
960	Vibrations of non-bridging oxygen (NBO)
1050–1120	Anti-symmetric stretching of Si-O-Si linkages
1400–1460	Carbonate group
1630–1645	Molecular water

Conclusion

Commercial window glass was recycled and prepared in 90RWG – $10Na_2O$ – $xMnO_2$ system with x = 0.001, 0.01, 0.1, and 1.0 mol% by melt-quenching technique. The density of the glass increased with increasing MnO_2 concentration. The lower molecular weight was substituted with a higher molecular weight (MnO_2). The ultrasonic velocity measurement results showed that the longitudinal and shear velocities increased with the concentration of MnO_2. The increase in the ultrasonic velocity might be due to the Mn^{2+} ions doped in the glass system, which leads to the creation of bridging oxygen atoms in the glass network. The elastic moduli were calculated and improved with increasing MnO_2 content in the glass system. Poisson's ratio was decreased, indicating an increase in cross-link density and rigidity. The microhardness increased with the concentration of MnO_2 doped in the glass system. This is due to the addition of Mn^{2+} ions, which are involved in the glass network by creating ionic bonds between the Mn ions and singly bonded oxygen atoms. FTIR results revealed that increasing the content of MnO_2 can create the formation of bridging oxygen (BOs). The FTIR data corroborated and confirmed the ultrasonic velocity and elastic moduli data.

Acknowledgement

The author wants to express their deep gratitude to Assoc. Prof. Jintana Laopaiboon and Assoc. Prof. Raewat Laopaiboon, Glass Technology Excellent Center (GTEC), Department of Physics, Faculty of Science, Ubon Ratchathani University for their great support in providing all facilities necessary during this work.

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