บทความวิจัย

การสร้างแก้วสีเขียวมรกตจากทรายทะเลจังหวัดกระบี่

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บทคัดย่อ

งานวิจัยนี้เป็นความร่วมมือระหว่างเทศบาลนครกระบี่ และศูนย์ความเชี่ยวชาญเทคโนโลยีแก้ว คณะวิทยาศาสตร์ มหาวิทยาลัยอุบลราชธานี เพื่อศึกษาความเป็นไปได้ในการใช้ทรายทะเล จากเกาะปอดะ จังหวัดกระบี่ เพื่อผลิตแก้วคล้ายอัญมณี โดยเริ่มต้นจากการวิเคราะห์องค์ประกอบของทรายทะเลกระบี่ด้วยเทคนิคการเรืองรังสีเอกซ์ ผลการศึกษาแสดงให้เห็นว่าทรายทะเลนี้มีองค์ประกอบหลักเป็นซิลิกา และมีอลูมิเนียมออกไซต์เป็นสารเจือปนอยู่เล็กน้อย ทรายทะเลที่มีสารเจือปนอยู่เล็กน้อยนี้สามารถนำมาสร้างแก้วสีเขียวมรกตในระบบ (52-x)KS-17Na_2O-4Al_2O_3-6CaO-7K_2O-xCr_2O_3 เมื่อ KS คือ ทรายกระบี่ และ x เท่ากับ 0.00 0.10 0.30 0.50 และ 0.70 เปอร์เซนต์โดยน้ำหนัก ซึ่ง Cr_2O_3 ถูกเติมเข้าไปเพื่อทำให้แก้วเป็นสีเขียว และได้ทำการศึกษาสมบัติต่าง ๆ ของแก้ว ผลการศึกษาพบว่าเมื่อมีการเติม Cr_2O_3 ตัวอย่างแก้วที่ได้เปลี่ยนจากแก้วใสไปเป็นสีเขียวคล้ายกับอัญมณีมรกต และมีสีเขียวเข้มขึ้นเรื่อย ๆ เมื่อปริมาณของ Cr_2O_3 เพิ่มขึ้น นอกจากนี้ค่าความหนาแน่นและปริมาตรโดยโมลของตัวอย่างแก้วที่ได้ก็เปลี่ยนแปลงตามปริมาณของ Cr_2O_3 ด้วย ดัชนีหักเหของแก้วตัวอย่างมีค่าน้อยกว่าของอัญมณีมรกตในธรรมชาติประมาณ 9-20% ขึ้นกับปริมาณของ Cr_2O_3 และเมื่อศึกษาสเปคตรัมการดูดกลืนแสงของแก้วพบว่าแถบช่องว่างพลังงานมีค่าลดลงจาก 2.66 eV ไปยัง 2.31 eV ตามปริมาณของ Cr_2O_3 ที่เพิ่มขึ้น ซึ่งแสดงว่าตัวอย่างแก้วที่ได้ทั้งหมดมีความเป็นฉนวน และเมื่อศึกษาตัวอย่างแก้วโดยใช้เทคนิคอัลตร้าโซนิก พบว่าเมื่อเพิ่มปริมาณของ Cr_2O_3 โมดูลัสตามยาวของตัวอย่างแก้วจะลดลง และการเปลี่ยนแปลงปริมาณของ Cr_2O_3 ยังส่งผลให้ความแข็งวิกเกอร์ของแก้วเปลี่ยนแปลงด้วย สมบัติต่าง ๆ ของแก้วที่เปลี่ยนไปนี้สามารถอธิบายได้จากการแตกของโครงข่ายแก้วเนื่องจากการแทนที่ของโครเมียมไอออนที่มีขนาดใหญ่กว่าและหนักกว่าที่ตำแหน่งของซิลิกอนไอออนที่เป็นโครงข่ายหลัก ผลการวิจัยนี้แสดงให้เห็นว่า ทรายทะเลจากจังหวัดกระบี่ สามารถนำมาทำเป็นแก้วสีเขียวที่มีสมบัติคล้ายอัญมณีมรกตในธรรมชาติและสามารถนำไปขึ้นรูปต่อเป็นลูกปัดแก้วได้

คำสำคัญ: แก้วสีเขียวมรกต ลูกปัดแก้วโบราณ โมดูลัสยืดหยุ่น สมบัติทางแสง

อ้างอิงบทความนี้

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Research Article

Fabrication of emerald green glass from Krabi sand

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Abstract

The possibility to use sea sand in Koh Poda, Krabi Province as raw material for fabricating synthetic glass jewelry was conducted in cooperation between municipal administration of Krabi and Glass Technology Excellent Center (GTEC), Faculty of Science, Ubon Ratchthani university. Firstly, composition analysis by EDS technique showed that the main constituent of Krabi sand was silica with the existence of low alumina impurity. This low impurity sand was then used to fabricate green emerald glass in system of (52-x)KS-17Na_2O-4Al_2O_3-6CaO-7K_2O-xCr_2O_3, where KS stands for Krabi sand and x = 0.00, 0.10, 0.30, 0.50 and 0.70 wt%. The Cr_2O_3 was added as a green-coloring agent and its effects on glass properties were evaluated. Many properties of the glass samples were then studied. The glass samples showed that an addition of Cr_2O_3 turned white clear glass to emerald green color and with increasing Cr_2O_3 content, the green color became darker. The density and molar volume of the glass sample were found to varied with Cr_2O_3 content. The reflection index of the glass sample was found to be 9-20% lower than that of natural emerald jewelry. Absorption spectra of the glass samples was used to calculate their band gaps and it was found to reduce from 2.66 to 2.31 eV with increasing Cr_2O_3 content. By means of these calculated band gap, the glass samples were insulator. By using ultrasonic technique, a decrease of longitudinal modulus with increasing Cr_2O_3 content was observed. On the other hand, their Vicker hardness varied with Cr_2O_3 content. The changes of glass properties could be explained by a breakdown of the network structure due to a substitution of larger and heavier chromium ions for silicon-network forming ions. The results report here show that Krabi sand can be used as raw material for fabricating green glass that look like natural emerald jewelry. Moreover, this glass system can be a potential material for making glass beads.

Keywords: Emerald green glass, ancient glass beads, elastic moduli, optical properties

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Introduction

Glass is one type of ceramic materials that exhibits many outstanding properties such as transparency, transmission of light and water proofing. Therefore, it has been widely used in many applications. In former times, ancient human used the natural glass to make hunting weapons. Later, glass had become widespread in household and decorative applications as many ancient glass beads were found around the world. This indicates that human had acquired knowledge on glass making since ancient time. In Thailand, a historical evidence of ancient glass melting furnace and many antique beads were found in Amphoe Klong Tom, Krabi Province in southern part of Thailand. Therefore, municipal administration of Krabi had an idea to duplicate ancient beads as a means of preserving them. Glass technology excellent center (GTEC) at Physics department, Faculty of sciences, Ubon Ratchatani University, established in 2006, made a research on the use of local river sand in Ubonratchatani Province such as Mun River sand, Mekong River sand and Chi River sand in manufacturing of colored glass. GTEC also has studied on ancient Andaman glass-making process by a grant from national research council of Thailand. Currently, GTEC has passed on this knowledge to municipal administration of Krabi. With cooperation between GTEC and municipal administration of Krabi, our research studied the properties of emerald green glass made from sea sand in Krabi Province. The glass in system (52-x)KS-17Na_2O-4Al_2O_3-6CaO-7K_2O-XCr_2O_3, where KS stands for Krabi sand and x = 0.00, 0.10, 0.30, 0.50 and 0.70 wt% were prepared by melted quenching technique. In order to obtain emerald green color, Cr_2O_3 were also added as coloring-agent. Effect of Cr_2O_3 content on many properties of emerald green glass was evaluated such as coloring, density, refractive index, elastic modulus, and hardness. This emerald green glass from Krabi sand was then used in fabrication of antiques glass beads.

Objective

In this research various tests were carried out to consider the effect of Cr_2O_3 content on properties of emerald green glass made from Krabi sand, which is sea sand from Koh Poda, Khao Phanom district, Krabi Province in southern part of Thailand. Eventually, fabricated glasses from this research was used for making ancient glass beads.

Materials and Methods

(1) Elemental analysis of the sea sand

National Metal and Materials Technology Center (MTEC) of Thailand conducted an elemental analysis of Krabi sand using Energy-dispersive X-ray fluorescence technique. Prior to this analysis, the sand was washed and dried in oven at 100°C for 4h.

(2) Preparation of Glass sample

Glass samples in system of (52-x)KS-17Na_2O-4Al_2O_3-6CaO-7K_2O-XCr_2O_3, where KS stands for Krabi sand and x = 0.00, 0.10, 0.30, 0.50 and 0.70 wt% were prepared by melted quenching technique. Good purity grade Na_2O, Al_2O_3, CaO and K_2O were added to lower the melting point and viscosity of the glass, as well as to modify the glass properties. Green color of the glass was achieved by adding Cr_2O_3. All chemicals were mixed thoroughly and melted in alumina crucible with electric furnace at around 1250 °C for 4 h. The melt was poured into warmed stainless-steel molds and annealed in another furnace at around 550 °C for 2 h. It was naturally cooled to room temperature to remove its thermal stress. After that, properties of prepared glass samples according to amount of added Cr_2O_3 were investigated.

(3) Density and Molar volume

Density (ρ) of glass samples was obtained using Archimedes' principle and calculated by using the following relation (Gaafar and Marzonk, 2007):

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

where ρ_l is the density of n-hexane. W_a and W_b are weight of glass samples, using an electronic balance with sensitivity of 0.0001 g, in air and n-hexane, respectively. The experiments were repeated 3 times for more accurate value. The estimated errors in these measuring were approximately 0.0001 g/cm^3.

Molar volume (V_a) of glass, which can be defined as the volume occupied by the unit mass of the glass, was obtained by following equation (Laopaiboon et al., 2011):

$$V_a = \frac{M_w}{\rho} \quad (2)$$

where M_w is the molar mass of the glass samples.

(4) Refractive index

The refractive index of glass samples with different content of Cr_2O_3 has been measured using refractometer (Abbe Refractometer, ATAGO) with an accuracy of ±0.0002.

(5) Ultrasonic velocities and longitudinal modulus

Measurement of longitudinal ultrasonic velocity in the prepared glass samples was conducted by using an ultrasonic flaw detector, (SONATEST Sitescan 230). The ultrasonic waves were originated from a ceramic transducer (Probe model: SLG4-10 for longitudinal velocity) with a resonant frequency of 4 MHz. This transducer can be used as a transmitter and a receiver at the same time. This technique was calibrated with calibration block V1 and Ultragel II (MAGNAFLUX) as couplant. The longitudinal ultrasonic velocity (v_L) was estimated by the following equation (El-Mallawany El-Khoshkhany and Afifi, 2006; Marzouk and Gaafar, 2007):

$$v_L = \frac{2x}{\Delta t} \quad (3)$$

where Δt is the time interval (s), x is the samples thickness (cm). The measurements were repeated 3 times for more accuracy. Then, the longitudinal modulus (L) was estimated using the following equations (Afifi and Marzonk, 2003):

$$L = {v_L}^2\rho \quad (4)$$

(6) Optical Absorption measurement

Optical absorption measurements are widely used to characterize the electronic properties of materials, through the determination of parameters describing the electronic transitions such as band gap. The optical absorption measurement of glass samples was carried out on Perkin Elmer lambda-25 UV/Vis spectrometer system, in the wavelength range 200 nm to 800 nm. As the optical absorption relates to the optical transition of the electrons from valence band to conduction band, band gap of the prepared glass samples was also evaluated using this UV-Vis study. The band gap values for glass samples were estimated based on the following equation:

$$(\alpha h v)^m = C\left(hv - E_g\right) \quad (5)$$

where α is the optical absorption coefficient, E_g is optical band gap of the glass samples and C is a constant. The parameter m depends on the characteristics of the band gap as $m = 2$ for direct band gap and $m = 1/2$ for indirect band gap. From the equation (5), the direct and indirect band gap of the glass samples can be obtained by the plots of variation of $(\alpha h v)^2$ versus photon energy (hv) and $(\alpha h v)^{1/2}$ versus photon energy (hv), respectively. Band gap could be evaluated using the extrapolation of the linear section of the plot. The intercept on hv-axis, where $(\alpha h v)^2 = 0$ or $(\alpha h v)^{1/2} = 0$, gives the value of band gap energy. In order to determine category of optical band gap (direct or indirect) of our glass samples, R-squared value of the linear extrapolation must be considered (Srinivasa Reddy Murali Krishna Veeraiah, 2006; Novatski et al., 2008, Chauhan, 2015; Govindasamy Murungasen and Sagadevan, 2017;)

(7) Vicker micro-hardness

For Vickers microhardness measurements, indentations were made on the glass using indentation loads of 9.8 N. Then Indentation diagonal lengths (d_1,d_2) in meter were measured for each impressions. Vickers micro-hardness was calculated using the following equation:

$$HV = \frac{F}{Area} = \frac{2Fsin\left(\frac{\theta}{2}\right)}{d^2} = \frac{1.854F}{d^2} \quad (6)$$

where HV is the Vickers microhardness in Pascal, F is the applied load in Newton, and $d = \frac{d_1+d_2}{2}$.

Results and Discussion

(1) Elemental Analysis of Krabi sand

The Krabi sand was collected from Koh Poda, Krabi Province in southern part of Thailand. This sand is almost white in color as in figure 1. The result by EDS analysis reveals silicon (49.75 wt%) and oxygen (49.46 wt%) as main component with small amount of aluminum (0.78 wt%). Therefore, the major constituent of this sand could be Quartz (SiO_2). Moreover, the significantly low amount of alumina (Al_2O_3) appearing in the sand insures that this sea sand is low-impurity sand which is suitable to be a raw material for glass-making process.

Figure 1: Krabi Sand

(2) Coloring of prepared glass sample

Glass samples were prepared and their color was analyzed and compared. It is found that glass sample without addition of Cr_2O_3 exhibits clear white. However, the addition of Cr_2O_3 turns the glass into transparent green color as can be seen in Table 1. With higher Cr_2O_3 content, glass samples become darker green tone.

Table 1: Comparison of prepared glass samples with different Cr_2O_3 content.

				
0.0 wt%	0.1wt%	0.3 wt%	0.5 wt%	0.7 wt%

(3) Density and Molar Volume

Variation of density and molar volume of glass samples with content of Cr_2O_3 is shown in figure 2. The density of prepared glass samples increases with increasing content of Cr_2O_3. As density of Cr_2O_3 (5.22 g/cm^3) is higher than that of SiO_2 (2.196 g/cm^3), the increase of glass density with increasing Cr_2O_3 content implied the substitution of Cr_2O_3 in the place of SiO_2, which is the main structure of the glass system. Figure 2 also shows that the molar volume of glass samples without addition of Cr_2O_3 is 14.4498 cm^3/mol and with an addition of 0.1 wt% Cr_2O_3, the molar volume drops to 14.3845 cm^3/mol. A drop of molar volume with a rise of density may be mainly caused by an increase of molecular weight of the glass system. However, further increase in content of Cr_2O_3 leads to an increase of molar volume of the glass as well as an increase of its density. This reveals a less compact glass structure due to partial replacement of SiO_2 by Cr_2O_3 in the glass network. This result could be explained by the fact that ionic radius of Cr^{3+} (0.80 Å) are larger than that of Si^{4+} (0.54 Å) which are main network structure interstices. The larger Cr^{3+} ions modify the interstices and form the larger one, leading to the increase in molar volume of the glass. Moreover, the Cr-O bond is more ionic and as well much weaker bond than Si-O bonds. This implied the insertion of Cr^{3+} ions in the glass network could rupture the glass network and form non-bridging oxygen (NBOs) atoms (Venkateswara Rao and Shashikala, 2014). Therefore, the addition of Cr_2O_3 in the glass samples could be responsible for increasing the number of NBOs and this leads to an expansion of the glass network.

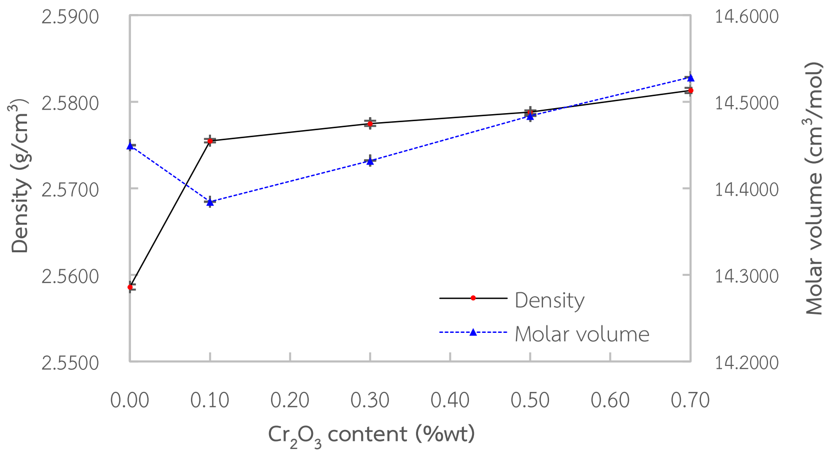

Figure 2: Variation of density and molar volume of glass samples as a function of Cr_2O_3 content.

(4) Refractive index

From refractometer, glass samples with Cr_2O_3 addition of 0.00, 0.10, 0.30, 0.50 and 0.70 wt% have refractive indexes of 1.2119, 1.3221, 1.3377, 1.3519 and 1.3559, respectively. It can be seen in figure 3 that the refractive index of glass increases with increasing Cr_2O_3 content. As NBOs are more polarizable than bridging oxygen (BOs), NBOs can cause the refractive index of the glass samples to increase significantly. Therefore, the increase in refractive index support the formation of NBOs due to the addition of Cr_2O_3 into the glass system (Seo et al., 2014). Moreover, the refractive indexes of all glass samples are found to be lower by 9-20% than that of emerald jewelry, which is in a range of 1.568 to 1.590. This could be possibly explained by the absence of Fe_2O_3, which is one of green coloring-agents, in Krabi sand.

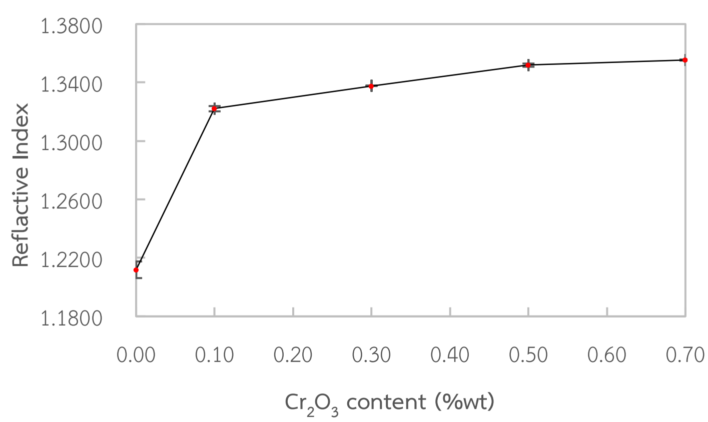

Figure 3: Variation of reflective index of glass samples as a function of Cr_2O_3 content.

(5) longitudinal ultrasonic velocity and modulus

It is found in figure 4 that an increase in concentration of Cr_2O_3 results in a reduction in ultrasonic velocity in the glass samples, and consequently reduction in their longitudinal modulus from 102.19 to 89.59

GPa. Generally, a reduction of ultrasonic velocity is related to the formation of a greater number of non-bridging oxygens (NBOs) and consequently, the decrease in connectivity of the glass network (Gilman, 2009). Thus, the substitution of Cr_2O_3 to the main glass network structure break the glass network and consequently reduce an energy transformation of oxygen, resulting in a reduction in longitudinal ultrasonic velocity and modulus of the glass samples.

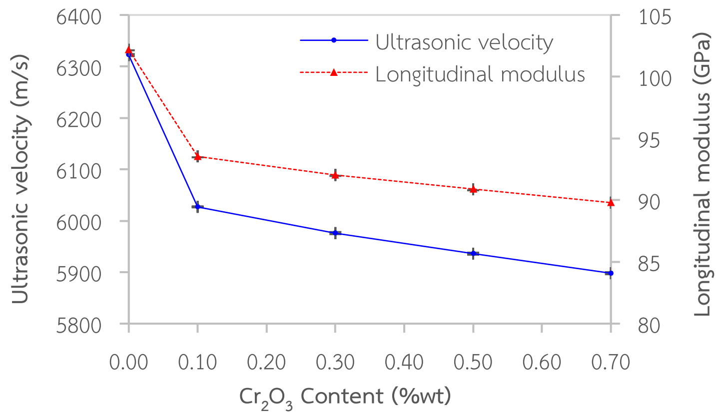

Figure 4: Variation of ultrasonic velocities and longitudinal modulus of the glass samples with different Cr_2O_3 content.

(6) Optical Absorption

Optical absorption spectra of the glass samples measured at room temperature by UV-Vis spectrophotometer over the wavelength of 200-800 nm is shown in figure 5. For the glass sample without the addition of Cr_2O_3, absorption occurs at below 350 nm and no absorption of visible light occur, therefore it appears white or colorless. On the other hand, the color of glass sample with the addition of Cr_2O_3 become green since no absorption of visible light occur only at around 500-600 nm. With increasing Cr_2O_3 content, the absorbance increases, as the glass samples become darker green tone.

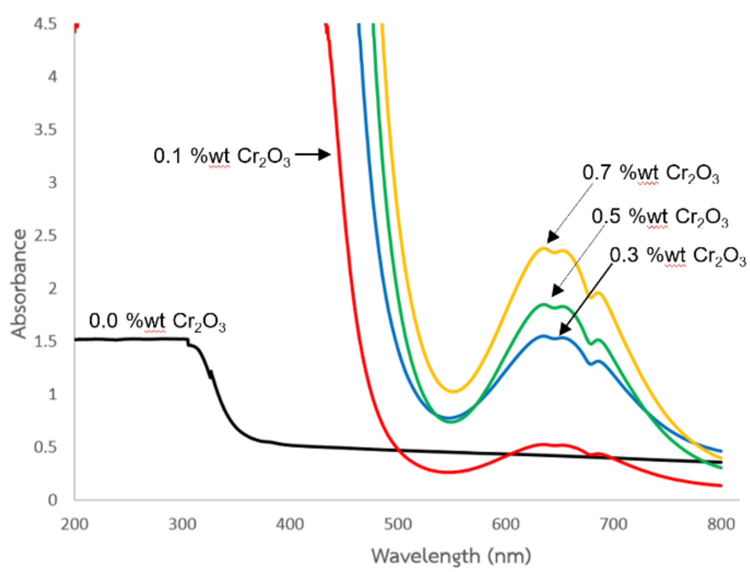

Figure 5: UV-Vis absorption spectra of prepared glass samples

(7) Optical Band gap

The measured absorption spectra were then further used to calculate optical band gap of glass samples. Plots of $(\alpha h\nu)^2$ versus $h\nu$ and $(\alpha h\nu)^{1/2}$ versus photon energy $(h\nu)$ were conducted and their R-square values were compared. From the results, R-squared value for direct transition is closer to 1 than the indirect. Hence, it could be concluded that our glass samples could be direct band gap material. The direct band gap for all glass samples are obtained and shown in figure 6. As the calculated direct band gap is higher than 2 eV, our glass samples are all insulators. The direct band gap gradually decreases from 2.69 eV to 2.31 eV, indicating a modification of band gap by addition of Cr_2O_3 in the glass system (Hammad and Abdelghany, 2016; Govindasamy Murungasen and Sagadevan, 2017). The decrease of the band gap could be explained by the formation of NBOs. It is known that, in the glass network, BOs are bonded to two cations, while NBOs are bonded to only one network cation. The formation of the NBOs leads to the reduction in network connectivity of the glass. Therefore, the increase in the Cr_2O_3 content leads to the formation NBOs, and consequently decreases the optical band gap of the glass (Bale et al., 2008).

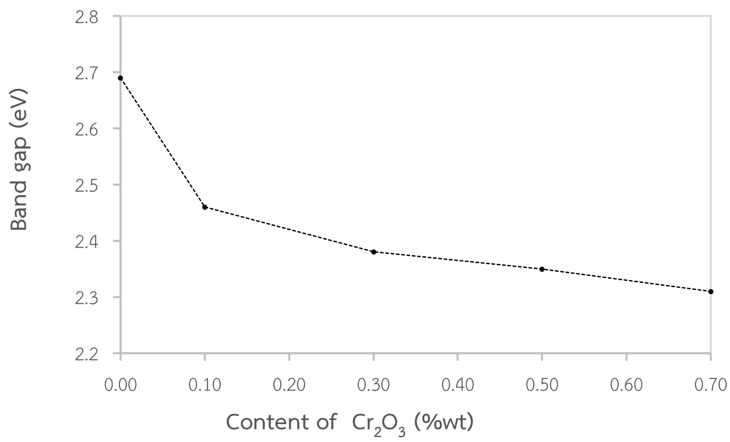

Figure 6: Direct band gap of glass samples with different content of Cr_2O_3

(8) Miro-Hardness Testing

Micro-hardness testing of the glass samples was carried out using Vickers micro-hardness and the result is shown in figure 7. It can be seen that the Vickers hardness decreases abruptly from 23.18 GPa for glass sample without addition of Cr_2O_3 to 14.56 GPa for the glass sample with addition of 0.10 wt% Cr_2O_3. However, with further increasing in addition of Cr_2O_3, the hardness increases slowly. The drop at the beginning of this hardness is due to that fact that chromium ion is larger than silicon ion which is the main network structure atom of the glass. The substitution of chromium ion for silicon leads to a breakdown of the network structure and consequently, resulting in a loose packed structure. The drops to the lowest micro-hardness can be supported by ultrasonic velocity in the glass samples (Gunhakoon, 2020). However, when the addition of Cr_2O_3 is higher than 0.10 wt%, the hardness of the glass samples increases with an increase of Cr_2O_3 content. This could be explained by the effect of higher densities of Cr_2O_3 (5.22 g/cm^3) than SiO_2 (2.196 g/cm^3). Moreover, the correlation between micro-hardness and molar volume support the substitution of larger radius Cr^{3+} ions for Si^{4+}network forming ions due to partial replacement of SiO_2 by higher density Cr_2O_3 in the glass system (Laopaiboon et al., 2020).

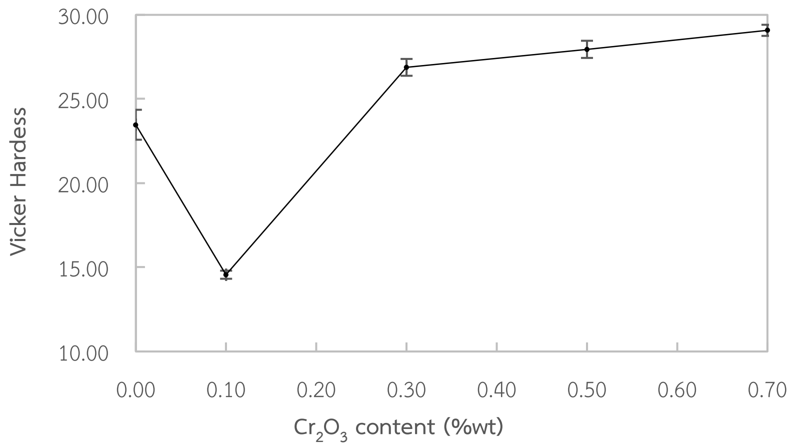

Figure 7: Variation of Vickers hardness of glass samples with different Cr_2O_3 Content.

(9) Glass bead-making testing

To test the possible use of the glass derived from Krabi sand for fabrication of ancient glass beads, glass rods from this emerald green glass were prepared as seen in figure 8 (left). Then these emerald green glass rods were used for making beads. During the bead-making process, the glass was heated, stretched, and cooled, however, no crack and bubble was observed on a beaded necklace as seen in figure 8 (right). Therefore the glass in system (52-x)KS-17Na_2O-4Al_2O_3-6CaO-7K_2O-XCr_2O_3, where KS stands for Krabi Sand and x = 0.00, 0.10, 0.30, 0.50 and 0.70 wt% can be a potential candidate for glass bead-making process.

Figure 8: (Left) emerald green glass rods from Krabi sand and (right) beaded necklace made from the emerald green glass rods.

Conclusion

The Cr_2O_3 green coloring agent was added to the glass system. Then, the properties of the prepared glass samples were examined and found to varied according to Cr_2O_3 content. Refractive index of the glass samples increased while their longitudinal modulus and optical band gap decreased with increasing Cr_2O_3 content. These results indicate that the added Cr_2O_3 caused the formation of NBOs and also the decrease of BOs in the glass network structure. The observed changes support the role of Cr_2O_3 as network modifier. The correlation between the results from molar volume and microhardness as well indicates a breakdown of the network structure due to a substitution of larger and heavier chromium ions for silicon-network forming ions. Moreover, this study shows the possible use of Krabi sand as raw material for glass bead-making process.

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