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# $Er^{3+}$-Doped Soda-Lime Silicate Glass: Artificial Pink Gemstone

**[1]Weeranut Kaewwiset, [1]Kheamrutai Thamaphat,**
**[2,3]Jakrapong Kaewkhao and [1,3]Pichet Limsuwan**

[1]Department of Physics, Faculty of Science,
King Mongkut's University of Technology Thonburi, Bangkok 10140, Thailand
[2]Center of Excellence in Glass Technology and Materials Science (CEGM),
Faculty of Science and Technology,
Nakhon Pathom Rajabhat University, Nakhon Pathom 73000, Thailand
[3]Thailand Center of Excellence in Physics,
CHE, Ministry of Education, Bangkok 10400, Thailand

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## ABSTRACT

$Er^{3+}$-doped soda-lime silicate glasses of the composition (in mol%) (65-x)$SiO_2$:25$Na_2O$:10CaO:x$Er_2O_3$ (where x = 0, 1, 2, 3, 4 and 5) were fabricated by conventional melt quenching technique. The physical and optical properties were measured and investigated. The erbium oxide enters the glass network as a modifier by occupying the interstitial space in the network and generating the NBOs to the structure. The molar volume increases with an increase in $Er_2O_3$ content, which is attributed to the increase in the number of Non-Bridging Oxygen (NBOs). The increase of NBOs in the structure generally leads to an increase in average atomic separation. The density, molar volume and refractive index of glasses increased linearly with increasing $Er_2O_3$ concentration. The color of glasses was changed from light pink to intense pink as the $Er_2O_3$ concentration was increased from 1 to 5 mol%. The Vickers hardness of $Er^{3+}$-doped glasses was found to be in the range of 450-500 HV. In this study, it can be concluded that the soda-lime silicate glasses doped with high $Er_2O_3$ concentration has intense pink color and high value of hardness which is suitable to be cut as gems.

**Keywords:** $Er^{3+}$-Doped Glass, Pink Gemstone, Color Measurement, Optical Properties, NBOs

## 1. INTRODUCTION

A gemstone is a piece of attractive mineral which is used to make jewelry or other adornments. Color is the most obvious and attractive feature of gemstones. In gem trade, the natural color and clarity of gem render non-fashionable for over the years, so there have been various attempts such as heat treatment, irradiation treatment and chemical treatment to improve the appearance and stability of inferior samples (Limsuwan *et al*., 2008; Gutierrez *et al*., 2010; Nassau, 1994). Depending on the type and extent of treatment, they can affect the value of the stone. Some treatments are widely used because the resulting color is stable, while others are not well accepted because the gem color is unstable and may revert to the original tone (Nassau, 1994).

Various pink gemstones include rose quartz, pink sapphire, pink topaz, morganite (pink beryl), Kunzite, pink tourmaline and pink opal (Cipriani and Borelli, 1986). However, some pink gemstones such as morganite is very pale pink color and Kunzite can be found in pale pink to lavender-pink color shade. Therefore, the price of these gemstones is much cheaper as compared to pink sapphire. Furthermore, Kunzite color is faded by light exposure. As all above mentioned, we were ignited to find the new method for fabrication of artificial pink gemstone that is not expensive but color is gorgeous, vivid, clarity and stability.

Silicate glass is an attractive host matrix for rare-earth ions because of its fine optical and mechanical properties such as good chemical stability, high UV transparency, high surface damage threshold, large tensile fracture strength and good durability (Xu *et al*., 2004; Yanbo *et al*., 2006; Sharma *et al*., 2007). In addition, the most common type of glass is the soda-lime silicate glass. These glasses are cheap, chemically durable and relatively easy to melt and fabricated in various shapes (Xu *et al*., 2004). It is well known that glass doped with a small amount of $Er_2O_3$ has light pink color (Padlyak *et al*., 2008). In this work, the artificial pink gemstones were therefore fabricated

**Corresponding Author:** Weeranut Kaewwiset, Department of Physics, Faculty of Science, King Mongkut's University of Technology Thonburi, Bangkok 10140, Thailand

from soda-lime silicate glass doped with high $Er_2O_3$ concentrations ranging from 1-5 mol%. The physical and optical properties were measured and investigated. The hardness and color measurements were also carried out.

## 2. MATERIALS AND METHODS

### 2.1. Sample Preparation

The $Er^{3+}$-doped soda-lime silicate glasses of the composition (65-x) $SiO_2$:25$Na_2O$:10CaO:x$Er_2O_3$ were prepared by the melt quenching technique. All chemicals used in the present work, $SiO_2$, $Na_2O$, CaO and $Er_2O_3$ were of high purity (Fluka, 99.99%). Appropriate amounts of the raw materials were thoroughly mixed and ground in a pestle and mortar for half an hour. The prepared mixture was then heated in a high purity alumina crucible at 1200°C by an electric furnace for about 3 h to ensure complete melting of all components. The melt was then quickly poured into a preheated stainless steel mold and annealed at 500°C for 3 h and let it cooled down slowly to room temperature. The amount of the glass batch is about 50 g/melt. The obtained glass was cut and finely polished into a size of 5×10×3 mm. The chemical compositions of the glasses, prepared in the present work, are summarized in **Table 1**.

### 2.2. Density, Molar Volume and Refractive Index Measurements

By applying Archimedes principle, the weight of the prepared glass samples was measured in air and in xylene using a 4-digit sensitive microbalance (Denver, Pb214). Then, the density, p, was determined from the relation (Chimalawong *et al*., 2010a; Kaewkhao *et al*., 2011) Equation 1:

$$\rho = \frac{W_a}{W_a - W_b} \times \rho_b \quad (1)$$

where, $W_a$ is the weight in air, $W_b$ is the weight in xylene and $P_b$ is the density of xylene ($P_b$ = 0.863 g $cm^{-3}$).

The corresponding molar volume, $V_m$, was calculated using the following formula Equatin 2:

$$V_m = M / p \quad (2)$$

where, M is the molecular weight of the multi-component glass system given by Equation 3:

$$M = x_{sio_2} Z_{sio_2} + x_{Na_2O} Z_{Na_2O} + x_{Cao} Z_{cao} + x_{Er_2O_3} Z_{Er_2O_3} Z_{Er_2O_3} \quad (3)$$

where, $x_{SiO_2}$, $x_{Na_2O}$, $x_{CaO}$ and $x_{Er_2O_3}$ are the mole fractions of the constituent oxides and $Z_{SiO_2}$, $Z_{Na_2O}$, $Z_{CaO}$ and $Z_{Er_2O_3}$ are the molecular weights of the constituent oxides.

**Table 1.** Chemical compositions of the glasses studied in the present work

| $Er_2O_3$ (mol%) | Glass system (mol%) |
|---|---|
| 0 | $65SiO_2$-$25Na_2O$-10CaO |
| 1 | $64SiO_2$-$25Na_2O$-10CaO-$1Er_2O_3$ |
| 2 | $63SiO_2$-$25Na_2O$-10CaO-$2Er_2O_3$ |
| 3 | $62SiO_2$-$25Na_2O$-10CaO-$3Er_2O_3$ |
| 4 | $61SiO_2$-$25Na_2O$-10CaO-$4Er_2O_3$ |
| 5 | $60SiO_2$-$25Na_2O$-10CaO-$5Er_2O_3$ |

The refractive index (n) of the glass samples was measured using an Abbe' refractometer (ATAGO) with mono-bromonaphthalene as a contact layer between the sample and prism of the refractometer. A sodium vapor lamp, nm (D line), was used as the light source.

### 2.3. Vickers Hardness Measurement

Vickers hardness of glass sample was measured with a micro hardness tester (Huatec Industry Instrumentation, DHV 1000).

The indenter is a square-based diamond pyramind with included angle between face of 136°. Load used in these measurements was 0.1 kg. Vickers hardness is defined as the load (kg) divided by the surface area (square millimeters) of the indentation. The area of indentation is calculated from the lengths of the diagonals. DHV-1000 micro Vickers hardness tester is equipped with optics, a light source, a digital microscope, a digital camera, a CCD video camera and a LCD screen and therefore it is able to produce a clearer indentation and hence a more precise measurement.

### 2.4. Color Measurement

The color of glass samples was measured using a spectrophotometer (Shimadzu 3100) with CIE L*a*b* system (Baydogan, 2004; Nassau, 2001). It provides a three-dimensional color space in which the numerical positions of colors more closely match to their perceived relative spacing. The central vertical axis (z-axis) represents lightness designated as L* whose values run from 0 (black) to 100 (white). The a* and b* axes have no specific numerical limits. The x-axis is a* value which positive a* is red and negative a* is green. Perpendicular to this and going toward the positive y-axis is yellow, which is given the name+b* while the opposite-b* is blue.

### 2.5. Optical Absorption Measurement

The optical absorption spectra of the prepared glass samples in the UV-Visible range from 190-750 nm were recorded at room temperature using a UV-Vis spectrophotometer (Perkin Elmer, Lambda 35).

## 3. RESULTS

### 3.1. Density, Molar Volume and Refractive Index

The molecular weight and measured density and molar volume of $Er^{3+}$-doped soda-lime silicate glass samples for different $Er_2O_3$ concentrations are listed in **Table 2**.

**Table 2.** Various physical and optical properties for the glass system (65-x)$SiO_2$: 25$Na_2O$: 10CaO:x$Er_2O_3$

| $Er_2O_3$ -(mol%) | Molecular weigth M (g) | Density, ρ (g/cm$^3$) | Molar volume, $V_M$ (cm$^3$/mol) | Refractive index (n) | Color scale $L^*$ | $a^*$ | $b^*$ |
|---|---|---|---|---|---|---|---|
| 0.00 | 60.157 | 2.5314±0.0013 | 23.764 | 1.5247±0.0001 | 84.11 | -0.31 | 0.07 |
| 1.00 | 63.382 | 2.6496±0.0020 | 23.921 | 1.5354±0.0003 | 73.00 | 8.59 | -2.50 |
| 2.00 | 66.606 | 2.7740±0.0017 | 24.111 | 1.5448±0.0001 | 64.44 | 11.86 | -3.41 |
| 3.00 | 69.830 | 2.8750±0.0027 | 24.289 | 1.5519±0.0004 | 67.34 | 14.92 | -4.65 |
| 4.00 | 73.055 | 2.9945±0.0014 | 24.425 | 1.5599±0.0002 | 60.15 | 16.34 | -4.89 |
| 5.00 | 76.279 | 3.0940±0.0010 | 24.654 | 1.5663±0.0005 | 63.29 | 18.31 | -5.46 |

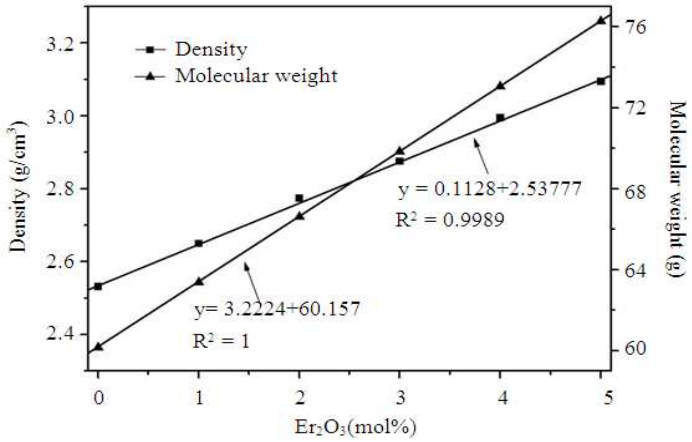


**Fig. 1.** Variation of density and molecular weight with $Er_2O_3$ concentration

The variation of the density and molecular weight with $Er_2O_3$ concentration is shown in **Fig. 1**. As seen in **Fig. 1** both density and molecular weight increase linearly with additional content of $Er_2O_3$ into the network. This indicates that replacing $SiO_2$ by addition of a small amount of $Er_2O_3$ results in the increase of the average molecular weight due to $Er_2O_3$ has a higher relative molecular weight than that of $SiO_2$. The molar volume depends on both density and molecular weight. However, it is clearly seen from **Fig. 1** that the increasing rate of molecular weight is greater than that of density.

**Figure 2** shows the variation of the molar volume with $Er_2O_3$ concentration. As shown in **Fig. 2**, the molar volume increases linearly with an increase in $Er_2O_3$ content. However, in the present work, the addition of $Er_2O_3$ in the glass system results in the opened glass network structure.

The measured refractive index of $Er^{3+}$-doped soda-lime silicate glass samples is also listed in **Table 2**. The plot between refractive index and $Er_2O_3$ concentration is shown in **Fig. 3**. It is seen that refractive index increases linearly with increasing $Er_2O_3$ concentration.

## 3.2. Vickers hardness

For Vickers hardness measurements in this work, the load used was 0.1 kg and dwell time of loading was 10 s. The hardness of each glass sample was measured at three different positions on the glass surface. The measured Vickers hardnesses and average values with standard deviations for undoped glass and all glasses doped with different $Er_2O_3$ contents are listed in **Table 3**.

The relation between average Vickers hardness and $Er_2O_3$ concentration is shown in **Fig. 4**. It is seen in **Table 3** that the average Vickers hardness of undoped and doped soda-lime silicate glasses with $Er_2O_3$ from 1 to 5 mol% are 384.6, 514.4, 482.2, 462.5, 454.6 and 442.5 HV, respectively. As seen in **Fig. 4**, the Vickers hardness value increased abruptly from 384.6 HV for undoped glass to 514.4 HV for 1 mol% $Er^{3+}$-doped glass, then it decreased slowly with increasing $Er_2O_3$ concentration.

This result indicates that the Vickers hardness value for $Er^{3+}$-doped soda-lime silicate glass is approximately ranged from 450-500 HV. In gem markets, only the Mohs scale is used for gem hardness scale. Therefore, to compare the Vickers hardnesses of glasses obtained in this study with those of natural gemstones which reported in Mohs scale, the relative hardnesses of gemstones in Vickers and Mohs are given in **Table 4** (Tabor, 2000).

**Table 3.** Vickers hardness of $Er^{3+}$- doped soda-lime silicate glass samples for various $Er_2O_3$ contents

| $Er_2O_3$ (mol%) | 1 | 2 | 3 | Ave. | SD |
|---|---|---|---|---|---|
| 0 | 386.7 | 369.6 | 397.5 | 384.600 | ±14.068 |
| 1 | 525.9 | 505.0 | 512.3 | 514.400 | ±10.607 |
| 2 | 460.5 | 492.1 | 494.0 | 482.200 | ±18.817 |
| 3 | 459.9 | 444.1 | 483.4 | 462.467 | ±19.775 |
| 4 | 466.5 | 441.0 | 456.2 | 454.567 | ±12.828 |
| 5 | 432.0 | 443.1 | 452.4 | 442.500 | ±10.213 |

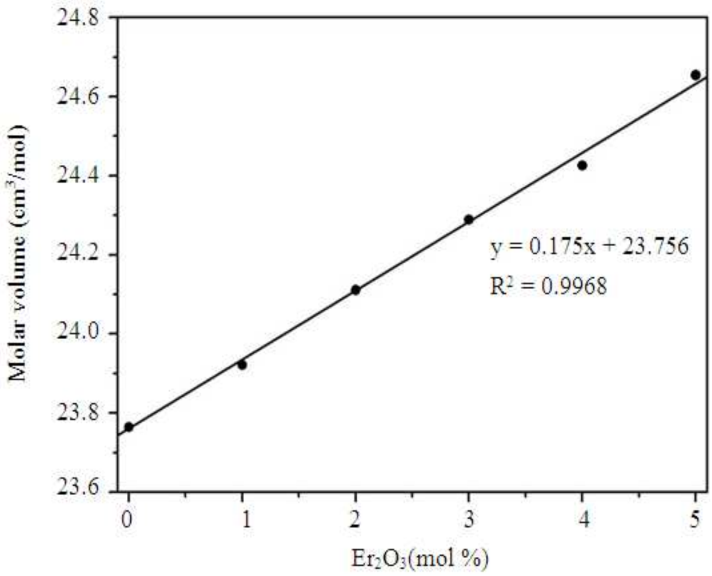


**Fig. 2.** Variation of molar volume with Er2O3 concentration

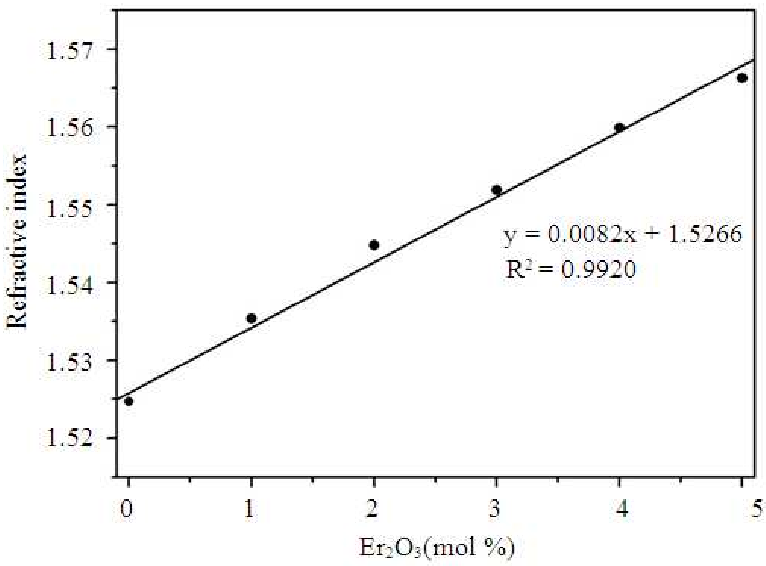


**Fig. 3.** Variation of refractive index with Er2O3 concentration

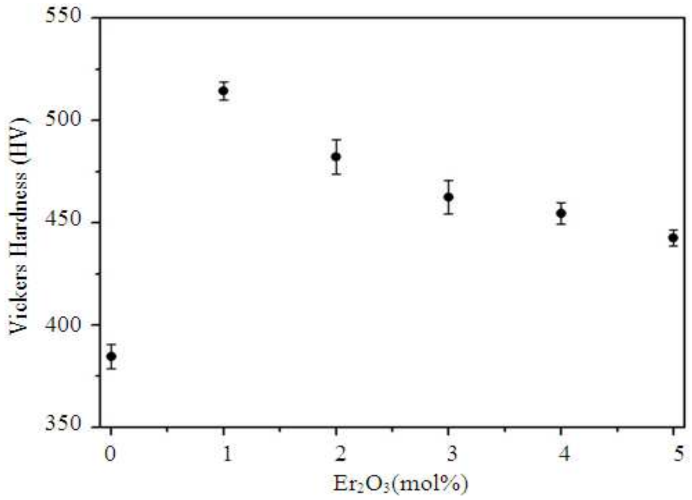


**Fig. 4.** Variation of Vickers hardness as a function of $Er_2O_3$ concentration

**Table 4.** Approximate relative hardnesses of gemstones for Vickers and Mohs scales

| Gemstone | Hardness Mohs | Vickers |
|---|---|---|
| Diamond | 10 | 3000 |
| Corundum | 9 | 2000 |
| Topaz | 8 | 1000 |
| Quart | 7 | 500 |
| Feldspar | 6 | 300 |
| Apatite | 5 | 200 |
| Fluorite | 4 | 100 |
| Calcite | 3 | 50 |
| Gypsum | 2 | 30 |
| Talc | 1 | 20 |

As seen in **Table 4**, the Hardness Value of 500 HV for $Er^{+3}$-doped glass is the same as that of quartz (7 Mohs) and about one-and two-Mohs scales less than those of semiprecious stones (such as Topaz) and corundums, respectively. A hardness of 7 on the Mohs scale is normally taken as the minimum for acceptable stones to use in jewellery. According to the hardness values discussed above, the $Er^{+3}$-doped soda-lime silicate glasses fabricated in this study are possible to be cut and polished as artificial pink gems.

## 3.3. Color

**Figure 5** shows the photographs of undoped and $Er^{3+}$-doped soda-lime silicate glasses. The measured values of L*, a*, b* in CIE L*a*b* color index of undoped and $Er^{3+}$-doped soda-lime silicate glasses for various $Er_2O_3$ concentrations are given in **Table 1**. The plot of a* and b* in chromaticity coordinates is shown in **Fig. 6**. It is seen that, for undoped glass, the coordinates (a*, b*) are (-0.31, 0.07), hence it is colorless. For $Er^{3+}$-doped glasses, the value of a* (red) increased at higher rate than that of b* (blue) as the $Er_2O_3$ concentration was increased from 1 to 5 mol%. As a result, the color of glass samples was changed from pale pink to vivid pink.

## 3.4. Optical Absorption

The absorption spectra of $Er^{3+}$-doped soda-lime silicate glasses in UV-Visible region at room temperature are shown in **Fig.** 7. The spectra obtained for all $Er^{3+}$-doped oxide glasses are similar in nature except for the band intensities. It is clearly observed that the absorption intensity of the absorption bands increases with increasing $Er_2O_3$ concentration.

Six absorption bands peaked at 380, 408, 450, 490, 525 and 655 nm were observed. All absorption band spectra are characteristics of $Er^{3+}$-doped oxide glasses (Sharma *et al*., 2007). In accordance with the energy level diagram and the literature data (Padlyak *et al*., 2006; 2008; Moorthy *et al*., 2000), all the observed absorption bands were assigned to appropriate f-f electronic transitions of $Er^{3+}$ ions from the $^4I_{15/2}$ ground state to the following excited states: $^4G_{11/2}$ ,$^4F_{3/2}$ , $^4F_{5/2}$, $^4F_{7/2}$, $^2H_{11/2}$ and $^4F_{9/2}$.

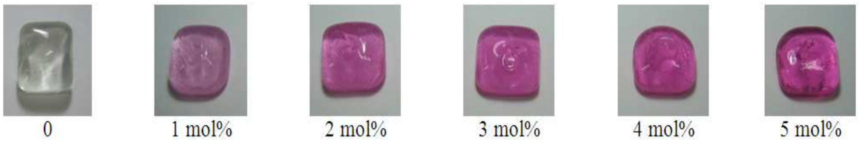


**Fig. 5.** The glass samples of $Er^{3+}$-doped soda-lime silicate glasses

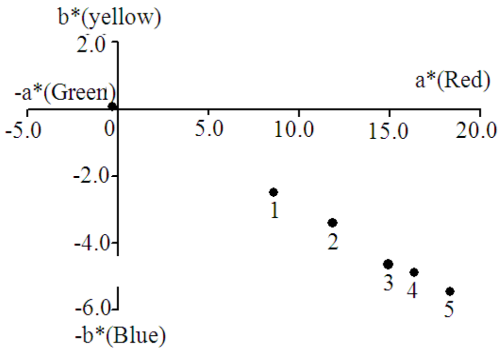


**Fig. 6.** The CIE L*a*b* color scale of $Er^{3+}$-doped soda-lime silicate glasses

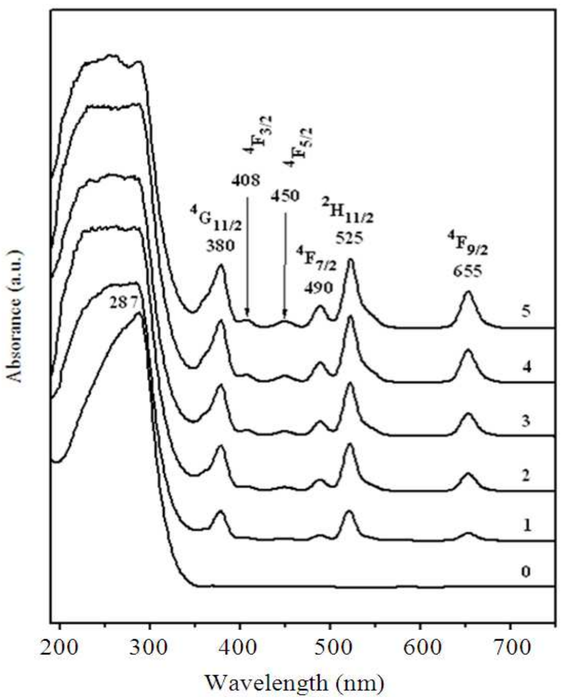


**Fig. 7.** The absorption spectra of $Er^{3+}$-doped soda-lime silicate glasses

As seen in **Fig. 7**, it should be pointed out that the absorption peak at 287 nm is due to the host glasses. In addition, the absorption spectra in visible region (400-750 nm) showed in **Fig. 7** reveal high absorption of light in the green region (525 nm) and yellow region (655 nm). On the other hand, it means that the light in the blue and red regions was transmitted. As a result, the $Er^{3+}$ -doped glass is colored purple. However, for glass samples with higher concentration of $Er_2O_3$, it is seen in **Fig. 7** that some light in the blue region was absorbed as indicated by the peaks at 450 and 490 nm. This leads to the more transmitted light in the red region, that is the glass samples are pink in color. The color of the glass samples as explained by the absorption spectra is in good agreement with the color of glass samples shown in **Fig. 5**.

## 4. DISCUSSION

In the present work, the density, molar volume and refractive index increase with additional content of $Er_2O_3$ due to the $Er_2O_3$ into the network which is attributed to the increase in the number of Non-Bridging Oxygen (NBOs). The increase of NBOs in the structure generally leads to an increase in average atomic separation. The results obtained indicate that the erbium oxide enters the glass network as a modifier by occupying the interstitial space in the network and generating the NBOs to the structure. It can also be concluded that the addition of $Er_2O_3$ may accordingly result in an extension of glass network (Abdel-Baki *et al*., 2007; Chimalawong *et al*., 2010b; Sindhu *et al*., 2005). It is well known that the alkali/alkaline oxides act as glass modifiers in the presence of glass formers like $SiO_2$, $B_2O_3$.

The increase in $V_m$ indicates that the volume of Non-Bridging Oxygen (NBOs) sites produced by the $Er_2O_3$ is greater than those produced by alkaline oxides.

## 5. CONCLUSION

Soda-lime silicate glasses of the composition (65-x)$SiO_2$:25$Na_2O$:10CaO were doped with high $Er_2O_3$ concentrations ranging from 1-5 mol%. The physical properties include density, molar volume and hardness were measured. It was found that the hardness of $Er_2O_3$-doped glass is high enough to be cut as gems. The optical properties include refractive index color and absorption spectra of glass samples were carried out. The results show that the refractive index increased linearly with increasing $Er_2O_3$ concentration. Furthermore, the results on color and absorption spectra measurements show that the pink color of fabricated glasses was increased from light pink to intense pink as the $Er_2O_3$ concentration was increased from 1 to 5 mol%.

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