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*Research Article*

# UV-visible-NIR study of $Er^{3+}$ doped soda lime silicate glass

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## Abstract

Erbium doped soda lime silicate glass with a composition of $25Na_2O{\cdot}10CaO{\cdot}(65-x)SiO_2{\cdot}xEr_2O_3$ (with $x = 0.00$, 0.02, 0.05, 0.10, 0.30 and 0.50 mol%) has been prepared using the normal melt-quench technique. The glass samples have been studied for colour, absorption, density and refractive index of $Er^{3+}$ ion in soda lime silicate glass as a function of the dopant concentration. The colour of glass samples was measured using a spectrophotometer with a reference to CIE L*a*b* colour index. It was found that the colour of glass samples is pink with doped $Er_2O_3$ in glass matrix. The optical absorption spectra of these glasses were recorded in the UV-VIS-NIR region. The fundamental absorption edge was identified from the optical absorption spectra. FTIR spectra were measured in order to estimate $OH^-$ stretching band around 3000 $cm^{-1}$. The density and refractive index were found to be in the range of 2.531 to 2.581 $g/cm^3$ and 1.525 to 1.530, respectively. The results show that the density and refractive index of glass samples increased with increasing of $Er_2O_3$ concentration.

**Keywords:** $Er^{3+}$ doped glass, optical properties, density, refractive index, Thailand.

## Introduction

It is well known that at present much attention has been devoted to the development of infrared lasers and amplifiers for telecommunication. Many rare earth doped glass matrixes such as silicates, phosphates, borates, fluorides, etc. have been used to produce such active optical devices [1, 2]. The rare earth ions doped glass is not only limited to infrared optical devices any longer but also a growing interest in visible optical devices. As a branch of visible luminescence, up-conversion fluorescence of rare earth ions received more and more attention. In order to obtain powerful visible up-conversion emission, $Er^{3+}$, $Tm^{3+}$, $Pr^{3+}$ and $Ho^{3+}$-doped chloride, sulphide and fluoride glasses have been developed [3]. However, poor chemical durability and thermal stability limit the application of these non-oxide glasses seriously. Thus, designing and optimizing novel and stable optical glasses becomes more and more significant [4]. Then the erbium doped glasses are attracted much interest because of the $^4I_{13/2} \rightarrow {}^4I_{15/2}$ transition in $Er^{3+}$ at a wavelength around 1.5 mm, coinciding with the low-loss window of standard optical communications fibre [5].

The soda lime silicate glass has been found to be a suitable optical material with high transparency, low melting point, high thermal stability and good rare earth ions solubility. On the other hand soda lime silicate glasses have superior chemical resistance and are optically transparent at the excitation and lasing wavelength [6]. They are more compatible with the fabrication process in the development of optical devices.

The purpose of the present study is to investigate the optical and physical properties of $Er^{3+}$ doped soda lime silicate glass with various $Er_2O_3$ of concentration (0.00-0.50 mol%).

## Methodology

### *Glass preparation*

The $Er^{3+}$ ions doped glass systems with a composition of $25Na_2O{:}10CaO{:}(65\text{-}x)SiO_2{:}xEr_2O_3$ where $x$ = 0.00, 0.02, 0.05, 0.10, 0.30 and 0.50 mol%, using the normal melt-quench method. $Na_2O_3$, CaO, $SiO_2$ and $Er_2O_3$ of analytical regent grade with purity higher than 99.9% were used. Synthesis of glasses was made in a porcelain crucible in an electric furnace. The temperature of melting was 1,200°C. The duration of melting was 3 hours after the last traces of batches were disappeared. The amount of the glass batch is 30 g/melt. Synthesis process was continued for sufficient time to ensure complete homogeneity. Then the melt was poured onto stainless steel mold. The quenched samples were annealed at 500°C for 3 hours and then cooled inside the furnace down to room temperature. The as-prepared glass samples were cut and then finely polished into a size of 5 mm × 10 mm × 3 mm.

### *Absorbance measurements*

Optical absorbance spectra in the UV-VIS-NIR range of the glass samples were obtained at room temperature with a double-beam spectrophotometer (Shimadzu 3100).

### *Density* ($\rho$)

The density, $\rho$, of glass was measured at room temperature according to the Archimedes method using xylene as the immersion liquid. The density of xylene at the experimental temperature is 0.863 g/cm$^3$. The density of glass is calculated according to the formula:

$$\rho = \frac{W_A}{W_A - W_B} \times 0.863 g/cm^3 \tag{1}$$

where $W_A$ is the weight of the sample in air, $W_B$ is the weight of the sample in xylene.

### *Refractive index and molar refraction measurements*

The refractive index, $n$, of the glass specimens was measured using an Abbe′ refractometer (ATAGO 3T).

The refractive indices obtained for different glass compositions were used to calculate the molar refraction, $R_m$, by the well-known Volf and Lorentz- Lorenz formula [7, 8]:

$$R_m = \frac{(n^2-1)}{(n^2+2)}\left(\frac{M}{\rho}\right) \tag{2}$$

where $n$ is refractive index (at a wavelength 587.56 nm), $\rho$ is the density and $M$ is the molecular weight of the glass samples. $M/\rho$ is called the molar volume, $V_m$, and $R = (n^2-1)/(n^2+2)$ is known as the refraction loss.

### *Molar electronic polarizability* $(\alpha_m)$ *and electronic polarizability of oxide ion* $(\alpha_O^{2-})$

The molar refraction is related to the structure of the glass and it is proportion to the molar electronic polarizability of the material $(\alpha_m)$ according to the relation:

$$\alpha_m = \left(\frac{3}{4\pi N}\right)R_m \tag{3}$$

where $N$ represents Avogadro's number or more strictly, the number of polarizable ions per mole.

Dimitrov and Sakka [9, 10] proposed the following expression for calculation of the average electronic polarizability of oxide ion $(\alpha_O^{2-})$ in numerous single component oxides on the basis of linear refractive index and energy gap. For fourthly glasses with a common formula $x_1A_pO_q{\cdot}x_2B_rO_s{\cdot}x_3C_nO_m{\cdot}x_4D_uO_v$ are additive quantities, we can calculate the electronic ion polarizability of an average oxide ion in glass $\alpha_O^{2-}(n_o)$ using the equation:

$$\alpha_O^{2-}(n_o) = \left[\frac{R_m}{2.52} - \sum\alpha_{cat}\right] / N_O^{2-} \tag{4}$$

where $\sum\alpha_{cat}$ denotes molar cation polarizability given by $x_1p\alpha_A + x_2r\alpha_B + x_3n\alpha_C + x_4u\alpha_D$ and $N_O^{2-}$ denotes the number oxide ions in the chemical formula given by $x_1q + x_2s + x_3m + x_4v$.

The molar cation polarizability $\sum\alpha_{cat}$ of the glasses is calculated using the data on the polarizability of cation collected in Ref. [11, 12]. $Si^{4+}$ ions have the electronic polarizability above 0.033Å$^3$ while $Na^+$ ion is 0.469 Å$^3$ and $Er^{3+}$ ion is 2.253 Å$^3$.

### *Theoretical optical basicity* ($\Lambda_{th}$)

The optical basicity ($\Lambda$) is of an oxide medium does the average electron donor power of all the oxide atoms comprise the medium. On the other hand the polarizability of oxide ions is closely related to the optical basicity of oxide ions is closely related to the optical basicity of oxide materials. The optical basicity could be prophesied from the composition of the glass and the basicity moderating parameters of the various cations present. Theoretical optical basicity ($\Lambda_{th}$) is calculated using the following expression proposed by Duffy and Ingram [13]:

$$\Lambda_{th} = \sum_i x_i \Lambda_i \tag{5}$$

where $x_1$, $x_2$, … , $x_n$ are equivalent fractions based on the amount of oxygen each oxide contributes to the overall material stoichiometry and $\Lambda_1$, $\Lambda_2$, …, $\Lambda_n$ are optical basicity values assigned to the individual oxides. The values optical basicity in this glass is $\Lambda_{SiO_2} = 0.50$, $\Lambda_{Na_2O} = 1.15$, $\Lambda_{CaO} = 1.00$ and $\Lambda_{Er_2O_3} = 0.929$, respectively [11, 12].

The optical basicity value ($\Lambda_{th}$) of an oxide medium is a numerical expression of the average electron donor power of the oxide species constituting the medium so that it is used as a measure of the acid-base properties of oxides, glass, alloys, etc. On the basis of refractive data Duffy has established that an intrinsic relationship exists between the electronic polarizability of oxide ion $\left(\alpha_O^{2-}\right)$ and the optical basicity of oxide ions medium ($\Lambda_{th}$), the following correlation [11]:

$$\Lambda_{th} = 1.67\left(1 - \frac{1}{\alpha_O^{2-}}\right) \tag{6}$$

The theoretical optical basicity for this glass system under study can be calculated using Eq. (6).

### *Fourier Transform Infrared Spectroscopy*

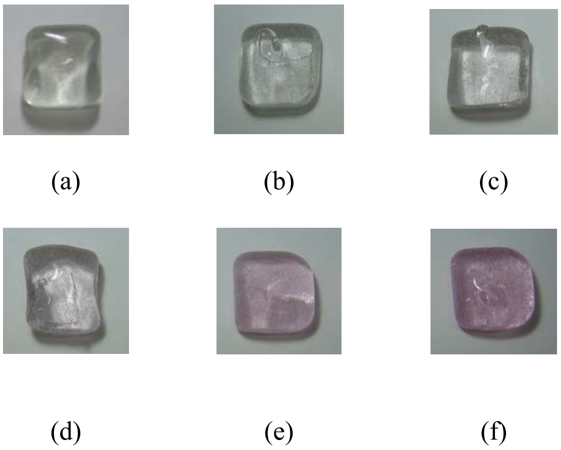
High resolution FTIR spectra were obtained on a Perkin Elmer model PE 2000 FTIR spectrometer. All FTIR were measured at room temperature cover the range from 800-4000 $cm^{-1}$ and using the KBr pellet technique.

## Results and Discussion

The glass samples with different concentrations of $Er_2O_3$ between 0.00 to 0.50 mol% were prepared as illustrated in Fig. 1. The colour of prepared glass samples was measured and the results are shown in Table 1. For undoped glass, it is colourless. On the other hand, the $Er_2O_3$ doped glasses are pink due to $Er^{3+}$ ions in the glass matrix [14]. The glass samples became pinker as the concentration of $Er_2O_3$ was increased.

**Table 1. $Er^{3+}$ concentration, CIE L*a*b* colour scale of $Er^{3+}$-doped $25Na_2O$:$10CaO$:$(65-x)SiO_2$:$xEr_2O_3$ ($x$ = 0.00, 0.02, 0.05, 0.10, 0.30, 0.50 mol%)**

| Sample name | $Er_2O_3$ (mol%) | Glass composition (mol%) | Colour scale | | |
|---|---|---|---|---|---|
| | | | L* | a* | b* |
| a | 0.00 | $65.00SiO_2 : 25Na_2O : 10CaO$ | 84.11 | -0.31 | 0.07 |
| b | 0.02 | $64.98SiO_2 : 25Na_2O : 10CaO : 0.02Er_2O_3$ | 68.96 | 0.09 | 1.31 |
| c | 0.05 | $64.95SiO_2 : 25Na_2O : 10CaO : 0.05Er_2O_3$ | 73.13 | 0.57 | 1.07 |
| d | 0.10 | $64.90SiO_2 : 25Na_2O : 10CaO : 0.10Er_2O_3$ | 72.40 | 1.58 | 0.58 |
| e | 0.30 | $64.70SiO_2 : 25Na_2O : 10CaO : 0.30Er_2O_3$ | 72.40 | 3.57 | -0.43 |
| f | 0.50 | $64.50SiO_2 : 25Na_2O : 10CaO : 0.50Er_2O_3$ | 70.52 | 5.34 | -1.05 |



(a) (b) (c)

(d) (e) (f)

**Figure 1. Glass samples of soda lime silicate doped with different concentrations of $Er_2O_3$.**

($Er_2O_3$ = 0.00, 0.02, 0.05, 0.10, 0.30, 0.50 mol%)

Table 1 shows the CIE L*a*b* colour index of glass samples with different $Er^{3+}$ concentrations. It is seen that the value of L* and b* decreased with increasing of $Er_2O_3$ concentrations whereas a* increased with increasing of $Er_2O_3$ concentrations. Furthermore, it is seen that a* is between 0.09-5.34 and b* is between -1.05 to 1.31 which corresponds to the pink regions colour scale.

Fig. 2 shows the absorption spectra of $Er^{3+}$ doped soda lime silicate glasses in UV-VIS-NIR region at room temperature. The inhomogeneously broadened bands are attributed to the transitions from the $^4I_{15/2}$ ground state to the various excited states of the $Er^{3+}$ ions [6].

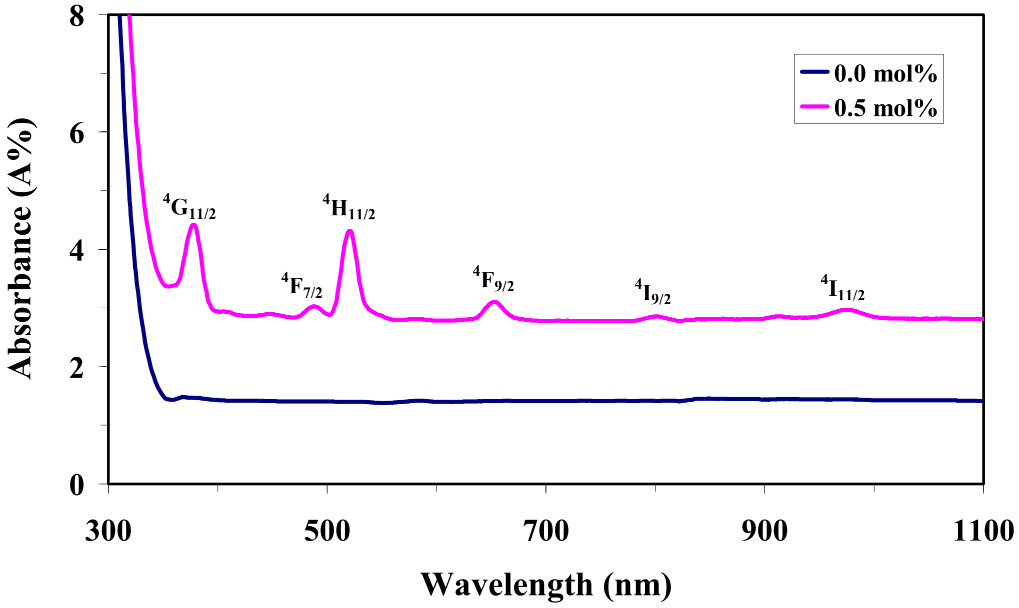


**Figure 2. The absorption spectra of $Er^{3+}$ doped soda lime silicate glasses.**

As can be seen in Fig. 2, the undoped glass exhibited absorption edge at 308 nm and another peak appeared with the doping of $Er_2O_3$ from 0.00 to 0.50 mol%. In Fig. 2, the transmission spectra consisting of 6 bands were observed at 378, 488, 520, 653, 801, 975 nm corresponding to the excited levels, $^4G_{11/2}$, $^4F_{7/2}$, $^4H_{11/2}$, $^4F_{9/2}$, $^4I_{9/2}$ and $^4I_{11/2}$ respectively. Furthermore, each absorption spectrum increased with increasing of $Er_2O_3$ concentration, which corresponds to glass colour.

The density ($\rho$), refractive index ($n$), molar refraction ($R_m$) and optical basicity ($\Lambda_{th}$), molar polarizability ($\alpha_m$) and electronic oxide ion polarizability ($\alpha_O^{2-}$) of the soda lime silicate glasses with different $Er_2O_3$ concentrations are shown in Table 2.

**Table 2. The density ($\rho$), refractive index ($n$), molar refraction ($R_m$) and optical basicity ($\Lambda_{th}$), molar Polarizability ($\alpha_m$) and electronic oxide ion polarizability ($\alpha_O^{2-}$) of the soda lime silicate glasses.**

| $Er_2O_3$ (mol%) | Density, $\rho$ (g/cm$^3$) | Refractive index, $n$ | Molar refraction, $R_m$ (cm$^3$/mol) | Optical basicity, $\Lambda_{th}$ | Molar polarizability , $\alpha_m$ (cm$^{-3}$) x10$^{-24}$ | Electronic oxide ion polarizability, $\alpha_O^{2-}$ (Å$^3$) |
|---|---|---|---|---|---|---|
| 0.00 | 2.5314 | 1.5247 | 7.2793 | 0.6288 | 2.8858 | 1.6544 |
| 0.02 | 2.5342 | 1.5249 | 7.2814 | 0.6291 | 2.8866 | 1.6542 |
| 0.05 | 2.5390 | 1.5259 | 7.2909 | 0.6294 | 2.8904 | 1.6553 |
| 0.10 | 2.5454 | 1.5268 | 7.3028 | 0.6301 | 2.8951 | 1.6563 |
| 0.30 | 2.5584 | 1.5277 | 7.3533 | 0.6327 | 2.9151 | 1.6611 |
| 0.50 | 2.5812 | 1.5301 | 7.3933 | 0.6353 | 2.9309 | 1.6632 |

The dependence of density and refractive index on $Er_2O_3$ concentration is shown in Figures 3 - 4, respectively. It can be seen that, for all $Er_2O_3$ doped glasses, the density and refractive index values increased with increasing $Er_2O_3$ concentration. It is due to the substitution of $Er_2O_3$ in the place of $SiO_2$. Theoretically, the refractive index is a function of density and mean polarizability of the medium [15].

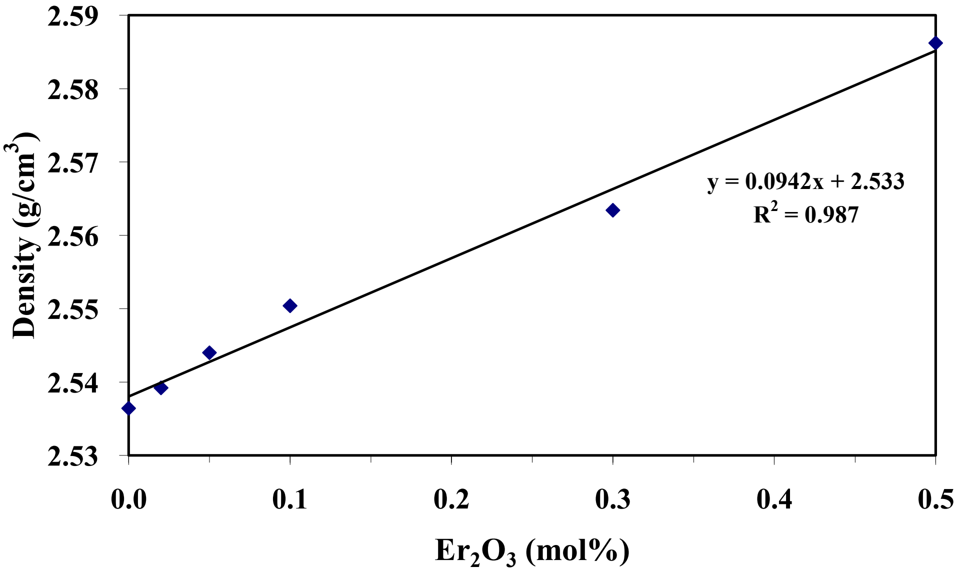


**Figure 3. The density with concentration of $Er_2O_3$ for the soda lime silicate glasses.**

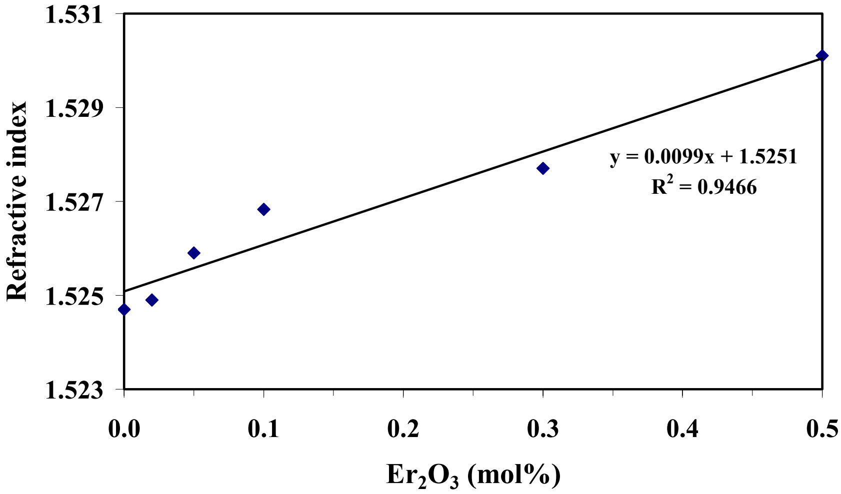


**Figure 4. Refractive index with concentration of $Er_2O_3$ for the soda lime silicate glasses.**

The value of the molar refraction ($R_m$) and molar polarizability ($\alpha_m$) of the $Er^{3+}$ doped soda lime silicate glasses shown in the Table 2 were evaluated using Eqs. (3) and (4), respectively. The increase in molar refraction and increases in refractive index accompany the increase in molar polarizability. These findings confirm that the refractive index of the glasses don't only depend on the density but moreover on the molar polarizability of the glass [16-17].

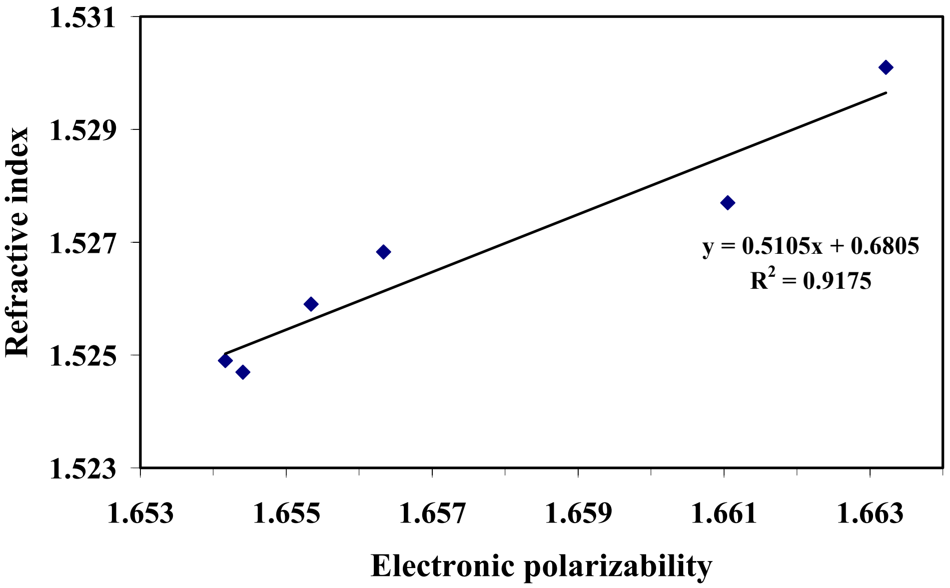


**Figure 5. The refractive index versus the electronic polarizability of oxide ions $\alpha_O^{2-}$ for the soda lime silicate glasses.**

The refractive index ($n$) versus a function of the electronic polarizability of oxide ions ($\alpha_O^{2-}$) was plotted in Fig. 5. It can be seen that increasing in the oxide ion polarizability along with the refractive index.

The relation between electronic polarizability of oxide ions ($\alpha_O^{2-}$) and optical basicity ($\Lambda_{th}$) in this glass system is shown in Fig. 6.

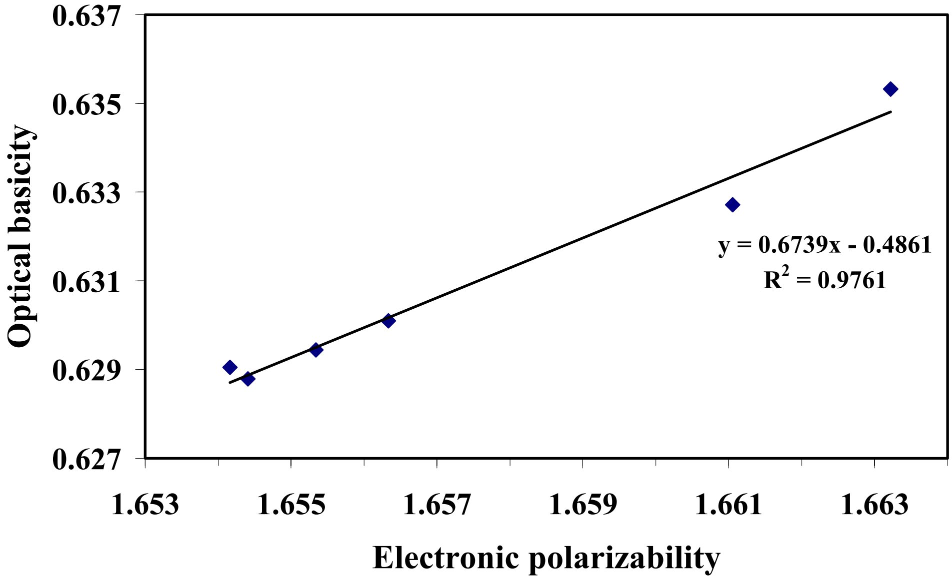


**Figure 6. The optical basicity versus electronic polarizability of oxide ions for the soda lime silicate glass.**

It can be observed that the optical basicity increases with increase in $Er_2O_3$ concentration. This relationship presents a general trend toward and increases in the oxide ion polarizability with increase in optical basicity.

These results demonstrate that the optical basicity is closely related to electronic polarizability. The optical basicity of the oxidic medium is the average electron donor power of all the oxide atoms comprising the medium. The optical basicity increase shows that with an increasing negative charge on the oxygen atoms there is an increasing covalency in the cation-oxygen bonding [18].

Fig. 7 shows FTIR spectra of $Er^{3+}$ doped soda lime silicate glasses in 4000-800 $cm^{-1}$ ranges. The FTIR spectrum of the undoped glass exhibits two characteristic $H_2O$ ($OH^-$ stretching vibration) bands [19]. The FTIR band observed about 3450 $cm^{-1}$ is a $H_2O$. The band around 1060 $cm^{-1}$ can be attributed to asymmetric stretching vibration of Si-O bond in [$SiO_4$] network [20]. The shoulder around 960 $cm^{-1}$ can be attributed to symmetric stretching vibration of Si-O bond in [$SiO_4$] network. FTIR spectra of $Er^{3+}$ doped soda lime silicate glasses from 0.02 to 0.50 mol%, corresponding to $OH^-$ stretching band around 3000 $cm^{-1}$, were observed.

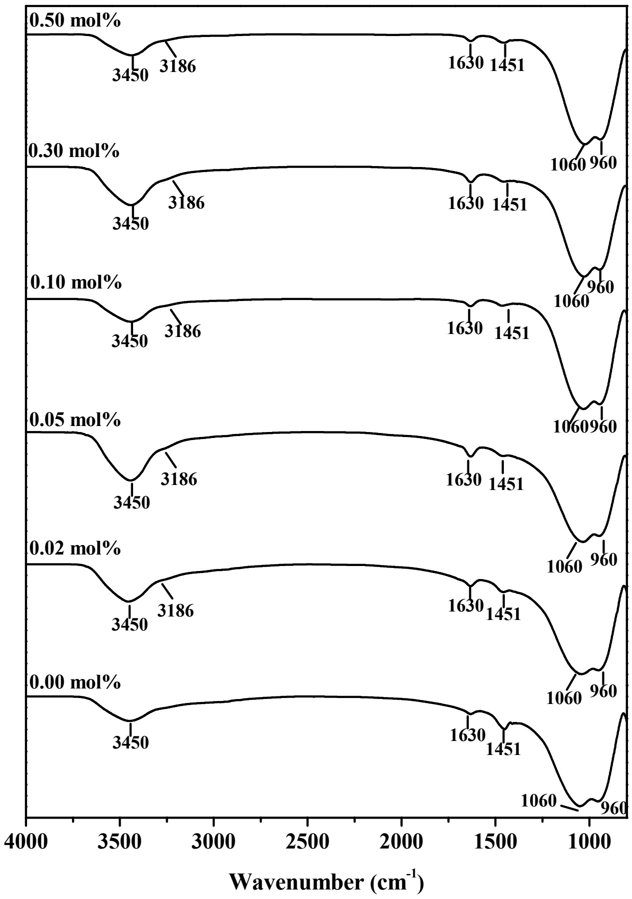


**Figure 7. The FTIR spectra of $Er^{3+}$ doped soda lime silicate glass.**

## Conclusion

Glass systems of composition $25Na_2O:10CaO:(65\text{-}x)SiO_2:xEr_2O_3$ ($x = 0.00$, 0.02, 0.05, 0.10, 0.30 and 0.50 mol%) have been synthesized by normal melt-quench technique. It has been found that the glass samples are pink colour with doped $Er_2O_3$ in glass matrix. The density and refractive indices of glasses were increased with increasing of $Er_2O_3$ concentration and also found the refractive indices could be increased linearly with glass densities. In the absorption spectra 6 bands were observed at 378, 488, 520, 653, 801 and 975 nm correspond to the excited levels, $^4G_{11/2}$, $^4F_{7/2}$, $^4H_{11/2}$, $^4F_{9/2}$, $^4I_{9/2}$ and $^4I_{11/2}$, respectively. FTIR spectra of $Er^{3+}$ doped soda lime silicate glass from 0.02 to 0.50 mol%, corresponding to $OH^-$ stretching band around 3000 $cm^{-1}$, were observed. The molar polarizability, optical basicity and electronic polarizability of oxide ion increases with an increase in $Er_2O_3$ concentration. Furthermore, the refractive index of the glass does not only depend on the density but more so on the optical basicity and electronic polarizability of oxide ion.

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