Optical and Electrical Properties of Nd$^{3+}$doped Na$_2$O-ZnO-TeO$_2$ Material

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1. Introduction

Tellurite glasses are extremely attractive materials for linear and non-linear application in optics due to their important aspects such as their low melting temperature, low phonon energy, and high refractive index, high dielectric constant, good chemical durability, high thermal stability, non-hygrosopic, with a large transmission window and the possibility to integrate a large amount of rare-earth ions [1-8]. It can be used as micro-lenses, IC photo masking glass, hard disks, press modeling of spherical lenses, glass substrates for solar cells, artificial bones, dental implants, and crowns. The optical property of rare-earth ions in tellurite glasses depends on the chemical composition, which determines the structure and the nature of the bonds of the glass matrix nature of the glass matrix bonds. Besides, understanding their microscopic mechanism of structural and optical behavior gave much thrust and basic interest for both academia and the industries. Tellurite glasses doped with rare-earth ions have attracted researchers for their broad spectrum of applications in optoelectronic and photo-electronic devices viz solid-state lasers optical switches, broad-band amplification, non-linear optical devices, infra-red (NIR) laser windows, optical fibers[9,10]. The doping of rare-earth ions in tellurite glasses has shown interesting properties like an amplification of optical signal in the
visible and NIR region, optical data storage, white light emission, and planner waveguides which are applicable to microchip lasers, biomedical diagnostics, light-emitting diodes, and high-density optical data reading [11-13]. Recently, the precise properties of tellurite glass have been investigated for the demonstration of various spectroscopic and non-linear optical device applications as broadband light emission and optical communications networks.

The applications of neodymium-doped glass materials are most commonly useful than the different types of rare-earth ions doped glasses [14]. In 1961, Snitzer initiated the application of glass material as a medium for containing neodymium ions (Nd$^{3+}$) [15]. Neodymium doped lasers have been used in various applications to perform within a high-efficiency range at room temperature. The phonon energy is decreased due to the amalgamation of heavy metal oxides into the tellurite glass system [16]. Also, alkaline earth metal oxides such as Na$_2$O, CaO, and MgO reduce the crystalline structure and increase the amorphous nature of the glass system [17-19]. For this result, the thermal stability of glasses is increased [20]. The fundamental structural units of tellurite glass are adapted to the rare-earth oxides like Nd$_2$O$_3$ from 4-coordination to 3-coordination by exchanging TeO$_4$ trigonal-bipyramid units to the TeO$_3$ trigonal pyramid units [21].

In this present work, the focus has been initiated to systematically study the glass formation, structural, optical, and electrical properties constant at room temperature with frequency variation and thermal conduction mechanism of the Nd$^{3+}$ doped Na$_2$O-ZnO-TeO$_2$ (NZT) glass materials with different doping concentrations.

2. Materials and Methods

The conventional melt quenching technique has been used to prepare Na$_2$O-ZnO-TeO$_2$ (NZT) glasses using research great initial ingredients Zinc Oxide (ZnO), Tellurium di-Oxide (TeO$_2$), Sodium Carbonate (Na$_2$CO$_3$) manufactured by Merck, and Neodymium Oxide (Nd$_2$O$_3$) made by Loba Chemie. The mixing ratio of Na$_2$O, ZnO, TeO$_2$ is maintained as 1:2:7 to prepare the host glass. Neodymium Oxide (Nd$_2$O$_3$) was added to the host glass as a dopant for (0-2) wt%. The homogeneous mixture of these compounds was obtained by grinding the ingredient powders in an agate mortar. The mixture was kept in an alumina crucible, and the crucible was placed in an electrical box furnace. The melt quenching process was obtained by two stages of heating with the temperature at 400 °C for 1 hour and temperature at 475 °C for the next half an hour to produce the quality tellurite glass. The cylindrical stainless steel plate was used to hold the melted sample for quenching, and the prepared glass was placed again in the furnace at 400°C for 1 hour to anneal the sample. The annealed glass was allowed to reach room temperature gradually through the slow cooling process to avoid thermal stress.

The thermal properties, differential thermal analysis, and thermo-gravimetry of the initial mixtures (raw materials in the powder form) were analyzed in the argon environment by using PerkinElmer Instrument (Pyris Diamond TG/DTA, thermo-gravimetric/differential thermal analyzer). These were studied properties for the temperature range of 30 °C to 650 °C with a scanning rate of 10°C/min. X-ray diffraction patterns were obtained using an X-ray diffractometer (RIGAKU model: Japan, XRD 6000, λ = 1.5418 Å) with a slow-scanning rate 3°/min between the angles 10º and 70º for all the samples. FTIR spectrometer (HITACHI Model F-700) was used to identify the nature of pure and doped glasses in the wavenumber range 400-3000 cm$^{-1}$. Optical absorption and emission spectra of all the glasses were obtained using UV/VIS/NIR spectrophotometer (PerkinElmer Lambda-35) for the wavelength range 400-800 nm and fluorescence spectrophotometer (HITACHI Model F-7000) for 300-600 nm.
at room temperature. The dielectric constants (ε) of prepared glass samples were measured using LCR- HiTESTER (HIOKI, Japan) for the large frequency range of 200 Hz-4MHz at room temperature. The temperature-dependent DC conductivity of these glasses was studied for the temperature range 36 °C-227 °C using a constant voltage supply and current meter.

3. Results and Discussion

The color of the pure NZT glass is white, while Nd³⁺ doped NZT glasses are revolved into purple color due to the doping of Nd³⁺ ions in the host glass NZT. The prepared transparent NZT glasses are shown in Figure 1. The color of the glass samples varies from bluish to violet with the increase of doping concentration. It has been found that there are no visible crystallites present in these transparent samples [22].

![Figure 1. Pictorial view of Nd₂O₃ dipping NZT glasses different samples.](https://biointerfaceresearch.com/)

3.1. Thermal analysis.

Figure 2 shows the DTA curve of the mixed precursor of pure and Nd³⁺ ions doped NZT glasses for the temperature range 35 °C to 650 °C. There are three successive endothermic peaks evolved in the temperature range of 72 °C to 160 °C for all the samples. These endothermic peaks in this specified temperature range reveal the desorption or evaporation of the moisture from the precursor ingredients [23]. It has also been observed that there are exothermic peaks present in the DTA curve within the temperature range 330 °C to 463 °C. These peaks demonstrate the removal of CO₂ through the decomposition of Na₂CO₃ present in the initial ingredients and the phase transition from solid powders to liquid form through melting. The phase transition has come about through the melting at the temperature of 447 °C. The melting point of TeO₂ (730 °C) is reduced to 447 °C due to the existence of Na₂CO₃ and ZnO in the initial mixture [24, 25]. It is also observed that the melting point of the mixture slightly decreases with increasing the concentration of Nd₂O₃. TG and its differentiation curves of the precursor powders of the samples are shown in Figure 3. These curves for all the samples depict the weight loss during heating at different temperatures. The total weight loss in the measured temperature range 35 °C to 650 °C is 10.71%, and this amount of losses occurred from the removal of moisture, evaporation of the volatile substances, and decomposition of Na₂CO₃ by releasing CO₂, etc. The major mass loss in the temperature range 64-125 °C is taken place due to the desorption of water, removal of moisture and other volatile materials, which is also confirmed from DTA curves [26]. The emission of CO₂ is observed at 385 °C to 395 °C, which is displayed in DTG curves for pure and doped samples. The small amount of mass is reduced within the temperature range of 442 °C to 460°C during the melting of solid powders. No weight loss is observed beyond the temperature of 460 °C.
Figure 2. DTA graphs of pure, 1.0 wt% and 2.0 wt% of Nd$_2$O$_3$ doped with NZT glasses.

Figure 3. TGA and DTG vs. temperature graph of pure, 1.0 wt% and 2 wt% Nd$_2$O$_3$ doped with NZT glass samples.

3.2. X-ray diffraction.

Figure 4 shows the XRD pattern of the glass samples. The XRD peaks of pure NZT glass are observed at 10.86°, 21.92°, 23.26°, 29.03°, 29.86°, and 31.39° for the corresponding planes (010), (110), (011), (021), (111) and (030) respectively. It has been found that the sharp peak at 13.24° signifies new hybrid compounds, and it may develop due to the presence of Na$_2$Te$_2$O$_5$, 2H$_2$O [27]. This new hybrid compound has not been formed in the case of the addition of doping material Nd$_2$O$_3$, as the peak position at 13.24° is absent in the doped NZT glass samples. The broadening of XRD peaks in higher doping concentrations reveals the amorphous nature of the doped samples. The shifting of the peak positions is observed for the higher concentration of Nd$_2$O$_3$ doped NZT glass samples. This shifting has ascribed to the modification of crystalline pattern embedded in the glass samples due to the incorporation Nd$^{3+}$ ions.
3.3. Optical properties.

3.3.1. FTIR.

The FTIR spectra for pure and doped NZT glasses are shown in Figure 5. Analyzing these spectra is a useful process to investigate structural studies with functional groups and bonding information in the crystalline and non-crystalline systems [28]. The transmission spectra of the various concentration of neodymium oxide doped glass samples are recorded for the region 400-3000 cm\(^{-1}\). The position of the structural unit of ZnO is observed in the band range 424–440 cm\(^{-1}\). The absorption band at 426 cm\(^{-1}\) is appeared due to the symmetric stretching vibration of the Zn–O bond [29]. The characteristics of tellurite oxide found the structural unit in the range 600–800 cm\(^{-1}\). In this broad range, the pure TeO\(_2\) is characterized by an infrared absorption at around 644 cm\(^{-1}\). The formation of tellurite glass contains two types of fundamental structural units as TeO\(_4\) trigonal bipyramidal and trigonal pyramidal. Symmetrical stretching vibration of Te–O bond in trigonal bipyramids (TeO\(_4\)) and Te–O bending vibrations in trigonal pyramids (TeO\(_3\)) in the tellurium network were observed around 644 cm\(^{-1}\) [30-33]. The broad peaks can be attributed to the mixing of two groups, and the absorption peaks broaden with the addition of Nd\(_2\)O\(_3\). This broadenings of peaks and increase of intensity confirm Nd\(^{3+}\) ions in the host glass matrices. The band at 771 cm\(^{-1}\) is evolved for pure and doped glasses due to trigonal pyramidal structural units. This band is attributed to the stretching vibration within the tellurium and the non-bridging oxygen of the trigonal pyramidal structure [34, 18]. The absorption peaks around 1658cm\(^{-1}\) and 2922 cm\(^{-1}\) are ascribed to stretching vibration of hydrogen bonding and mental bonding with hydroxyl group as the hydroxyl group is coupled with the Te\(^{4+}\) glass network [35].
3.3.2. Optical absorption.

The absorption spectra of pure and Nd$_2$O$_3$-doped NZT glasses are shown in figure 6 for the visible region (400-800 nm) at room temperature. It has been found that the absorption transition occurs only for Nd$^{3+}$ ions doped glasses, and the intensity of absorption spectra increases with the increase of doping concentrations. There is no transition of the pure glass sample.
The peak positions of absorption spectra for Nd$^{3+}$ ions doped glasses are designated as similar to $^4$I$_{9/2} - ^4$F$_{J}(J= 9/2,7/2)$ and $^4$I$_{9/2} - ^4$G$_{J}(J=11/2,9/2,7/2,5/2)$ transitions corresponding to the six absorption band at 430 nm ($^4$I$_{9/2} - ^4$G$_{11/2}$), at 512 nm ($^4$I$_{9/2} - ^4$G$_{9/2}$), at 525 nm ($^4$I$_{9/2} - ^4$G$_{7/2}$), at 583 nm ($^4$I$_{9/2} - ^4$G$_{5/2}$), at 683 nm ($^4$I$_{9/2} - ^4$F$_{9/2}$) and 746 nm ($^4$I$_{9/2} - ^4$F$_{7/2}$). Though the absorption band of rare-earth-doped tellurite glasses arises in the ultraviolet region in general, the current glass network displays the absorption in the visible region with large intensity due to the presence of Nd$^{3+}$ions as a dopant in the glass samples. The introduction of Nd$^{3+}$ions behaves as crystalline material, confirming the sharp absorption band, also confirmed from XRD. The different forbidden transitions concerning 4f levels are also involved in the exhibition of absorption bands in the visible region [20, 36]. It has also been found from figure 6 that the peak positions of absorption bands for different dopant concentration of Nd$_2$O$_3$ is slightly shifted towards a lower wavelength. This displacement of the absorption band may be occurred due to the change of structural arrangement and various fundamental units of the present glass materials. The peak position of the absorption band may also be shifted due to the change of strength in the oxygen bond in the glass materials. Similar absorption spectra are observed for the addition of rare-earth oxide of the same type in the other tellurite glasses [10,37-40].

Figure 7 shows the variation of $(\alpha h\nu)^{1/2}$ with $h\nu$ (Tauc plot) for all glass-ceramics at room temperature. Here, the coefficient of absorption $\alpha(\nu)$ of optical absorption spectra is obtained according to the formula of Davis and Mott:

$$\alpha(\nu) = (A/d)$$  \hspace{1cm} (1)

where A is the absorbance and d is the thickness of the sample [41, 42, 16].

The indirect bandgap energy is decreased from 2.63 eV and 1.32 eV due to the higher concentration of the Nd$^{3+}$ions. The bandgap energy and other physical properties of pure and doped glass samples are tabulated in Table 1. The physical properties perform an important part in estimating the optical property for rare-earth-doped glasses. The structural efficiency of the glass samples is developed in the glass network. The physical parameters of prepared glasses like concentration ions, inter-ionic distance ($r_i$), polaron radius ($r_p$), and field strength (F) are calculated and displayed in Table 1. The variation of inter-ionic distance ($r_i$) and field strength (F) with different ion concentrations of rare-earth ion (Nd$_2$O$_3$) is shown in figure 8. It has been found that the Inter-ionic distance ($r_i$) decreases, and field strength (F) increases with the increase of doping concentration of Nd$_2$O$_3$. The Stronger field strength is evolved around Nd$^{3+}$ ions with the increase of Nd-O bond strength. The compactness of the glass structure is confirmed from the result of the density and increasing concentration of Nd$^{3+}$ ions in the doped glasses. The electron localization may be formed due to the decrease of $r_i$ and $r_p$. This formation of electron localization can directly affect bandgap energy according to the structural change of the glass system. The donor centers in the glass matrix increase with the increase of electron localization, and the outcome is the decrease of optical band gap energy ($E_g$) which is also confirmed from the Tauc’s plot for pure and Nd$^{3+}$ doped glasses [43-47].

| Table 1. Bandgap energy and physical properties of pure and Nd$^{3+}$-doped NZT glass materials. |
|-------------------------------------------------|-------------------------------|
| **Physical properties** | **Nd$_2$O$_3$ doped different samples** |
| Doping concentration of Nd$_2$O$_3$ in NZT (wt%) | 0.0 | 0.5 | 1.0 | 1.5 | 2.0 |
| Concentration of Nd$^{3+}$ions N(10$^{20}$ions/cm$^3$) | - | 1.49 | 2.08 | 2.19 | 3.31 |
| Inter ionic distance between the Nd$^{3+}$ ions ($r_i$ in Å) | - | 18.87 | 16.87 | 16.60 | 14.46 |
| Polaron radius ($r_p$ in Å) | - | 7.61 | 6.80 | 6.69 | 5.83 |
| Electric Field strength F(in 10$^{10}$ cm$^{-2}$) | - | 2.49 | 3.12 | 3.22 | 4.25 |
| Bandgap Energy $E_g$ indirect (\(n=2\)) eV | 2.63 | 1.90 | 1.63 | 1.50 | 1.32 |
3.3.3. Fluorescence spectra.

The fluorescence spectra of pure and \(\text{Nd}_2\text{O}_3\) doped with NZT glasses have shown in figure 9. The spectra have been obtained at room temperature for the wavelength range 355 nm to 600 nm with an excitation wavelength of 325 nm. No transition takes place for a pure NZT glass sample. The intensity of the emission spectra for all the doped glasses increases with increasing the doping concentration of \(\text{Nd}^{3+}\) ions. It has been found that a broad spectrum of wavelength 370-556 nm is emitted from the doped samples. This spectrum contains several emission lines, which are evaluated from the deconvolution of each spectrum. We have observed five emission bands emitted a broad spectrum \(2\text{P}_{3/2} \rightarrow 4\text{I}_{9/2}\) at 371 nm, \(2\text{D}_{3/2} \rightarrow 4\text{I}_{9/2}\) at 421 nm, \(2\text{P}_{1/2} \rightarrow 4\text{I}_{9/2}\) at 431 nm, \(4\text{G}_{11/2} \rightarrow 4\text{I}_{9/2}\) at 467 nm, and \(4\text{G}_{7/2} \rightarrow 4\text{I}_{9/2}\) at 546 with the excitation wavelength \(\lambda_{\text{exc}} = 325\) nm. The emission corresponding to the \(2\text{P}_{1/2} \rightarrow 4\text{I}_{9/2}\) transition is stronger than the other emissions bands [11, 48]. As the \(\text{Nd}^{3+}\) ions are populated in the \(2\text{D}_{5/2}\)
level, some of them relax radiatively to this level by emitting the fluorescence. The green emission of Nd$^{3+}$ ion in doped NZT glass is observed due to the decaying transition from $^4G_{7/2}$ $\rightarrow^4I_{9/2}$ at 546nm. The peak position of the transitions shifted towards a higher wavelength for 2% doping of concentration of Nd$_2$O$_3$. This shifting may arise due to the presence of excited Nd$^{3+}$ ions in $^2D_{5/2}$ and $^4G_{11/2}$ state for the respective transitions with blue emissions [11].

**Figure 9.** Fluorescence spectra of various concentrations of Nd$^{3+}$ions doped with NZT glasses, excited at 325 nm.

3.3.4. Cross-section.

The absorption cross-section of the glass samples for the $^4G_{5/2}$ $\rightarrow^4I_{9/2}$ transition have been calculated using the Lambert-Beer formula:

$$\sigma_{ab}(\lambda) = 2.303 \frac{A}{(Nd)}$$

(2)

Where $A$ is absorbance, $d$ is the thickness of the sample, and $N$ is the density (ions/cm$^3$) of Nd$^{3+}$ ion of the telluride glass. Figure 10 shows the absorption cross-section is decreasing for increasing the concentration of Nd$^{3+}$ doped NZT glass samples. The emission cross-section is measured using MaCumber's (1964) theory [49].

The absorption and emission cross-section are associated with the formula:

$$\sigma_{em}(\lambda) = \sigma_{ab}(\lambda) \exp[(E - h\nu)/kT]$$

(3)

where $\nu$ be the phonon frequency, $E$ is the free energy needed to excite Nd$^{3+}$ from $^4I_{9/2}$$\rightarrow^4G_{5/2}$ state at temperature $T$, $h$ is the Planck’s constant, and $k$ is the Boltzmann constant. The emission cross-section for the $^4I_{9/2}$$\rightarrow^4G_{5/2}$ transition of Nd$^{3+}$ doped glasses is shown in Figure 11. The emission cross-section is decreased for a higher concentration of Nd$^{3+}$ions of NZT glasses.
Figure 10. Absorption cross-section vs. wavelength (nm) for 0.5 to 2 wt% doped Nd$_2$O$_3$ with NZT glass materials.

Figure 11. Wavelength (nm) dependence emission cross-section for NdO$_3$ doped with NZT glass materials.
3.4. Dielectric property.

3.4.1. Dielectric constant.

Figure 12 shows the real part of the dielectric constant ($\varepsilon'$) of Nd$^{3+}$ doped NZT at room temperature in the frequency range 200 Hz to 4 MHz. The value of $\varepsilon'$ of Nd$^{3+}$ doped NZT glass samples is decreased with increasing frequency from 200 Hz to ~2 kHz. At lower frequencies, the value of $\varepsilon'$ is high due to the absence of spontaneous polarization in oxide glass materials [50]. The values of $\varepsilon'$ of our samples are almost frequency independent in the frequency range ~3 kHz to ~1 MHz due to a decrease of ionic, space charge, and orientation polarization. The dielectric constant is increased in the frequency range ~1 MHz to 4 MHz with a higher concentration of Nd$^{3+}$ ions. A similar result has been observed in Eu$^{3+}$ ions doped NZT glass samples [51].

![Figure 12](https://biointerfaceresearch.com/)

**Figure 12.** The variation of dielectric constant ($\varepsilon'$) as a function of frequency for Nd$^{3+}$ doped NZT glasses.

3.4.2. DC conductivity.

Figure 13 shows the variation of electrical conductivity with temperature for pure and Nd$^{3+}$ doped NZT glass materials. It has been found that the conductivity of all the glass samples is increased with increasing temperature and also with the increase of doping concentration in the host glass. This increment of conductivity with temperature reveals that the electrical conduction mechanism is Arrhenius type. This Arrhenius mechanism of electrical conduction can be used and estimated activation energy of the samples using the relation:

$$\sigma_d = \sigma_0 \exp(-\frac{E_a}{kT})$$

(4)

where, $E_a$ is the activation energy, $\sigma_0$ is the pre-exponential factor, $T$ is the absolute temperature, and $k$ is the Boltzmann constant. The estimated activation energies for all the samples have been calculated from figure 13. The activation energy decreases (666 meV to 552 meV) with increasing concentrations of Nd$_2$O$_3$ doped glass samples [50].

The increment of conductivity due to the increase in the doping concentration of rare-earth ions can be explained based on the interaction between the rare-earth ions and the structural units of host glass. The atomic weight, doping concentration, and location of Nd$^{3+}$ ions describe the mechanism of conductivity in the glass structure. The addition of rare-earth
ions in the host glass creates a large number of non-bridging oxygen in the glass materials. This is also confirmed from the analysis of obtained FTIR spectra for all the samples. So, the increases of Nd$^{3+}$ ions in the NZT glasses enhance the creation of non-bridging oxygen atoms, and hence the conductivity is increased for all the doped glass samples [52].

Figure 13. Arrhenius plot of ln$\sigma$ vs 1000/T for Nd$^{3+}$ ions doped NZT glasses.

4. Conclusions

Neodymium (III) doped NZT glasses were effectively manufactured by the melt quenching method. The X-ray diffraction characterization sturdily signified the amorphous nature of the prepared glasses for the higher concentration of Nd$^{3+}$ ions. The results of the preparation and characterization of the luminescent system are obtained for the addition of various concentrations (0.5 wt%, 1.0 wt%, 1.5 wt%, 2.0 wt%) of NZT glass samples. Six absorption bands are displayed corresponding to 430 nm ($^{4}I_{9/2}$$-^{4}G_{11/2}$), at 512 nm ($^{4}I_{9/2}$$-^{4}G_{9/2}$), at 525 nm ($^{4}I_{9/2}$$-^{4}G_{7/2}$), at 583 nm ($^{4}I_{9/2}$$-^{4}G_{5/2}$), at 683 nm ($^{4}I_{9/2}$$-^{4}F_{9/2}$), and 746 nm ($^{4}I_{9/2}$$-^{4}F_{7/2}$). The peak value of photoluminescence spectra is slightly changed due to the addition of the higher concentration of rare-earth ion of the NZT glass sample. It specifies that the excitation wavelength simply depends upon the glass composition. Field strength (F) was observed to increase with increasing the Nd$^{3+}$ ions concentration. Inter ionic distance ($r_i$) and polaron radius ($r_p$) were found to decrease with increasing the Nd$^{3+}$ ions. The role of Nd$^{3+}$ ions in modifying the structural and optical properties has been understood. The decrease in the value of direct optical band gap energy was ascribed to the change in the structure of such glasses. The dielectric constant of Nd$_2$O$_3$ doped NZT glasses was measured for various frequencies and seen as a stable substance within the frequency of 3 kHz to 1 MHz. So Nd$_2$O$_3$ doped NZT glass samples have a large prospect for applying optoelectronic devices.

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Conflicts of Interest

All the authors have no interest in any financial and personal relationships with other people or organizations that could inappropriately influence (bias) their work. Authors are interested only for academic purposes.

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