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## Influence of ZrO<sub>2</sub> additions on the structural and optical properties of Na-B-Te Glass for Optoelectronic Applications

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#### Abstract

This study is to estimate the effect of the replacement of  $TeO_2$  by  $ZrO_2$  on the sodium borotellurite glass. Five samples have been prepared according the chemical formula; 30 mol % Na<sub>2</sub>CO<sub>3</sub>- 50 mol % B<sub>2</sub>O<sub>3</sub>- (20-x) mol% TeO<sub>2</sub>- x% ZrO<sub>2</sub> where x =0, 2, 4, 6, and 8mol% using high purity chemicals. The raw materials of each sample were mixed well in an agate mortar and were then transferred into a porcelain crucible. The batches were melted using an electric muffle furnace at a temperature of 1200°C for one hr. The melts were stirred several times to ensure homogeneity. Each melt was poured on a brass plate at RT and another brass plate was put on the sample melt to obtain thin plate glass sample. Archimedes principal was used to determine the density of each glass sample. The molar volume of each glass sample was calculated. The results of experimental density and the calculated molar volume showed that the samples density decreased, while the molar volume increased with increasing ZrO<sub>2</sub> content. The five glass samples were measured by XRD to confirm their amorphous structure. The FTIR spectra of the studied glass samples were obtained at RT in the range from 4000 to 400 cm<sup>-1</sup>. The FTIR spectra showed that as Zr ions were increased the BO<sub>3</sub> changed to BO<sub>4</sub> then the BO<sub>4</sub> and the nonbridging oxygen increased. The transmission spectra using UV, Vis. Spectra in the range of 190-1100 nm were measured, then optical parameters could be obtained. The optical measurements results that obtained from UV and visible region and the optical properties that calculated using different equations demonstrated the ability of using these samples for different optoelectronic and energy storage applications.

#### Keywords, ZrO<sub>2</sub> borotellurate glass, FTIR groups, Optical parameters.

#### 1. Introduction

Oxide glasses have very important attention due to its different applications in different fields [1-6]. Moreover, glassy materials have different types according to their composition. Oxide glass have different fields of application such as optoelectronic, sodium ion batteries, smart windows, optical filter, solar energy .... etc. When two alkali oxides or alkaline earth oxides are added to borate glasses, their characteristics exhibit a non-linear behavior when the oxides' concentrations are gradually adjusted. The term "borate anomaly" refers to this deviation from linearity [7]. Numerous investigations on the so-called borate anomaly have been conducted using a variety of methods, including electrical conduction, infrared spectroscopy, and neutron diffraction research .... etc. [8-10]. However, it is interesting to follow the structural groups forming borate glass networks [11, 12]. Such structural changes may conduct to the correct occupation of an ion, whether it participates either as former or modifier cation. This article aims to is to estimate the effect of the replacement of TeO<sub>2</sub> by ZrO<sub>2</sub> on the structural and optical parameters of the sodium borotellurite glass doped with ZrO<sub>2</sub>, applying IR spectroscopic analysis as well as the measurement or calculation of density, molar volume and optical parameters. These measurements were done to examine the effect of the gradual increase of Zr ions content at the expense of Te ions in the prepared glass properties.

#### 2. Experimental Work

Five glass samples have been prepared based on the chemical composition; 30 mol% Na<sub>2</sub>CO<sub>3</sub>-50 mol % B<sub>2</sub>O<sub>3</sub>- (20-x) mol% TeO<sub>2</sub>- xmol % ZrO<sub>2</sub> where  $0 \le x \le 8$  mol% using high purity chemicals. The raw chemical of each sample were mixed in an agate mortar and then moved to a porcelain crucible. Batches were melted using an electric muffle-furnace at a temperature of 1200°C for one hr. The melts were stirred several times to ensure homogeneity. Each melt was poured on a brass plate at RT and another brass plate was put on the sample melt to obtain thin plate glass sample. Archimedes technique was used to determine the experimental density value of each glass sample by weighing it in air and in carbon tetrachloride as an immersion liquid at RT. The molar volume of each glass sample was calculated from the mean molecular weight of such glass sample divided by its density, or the volume occupied by one gram mole of the glass. It is considered to be more sensitive to investigate and to reflect the structural differences between glass and crystalline materials. This is because it normalizes the atomic weights of different glass constituents [13]. Equation (1) can be used to calculate the experimental molar volume  $(V_m)_{exp}$  of the studied glass, where  $W_m$  is the mean molecular weight of the sample, and  $\rho_{exp}$  is its experimental value of the density [13].

$$(V_m)_{exp} = \frac{W_m}{\rho_{exp}} \tag{1}$$

X-ray diffractometer was used to check the prepared samples to confirm their amorphous nature,  $CuK_{\alpha}$  of  $\lambda$ =0.1541 nm was used in XRD measurements. The FTIR was used to detect some information about the network of the prepared glass system. The FTIR measurements were obtained in the range from 400 to 4000 cm<sup>-1</sup> using spectrometer of type Perkin Elmer, model RTX, and KBr discs were used in FTIR measurements. Carrel Zeiss-PMQ spectrometer was used to measure the optical transmission (T) spectra which in turn used to obtain the optical parameters using at room temperature in the range 190 - 1100 nm.

#### 3. Results and Discussion

#### 3.1 Structure characterization

Fig. (1) represents the obtained XRD patterns for all the studied samples, where a broad hump centered around  $2\theta = 26$  degrees can be observed with no indication of any diffraction peaks in all the XRD patterns which reveals the amorphous nature of the prepared samples.



Fig. (1), XRD patterns for the studies samples

The bulk density and the molar volume values are plotted in Fig. (2) as a function of  $ZrO_2$  content. As seen in Fig. (2), the experimental density values decreased gradually with the gradual increase of  $ZrO_2$ . The decrease of density value can be attributed to the replacement of one mole of  $TeO_2$  (mean molecular weight = 159.5988 g/mol) by one mole of  $ZrO_2$  (molecular

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weight = 123.2228 g/mol). Also, in Fig. (2) the molar volume values increased gradually with the gradual increase of  $ZrO_2$  content. The observed increase of molar volume may be due to the ionic radius of Zr ion (160 pm) is greater than the ionic radius of Te ion (140 pm), so the replacement of one mol of TeO<sub>2</sub> by one mol of ZrO<sub>2</sub> caused gradual increase in the molar volume values.



Fig. (2), The variation of density and molar volume as a function of  $ZrO_2$  content

Fig. (3) Shows the FTIR charts of all samples where each chart consists of four regions located at  $1600 - 1200 \text{ cm}^{-1}$ ,  $1200 - 800 \text{ cm}^{-1}$ ,  $800 - 500 \text{ cm}^{-1}$  and  $500 - 420 \text{ cm}^{-1}$  [14]. Each spectrum was deconvoluted [15]. The position and the area of each peak was calculated and assigned, table (1). On the other side, Fig. (4) Illustrates the deconvoluted FTIR peaks for 4 mol% ZrO<sub>2</sub> sample as a representative sample.



Fig. (3). FTIR absorbance spectra of the prepared glass samples



**Fig. (4).** The deconvoluted spectrum of the glass sample of 4 mol % ZrO<sub>2</sub>, as a representative sample.

There are many FTIR features indicating the different vibration modes of the structural chains of the building units in the network of the prepared glass samples as the following points and table (1),

1. The band at the range about  $450 - 470 \text{ cm}^{-1}$  is assigned to the bending mode of Te–O–Te in TeO<sub>4</sub> structural units [16].

2. The band at 531 cm<sup>-1</sup> due to Zr–O vibrations of ZrO<sub>4</sub> units. The summary of various band positions of IR spectra is indicating growth of the band related to tetrahedral BO<sub>4</sub> units and ZrO<sub>4</sub> units at the expense of triangular BO<sub>3</sub> units and asymmetric groups with the increase of ZrO<sub>2</sub> content [17].

3. The bands located at 620 and 699 cm<sup>-1</sup> are assigned to Te–O–Te and O–Te–O linkages which represents the stretching mode of  $[TeO_4]$  trigonal pyramidal and the stretching mode of  $[TeO_3]$  trigonal pyramidal units respectively [17-19].

4. The peak at 700 cm<sup>-1</sup> due to bending vibrations of B-O-B linkages Zr–O–Zr vibrations of ZrO<sub>4</sub> units. [20]

5. The peak around 863 cm<sup>-1</sup> is attributed to the vibration of tri, Penta and diborate groups of  $BO_4$  tetrahedra [21].

6. The FTIR bands in the range 950–990 cm<sup>-1</sup> are noticed for all borate glass samples, attributed to stretching vibrations of B–O–B linkages [22].

7. Furthermore, the peaks detected in the second region at 1025 and 1050 cm<sup>-1</sup> are assigned to the B–O–B stretching vibration of BO<sub>4</sub> tetrahedral in penta-borate units [23].

8. The intense band at 1089 cm<sup>-1</sup> is assigned to the asymmetric stretching of BO<sub>4</sub> units in the BO<sub>4</sub>–O linkages [24].

9. The peaks at 1374 and 1262 cm<sup>-1</sup> in the first spectral region are attributed to O-B-O stretching of trigonal BO<sub>3</sub> units [23, 25].

10. An additional FTIR band between 1305 and 1340 cm<sup>-1</sup> is due to the asymmetric stretching vibration of BO<sub>3</sub> triangular units in various borate groups [26].

11. The bands located at 1420 and 1490 cm<sup>-1</sup> may be due to B-O stretching vibration in BO units metaborate, pyroborate, and orthoborate groups [27, 28].

12. The band centered around 1644  $\text{cm}^{-1}$  is due to the B – O stretching vibration of borate triangles with non-bridging oxygen (NBO) [29].

From the obtained IR results, boron ions appeared in both BO<sub>3</sub> and BO<sub>4</sub> structural groups, and some slight difference in the area under their IR bands are also observed. Since the N<sub>4</sub> function represents the relative ratio between the formed BO<sub>4</sub> to the total boron present in a glass sample, however it can be calculated according to equation (2), [30, 31].

$$N_4 = \frac{\text{The area under B04 bands}}{\text{Total area under all boron}} = \frac{[B04]}{[B03]+[B04]}$$
(2)

The area of each peak and its position, also its assignment is listed in Table (1), while Fig. (5) shows the relation between the calculated N<sub>4</sub> and ZrO<sub>2</sub> content (mol %). One can see that N<sub>4</sub> is decreasing markedly as ZrO<sub>2</sub> mol % was increased at fthe expense of TeO<sub>2</sub> in the structure of the glass system. It is well known that B<sub>2</sub>O<sub>3</sub> in glass network became BO<sub>3</sub> and BO<sub>4</sub> which in turn connected with each other to form B - O - B [29]. While TeO<sub>2</sub> in glass structure became in the form of Te – O – Te bond [31].



Fig. (5), The variation of N<sub>4</sub> ratio as a function of ZrO<sub>2</sub> content.

**Table (1),** The vibration absorption modes in the range from 400 cm<sup>-1</sup> to 1700 cm<sup>-1</sup>, and their relative area of each peak.

Sample code		0 mol % ZrO <sub>2</sub>	2 mol % ZrO <sub>2</sub>	4 mol % ZrO <sub>2</sub>	6 mol % ZrO <sub>2</sub>	8 mol % ZrO <sub>2</sub>
Wave number (cm <sup>-1</sup> ) for each peak	Peak 1	461.10	457.79	462.90	460.44	461.76
	Area	0.70	0.65	0.49	0.30	0.21
	Peak 2	-	530.03	533.39	528.04	530.42
	Area	-	0.45	0.57	1.20	2.43
	Peak 3	604.03	604.17	606.04	608.07	614.82
	Area	0.64	0.57	0.51	0.33	0.23
	Peak 4	696.61	696.36	706.32	704.51	714.62
	Area	5.42	5.19	4.64	2.58	1.81
	Peak 5	883.87	885.34	885.17	893.64	880.86
	Area	3.74	3.94	4.31	6.21	10.43
	Peak 6	941.78	955.81	950.68	955.20	953.96
	Area	11.30	12.24	14.72	13.73	14.34
	Peak 7	1057.65	1054.23	1057.07	1052.16	1050.19
	Area	2.66	2.49	2.07	1.51	1.29
	Peak 8	1169.55	1162.84	1152.62	1146.37	1140.09
	Area	3.00	2.87	2.18	1.16	0.51
	Peak 9	1272.15	1276.43	1262.60	1259.23	1276.03
	Area	3.60	4.06	6.46	7.34	9.70
	Peak 10	1363.96	1366.84	1362.55	1380.30	1360.53

Area	2.39	2.97	4.85	5.17	6.53
Peak 11	1473.92	1479.50	1456.88	1460.33	1461.35
Area	8.00	8.31	9.11	10.33	12.98
Peak 12	1617.14	1625.52	1635.41	1637.49	1669.46
Area	1.03	1.14	2.19	3.96	4.60

3.2. Optical properties results,

The UV–Vis. spectra are used for measuring the spectral transmission behavior of the studied glass samples. In Fig. (6), the optical transmission behavior versus the applied wavelength was drawn in the range of 190 to 1100 nm at room temperature. From Fig. (6), the optical absorption edge ( $\lambda_{cut-off}$ ) could be obtained from the point of intersection with  $\lambda$  axis. Table (2) showed the values of  $\lambda_{cut-off}$  for all the studied samples of different ZrO<sub>2</sub> content. As the absorption edge could be calculated using the strength of oxygen bond in the glass network [32] so, the increasing of ZrO<sub>2</sub> content causes more strengthening of all networks of the glasses. This confirms the finding of M. G. Moustafa et al. [33] about the increase of the strength of the network of the glass with the increase in Zr<sup>4+</sup> cations.



Fig. (6). The spectral transmittance behavior of the studied glass samples Vs. the applied wavelength ( $\lambda$ )

The absorption coefficient,  $\alpha$ , near the edge of each spectrum was calculated by equation (3), where (d) is the thickness,  $I_o$  and  $I_t$  are the intensities of the sample incident and transmitted radiations respectively.

$$\alpha = \frac{1}{d} \ln \left( \frac{I_0}{I_t} \right) \tag{3}$$

$$\alpha \, h\upsilon \,=\, \mathbf{B} \, (h\upsilon \,-\, E_{opt})^r \tag{4}$$

For amorphous materials, Mott and Davis have given equation (4) [33–35]. Where hu is the incident photon's energy, E<sub>opt</sub> is the optical band gap, B is a constant known as the band tailing parameter, and r is an index. The values (2, 3, 1/2, or 3/2) denote the corresponding indirect allowed, indirect forbidden, direct allowed, and direct forbidden transitions [35]. Although the latter incorporates phonon interactions, both of the band gaps of the aforementioned dependence are buried band transitions [36]. The associations between  $(\alpha h \omega)^{1/2}$  vs photon energy (h $\omega$ ) and ( $\alpha$ h $\omega$ )<sup>2</sup> versus photon energy (h $\omega$ ) were displayed in Figs. (7) and (8), respectively, to calculate the indirect or direct band gap. The value of optical band gap E<sub>opt</sub> for either direct or indirect band gap can be determined by extrapolating the linear parts of the curves [36], see Table (2). The direct and indirect band gap energies decreased from 3.17 eV to 2.95 eV and from 3.21 eV to 2.36 eV with increasing of ZrO<sub>2</sub> content. The disorder of the glass structure exhibits larger extension of the localized states within the gap according to Mott and Davies relation [37]. The generation of bridging oxygen and density fluctuation can both be responsible for the rise in the direct and indirect band gap energies [37]. The electrical flaws in the glass network may be the cause of the rise in both the direct and indirect band gap energy [38]. The Urbach rule is typically followed by the fundamental absorption edge, as in the following,



Fig. (7). Indirect band gap energy of glass samples.



Fig. (8). Direct band gap energy of glass samples.

Table (2), optical parameters of all the glass samples as a function of ZrO<sub>2</sub> content.

Optical parameters	0 mol %	2 mol %	4 mol %	6 mol %	8 mol %
	ZrO <sub>2</sub>				
Cut-off wavelength, $\lambda c$ (nm)	332	349	365	394	407
Optical band gap, direct Eg (eV)	3.22	3.10	2.96	2.87	2.63
Optical band gap, indirect Eg (eV)	3.23	2.98	2.71	2.39	2.27
Optical band gap by dielectric loss (eV)	3.20	3.03	2.81	2.69	2.49
Urbach energy (eV)	0.32	0.39	0.43	0.55	0.61
Linear refractive index, n	1.99	2.02	2.07	2.11	2.16
molar refraction (R <sub>m</sub> )	16.656	18.260	19.068	20.677	21.300
molar polarizability $a_m$	0.238	0.247	0.265	0.293	0.304
electronic polarizability $(\alpha_0^{-2})$	2.8746	3.177	3.335	3.639	3.7639
Linear susceptibility, c1 (esu)	0.76	0.68	0.57	0.51	0.42
<b>3rd non-linear susceptibility x10<sup>-11</sup></b> esu)	5.56	3.65	1.76	1.11	0.54
Non-linear refractive inde x 10 <sup>-10</sup> (esu)	10.51	6.8	3.2	1.98	0.94
optical basicisity A	1.31	1.34	1.36	1.39	1.41
optical basicisity $\Lambda_{th}$	0.753	0.749	0.745	0.741	0.737
metallization character	0.501	0.491	0.474	0.448	0.440

To calculate the width of the energy band tail (Urbach energy  $(\Delta E_U)$ ) of the density of states, the model proposed by Urbach can be followed [32] using equation (5), where  $\alpha_0$  is a constant,  $\Delta E_U$  is Urbach energy which represents the width of the band tails of the localized states and the degree of disorder.

$$\alpha = \alpha_0 e^{\left(\frac{h\nu}{\Delta E_u}\right)}$$
(5)

By drawing the relationship between  $(\ln \alpha)$  against  $(h\upsilon)$  for the studied samples, the Urbach energy  $(\Delta E_U)$  has been calculated from the reciprocal of the slope of the linear part of these curves [39]. The results were recorded in Table (2), it was found that there is variation in  $(\Delta E_U)$ as a function of ZrO<sub>2</sub> content. The origin of Urbach energy in amorphous materials may be the stronger broadening of absorption edge compared with crystals; this can be referred to the decrease of long-range order. Accordingly, Urbach energy decreases linearly with increasing ZrO<sub>2</sub>. The decrease in E<sub>opt</sub> and the increase in Urbach energy with ZrO<sub>2</sub> content, Table (2) may

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be due to the gradual increase of the degree of disorder in the studied samples [32, 34]. This may be a promising result that makes these glass samples could be used as energy storage material.

$$\frac{n^2 - 1}{n^2 + 2} = 1 - \sqrt{\frac{E_g}{20}} \tag{6}$$

Another crucial factor in the analysis of the optical characteristics of glass systems is the refractive index (n), which is employed to assess whether or not these glass samples are suitable for use in optical devices [41]. It is dependent upon the cationic polarizability [42] and oxides included in the glass sample, both of which are impacted by light's interaction with the atoms' electrons in glass [43]. Using Eq. (6) [44], the refractive index (n) was calculated based on the optical band gap. The refractive index values (n) are decreased with increasing ZrO<sub>2</sub> content for all the samples as illustrated in Table (2). This may be due to an increase in the ratio of NBOs to bridging oxygens that shown in the FTIR results [45, 46]. The increase of NBOs gives extra ionic bonds that exhibit large polarizability over covalent bonds of bridging oxygen.

$$R_m = \frac{n^2 - 1}{n^2 + 2} V_m \tag{7}$$

$$\alpha_m = \left(\frac{3}{4\pi N_A}\right) R_m \tag{8}$$

The molar refraction  $R_m$  in cm<sup>3</sup> for isotropic substance as glass can be determined by using Lorentz–Lorenz equation [47, 48], Where the factor  $(n^2-1)/(n^2+2)$  is called the refraction loss. The calculated values of  $R_m$  are listed in Table (2). The molar polarizability  $\alpha_m$  of a material is measured by Å<sup>3</sup> and is proportional to  $R_m$  which is accompanied by the structure of the glass sample through the following Clasius – Mosotti relation (Eq. 8) [49], where  $N_A$  is Avogadro's number. The behavior of  $\alpha_m$  with the gradual increase of ZrO<sub>2</sub> content is illustrated in Table (2). It can be seen that  $\alpha_m$  increases with the gradual increase of ZrO<sub>2</sub> content [50].

$$\alpha_{o^{-2}} = \frac{\left(\left(\frac{R_m}{2.52}\right) - \sum \alpha_{cat}\right)}{N_{O_2}} \tag{9}$$

The electronic polarizability  $(\alpha_0^{-2})$  of oxide ions is considered to be one of the essential properties of a substance, due to its utilization in the field of optics or electronics [51,52]. It can be computed based on the refractive index using eq. (9) [53], Where  $\sum \alpha_{cat}$  denotes the molar cation polarizability given by  $X_A \alpha_A + X_B \alpha_B + X_C \alpha_C + X_D \alpha_D$ ; X points out the molar fraction per each oxide, and  $N_{O_2}$  refers to the percentage of the number of the oxygen ions that exists in each component in the glass sample. The calculated values of  $(\alpha_0^{-2})$  are tabulated also in Table (2). The increase of ZrO<sub>2</sub> cations lead to a rise in the values of  $(\alpha_0^{-2})$  with a rise in (n). This result may be referred to the increasing number of NBOs in the glass system as increasing of the content of ZrO<sub>2</sub> [54, 55].

The optical basicity  $\Lambda$  is the rising ability of transport oxide ions to cations which leads to a rise in covalency of oxygen-cation bonding [56, 57]. So,  $\Lambda$  was used as a measurement of acid-

base property of the oxide glasses and is explained the electron density carried by oxygen [58]. Duffy founded an essential relation among ( $\alpha_0^{-2}$ ) and ( $\Lambda$ ) [58] as shown in Eq. (10);

$$\Lambda = 1.67 \left( 1 - \frac{1}{\alpha_0 - 2} \right) \tag{10}$$

Also, the theoretical optical basicity  $\Lambda_{th}$  can be calculated based on the following relation suggested by Duffy and Ingram [58] Eq. (11), where (x<sub>i</sub>) is the molar fraction of each oxide (i) and ( $\Lambda_i$ ) refers to the value of the basicity of such oxide i. [58].

$$\Lambda_{th} = \sum x_i \Lambda_i \tag{11}$$

The basicity of TeO<sub>2</sub>, B<sub>2</sub>O<sub>3</sub>, Na<sub>2</sub>O, and ZrO<sub>2</sub> are 0.33, 0.420, 1 and 1.18 respectively. The electronic polarizability values of the ions B, Na, Te, and Zr are 0.002, 0.175, 1.595 and 0.357 Å<sup>3</sup> respectively [58]. The computed optical basicity values of oxide ions are listed in Table (2). It is observed that the increase of ZrO<sub>2</sub> content causes a rise in the values of the optical basicity. This may be due to the rise of covalency force in the cation–oxygen bonding that referred to the rise of the negative charges on oxygen atoms. The behavior of ( $\Lambda$ ) for the studied glasses implies the higher capability of oxide ions to move the electrons to the surrounding cations [58]. As a conclusion of this part of study, it is observed that there is a common tendency of increasing electronic polarizability and basicity with the rising of the refractive index and reducing in the energy gap, which are considered an important results for different applications in the field of optoelectronics and energy storage.

#### 4. Conclusion

Archimedes technique was used to determine the experimental density value of each glass sample. Then the density was calculated according to Archimedes equation. The molar volume of each glass sample was calculated. The results of experimental density and the calculated molar volume showed that the samples density decreased, while the molar volume increased with increasing ZrO<sub>2</sub> content. The glass structure of the prepared samples were confirmed by XRD. The FTIR spectra showed that as Zr ions were increased the BO<sub>3</sub> changed to BO<sub>4</sub> then the BO<sub>4</sub> and the nonbridging oxygens increased. The optical parameters were obtained by measuring the transmission (T) spectra. The optical results that obtained from UV and Vis. region and, that calculated using different equations demonstrated that these glass samples are promising for different applications in optoelectronic and energy storge fields.

#### Declarations

- o Ethics approval and consent to participate, Not applicable
- o Consent for publication, Not applicable
- o Availability of data and materials, Not applicable

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o **Competing interests**, The authors have no relevant financial or non-financial interests to disclose.

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o **Authors' contributions,** All listed authors contributed to the study conception and design. Samples preparation, data collection and analysis were performed by prof. Mohamed yousry hassaan, dr. ayman abdel-karim bendary, dr. mohsen hasan abdel-wahed and dr. mirvat ragab atta. The first draft of the manuscript was written by dr. ahmed saber abdel-moety and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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