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Effect of gamma irradiation on thespectroscopic studies of CeO2-doped Zinc Phosphate Glass

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Abstract:*UV-visible and FTIR spectroscopic properties of some prepared undoped zinc phosphate and CeO2-doped glasses of the same composition were measured before and after successive gamma ray irradiation. The undoped zinc phosphateglass exhibits strong ultraviolet absorption, which is correlated with the presence of trace iron impurities within the raw materials used for the preparation of such glass.The undoped glass shows after gamma irradiation with 10.3 kGy/hr, the broadness of the UV absorption extending from* 200 to 339 nm *and permanence band centered at 728 nm. FOR 7CeO2-doped glasses produces absorption extending from 200- 354 nm and showing a kink at about 367 nm and existence visible band centered at about 728 nm, but the UV absorption remains unaffected with one peak at about 728 nm. Infrared absorbtion spectra of the prepared glasses reveal characteristic IR vibration due to characteristic phosphate groups according to glass composition and the effects of CeO2 or gamma irradiation are observed to be limited.*

Keywords *UV-visible; IR spectra;Zincphosphateglass;CeO$_2$; Gamma irradiation .*

1.INTRODUCTION

The zinc phosphate glasses are becoming more and more attractive because they can be prepared and easily modified with ZnO to obtain the depolypatemerized phosphate structures by exceeding the pyrophosphate composition limit. When zinc was further incorporated into the binary phosphate systems such as $BeO.P_2O_5$ and $MgO.P_2O_5$,the composition of metaphosphate (Q^2) chain and zinc coordination changes become complicated and show anomalous composition/properties behavior[1].The binary zinc ultra-phosphate glass, xZnO. (1-x)P2O5 with $0 \leq x \leq 0.5$and zinc polyphosphate glass, xZnO. (1-x)P2O5 with $0.5 \leq x \leq 0.71$, have been prepared by Brow et al.[2] and Meyer [3], respectively, and the structure and the distribution of Q^i-phsphate sites were investigated by Raman and NMR spectroscopy. Their results show that the addition of low ZnO content leads to the increase in Q^2-phosphate sites [2,3], and as x>o.6, Q^0tetrahedra is formed, i.e, chaine phosphate unit dominates the glass structure at lower ZnO content, and pyrophosphate (Q^1)and orthophosphate (Q^0)units become dominant in the glass network at higher ZnO composition [2]. Aluminum is one of the most widely used oxides which could play an intermediate role between glass former and glass modifier in the glass structure due to three different coordination numbers[4]. The formation of P-O-Al chains in $Al(OP)_4$ plays as the glass former, while excess Al^{3+} ions lead to $Al(OP)_6$ and play as the glass modifier. Al_2O_3 also shows high solubility with rare earth ions. In addition, it has been shown that the addition of Al_2O_3 and ZnO mixed in the phosphate glasses may produce $Al(OP)_4$ and $Zn(OP)_4$ in the glass network to increase the number of the bridiging oxygen (BO), thus the chemical durability can be enhanced [5,6]. Phosphate glasses enjoy a range of compositional and structural possibilities (ultra, meta, pyro, and ortho) that facilitate tailoring chemical and physical properties of interest for specific technological applications [7]. The introduction of transition metal oxides, lead oxide as well as alumina can provide a much higher chemical durability to phosphate glasses. Phosphate glasses can accept high percentages of transition metal ions or rare earth ions and remain clear with brilliant colors and it is known that the phosphate glasses favor the lower oxidation or octahedral coordination states of transition metal or rare earth ions [8,9]. The lanthanides ions are considered to be well shielded owing to the screening of the 4f- electrons by the outer $5s^2 5p^6$electrons. However, cerium as one from the lighter group of lanthanides can be present in glass in the trivalent and tetravalent states and the ratio of each depends on the cerium concentration, the condition of melting and the type and composition of glass involved. In alkali borate and alkli silicate glasses, cerium ions appear mainly as Ce^{4+} ($4f^0$) ions having a broad absorption band at 260 nm which belongs to the character of charge transfers and has no fluorescence[10]. In sodium phosphate glass, Ce^{3+} ($4f^1$) ions are dominantly present and assumed to possess numerous UV peaks at 290, 250, 228, 208, and 192 nm which are assigned to $4f \rightarrow 5d$ transitions of Ce^{3+} [10,11]. These bands are assumed [11,12] to be moderately broad and intense, as expected from electric dipole allowed $4f \rightarrow 5d$ transitions. The occurrence of more than one Ce^{3+} absorption band in the region between 25,ooo cm^{-1} (400 nm) and 50,000 cm^{-1}(200 nm) is assumed to be due to the crystal field splitting of the 5d(2p) state [13]. The sharper splitting of the d-orbitals in phosphate glasses indicates that the asymmetric part of the crystal field is higher in phosphate anions. Blait and Duffy [14] have also observed a broad absorption band in the visible region peaking at 666 nm. The present work reports UV-visible and and FT infrared absorption spectra of undoped and and CeO2-doped Zinc-Aluminum Phosphate Glass. The same spectral measurements were repeated to the same samples after successive gamma irradiation. This study aims to obtain detailed spectral properties of cerium ions in a host Zinc-Aluminum Phosphate Glass and to deduce the induced defects generated by gamma irradiation.

2.Experimental details

2.1.Glass preparation:

Targeting glasses have the nominal molar compositions [$(35-x)ZnO:5Al_2O_3$: $60P_2O_5:xCeO_2$] where x takes the values (x=0, 7 mol%). The above constituents in given proportions were melted in band crucible furnace to obtain the host ZAP glasses. The batches were first preheated to 800 ^{0}C to remove H2O with a heating rate of 10 ^{0}C/min. The above materials were then melted for 2h in air ambient at temperatures 1100 ^{0}C depending on the glass compositions. Square samples with dimension of 1mm×1mm×0.7mm .

This work reports experimental results of an effort undertaken to identify and characterize the radiation-induced defects created during gamma (γ)irradiation of Zinc Phosphate Glass doped with the Cerium Ionson the basis of optical absorption measurements performed before and after irradiation. It is assumed that the induced absorption band formed after γ-irradiation of Zinc – Aluminum Phosphate Glass (ZAPG) glasses are created by some hole-type color centers related with non-bridging oxygen ions (NBO) located in different surrounding.

2.2.X-ray diffraction (XRD):

X-ray diffraction studies were carried out by using TOE STADIP, Diffractometer: Transmission, Generator: 40kV/30 mA, Radiation: λ [Cu $K\alpha_1$] = 0.154060 nm, Scan mode: Transmission, Scan type 2θ= 15-85^0 ,$\Delta 2\theta$= 0.02^0 , PSD step=0.50 (Linear PSD), Time/step=30 sec, sample with spinner, largest slit (6mm).

2.3.UV-visible absorption measurements:

Ultraviolet and visible absorption spectra were measured for perfectly polished glasses of equal thickness (0.799 cm± 0.1cm) before and after each gamma dose of irradiation using a recording double beam spectrophotometer (type JASCO corp. v-630, Rel-00Japan) covering the range from 200 to 1100nm.

2.4. Infrared absorption measurements:

The infrared absorption spectra of undoped and CeO$_2$ –doped Zinc Phosphate Glass were measured at room temperature in the wave number rang 400-4000 cm^{-1}. The prepared glasses were used in the form of fine powders, which were mixed with KBr, in the ratio 1:100, respectively. The weighed mixture was then subjected to a pressure of 5 tons/cm^2 to produce clear homogenous discs.

2.5.Gamma irradiation facility :

A^{60}CO gamma cell was used as γ-ray source with a dose rate 10.30Gy/hrat room temperature in air. The samples were placed in the dosimetric point corresponding to the dose rate of 10.30Gy/h and each sample was subjected to same sequence of total absorbed doses.

3.RESULTS

3.1. X-ray diffraction:

x-ray diffraction patterns (XRD) of the studied glasses showed a broad halo pattern, which is the characteristic of amorphous nature and XRD pattern of samples are shown in Fig.1.

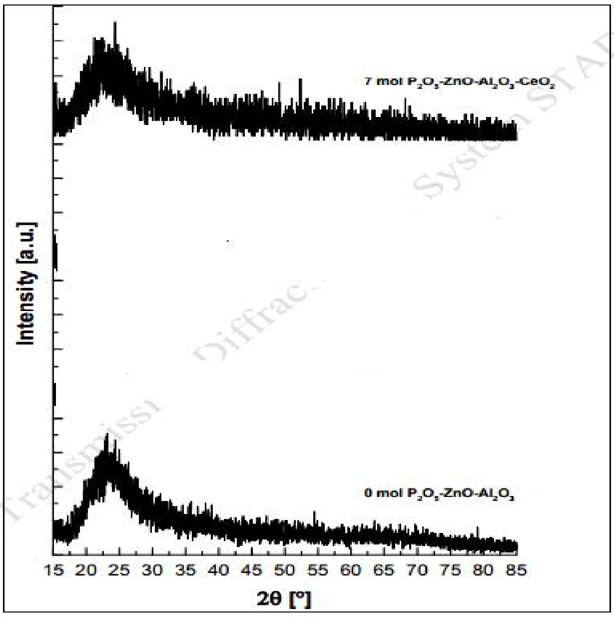

Figure 1. Typical XRD pattern for glass samples

3.2.Ultraviolet –visible absorption spectra of undoped and CeO$_2$-dopedZinc – Aluminum Phosphate Glass(ZAP)and Effect of gamma irradiation on the UV-visible absorption spectra :

Fig.2 illustrates the optical absorption of the base undopedzinc phosphate glass before and after gamma irradiation . The spectrum shows band edge broadabsorptionextending from 200 to 339 nm and visible band is observed at 728 nm. The base glass after irradiationwith a dose of 1.044Gy/hr shows an extra inducedband edge centered at about 351nm and permanence band centered at 728 nm and increase absorption.

Fig.3 illustrates the optical absorption spectra of the CeO2-doped sample after gamma irradiation with a dose of 1.044Gy/hr and 10.3kGy/hr.The glass contatinig 7%CeO2 shows strong ultraviolet absorption extending from 200 to about 354 nm and showing a kink at about 367 nm and existence visible band centered at about 728 nm. The low gamma irradiated 7%CeO_2 glass exhibits a broad UV absorption extending from 200 to about 364 nm and showing a kink at about 377 nm and existence visible band centered at about 728nm. The high gamma irradiated 7%CeO_2 glass exhibits a broad UV absorption extending from 200 to about 370 nm and showing a kink at about 396 nm and existence visible band centered at about 728nm.

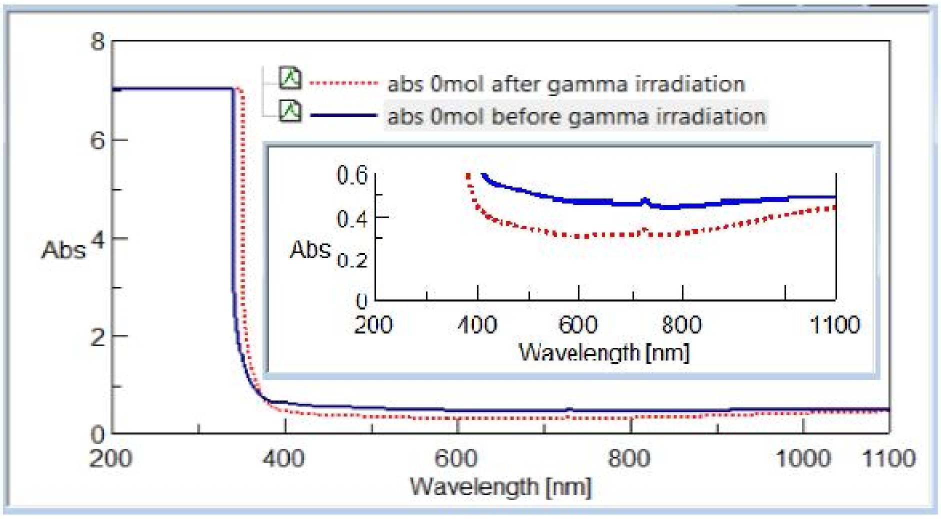

Figure 2. UV-visible absorption spectra of undoped(ZAP) glass before and after gamma irradiation

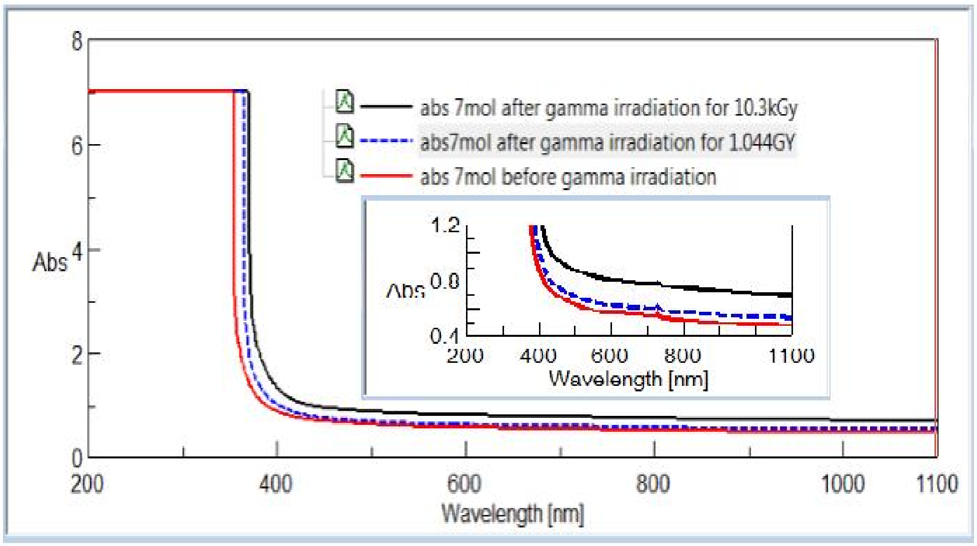

Figure 3.UV-visible absorption spectra of 7%CeO_2-doped (ZAP) glass before and after gamma irradiation

3.3. Infrared transmittance spectra of the Studied Glasses:

Figure 4 illustrates the FTIR spectra of the undoped and doped (ZAP) glass which reveal almost repetitive spectral vibrational bands with some limited variations. The IR spectrum of the base undoped glass shows the following spectral features:

(a) a small band is observed at about 410 cm^{-1}
(b) a small band is observed at about 481 cm^{-1}
(c) band centered at about 789 cm^{-1}
(d)a first strong broad with two identified peaks at 920 and 1087 cm^{-1}
(e)a broad band at 1271 cm^{-1} and small peak is observed at 2335 cm^{-1}
(f) a small peak is at 2926 cm^{-1} and a broad near IR medium band centered at 3414 cm^{-1}

Figure 4 also revels that all the glasses containing Ce ions exhibit the same fundamental and characteristic IR transmittance bands observed in the spectrum of the undoped glass.The intensities of all the IR bands are observed to slightly decrease except for two specific bands at about 2332 cm^{-1} and 3451 cm^{-1}. The most bands are retained in them position except the final broad near IR band centered around from 3414 to 3451 cm^{-1} is due to bending and stretching vibrations of contaminated water having no role in glass structure.

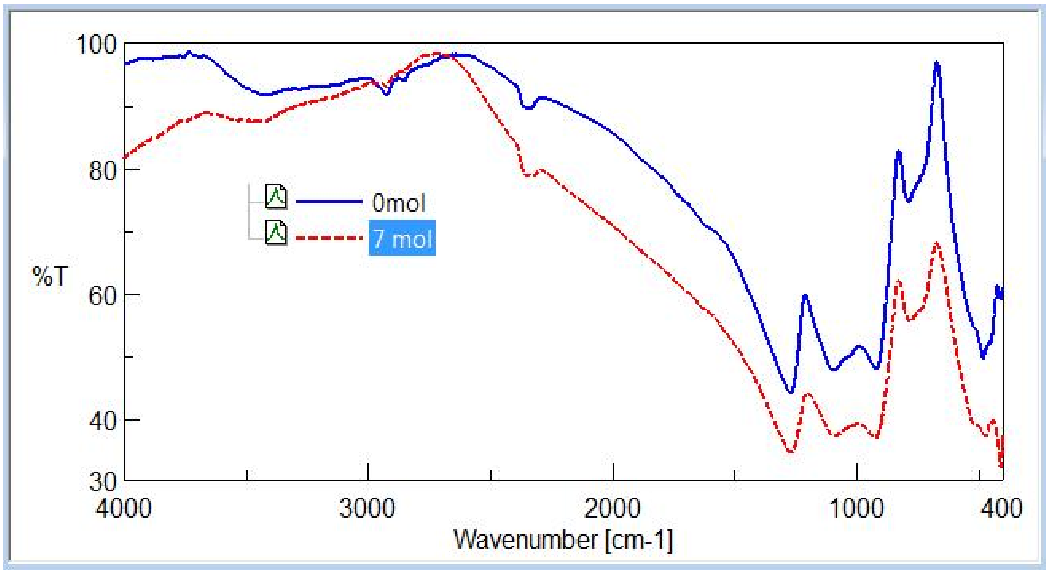

Figure 4. FTIR infrared transmittance spectra of undoped and CeO$_2$-doped (ZAP) glass before gamma irradiation

3.4 Infrared Transmittance Spectra after Gamma Irradiation:

Figure 5 illustrates FTIR spectra of the studied glasses after being subjected to gamma irradiation with a dose of 10.3kGy/hr for sample 7mol.Inspection of the IR spectral curves indicates that the main characteristic bands remain almost unaffected except by minor or limited changes of the intensities of bands. The specific new bands at 464-484-793 cm^{-1} for 7% CeO$_2$-doped zinc – aluminum phosphate glass after gamma irradiation.

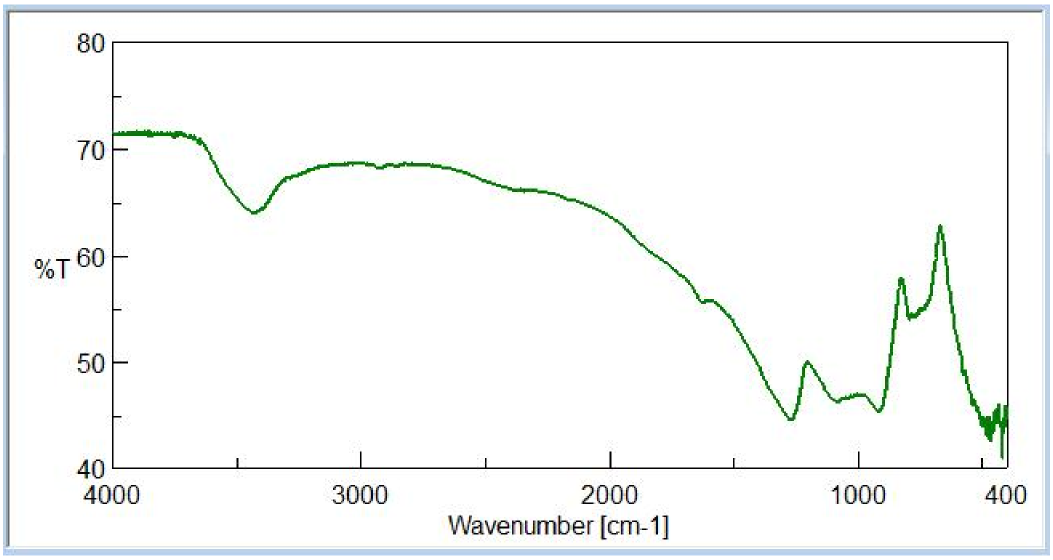

Figure 5. FTIR infrared transmittance spectra of CeO_2-doped zinc – aluminum phosphate glass after gamma irradiation

4.DISCUSSION

4.1. Interpretation about the origin of UV absorption in the undoped base zinc – aluminum phosphate glass

Sigel and Ginther [15] and cook and Mader [16] have separately observed ultraviolet absorption bands in some commercial glasses and they have related such UV absorption bands to originate from the presence of unavoidable trace iron impurities within the raw materials used for the preparation of such commercial glasses.

Duffy and Ingram [17] and Duffy [18] have arranged and characterized differently originated ultraviolet absorption spectra usually observed in various glasses. Some transition metal ions (e.g. Fe^{3+}, Cr^{6+}) in glasses exhibit characteristic charge transfer UV spectra which are related to an electron transfer mechanism. But certain other metal ions including Ce^{3+}, Th^{3+}, U^{4+} as well as $d^{10}s^2$ ions (such as pb^{2+} and Bi^{3+}) absorbed radiation through electronic transitions involving orbitals essentially of the metal ion only, and the name "Rydberg" has been suggested for such spectra to distinguish them from the common charge electron transfer spectra.

4.2. Interpretation of the visible absorption of of cerium ions in zinc phosphate glass

It is accepted that cerium ions can exist in glasses [19,20] in two valences, namely Ce^{3+} and Ce^{4+} ions. Trivalent cerium ($4f^1$)ions possess parity- allowed transitions between 4f and 5d electronic configuration. The energy separation from the lowest 5d levels to the levels of 4f are very large. Because of the large energy gaps, non-radiative decay is less probable and 5d fluorescence can be observed [21]. On the other hand, in the case of tetravalent cerium ($4d^0$), the absorption belongs to the character of charge transfer and has no luminescence [22]. Weber [19] shows that Ce^{3+} in silicate glasses absorb at 320nm and emits at 450nm with a decay time of 120ns at room temperature, whereas Ce^{4+} ions show a broad absorption spectrum due to charge transfer band ($O^2 \rightarrow Ce^{4+}$) in the UV range and with no fluorescence .A number of authors have attempted to explain the appearance of the visible absorption of ofCe-containing glasses and have proposed that the cause of its appearance is associated with the formation of clusters with exchange interaction between the cerium ions in the cluster and some impurity [23]. Such type of absorption spectra in cerium –glasses was interpreted by Diekeand Crosswhite [24] to be due to inter-configurational transition of doubly charged cerium.

4.3.Effect of gamma irradiation on the optical absorption spectra

4.3.1. Interpretation of gamma irradiation on the optical spectra of undoped (ZAP) glass

The gamma-ray irradiation on glasses creates secondary electrons from the sites where they are in a stable state and have an excess energy. When glass is exposed to high-energy radiation, a number of defects can be formed [25]. These defects are basically formed due to trapped electrons or holes in either the pre-existing sites or voids of glass created by bond breaking due to irradiation [26]. Three basic processes are proposed by El-Batal: (a) radiolytic processes, (b) atomic displacement and ionization and (c) charge trapping or elecrtron rearrangements [27]. The properties of glasses are usually subjected to a variety of changes under the influence of gamma ray irradiation due to partial rupture of chemical bonds or destruction of the network as well as introduction of defects. It has been repoted that defects in glasses are produced in pairs of positive hole centers (HC) and negative electron centers (EC) [28]. The electronic transitions of these defects regularly cause high absorbance in the UV and visible region. Cerium readily changes its oxidation state from Ce^{3+} to Ce4+ and vice versa. Cerium ion has been used earlier as a photosensitive or as defect-Scavenger in order to suppress the photo-ionization in glasses in the visible range [29,30]. Shows the optical absorption spectra undoped (ZAP) glass and doped (ZAP) glass after gamma ray exposure. It is observed that band edge of the glasses has been progressively shifting towards the longer wavelength after irradiation and band edge is more pronounced in visible region. The influence of irradiation has also been explained by the following reaction $Ce^{3+} + HC \rightarrow Ce^{4+}$ and $Ce^{4+} + EC \rightarrow Ce^{3+}$, where HC is the hole center captured by the cations and EC is the electron center captured by the anions [25,26,31]. The continuous shifting of band edge in these glasses confirms that $Ce^{3+} + HC \rightarrow Ce^{4+}$reaction .When Ce3+ is oxidized to Ce4+, it loses its 4f electron and this ion has no definite peak, it has also been confirmed by some studied that the presence of Ce3+ in glass network plays the role of positive radiation protection. The properties of glasses usually exhibit variety of changes under the exposure to gamma rays which is due to breakage of chemical bonds and as well as introduction of defects. In glasses are generated in pairs of negative electron centers (EC) and positive hole centers (HC). As the absorption of EC occurs in the ultraviolet part and the absorption of HC in the visible region [32]. Increase in the absorption of the glasses in the visible region is attributed to the hole center.

4.4. Interpretation of the infrared transmittance spectroscopic data

Previous extensive studies on the IR spectra of phosphate glasses have arrived to several important parameters concerning interrelation between IR spectra and glass constitution [33,34].

(a) The network structure of phosphate glasses is accepted to contain a polymeric arrangement of phosphate groups with variable lengths depending on the other partners and ratios of the various constituents.
(b) Like silicate glasses, the phosphate glasses are constructed from corner-sharing tetrahedral, with the important difference that these are three-fold rather than four-fold coordinated.
(c) Recent neutron diffraction studies of these glasses revealed that vitreous P_2O_5 contains two types of P-O bonds, i.e. P-TO (TO = terminal oxygen bond) or P-NBO (NBO bond) and P-BO (BO=bridging oxygen bond). The former is slightly shorter than the latter. Infrared and Raman spectroscopic studies carried out on vitreous P_2O_5, also distinguish between bridging and terminal oxygen atoms.Similar to binary silicate glasses, addition of modifying oxides to vitreous P_2O_5 results in the conversion of BO to NBO.

(d) In recent years there has been much research on improving the physical properties and chemical durability of phosphate glasses by introducing intermediate type metal oxides (MO) like AL_2O_3, Fe_2O_3, TiO_2, ZnO, which generate additional O-M-O type cross-linkages among different polyphosphate anions.
(e) Most research studies on the vibrational spectroscopy of phosphate glasses treat information on their structure obtained in terms of the oxygen bridges and terminal groups rather than in terms of polyhedral, or Q^n species. Notably, it is the (PO_2), (PO_3) and (PO_4) groups.

From previous IR studies on phosphate glasses and related crystalline phosphates, the present experimental IR spectrum of zinc phosphate glass can be interpreted as follows:
(a) It is reported [35] that the band at 410 $cm^{-1}$is assigned to the bending vibrations of O-Al-O
(b) It is reported [33, 36, 37] that the band at 475-480cm^{-1} is assigned to the bending vibrations of O-P-O
(c) It is reported [38] that the band at 780 cm^{-1} is due to asymmetric stretching vibrations of P-O-P bonding, denoted as $(POP)_S$
(e) It is reported [39] that the band at 920 cm^{-1}can be attributed to antisymmetric and symmetric stretching modes of the P-O-P bonds in the chains.
(f) It is reported [40] that the band at 1090 $cm^{-1}$is attributed to the asymmetric stretching of V_{as} (P-O-P) modes linked with small metaphosphate rings.
(g) It is reported [41] that the band at 1270 $cm^{-1}$is assigned to the asymmetric stretching vibrations of O-P-O
(h) It is reported [42] that the band at 2335 cm^{-1} which correspons to the stretching vibration mode of OH- groups in several oxide glasses.
(i)It is reported [41] that the band at 2925 $cm^{-1}$is assigned to the stretching vibration of the P-O-H
(k)It is reported [43] that the band at 3450 cm^{-1} which is related to water and OH vibrations.

The effect of increasing CeO_2 content is observed to cause some changes in the intensities of the IR bands. It seems that the increase of CeO_2 contents causes some polymerization in the phosphate network leading to the dominance of the metaphosphate groups.

4.5. Interpretation of the effect of gamma irradiation on the IR spectral data

Carful inspection of the IR spectra after irradiation indicates that the overall relative intensities of most bands increase than before irradiation. These observations can be interpreted on the following basis:

(1) The slight decrease of the bands can be related to the possible more randomness of the network or the disruption or changes of some bond angles or bond lengths by irradiation.

(2) Early primak [44] and followed by Hobbs et al. [45] have assumed the generation of defects or breaking of the connectivity of the network by changing in the bond angles and/or bond length of the structural building groups.

(3) Later, Piao et al. [46] have assumed that during irradiation, the ionization processes produce electron- hole pairs, providing paths for bond rearrangements.

(4) It is observed that the main metaphosphate bands (≈1270 cm-1) remain unaffected and the irradiation is affecting other subsidiary groups and even increasing the intensities of the vibrations due to weakly bonded surface water or OH groups.

(5) On the concentration of CeO_2 (7%) the mentioned IR bands are observed to be quite identified and are no affected by irradiation. This can be related to some shielding effect of Ce ions present in increasing content (7%) sample. While the band at 790 cm^{-1} is attributed to the sym-metric stretching

vibrations (P-O-P)rings [36,37] and band at 485cm^{-1} is attributed to bending vibration. The band at 464 cm^{-1} is attributed to bending vibrations of bridging phosphorus, δ(O-P-O) and/or δ(P=O) [47].

5.CONCLUSION

Ultraviolet –visible measurements of the base zinc – aluminum phosphate glass show strong UV absorption which is attributed to the presence of trace iron impurities contaminated within the raw materials for glass preparation. CeO_2-doped samples show distinct visible absorption bands which are related to the presence of Ce^{3+} or Ce^{4+} ions in distorted octahedral symmetry. Gamma irradiation produces induced defects which are related to electron defects within the UV region and positive hole defects in the visible region. It is observed that the presence of CeO_2 causes some shielding effects towards successive gamma irradiation.

Infrared absorption spectra show characteristic IR bands due to phosphate groups in accordance with the limit of glass composition studied. The presence of CeO_2 in glasses causes no effect on the number and position of the IR bands. Gamma irradiation causes minor changes in the intensities of IR spectra, which are correlated with the changes in bond angles and/or bond lengths for the structural phosphate groups together with the shielding effect due to the presence of CeO_2.

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