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# UV–visible and infrared absorption spectra of gamma irradiated CuO-doped lithium phosphate, lead phosphate and zinc phosphate glasses: A comparative study

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ABSTRACT

Undoped and CuO-doped lithium phosphate, lead phosphate and zinc phosphate glasses were prepared. UV–visible and infrared absorption spectra of the prepared samples were measured before and after successive gamma irradiation. Experimental optical spectra of the undoped samples reveal strong UV absorption bands, which are attributed to the presence of trace iron impurities in both the lithium and zinc phosphate glasses while the lead phosphate glass exhibits broad UV bands due to combined absorption of trace iron impurities and divalent lead ions. The CuO-doped glasses reveal an extra broad visible band due to $Cu^{2+}$ ions in octahedral coordination. The effects of gamma irradiation have been analyzed for both the sharing of all constituent components including trace iron impurities. Infrared absorption spectra of the prepared samples were investigated by the KBr disk technique. The FTIR spectra reveal main characteristic absorption bands due to different phosphate groups. The IR spectra are observed to be slightly affected by the increase of CuO in the doping level (0.2–3%) indicating the stability of the main network units.

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## 1. Introduction

Phosphate glasses usually possess low melting temperatures, high thermal expansion coefficients ($\alpha$), low glass transition temperatures ($T_g$), low softening temperatures ($T_s$), and are of increasing interest for many applications, e.g. glass to metal seals, low temperature enamels for metals, biomaterials, fertilizer agriglass and even for immobilization of radioactive wastes [1–7]. The poor chemical durability data of alkali and alkaline earth phosphate glasses have been improved by the addition of one or more of multivalent oxides:$Al_2O_3$, PbO, ZnO, SnO and $Fe_2O_3$, etc., which results in the formation of newer bonds such as Pb–O–P, Al–O–P, Zn–O–P, Sn–O–P, Fe–O–P, which increase the chemical durability to a measurable extent.

Oxide glasses containing transition metal ions have been shown to exhibit interesting spectroscopic and electrical properties [8,9]. In various glasses, copper can exist as divalent $Cu^{2+}$ ions, monovalent $Cu^{+}$ ions and metallic copper ($Cu^0$) although most glasses melted under ordinary atmospheric conditions are usually assumed not to contain metallic copper [10–14]. Colors produced by $Cu^{2+}$ ions have been investigated and interpreted from the view of ligand field theory [8,10–14].

In oxide glasses, the transition metal ions form coordination complexes with double charged oxygens as the ligands are usually in 4-fold or 6-fold coordination. As a general rule, octahedral symmetry is favored over tetrahedral; but other factors, such as ionic radius, polarizability of the transition metal (TM) ion and Jahn–Teller distortion can modify the spectral results. The actual symmetry taken up will also depend on glass composition. In phosphate glasses, transition metal ions are shown to exist in a reasonable concentration of the reduced state or octahedral coordination [8,11–17].

Gamma irradiation of glasses has been shown to give rise to induced optical absorption bands [18,19]. The induced defects generated by gamma irradiation virtually originate from intrinsic defects within the glass matrix itself and from the sharing of extrinsic defects due to dopants or impurities [20,21].

The main objective of this work is to investigate the UV–visible optical absorption spectra of copper ions in the three specific and different phosphate glasses, namely lithium phosphate, lead phosphate, zinc phosphate before and after gamma irradiation. A second objective of the present work is to measure the FT infrared absorption spectra of the prepared undoped and CuO-doped samples before and after being subjected to a high gamma dose of 8 Mrad ($8 \times 10^4$ Gy).

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## 2. Materials and methods

### 2.1. Preparation of glasses

Copper-doped glasses of varying basic hosts including lithium phosphate, lead phosphate and zinc phosphate (Table 1) of the nominal composition 50 mol% of each constituent oxide were prepared. Reagent grade $Li_2CO_3$, $Pb_3O_4$, ZnO, $NH_4H_2PO_4$ and CuO were used as raw materials. In porcelain crucibles with lids, the batches were calcined at 500 °C for 30 min to remove $NH_3$ and $H_2O$; the furnace was then raised to 1000–1100 °C for 1 h. The melts were poured into warmed stainless steel molds for the required dimensions. The prepared samples were immediately transferred to an annealing muffle furnace adjusted at 350 °C for lithium phosphate and zinc phosphate samples and at 250 °C for lead phosphate glasses. The annealing muffle furnace was left to slowly cool to room temperature after 1 h at a rate 30° C/h.

### 2.2. UV–visible absorption measurements

Optical (UV–visible) absorption spectral measurements for the undoped and CuO-doped samples within the range 200–1100 nm were carried out using a recording double beam spectrophotometer (Jasco Corp., V-570, Rel.60, Japan) for samples of equal thickness ($2 \pm 0.1$ mm) before and after each successive gamma irradiation.

### 2.3. Infrared absorption spectra measurements

Fourier-transform infrared (FTIR) spectra of the prepared glasses were obtained with an FTIR spectrometer (type Mattson 5000, USA). Powdered glass samples (2 mg) were mixed with KBr powders (200 mg) and pressed to 5 tons/cm$^2$ to form thin transparent disks whose IR spectra could be observed. Infrared absorption spectra within the range 400–4000 cm$^{-1}$ were recorded at room temperature.

### 2.4. Irradiation facility

A $^{60}$Co gamma cell (2000 Ci) was used as a gamma ray source with a dose rate of 1.5 Gy (150 rad/s) at a temperature of 30 °C. The investigated glass samples were subjected to the same gamma dose every time. Using a Fricke dosimeter, the observed dose in water was utilized in terms of dose in glass. No cavity theory correction was made. Each glass sample was subjected to a total final dose of $8 \times 10^4$ Gy ($=8$ Mrad).

**Table 1**
Chemical Composition of the prepared glasses.

| Sample | $P_2O_5$ (mol%) | $Na_2O$ (mol%) | PbO (mol%) | ZnO (mol%) | CuO added (wt%) | $Al_2O_3$ added (wt%) |
|---|---|---|---|---|---|---|
| G1 | 50 | 50 | | | 0.2 | 0.5 |
| G2 | 50 | 50 | | | 0.5 | 0.5 |
| G3 | 50 | 50 | | | 1.0 | 0.5 |
| G4 | 50 | 50 | | | 3.0 | 0.5 |
| G5 | 50 | | 50 | | 0.2 | 0.5 |
| G6 | 50 | | 50 | | 0.5 | 0.5 |
| G7 | 50 | | 50 | | 1.0 | 0.5 |
| G8 | 50 | | 50 | | 3.0 | 0.5 |
| G9 | 50 | | | 50 | 0.2 | 0.5 |
| G10 | 50 | | | 50 | 0.5 | 0.5 |
| G11 | 50 | | | 50 | 1.0 | 0.5 |
| G12 | 50 | | | 50 | 3.0 | 0.5 |

## 3. Results

### 3.1. UV–visible absorption spectra of undoped lithium phosphate, lead phosphate and zinc phosphate glasses

Fig. 1(a) illustrates the UV–visible absorption spectrum of undoped lithium phosphate glass, which reveals strong and broad UV absorption band centered at about 235 nm with two connected minor peaks at about 220 and 250 nm and no visible bands could be observed. On subjecting this glass to a total gamma dose of 8 Mrad, the UV spectrum reveals an intense broad absorption extending from 200 to about 390 nm with three small peaks at about 230, 250 and 280 nm followed by a broad asymmetrical visible band centered at about 500 nm.

Fig. 1(b) reveals the UV–visible absorption spectrum of undoped lead phosphate glass showing a strong and broad ultraviolet absorption comprising two peaks at about 230 and 275 nm and followed by an obvious small peak at about 340 nm but with no obvious visible band. On subjecting the parent undoped lead phosphate glass to a total gamma dose of 8 Mrad, the UV spectrum reveals a broad prominent band extending from 200 to about 350 nm and showing three small peaks at about 230, 250, 280 nm and a slight curvature at about 340 nm and no visible bands are observed.

Fig. 1(c) shows the UV–visible spectrum of undoped zinc phosphate glass revealing a prominent ultraviolet quite broad band centered at about 235 nm with subsidiary closely related small kinks at about 225 and 250 nm and with no visible bands. On subjecting this glass to a gamma dose of 8 Mrad, the UV spectrum exhibits a broad and strong absorption extending from 200 to about 400 nm with three small peaks at about 230, 255 and 290 nm and with a shoulder at about 345 nm. The spectrum reveals no visible bands.

### 3.2. UV–visible absorption spectra of CuO-doped glasses

Fig. 2a–c illustrates the optical absorption spectra of CuO-doped three different phosphate glasses and the spectral data can be summarized as follows:

a. The CuO-doped lithium phosphate glass with 0.2% CuO shows a spectrum consisting of a high intense UV absorption with two peaks at about 220 and 275 nm followed by an unsymmetrical very broad visible band centered at about 820 nm. On increasing the CuO content to 0.5%, the UV absorption of this sample remains the same with very close intensity while the broad visible band exhibits high intensity with splitting to two peaks at about 780 and 825 nm. On further increasing the CuO content to 1%, the UV absorption remains the same as with 0.2% and 0.5% CuO and the visible broad band exhibits still higher intensity and the two peaks shifts the first band to about 760 nm and the second band to about 980 nm. With the last 3% CuO sample, the UV absorption slightly increases in intensity but with the same peaks position but the broad visible band shows still higher intensity but assumes two segment, the first segment reveals two peaks at about 650 and 770 nm and the second segment is broader with three peaks at about 1080, 1200 and 1300 nm.
b. The CuO-doped lead phosphate glass containing 0.2% CuO exhibits in general a spectrum similar to that for the undoped lithium phosphate glass except for two differences. The first difference is the resolution of a further UV band at about 360 nm and the second difference is that the broad visible band shows splitting to three peaks at about 820, 1120 and at 1350 nm in the last concentration (3% CuO) sample.
c. The CuO-doped zinc phosphate glass containing 0.2% CuO shows the same strong UV absorption as observed with the

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