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Fourier Transform Infrared and Electron Paramagnetic Resonance Spectral Studies of V2O5 Ion Doped Zinc –Lead- Lithium- Phosphate (ZNPBLI) Glasses

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Abstract

A glass composition $(P_2O_5)_{65} – (Pb_3O_4)_{10} – (ZnO)_5– (Li_2CO_3)_{20-x}$ - $(V_2O_5)_x$where x = 0, 0.2, 0.4, 0.6, 0.8 and 1 mol % is used to prepare vanadium ion dopedZnPbLiPglass samples V0 to V5 by melt quench technique method. The glass samples are non crystalline in nature, which is confirmedthrough X-ray diffraction. The physical properties like density and refractive index values are evaluated by using the Archimedes method and Abbe's Refract meter. The prepared glass samples are having differentphysical property valueswhen compared toother phosphate glasses and the values are found to be varied with respect to their composition. Fourier transform infrared (FT-IR) spectra exhibits few sharp and broad absorption bands which attributes to P-O-V bonds harmonicat 467cm^{-1},P-O-P symmetric stretches at 781 cm^{-1} and P-O-P asymmetric stretches at 880 cm^{-1}vibrations. Electron paramagnetic Resonance (EPR) spectra exhibits resonance signal at g~4.6612 and ~1.9603due to coupled pairs and Free State of vanadium ions. It is evident from the Spin-Hamiltonian Parameters (SHP) that the Vanadium ions are available in the sites ofoctahedral with tetrahedral composition and which is a part of o C_{4v}symmetry. These studies will be

helpful in understanding optical and dielectric properties of the prepared glasses.

Key words: Vanadium ion, phosphate glasses, Refractive index, Absorption bands, ` Spin-Hamiltonian parameters.

Introduction

Phosphate glasses usually exhibit high thermal expansion, transmission gain coefficient, dispersion, refractive index and wider band width capacity.[1] These glasses offer advantages in many applications such as nuclear waste storage glass, optical material molding, technical, biological, semiconducting applications and solid-state amorphous electrolytes in secondary batteries.[2-4]These glasses offer advantages in many applications such as nuclear waste storage glass, optical material molding, technical, biological, semiconducting and electrolytic applications in secondary batteries.[2-4]Current trendsshow that the addition of metal oxides with high valence cations like V_2O_5 increase the glass transition temperature and oppose aqueous corrosion also. The glasses doped with V_2O_5 are more beneficial as they exhibit semiconducting behavior due to electron hopping between V^{4+} and V^{5+} ions which leads to enhancementof conductivity.[5] The glasses mixed with Pb_3O_4 are expected to become highly stable against diversification andremain chemically inert.[6]The thermal stability of the oxide glassescan be enhanced by the addition of Li_2CO_3 due to the increase in NBO bonding and ZnO increases the chemical durability which promote the glasses as promising materials for photonic andoptoelectronic applications.[7,8]Earlier several investigations were conducted on Phosphate glasses by using Oxides in their composition but none of the researchers had made an attempt to study the physical, morphological and optical studies of Phosphate glasses from the simultaneous variation of Li_2CO_3 and V_2O_5 so far, further results have been viewed with respect to variation in the trend (increase/ decrease) of physical properties by virtue of dopent composition of matrix as cited in the references.[8,18]The physical, optical and morphological studies of V_2O_5 doped Zinc Lead Lithium Phosphate glasses are carried out for the composition $(P_2O_5)_{65}$ –$(Pb_3O_4)_{10}$ – $(ZnO)_5$– $(Li_2CO_3)_{20-x}$ - $(V_2O_5)_x$. The

Characterization techniques like X -ray diffraction (XRD), Electron Paramagnetic Resonance (EPR) and Fourier Transform Infrared (FTIR) spectroscopy are performed on these samples to investigate the role of doped ion in glass matrix and the corresponding structural changes.

Experimental

A unique glass $(P_2O_5)_{65}$ – $(Pb_3O_4)_{10}$ – $(ZnO)_5$– $(Li_2CO_3)_{20\text{-}x}$ - $(V_2O_5)_x$where (x = 0, 0.2, 0.4, 0.6, 0.8 and 1.0) is used for the preparation of six glass samples V0, V1,V2,V3,V4 and V5 by melt quench method. All the Compounds as mentioned in the composition are of analytical grade and their quantities for each sample are shown in Table 1. The composition mixture is taken in different crucible and s placed in a muffle furnace and heated up to ~950^{0}C for one hour and hot liquid is poured on brass plate. ThepreparedV_2O_5dopedZnPbLiP glasses shown in Fig.1. Samples are subjected to annealing process to remove mechanical stress, by maintaining a temperature of ~300^{0}C for 3 hours. In the present study,the procedure and formulae are taken from the previous works of authors [8], to determine the physical and optical properties of present prepared glass samples. The following techniques are used at room temperature to record and procure the spectral, elemental and morphological data.

- X-Ray Diffraction (XRD): RIGAKU mini flex 600 models with Cu-K_α 1.5418Å radiation
- Electro Paramagnetic Resonance spectrometer (EPR): BRUKER ERO73 series operated in X – band (9.4 GHz) .
- Fourier Transform Infra Red spectrometer (FTIR): CARY 630 model using KBr pellets.

Results and Discussion

XRD Spectra

The Non- Crystalline and amorphous nature of the preparedV_2O_5 doped ZnPbLiP glasses (V0 to V5) were confirmed by the recoded powder X-ray diffraction spectra at room temperature. The amorphous nature of the samples is revealed by the absence of Bragg peaks in the spectra as shown in Fig.2.

Physical Properties

The evaluated Physical properties of V_2O_5doped ZnPbLiP glasses are shown in

Table 2.

The refractive index of V_2O_5 doped ZnPbLiPglasses were measured by using Abbe'srefractometerat 589.3 nm The density ρ was determined byusing the conventional Archimedes method, with toluene as an immersion liquid of stable density (0.868 g/cn^3).[9]

$$\rho = \frac{W_{air}}{W_{air} - w_{liq}} \rho_0 \qquad (1)$$

Where $W_{air}$and$W_{liq}$are the weights of the glass sample in air and liquid respectively.

The molar volume was calculated from [10]

$$V_M = \frac{M_W}{\rho} (2)$$

The physical properties like Fe^{2+} ion concentration (N), dielectric constant (ε), polaron radius(r_p), Inter ionic distance (r_i), Field strength (F), molar refraction(R_m) and Reflection loss (R%) from the glass surface were evaluated by using the standard formulae.[11-15]

Fe^{2+} ion concentration was calculated using[11]

$$N(\text{ions /cm}^3) = \frac{Mole\ \%\ of dopantpart\ \times Avogadronumber\ \times GlassDensity}{Averagemolecularweig\ \ ht} (3)$$

The dielectric constant (ε) was calculated using[12]

$$\varepsilon = n_d^2 \qquad (4)$$

The polaron radiuses (r_p)inter ionic distance(r_i)and field strength(F)were calculated using the formula[13]

$$r_p = \frac{1}{2(\frac{\pi}{6N})^{1/3}} \quad (5)$$

$$r_i = (\frac{1}{N})^{1/3} \quad (6)$$

Where N is the value of V^{4+}ionconcentration.

$$\text{Field strength } F = \frac{z}{r_p^2} \qquad (7)$$

Where r_p is polaron radius and z is the oxidation number of doping ion.

Reflection loss percentage was calculated by using the Fresnel formula.[14]

$$R = [\frac{(n_d - 1)}{(n_d + 1)}]^2 (8)$$

The molar refractivity R_m for each glass was evaluated using[15]

$$R_m = [\frac{n_d^2 - 1}{n_d^2 + 2}]\frac{M}{D} \qquad (9)$$

Where M is the average molecular weight and D is the density in g/cm^3.

The optical dielectric constant ($p\frac{dt}{dp}$) was calculated by using the formula.[16]

$$p\frac{dt}{dp} = (\varepsilon - 1) = (n_d^2 - 1)(10)$$

The electronic polarizabilityα_e was calculated using the formula.[17]

$$\alpha_e = \frac{3(n_d^2 - 1)}{4\pi N(n_d^2 + 2)} \qquad (11)$$

Various physical properties of V_2O_5 doped ZnPbLiPglasses (S1 to S5) are shown in Table 2. In the present prepared glasses the average molecular weight of glass sample V1 to V5 are increasing from179.8845 to 180.7484 gram, the molar volume values are decreasing from 49.4691 to 49.2959 cm^3 and the density values are increasing from 3.6363 to 3.6666 g/cm^3 as the mol% of iron ion concentration increases in the glass sample. The effect of V_2O_5 on density and refractive index is shown inFig.3 (a). The mean atomic volume values are decreasing from 7.5548 to 7.5376 cm^3/mole but V^{4+} ion concentration increasing from 2.4357 x $10^{20}$to 12.2331 x 10^{20} ions/cm^3 , it is due to the increase in density of glass samples V1 to V5.The polaron radius decreasing from 2.9947 A^0 to 1.7487 A^0 , Inter atomic distance is decreasing from 7.4323 A^0 to 4.3399 A^0 and Molecular electronic polarizability α_e decreases from 4.0969x 10^{23}cm^3 to 0.8172 x 10^{23} cm^3, it is due to the increase of V^{4+} ions concentration but Filed strength F values are increasing from 3.3451 x 10^{15} cm^{-2} to 9.8103 x 10^{15}cm^{-2} due to the decrease values of polaron radius $r_p$of Vanadium ion doped phosphate glasses from V1 to V5. Fig. 3(b)representthe effect of V_2O_5 concentration on polaronradius and inter atomic distance of vanadium doped zinc lead lithium phosphate glasses. As the properties of dielectric constant, optical dielectric constant, Molar Refraction and Reflection loss depend upon the refractive index of glass material. In the present study the refractive index values of vanadium doped prepared glass samples (V1 to V5) is increasing from 1.5605 to 1.5620, Dielectric constant increases from 2.4352 to 2.4398, optical dielectric constant increases from 1.4352 to 1.4398, Molar Refraction decreasing from16.0076 to 15.9867 and also the reflection loss decreasing from 4.7918 to 438119 due to the increase of refractive index values. The effect of V_2O_5 concentration on dielectric constant and optical dielectric constant are shown in Fig. 3(C). Finally the trend in physical properties of V_2O_5 doped ZnPbLiPglass samples of the present study well support thestudies of.[8, 18]

The exact values of optical basicity can be inferred from the glass parameters of different cations.[19]It's values of present glasses V1 to V5 are slightly decreasing from 0.6736 to 0.6718. High electron donor capability of oxide ions to the cations signifies high optical basicity. It is very useful in designing in unique optical materials like polorizers, detectors and modulators with higher optical performance.[20-21]

***FTIR spectra*:**

FTIR spectra of Vanadium ion doped ZnPbLiPglasses are shown in Fig.4. Assignedvibrational frequencies are tabulated in Table3.

FIR spectroscopy set up the basic structural units and to investigate the impact of transition metal ion dopent with composition on structural changes. The vanadium ion doped ZnPbLiP glass samples V1 to V5 exhibit percentage transmittance band in the IR range 1500-400 cm^{-1} as mentioned in figure 4and frequency values are tabulated in Table3. Fourier Transform Infrared (FTI) spectroscopy used as thefoundation for qualitative identification structural units in phosphate glasses glasses.[22]

In figure 4 at ~1054 cm^{-1}frequencyto PO_4^{3} molecules are at fundamental stretch,it is also observed that the percentage of transmittance in glasses is decreasing and absorption levels are increased with the increase of vanadium dopant and PO_4^{3-} molecules are at fundamental stretch and at 880 cm^{-1}frequency P-O-P groups in Q_1 structure are in asymmetric stretching with a transmittance of sixty five percent for highest vanadium doped glass.[23]PO stretching absorption bands are observed at frequency of ~1220 cm^{-1}because of alkali oxides.[24] P-O-P symmetric stretching frequencies are observed at ~702-781 cm^{-1} and thebroad absorption bands.[25] Harmonic bending V-O-P frequencies are observed at ~462-467 cm^{-1} and transmittance percentage through the glass is moderate.[26]

EPR spectra

Figure 5 shows EPR spectra of vanadium doped ZnPbLiP glasses and they are good agreement with the previous works of phosphate glasses.[27-29]The spectra represents three hyperfine bands in I the magnetic field region in the form of parallel and perpendicular components. This signifies to SHP in both the parallel and perpendicular magnetic field directions i.e., $g_{\parallel}$ and $g_{\perp}$. The spectra of glass doped with Vanadium ions are experimented with using an axial SH equations [30,31]asmentioned below:

$$H= \beta[g_{\parallel}B_Z S_Z + g\perp(B_x S_x + B_y S_y)] + A_{\parallel}S_z I_z + A\perp(S_x I_x + S_y I_y) \quad (1)$$

Where β is the Bohr magnneton, $g_{\parallel}$and $g_{\perp}$components anisotropic g-tensor.$A_{\parallel}$and$A_{\perp}$ are components of the hyperfine components of the hyperfine tensor A, z for the symmetry axis B_x, B_y and B_z are the stati components of magnetic field, S and I are the electron and nuclear spin operators. The solution of the Spin- Hamiltonian provides the expression for the peak positions of the principal g and A terms as[32]

$$h\vartheta= g_{\parallel}\beta B + mA_{\parallel} + (\frac{15}{4} - m^2)\frac{A_{\perp}^2}{2g_{\parallel}\beta B} \quad (2)$$

$$h\vartheta= g_{\perp}\beta B + mA_{\perp} + (\frac{15}{4} - m^2)\frac{A_{\parallel}^2 + A_{\perp}^2}{4g_{\parallel}\beta B} \quad (3)$$

Where$m$ is the nuclear magnetic quantum number of the vanadium nucleus i.e. m= $\frac{3}{2}, \frac{1}{2}, \frac{-1}{2}, \frac{-3}{2}$ and ϑ is the microwave frequency.

EPR spectra of vanadium ion doped ZnPbLiP glasses are shown in the figure 5 and g values in parallel and perpendicular direction are tabulated in Table 4.

In the EPR spectra hyperfine signal found signals at g~ 4.45 -4.66 in parralell direction and g~ 1.96-1.99 in perpendicular direction of the field for all glass samples V1 to V5 as shown in Fig. 5.. The resonance at g ~4.45-4.66 is attributed to the isolated V^{4}+

ion mainly situated in rhombic fuzzy octahedral site where as g~ 1.96-1.99 resonance arise from axially distorted site respectively. It is evident that the iron ions are in the state of trivalent and the symmetry is distorted in octahedral sites of glasses.

Both the g~4.2 and g~2.0 values of resonance signal were analysed by many researchers.[34,35]Some researchers[33-38]highlighted that the value of g in glass containing V^{4+} ions were related to the coordination number. The absorption at g~4.2 and g~2.0 are from V^{4+} ions in tetrahedral and octahedral coordination's respectively.[35]As perWickman et al.,[39]V^{34+} ions in rhombic vicinities highlight the transitions with g~4.6 isotropic value corresponding to middle kramers doublet.

Conclusions

The prepared V_2O_5 doped ZnPbLiPglasseshave non crystalline and amorphous nature. The energy dispersion spectrum reveals that the prepared glass contains all the elements which are present in the composition. The physical property values of V_2O_5 doped ZnPbLiPglasses values are varying as the concentration mol% enhanced and also differentwhen compared to other phosphate glasses The density values are increasing from 3.6363 to 3.6666 g/cm^3, In the present study the refractive index values increasing from 1.5605 to 1.5620 but optical basicity values decreasing from 0.6736 to 0.6718 for all glass samples and is required for the design of the new optical functional materials like polorizers, detectors and modulators with higher optical performance. Spin–Hamiltonian parameters are evaluated from EPR spectra which exhibit two resonance intense signals at g ~ 4.66 and ~ 1.96 representing exchange of coupled pair and free state of Vanadium ions. It is evident from the Spin- Hamiltonian Parameters (SHP) that the Vanadium ions are available in the sites of octahedral with tetrahedral composition and which is a part of o C_{4v} symmetr.y FourierTransform Infrared (FT-IR) spectra exhibits few sharp and broad absorption bands which attribute to (P-O-V) at 467cm^{-1},P-O-P)$_s$ at 781 cm^{-1} and (P-O-P)$_{as,}$ at 880 cm^{-1}vibrations. These studies will be helpful in understanding optical and dielectric properties of the prepared glasses.

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Table 1: Compositions of the glass samples (mole %)

Sample	Composition (mole %)				
	P_2O_5	Pb_3O_4	ZnO	Li_2CO_3	V_2O_5
S0	65	10	5	20	-
S1	65	10	5	19.8	0.2
S2	65	10	5	19.6	0.4
S3	65	10	5	19.4	0.6
S4	65	10	5	19.2	0.8
S5	65	10	5	19	1.0

Table 2. Physical properties ofV_2O_5 doped ZnPbLiPglasses.

Physical properties	V1	V2	V3	V4	V5
Average Molecular weight M (g)	179.8845	180.1005	180.3164	180.5324	180.7484
Mean Atomic Volume (cm^3/mole))	7.5548	7.5583	7.5504	7.5489	7.5376
V^{4+}ion concentration N(x 10^{20} ions/cm^3)	2.4357	4.8718	7.3193	9.7664	12.2331
Density d (g/cm^3)	3.6363	3.6401	3.6494	3.6556	3.6666
Refractive Index (n_d) at 589.3 nm	1.5605	1.5611	1.5615	1.5618	1.5620
Polaron Radius r_p (A0)	2.9947	2.3768	2.0753	1.8850	1.7487
Field Strength F (x10^{15} cm^{-2})	3.3451	5.3103	6.9658	8.4427	9.8103
Inter atomic distance r_i(A^0)	7.4323	5.8988	5.1504	4.6783	4.3399
Dielectric constant (ε)	2.4352	2.4370	2.4383	2.4392	2.4398
Optical dielectric constant ($(p\frac{\partial t}{\partial p})$)	1.4352	1.4370	1.4383	1.4392	1.4398
Molar volume V_m (cm^3)	49.4691	49.4768	49.4099	49.3852	49.2959

Molar Refraction $R_m(cm^{+3})$	16.0076	16.0242	16.0119	16.0109	15.9867
Mol.r electronic Polarizability α ($x10^{+23}cm^3$)	4.0969	2.0498	1.3651	1.0234	0.8172
Optical Basicity (Λ_{th})	0.6736	0.6732	0.6727	0.6723	0.6718

Table 3. FTIR spectral peaks of vanadium ion doped ZnPbLiP glasses

FTIR frequencies (cm^{-1}) of samples V1 to v5	Vibration Assignments
~462-467	P-O-V linkages of harmonics bending frequencies
~702 -781	Vibration of P-O-P symmetric stretching
~ 880	Formation of P-O-P groups in Q_1 structure with asymmetric stretching
~ 1052	vibrations
~1220	PO_4^{3-} fundamental vibrational mode
	PO stretching vibration

Table 4: g- values at higher and lower magnetic fields of V_2O_5 doped ZnPbLiP glasses

Sample	$g_{\parallel}$	$g_{\perp}$	Δg_0	$A_{\parallel} X\ 10^{-4} cm^{-1}$	$A_{\perp}\ X\ 10^{-4} cm^{-1}$	$A_0 X10^{-4}$
V1	4.4517	1.9669	0.7928	407	169	135
V2	4.4928	1.9728	0.8105	423	168	141
V3	4.576	1.9636	0.8324	452	170	150
V4	4.6383	1.9943	0.8733	479	162	159
V5	4.6642	1.9603	0.8593	483	178	161

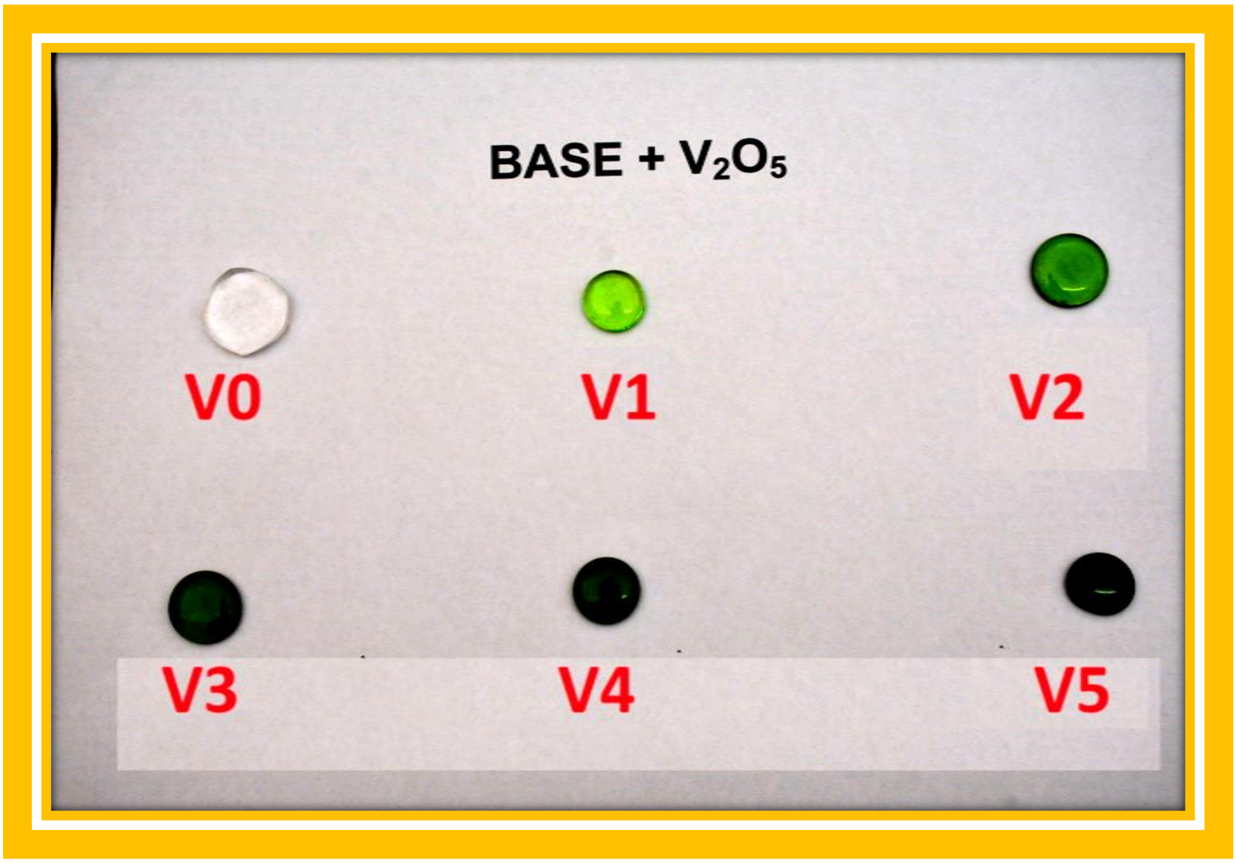


Fig.1 Physical appearance of glass sample.

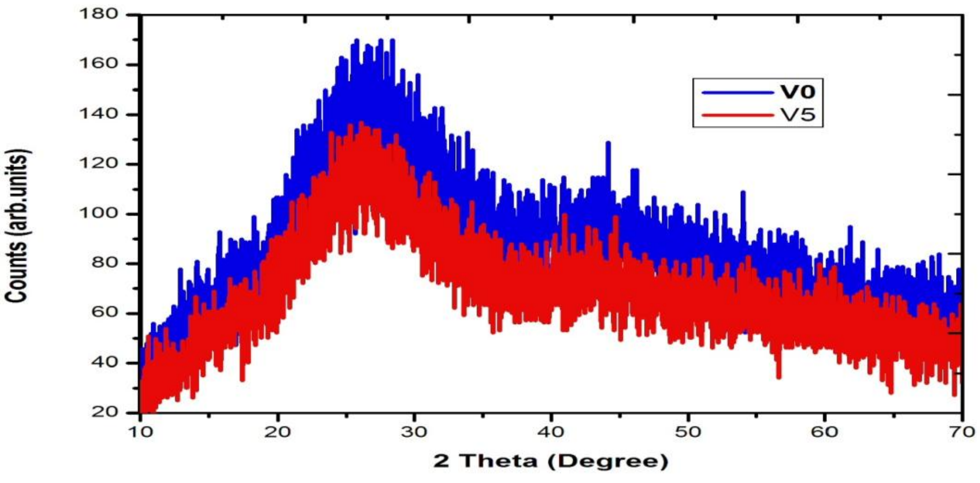
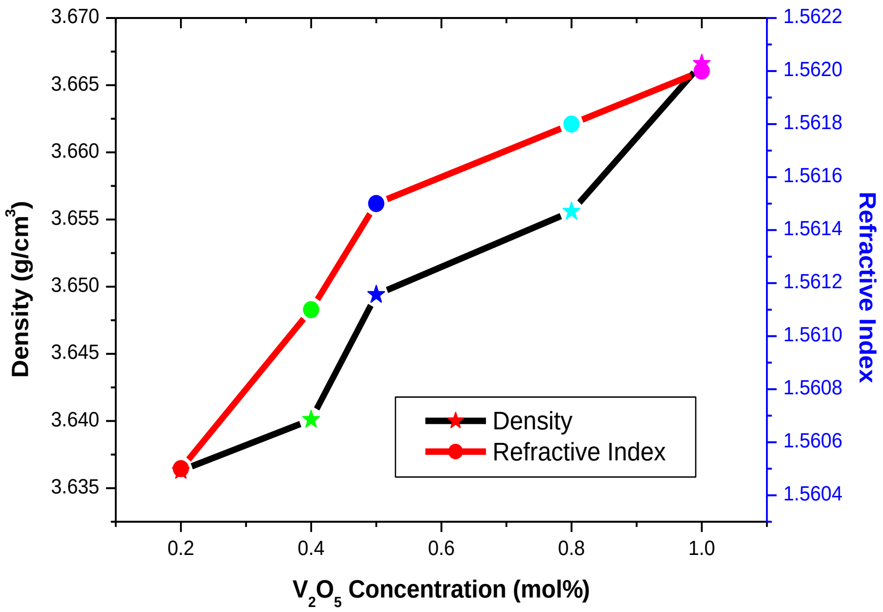
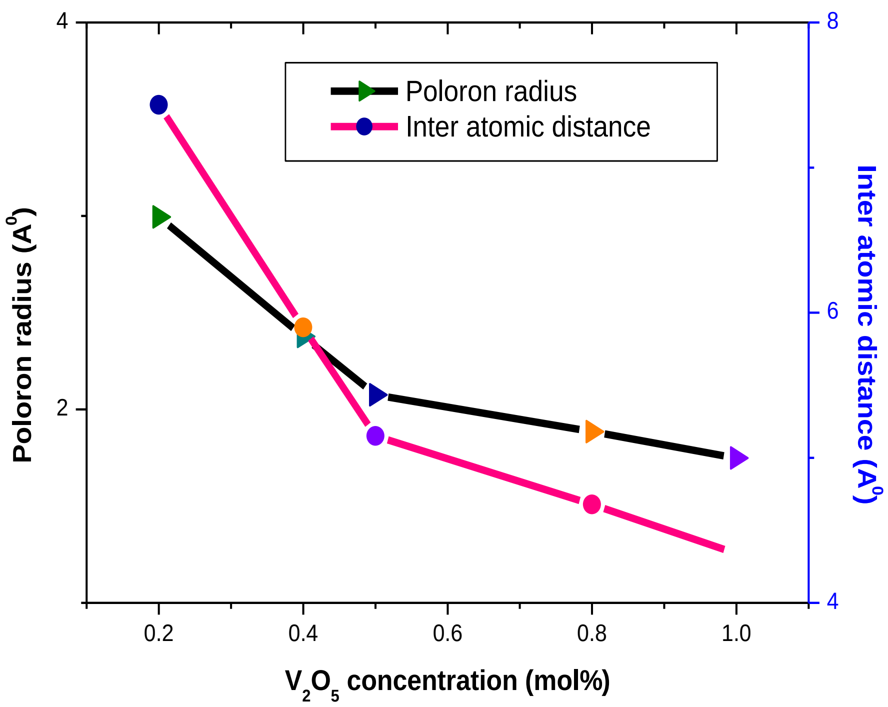


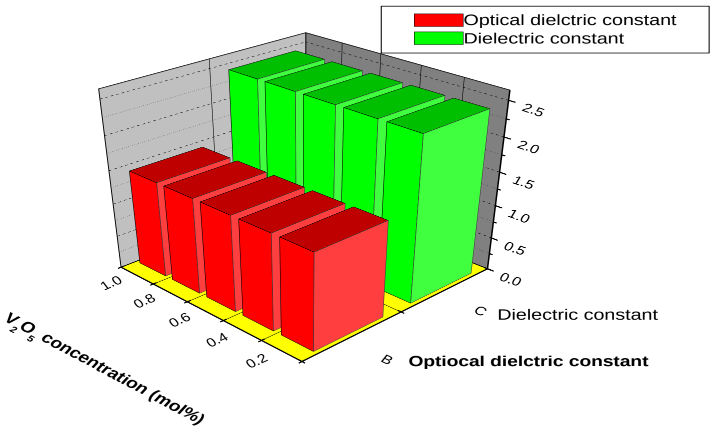
Fig.2 XRD spectra of V_2O_5 doped ZnPbLiPglasses.


3.670
3.665
3.660
3.655
3.650
3.645
3.640
3.635
Density (g/cm^3)
1.5622
1.5620
1.5618
1.5616
1.5614
1.5612
1.5610
1.5608
1.5606
1.5604
Refractive Index
Density
Refractive Index
0.2
0.4
0.6
0.8
1.0
V_2O_5 Concentration (mol%)

3(a)


4
2
Poloron radius (A^0)
8
6
4
Inter atomic distance (A^0)
Poloron radius
Inter atomic distance
0.2
0.4
0.6
0.8
1.0
V_2O_5 concentration (mol%)

3(b)



3 (c)

Fig. 3 Effect of V_2O_5on (a.) Density and Refractive (b) Polaronradius and Inter atomic distance(c) Dielectric constant and Optical dielectric constant of ZnPbLiPglasses.

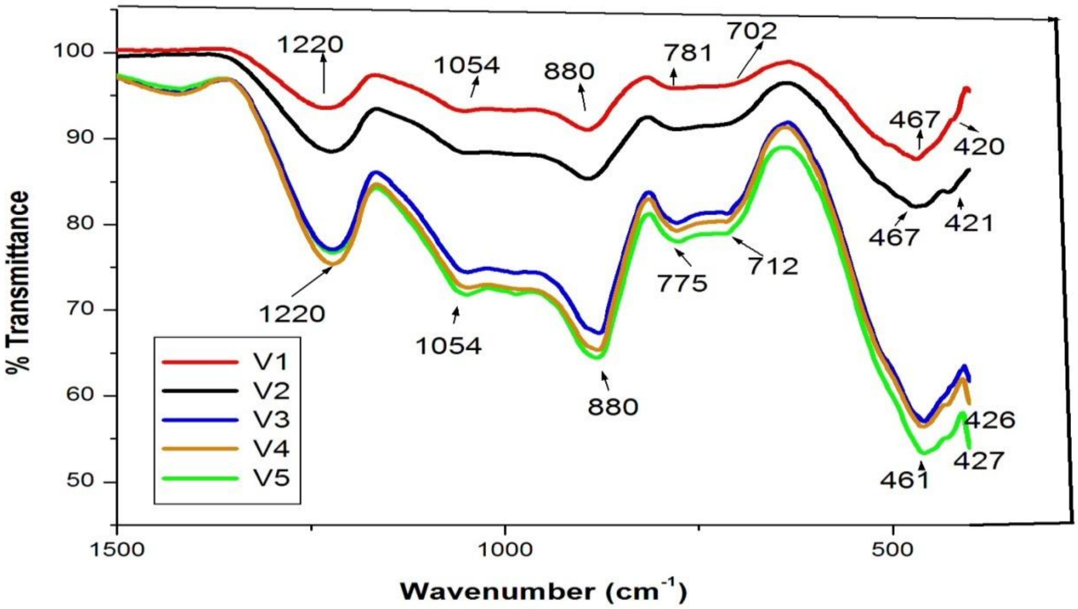


Fig. 4 FT-IR spectra of vanadium ion doped ZPbLiP glasses

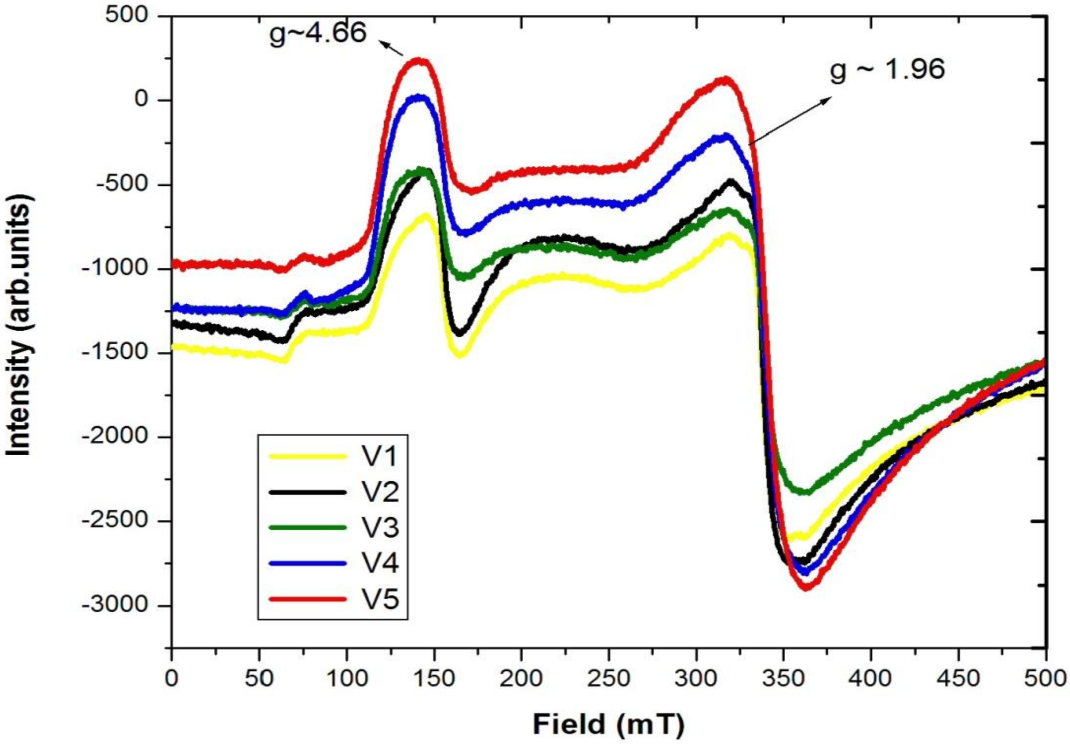


Fig. 5 EPR spectra of vanadium ion doped ZnPbLiP glasses