Metrological and Optical –Mechanical Study of a Band Pass Glass Filter Working as Photometric Filter.

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Abstract

In the present work, glass filters were obtained from three different glass (systems) components, phosphate glass, borate glass and Lithium borate glass with different concentration of transition metals. To characterize these filters, the mechanical properties of the known bond strength, the stability of the glass network, and rigidity of the prepared glass filter were measured.

Mechanical properties were investigated using ultrasonic by pulse echo technique using sound wave velocity measurements at 4MHz (both longitudinal and shear) at room temperature. The transmission measurements using precise spectrophotometer have been showed that, the three studied glass systems (components), could be used as band pass filter in visible region simulating V(λ) filter. It blocks radiation away from the Visible region (380-780) to better than 0.01%. A combination of a trap detector with the filters were used as precise V(λ) filtered trap detector. The system can be characterized to be used for photometric measurements. The uncertainty analysis indicates a relative expanded uncertainty for the illuminance measurements. V(λ) filtering is more expensive, in this paper, using limited capabilities, and cheap materials, we got an optical filter close to the standard V(λ) filter. With increasing laboratory capabilities, we can improve the optical properties and that is what we are aiming for in the future.

Keywords: phosphate glasses, borate glasses, ultrasonic.

1. Introduction

The glass structure is essential to understand the behavior of the materials. The velocity of ultrasonic waves and hence the elastic moduli are particularly suitable for characterizing glasses as function of composition [1-3].

Ultrasonic characterization of materials is versatile tool for the inspection of the microstructure and their mechanical properties. This is possible because of the close association of the ultrasound waves with the elastic and the inelastic properties of the materials. Since the choice of the most appropriate material for particular application requires knowledge of its mechanical properties [4, 5]. The measurement of ultrasonic parameters like velocity as a function of composition and calculation of the elastic moduli and other parameters are of great interest in glass. These ultrasonic parameters besides density and molar volume are sensitive and informative about the changes occurred within the structure of glass network [6-8].

In our previous work (under press), the structure properties of three glass systems were studied to use it as a band pass filter for a trap detector to match the sensitivity of human eye. It can be also used as a UV-laser protection. In the present work a complete study of the mechanical properties as ultrasonic was done.

2. Research Methodology

The glass filters were formulated from three glass systems the first one is, sodium zinc phosphate glasses [$40P_2O_5$-40ZnO-(19-x) Na_2O-(x) Cu_2O-1CaO] at (x=0.5,1, 2, 4, 6 & 8 mol%), the second one is, borate glass [$40B_2O_3$-40ZnO-(19-x) Na_2O-(x) Fe_2O_3-1CaO] at (x=0.5,1, 1.5 & 2 mol%) and the last one is lithium borate glass [$75Li_2B_4O_7$–xCu_2O-(25-x) PbO_2] at (x= 2.5, 5, 7.5 &10 mol%). All three systems were prepared by the conventional melt–quench technique. The chemical reagents were thoroughly mixed and ground for 30–40min in mortar pestle. Then the batch was melted in a porcelain crucible using a muffle furnace for 3–4 hours at a temperature ranging from 700 to 1100ºC depending on the composition. When the melt was thoroughly homogenized and attained desirable viscosity, it was poured onto the preheated brass plate. The prepared glass samples were annealed at appropriate temperatures (between 300 & 400 ºC) for 2hour to remove the internal stresses. Then the samples were left inside the annealing furnace to cool slowly at room temperature. The prepared samples were polished carefully for the different measurements.

2.1. Optical measurement

Spectral transmission for polished glass samples were measured by a recording spectrophotometer in the region (300–800) nm. The spectrophotometer used in this study, is computer-controlled SHIMADZU, UV-3101PC–UV–VIS–NIR Scanning Spectrophotometer.

2.2. Set up for filtered trap detector

A system consists of standard lamp FEL, a trap detector QED200, a band path filter and a precision aperture (11mm) were used for the measurements as shown in Figure (1,2). A photometric bench in NIS photometry lab was used, system with movable carriages is mounted on the table. Two visible laser diodes are needed for optimum adjustment of lamp filaments with respect to the optical axis. It can be adjusted in three coordinates until the laser beam passes through the center holes of the baffles. The lamp was aligned base down with the longitudinal axis of the glass envelope vertical. The lamp was aligned with the filament vertical and set normal to the line joining the centers of the filament and photometer aperture. Measurements were made at 2 meters from the mean plane of the filament using photometer with an aperture of 11 mm diameter which viewed the entire lamp [9,10].

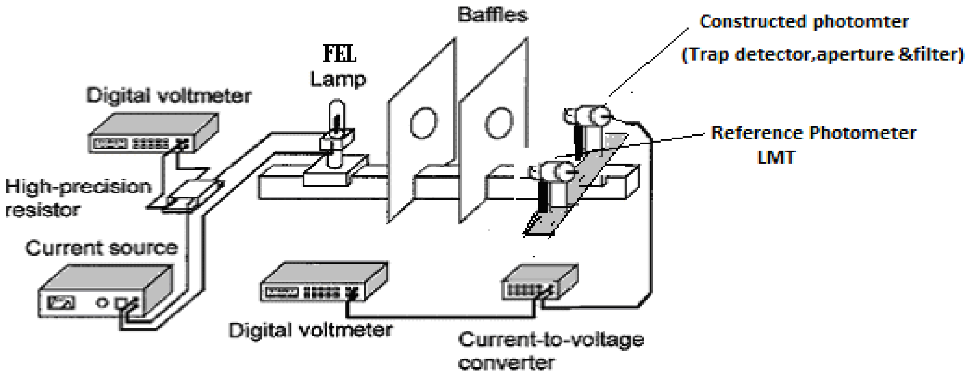

Fig. 1. Schematic diagram for the Photometric Measurement System.

Fig. 2. Set-up of Photometric Measurements system.

2.3. Ultrasonic measurement

The ultrasonic wave velocities (longitudinal V_L and shear V_s) measurements were carried out to highly polished samples by applying the pulse-echo technique at room temperature using transducer (Karl Deutsch) with fundamental frequency of 4 MHz to measure longitudinal ultrasonic wave velocity, and a digital ultrasonic flaw detector (USN60—Krautkramer).

3. Results and discussion

3.1. Optical and photometric measurement

In a previous work under publication, different filters were prepared. The filters were tested for matching the spectral sensitivity of the human eye. It was found that the most convenient filters that matched the human eye sensitivity, the glass system [$40P_2O_5$–40ZnO-(19-x)Na_2O–1CaO] at concentration $1Cu_2O$ and filter from the glass composition [$40B_2O_3$-40ZnO-(19-x)Na_2O-(x)Fe_2O_3-1CaO] at concentration $1.5Fe_2O_3$. Samples ($1Cu_2O$) in the phosphate system and sample (1.5 Fe_2O_3) in the borate collected together to obtain good band pass glass filter and the glass composition [75 $Li_2B_4O_7$ –xCu_2O-(25-x) PbO_2] at concentration $2.5Cu_2O$ optical glass filter.

The two filters were mounted in the spectrophotometer holder and spectrally scanned in the range 380-780nm. For 0.1 nm interval to produce a curve indicates a relationship between wavelength and regular transmittance percentage.

Figures (3&4) illustrates the relation between wavelength and normalization transmittance at 555 nm peak wavelength (at 1 nm bandwidth) of the two samples (glass components). Each filter has a thickness of 2.25 mm, and the diameter of the usable area is 12 mm. Figure (5) illustrates that filter ($1Cu_2O+1.5Fe_2O_3$) is close to standard V(λ) filter.

A photometer in photometry lab consists of a trap detector QED200, a band path filter -1 or -2, and a precision aperture. The band path filter modifies the spectral responsivity of the trap detector to coincide with the V(λ) curve, each band pass filter was close fitted to the entrance aperture and investigated individually with the trap detector. The illuminance values were once measured by the filtered trap detector & then measured by the standard LMT detector. The measurements were repeated for filtered trap detector using second band path filter. The ratio of illuminances obtained by the standard LMT and that obtained by the filtered detector was calculated in (Tables 2 and 3), as these tables show that the ratio of illuminances obtained by the standard LMT and filter 1 is better than the illuminances ratio obtained them by standard LMT and filter 2.

The uncertainty associated by the measurements is evaluated by combining the individual standard uncertainty of all the components that affected our measurement accuracy, such as uncertainty of the calibrated reference photometer, Long-term drift of the photometer (during one year), responsivity, uncertainty of distribution temperature measurement, Random noise from the lamp, Stray light, Trans impedance gain of the amplifier (Which is indicated in the catalog specifications) and repeatability of measurements. From Table (1) it was found the relative expanded uncertainty does not exceed 2%.

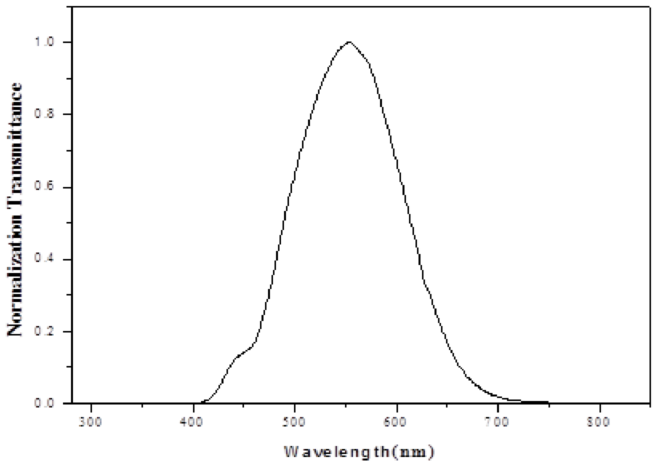

Fig. 3. Optical transmission spectra of collected sample ($1Cu_2O + 1.5 Fe_2O_3$).

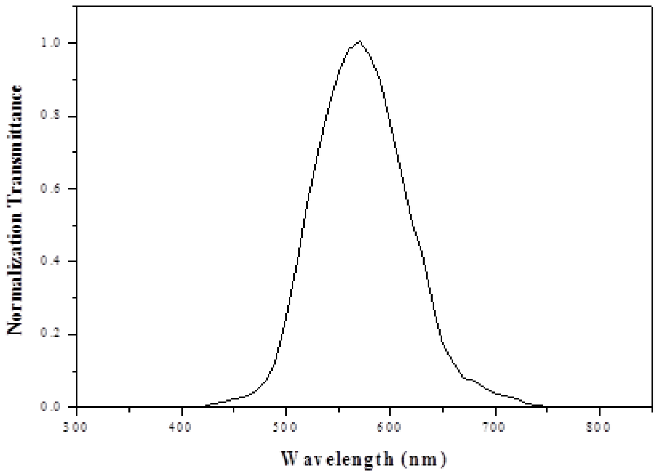

Fig. 4. Optical transmission spectra of $2.5Cu_2O$.

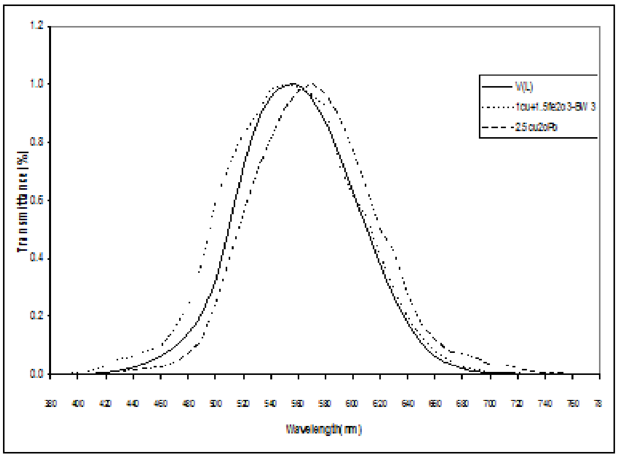

Fig. 5. Shows Normalized spectral responsivities graph as a function of wavelength for 2 composition $1Cu+1.5Fe_2O_3$& $2.5Cu_2OPb$ (at band width 1nm) compared with CIE V (λ) filter.

Table 1. Uncertainty analysis for illuminance measurement using a reference photometer with a 2856 K incandescent source.

Factor	**Distribution**	**Type**	**Relative standard Uncertainty [%]**
Uncertainty of the calibration of the reference photometer (in A/lx)	Rectangular	Type B	0.5
Long-term drift of the photometer (during one year)	Rectangular	Type B	0.289
Responsivity change within the controlled temperature range (±1°C)	Normal	Type A	0.2
Uncertainty of distribution temperature measurement (±50 K)	Rectangular	Type B	0.115
Random noise from the lamp	Normal	Type A	0.3
Stray light from the measurement set-up	Rectangular	Type B	0.058
Trans impedance gain of the amplifier	Rectangular	Type B	0.023
Repeatability of measurements	Normal	Type A	0.7
Combined Uncertainty			0.971
Expand uncertainty of illuminance measurements (k = 2)			1.941

Table 2. Ratios of illuminances measured by LMT standard detectors and Trap detector with first filter (good filter).

Detector serial number	**Luminous responsivity**	**Illuinanced calculated for Reference LMT detector**	**Illuminance calculated for trap detector using filter 1 ($1Cu_2O + 1.5 Fe_2O_3$)**	**Ratio of illuminance measured by LMT / illuminance measured by trap detector with the first filter**
LMT 02A7512	26.70 nA/Lux	213.158	217.198	0.9814
LMT 02A7513	26.70 nA/Lux	213.379	217.600	0.9806
LMT 10A9242	27.36 nA/Lux	212.986	216.780	0.9825
LMT 10A9243	27.40 nA/Lux	213.074	217.046	0.9817
Average				0.9816

Table 3. Ratios of illuminances measured by LMT standard detector and Trap detector with second filter (bad filter).

Detector serial number	Luminous responsivity	Illuminance calculated for Reference LMT detector	Illuminance calculated for trap detector using filter2 (2.5Cu_2O)	Ratio of illuminance measured by LMT / illuminance measured by trap detector with the second filter
LMT 02A7512	26.70 nA/Lux	213.158	204.566	1.042
LMT 02A7513	26.70 nA/Lux	213.379	205.528	1.0382
LMT 10A9242	27.36 nA/Lux	212.986	205.764	1.0351
LMT 10A9243	27.40 nA/Lux	213.074	204.800	1.0404
Average				1.0387

3.2. Ultrasonic measurement

The ultrasonic wave velocity was estimated by dividing the round-trip distance (twice the thickness of the sample) by the elapsed time according to the relation;

$$V = \frac{2X}{\Delta t} \tag{1}$$

Where, X is the thickness of the sample and Δt is the interval time

In an amorphous solid (such as glass), the elastic strain produced by a small stress can be described by the longitudinal modulus (L) and shear modulus (G) given as

$$\mathrm{L} = \rho \mathrm{V_L} \tag{2}$$

$$G = \rho V_s^2 \tag{3}$$

where V_L and V_s are the longitudinal and transverse sound velocities and ρ is the density of glass samples. The sound velocities also allow the determination of Young's modulus (E), the bulk modulus (K), the micro-hardness (H) and Poisson's ratio (σ) is defined for any structure as the ratio between lateral and longitudinal strain produced when tensile force is applied, using the following equations [11-13]:

$$\mathrm{K} = \mathrm{L} - (4/3)\mathrm{G} \tag{4}$$

$$\mathrm{E} = 2\mathrm{G}(1 + \sigma) \tag{5}$$

$$\sigma = (\mathrm{L} - 2\mathrm{G})/2(\mathrm{L} - \mathrm{G}) \tag{6}$$

$$\mathrm{H} = (1 - 2\sigma)\mathrm{E}/6(1 + \sigma) \tag{7}$$

The Debye temperature (θ_D) represents the temperature at which nearly all the vibrational modes are excited. For the studied glass samples, it was obtained from the relation given by:

$$\theta_D = \left(\frac{h}{k_B}\right)\left(\frac{3Z\mathrm{N_A}}{4\pi Va}\right)^{1/3} V_m \tag{8}$$

where h is the Planck's constant, K_B the Boltzmann's constant, N_A the Avogadro's number, Z the number of atoms in molecular formula, V_a the mean atomic volume and V_m the mean ultrasonic velocity defined by the relation

$$V_m = \left(\frac{1}{3}\left(\frac{1}{V_L^3} + \frac{2}{V_s^3}\right)\right)^{-\frac{1}{3}} \tag{9}$$

The softening temperature (T_s) is another important parameter defined as the temperature point at which viscous flow changes to plastic flow.

$$Ts = \frac{V_s^2 M}{nC^2} \tag{10}$$

Where C is the constant of the proportionality and equals $0.5074.10^5$ cm $s^{-1}k^{-1/2}$and n is number of atoms in the chemical formula.

Figure (6) illustrated that the velocities (V_L and Vs) are the longitudinal and shear velocities respectively for the three systems of glasses. In all present glass systems, their ultrasonic velocities were found to be enhanced by increasing of modifier oxide (transition metal) contents. In figure (6.a) ultrasound velocities increased with the increase in Cu_2O mol%, the increase is related to the decrease in the number of non-bridging oxygen and thus increases the glass network connection. So, the increase in ultrasound velocities is due to the fact that the addition of Cu_2O in sodium phosphate glass leads to the formation of P-O-Cu bonds when Cu_2O replaces Na_2O which increases the cross-linking density between the phosphate chains in glass network, thus, enhances chemical durability this in agreement with S.Y. Marzok [14].

Figure 6 (b), show the increase in ultrasound velocities with the increase in Fe_2O_3 mol%, is related to the increases in cross-linking density, decrease in the number of non-bridging oxygen atoms and thus increases the glass network connection this in agreement with the results obtained from the previous measurements (density, molar volume and IR) from IR the shift to higher wave number indicate that the strength of the bonds increase as Na_2O is replaced by Fe_2O_3.

Figure 6 (c), show the increase in ultrasound velocities increased with the increase in Cu_2O mol%, which is related to increases in cross-linking density, decrease in the number of non-bridging oxygen atoms and thus increases the glass network connection. These results were confirmed by those observed from (density, molar volume and IR) studies due to the conversion of BO_3 into BO_4 which increase the crosslink density and will contribute to the increase in the ultrasonic velocity.

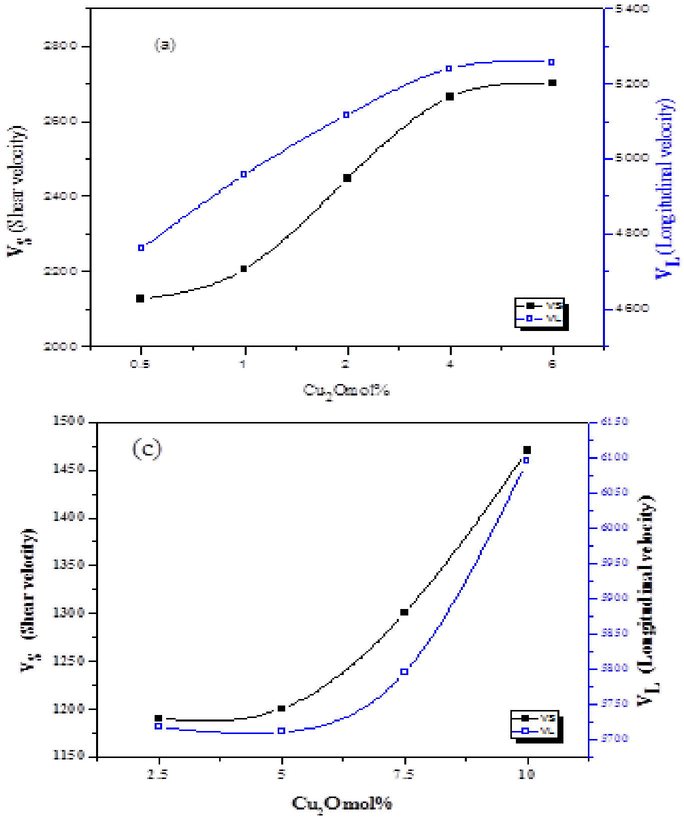

Fig. 6. (a-c): Variation of the longitudinal V_L and shear velocity V_S
a) with Cu_2O mol % **b**) with Fe_2O_3 mol % **c)** with Cu_2O mol %.

Figure (7) shows the behavior of elastic moduli (L, G, E and K). It can be seen from the Figure 7(a) that all the elastic moduli were increased with the Cu_2O concentration. the increase in elastic moduli is in agreement with the same reasons that increased density and ultrasound velocities. Moreover, as Copper ions have small ionic radii and a large charge, they go into a glass network in a position that creates a cross-link between the tetrahedral phosphate (Cu - O - P instead of P - O - P), and it improves the stability and mechanical properties of glass [15,16].

Figure 7(b) shows the elastic moduli increase with the Fe_2O_3 concentration. From IR the strength of bonds increase as Na_2O was replaced by Fe_2O_3 the increase in elastic moduli is in agreement with the same reasons that increased the longitudinal and shear velocities.

Figure 7(c) shows the elastic moduli decrease with increasing the Cu_2O concentration in expense of PbO. The decrease in elastic moduli may be due to decrease in the density in spite of the increase in the ultrasonic velocity.

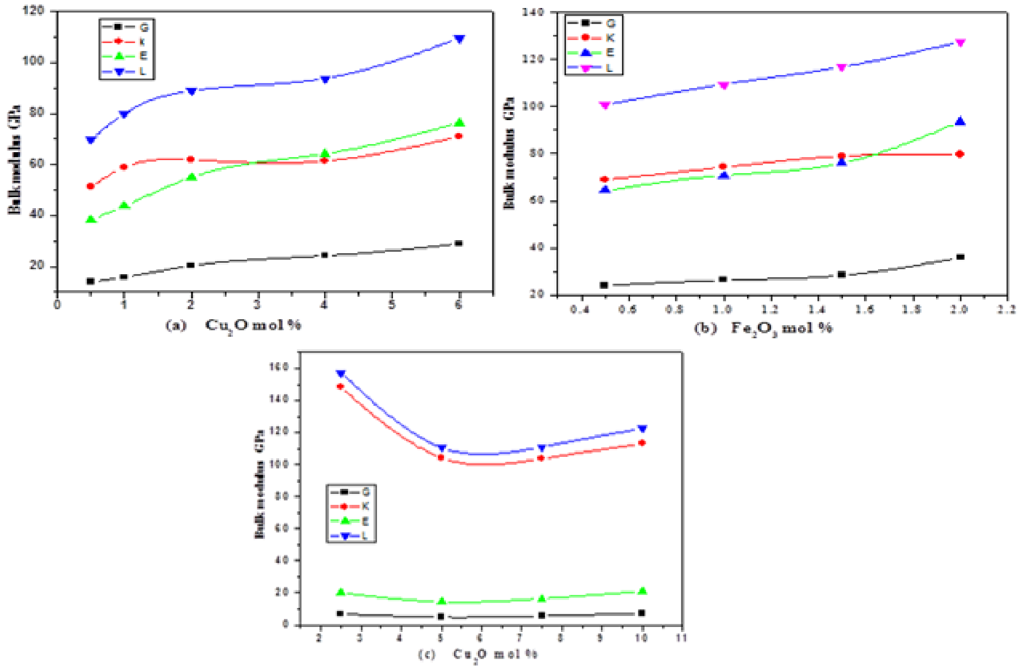

Figure 7 (a-c): The behavior of elastic moduli (L, G, E and K).

The Debye temperature Θ_D, softening temperature Ts with the Cu_2O content represented in Figure (8) Debye temperature represents the temperature at which almost all the high frequency (lattice)vibrational modes are excited and it is increase with system rigidity. From the figure, it is clear that increased with increasing Cu_2O content, which means that the rigidity of the glass system increases with the increase in Cu_2O. The softening temperature is also affected by rigidity of the system, rigidity increases as non-bridged oxygen decreased, cross-link density increases, and with stretching strengthening of bonds, all of these were increased with Cu_2O increasing in agreement with [17-19] and with the results of (IR, density, Hardness) [study under press].

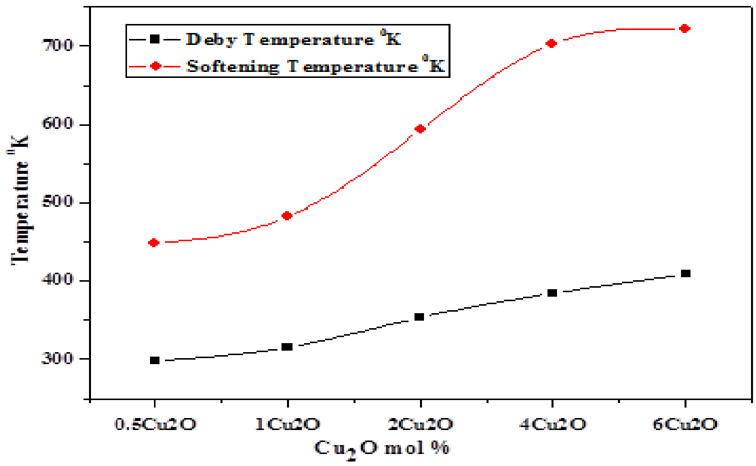

Fig. 8. The relation between Debye temperature, Softening temperature and Cu_2O content.

Poisson's ratio σ is defined for any structure as the ratio between lateral and longitudinal strain produced when tensile force is applied or function of the ratio of longitudinal and shear velocities. Bridge and Higazy [20] have suggested a close correlation between Poisson's ratios and cross-link density, which is defined as the number of bridging bonds per cation.

Poisson ratio from the glasses was affected by the crosslink density of the glass structure. Figure 9 (a, b and c) illustrated the variation of Poisson's ratio with composition we observed that Poisson's ration decreases in three system of glasses as the cross–link density increasing this is in agreement with all the results. The behavior of the variation of Poisson's ratio was nearly reverse of that observed for the variation in elastic moduli [21-22].

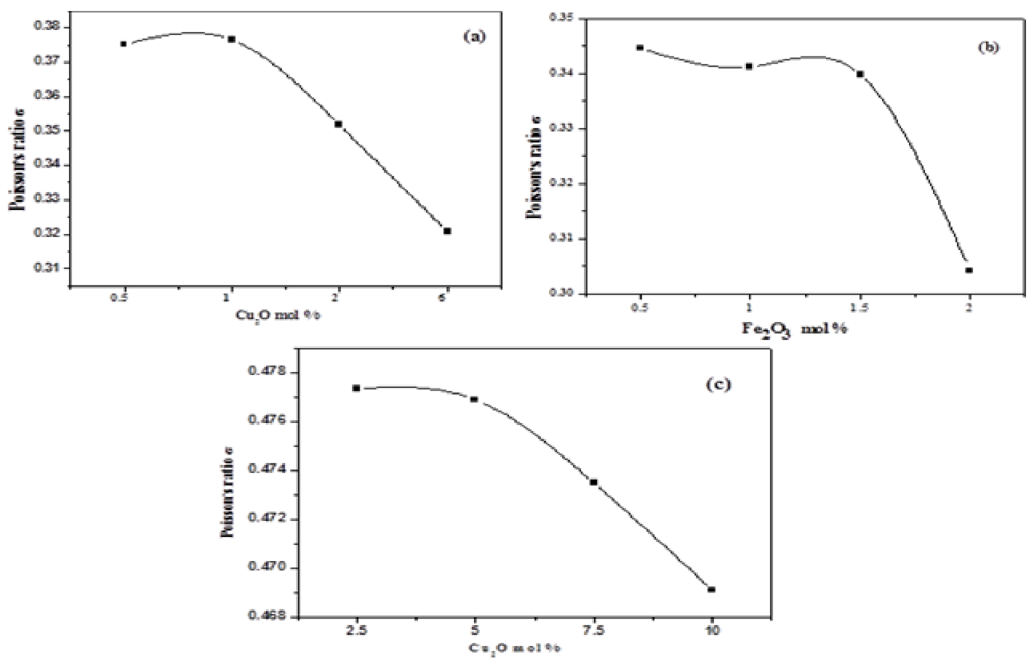

Fig. 9 (a-c) the variation of Poisson's ratio with composition
a) with Cu_2O mol% **b)** with Fe_2O_3 mol% **c)** with Cu_2O mol%.

4. Conclusion

The previous results illustrated that the two filters ($1Cu_2O+1.5Fe_2O_3$) and ($2.5Cu_2OPb$) are band path filter but the filter ($1Cu_2O+1.5Fe_2O_3$) a good filter is close to the V(λ) standard filter which is in a range of sensitivity of human eye. When calibration this filter on the photometric system in photometry lab in NIS (national institute for standard) and calculated the uncertainty budget which is equal to (1.94).

We obtained from studied the mechanical properties for three glass systems: -

1) The ultrasonic wave velocities increased in all glass systems by increasing of modifier oxide and the elastic moduli increased.

2) Debye temperature and softening temperature increased with increasing of modifier oxide due to the increase the rigidity of the glass network.

3) Poisson's ration decreases in three system of glasses which indicate that the crosslink density increasing.

Mechanical measurements showed an increase in elastic moduli, an increase in bond strength, and an increase in the rigidity of the glass network. This ensures that the properties of the prepared glass band pass filter are good.

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