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Electrical and Structural Properties of Nickel Oxide - Containing Phosphate Glasses

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PHOSPHATE glasses containing 0.05-7.5g NiO/100 glass were prepared using sodium di-hydrogen phosphate. The prepared glasse were investigated using IR, a.c. conductivity, dielectric constant (ε`) and the dielectric loss (ε``). IR spectra revealed that the Ni^{2+} ions are located near P=O. ♀♀Addition of Na_2O causes a breakdown of the ring type with the formation of more non-bridging oxygens and depolymerization of the network. The conductivity of Ni-containing glasses is more than that of Ni-free phosphate glass, and attain maximum value for glass containing 0.25 g NiO per 100g glass The relation between log σ and reciprocal of the temperature suggests that, the mechanism predominating in the high temperature region is ionic in nature, while it is electronic in origin in the low temperature region. Introducing NiO extends electronic conduction through hopping mechanism to the high temperature region particularly at high frequency. The dielectric constant ε' and the dielectric loss ε`` increase with increasing temperature, while a decrease in ε' with increasing frequency was observed.

Keywords: Phosphate glasses, Nickel oxide-containing glasses, Electrical conductivity, Dielectric constant and Dielectric loss.

Phosphate glasses are technologically important materials because they generally have higher thermal expansion coefficient, lower transition temperature and higher electric conductivity than silicate and borate glasses[1-3]. Phosphate glasses have very high transmission in the ultraviolet region which is a unique property when compared to that of silicate glasses. They are suitable materials for high power lasers because of low thermo-optical coefficient and large emission [4].

Glasses containing transition metal ions are of great interest due to their uses in memory , photo-conducting devices cathode materials in dry batteries and magnetic materials [5-8]. Transition metal ions when present in oxide glasses form coordination complexes with the doubly charged oxygens, usually in 4-fold or 6-fold coordination. The octahedral coordination is generally favored over the tetrahedral one, yet factors such as ionic radius, polarizability, glasses composition and Jahn-Teller distortion may modify the state of coordination in the glass[8]. In phosphate glasses transition metal ions exist in octahedral coordination [8-11] .

Phosphate glasses are generally formed by PO_4 tetrahedra connected by corners. Connection by three of four corners occurs in vitreous P_2O_5, leads to a three-dimensional network. The fourth corner is occupied by a terminal double bonded oxygen. Introduction of network modifiers, *e.g.* Na_2O, results in breaking P-O-P bonds with subsequent formation of terminal oxygens (P-O⁻) . Depending on the number of the bridging oxygen the phosphate structure can be described according to notations used by Lippma *et al.* [12] as Q°, Q^1 , Q^2 & Q^3 . Q^3 denotes a fully polymerized structure, while Q^2 unit gives only a two-dimensional structure based on chains or rings. Q^1 means two tetrahedra connected by a corner and Q^0 means isolated tetrahedral [3,4] .

The optical properties of nickel-containing glasses have been extensively studied[2,13]. The present work is concerning with giving more information about the electric properties and the structure characteristics of Ni-containing phosphate glasses.

Experimental

Preparation of glass specimens

Glasses were prepared using chemically pure sodium di-hydrogen phosphate as a starting basic material. NiO was introduced as finely powdered solid. Na_2O or P_2O_5 were introduced in the glasses in the form of anhydrous sodium carbonate and ammonium dihydrogen phosphate, respectively, and their amounts were expressed as g per 100 g glass. Table 1 lists the compositions of the glasses studied. Glass batches were introduced in porcelain crucibles and heated first at 300 $^{\circ}$C to expel water and ammonia and then sintered at 850°C for l hr. The melts were rotated twice to ensure homogeneity. The homogenized melts were cast in preheated stainless steel molds in the required dimensions. The prepared samples were immediately transferred to a muffle furnace at 300 $^{\circ}$C/hr, and then it was shut down to cool to room temperature.

Infrared spectra measurements

The KBr disc technique was used to measure the IR spectra of the prepared glass samples. The IR spectra were measured for the homogeneous discs at room temperature in the range 400-3000cm^{-1} using Jasco FT/IR-300 E Spectrometer, Japan. Measurements were carried out immediately after preparing the desired discs.

Electrical conductivity

The a.c. method was applied to determine the electrical conductivity. A measuring circuit consisting of a sample holder (cell), high temperature furnace, variac and LCR HiTester (HIOKI, 3532-50) was used.   Higher temperature was provided by increasing the input voltage of the variac transformer connected to

the wire-wound resistance heater. The temperature was determined using a copper/constantine thermocouple in close proximity to the sample. The above procedure was followed after checking the validity of Ohm's law behavior, where the volt-current relationship was shown to be linear. The conductivity measurements were carried out over a temperature range of 25°C up to 190°C. Moreover, the measurements were performed after sufficient time at each temperature, for establishing equilibrium values for conductivity. Conductivity σ (Ω^{-1}. cm^{-1}) was obtained after recording the resistance (R_{Ω}) by using the equations: $\sigma = 1/\rho$, $\rho = R_{\Omega}$. A_{cm}/d , where: R_{Ω} = measured resistance (Ω. cm), A_{cm} = sample surface area (cm^2), d = thickness of the sample (cm).

TABLE 1. Nominal composition of the studied meta-phosphate glasses Na$_2$O-P$_2$O$_5$.

Glass No.	Additions (g) per 100 g glass
0	None
1	0.05 NiO
2	0.25 NiO
3	0.5 NiO
4	7.5 NiO
5	0.25 NiO, 5 Na$_2$O
6	0.25 NiO, 10Na$_2$O

Dielectric constant (ε`) and dielectric loss (ε``)

The dielectric constant (ε`) and the dielectric loss (ε``) were measured at frequency range from 0.1 KHz to 5 MHz.. Measurements were carried out using LCR HiTester (HIOKI,3532-50). The temperature was determined using a copper/costantine thermocouple in close proximity to the sample. The following equations were used to obtain the dielectric constant (ε`) and dielectric loss (ε``): ε`= C.d / ε$_o$.A; ε``= ε`. D, where: C= measured capacitance of the sample, d = thickness of the sample, ε$_o$ = permittivity of free space equals 8.85×10^{-12}, A= sample surface area (m^2), D= the power factor which is obtained directly from the instrument.

Results and Discussion

Effect of NiO- content on the glass structure

The IR spectra of glasses studied are characterized by four groups of bands found at 1200-1300, 800-1200, 700-800 and 400-600 cm^{-1} (Fig.1). The

corresponding vibrations of structural groups giving rise to these bands and their assignments are listed in Table 2. The band at 1302 cm^{-1} in the base glass originates from the P=O double bonds vibrations in the PO_2^- terminal groups [3,4]. The position of this band slightly shifts from 1302 cm^{-1} to lower frequency as the Ni^{2+} ions concentration increases .It shifts to 1285 cm^{-1} in glass containing 7.5g NiO /100g glass. This shift could be attributed to elongation of P=O bond [4]. It is reasonable to assume that Ni^{2+} ions are located near P=O. It has been reported that IR absorption in the region 800-1200 cm^{-1} are sensitive for the different metaphosphate groups in the form of chain -, ring- terminal groups [3,14,15]. In this region the following bands were recorded in Ni- free glass (Glass no.0): 875,1000 and 1108 (cm^{-1}) .The absorption band at 875 cm^{-1} can be attributed to asymmetric stretching vibration of P-O-P groups linked with linear metaphosphate chain[16,17]. The band at 1000 cm^{-1} can be attributed to asymmetric stretching modes in phosphate tetrahedral linked with large metaphosphate rings[16,17]. This interpretation is in accordance with the assignment of the band at 935 cm^{-1} recorded in sodium phosphate glasses [3]. The band at 1108 cm^{-1} is also similar in position with that observed at 1130 cm^{-1} which shifted to 1098 cm^{-1} with changing glass composition[3]. This band has been assigned to asymmetric stretching of PO_2^- groups, with the formation of terminal PO_3^{2-} groups.

The introduction of NiO in the base phosphate glass seems to have no effect on the bands located at 800-1200 cm^{-1}. The bands at 762 and 722 (cm^{-1}) are connected with symmetric stretching vibrations of P-O-P. This attribution is in accordance with that suggested by other authors[3,18] . The band at 762 cm^{-1} shows tendency to shift towards higher frequencies as NiO content was increased. A sharp band was observed at 480 cm^{-1} with a shoulder at 521 cm^{-1}. The first band seems not sensitive to variation of NiO content. This band has been assigned to the bending variation of the O-P-O bond of $(PO_2)^n$ chain groups[8]. The second band at 521 cm^{-1} shifts to higher frequencies with increasing NiO content. (Fig.1). This latter band can be attributed to bending variation of P=O, which is in consistence with the assignment given to similar band found at about 500 cm^{-1} [8,16].   The shifts to higher frequencies mean a change in the strength of bond P=O with increasing NiO content. This finding may confirm the presence of Ni^{2+} ions near by the P=O bond. (Fig.2). The absorption bands shown in the region of frequencies higher than 1300 cm^{-1} were attributed to H_2O and P-OH vibrations[19].
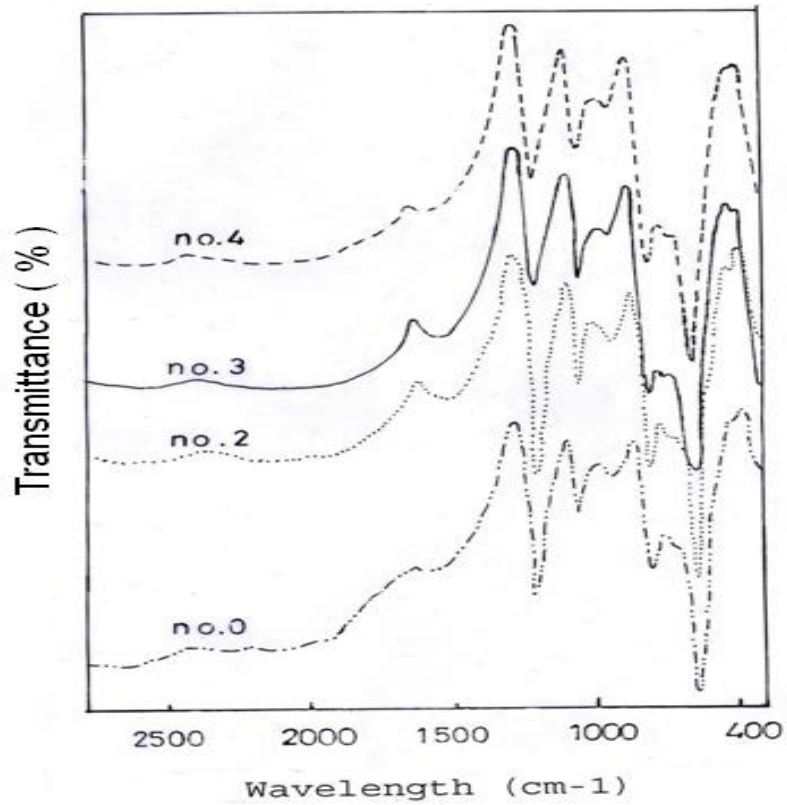
Fig. 1. IR spectra recorded in phosphate glasses with different NiO added per 100g glass, No.1=0, 2=0.25 , 3= 0.5 and 4=7.5.
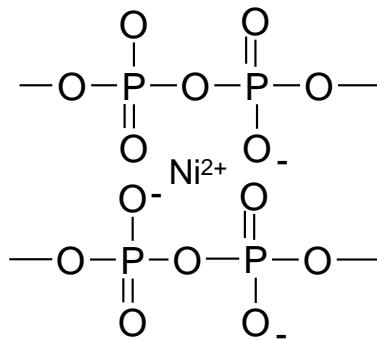

Fig. 2. A possible structural group in Ni-containing phosphate glasses.

Effect of glass composition on the glass structure

Figure 3 shows the effect of Na_2O on the IR spectra when added to the phosphate Ni-containing glass. The positions of IR absorption bands (cm^{-1}) recorded in metaphosphate glasses containing 0.25g NiO /100g glass with additional amounts of Na_2O per 100g glass and possible assignments are listed in Table 2.

TABLE 2 .Position of absorption IR bands (cm^{-1}) for the studied glasses and possible assignments.

Glass No.	$(PO_2)^-$	$(PO_3)^-$	P-O-P bridges				P=O Bonding	O-P-O Bonding
0	1301	1108	1000	(875)[1]	(762)[2]	(722)[3]	521	480
2	1302 1280	1092	1013	(875)[1]	(770)[2]	(732)[3]	525	480
3	1284	1100	996	(883)[1]	(778)[2]	(732)[3]	525 529	485
4	1285	1100	1004	(875)[1]	(770)[2]	(732)[3]	521	480
5	1388 1294	1152 1101	1020 977	(875)[1]	(770)[2]	(705)[3]	533	489
6	1382 1302	1165 1109	1028 980	(883)[1]	(762)[2]	(706)[3]	544	480

[1] asymmetric stretching. [2]symmetric stretching. [3]symmetric stretching of cyclic rings.

It is seen from Fig. 3 that, the addition of Na_2O causes a drastic change in the structure of the metaphosphate glass as reflected by the changes taking place in the IR spectra. The P-O-P bridges with ring structures with band at 1012 cm^{-1} decreases with increasing Na_2O content. This decrease was accompanied by an increase of the bands at the region $1280\text{-}1382$ cm^{-1} due to $P\text{-}O^-$ in the terminal PO_3^- groups, and formation of P-O-P with chain structure (the bands at the region $700\text{-}900$ cm^{-1}). The addition of Na_2O to phosphate glass causes a continuous breakdown of the ring type structure in the phosphate glass with the formation of more non-bridging oxygens and depolymerization of the network.

a.c. Conductivity

Figure 4 (a-e) shows the relation between log σ and reciprocal of temperature for different Ni-doped and un-doped phosphate glasses measured at different frequencies (1KHz to 5MHz). The dependence of log σ on 1000 /T is characterized by two temperature regions. The first is the lower temperature region where the conductivity increases slightly with temperature. The second region is the higher temperature region, where σ increases with higher rate as the temperature increases. It can be seen from Fig. 4 (a-e) that, (for all glasses) the first region extends towards higher temperature as the measurement is carried out at higher frequencies. For low frequencies (particularly 1KHz and 100 KHz) the relation between log σ and reciprocal of the temperature obeys the well known Arrhenius equation: $\sigma = \sigma_o\ e^{-\Delta E/kT}$, where σ_o is the pre-exponential factor, ΔE is

the activation energy , k is the Boltzmann constant and T is the absolute temperature. The activation energies of conduction at low and high temperature regions are calculated for those samples measured at 1KHz and listed in Table 3. It can be noticed that, the energy gap at low temperature region of samples is lower than that of high temperature region. This may be attributed to the different mechanisms in either region. The activation energy ,ΔE, has values in the range 0.06 - 0.4eV and 1.25 - 1.6 eV at the low and high temperature regions, respectively, for the free and Ni-containing glasses. These data suggest that, the mechanism predominating in the high temperature region is ionic in nature, while it is electronic in origin in the low temperature region.
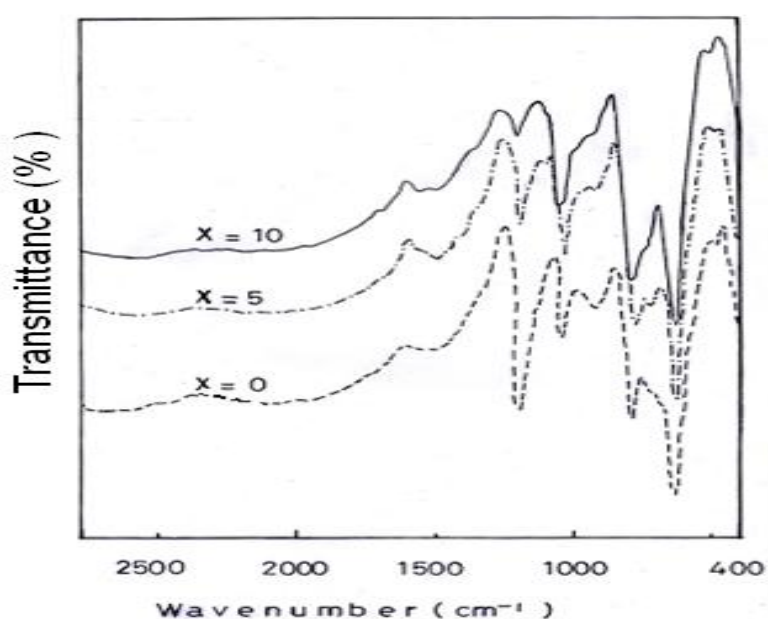

Fig. 3. IR spectra recorded in phosphate glasses with 0.25 NiO and additional x gram Na$_2$O (added per 100g glass).

TABLE 3. Activation energy (ΔE) of Ni-containing glasses measured at 1 KHz .

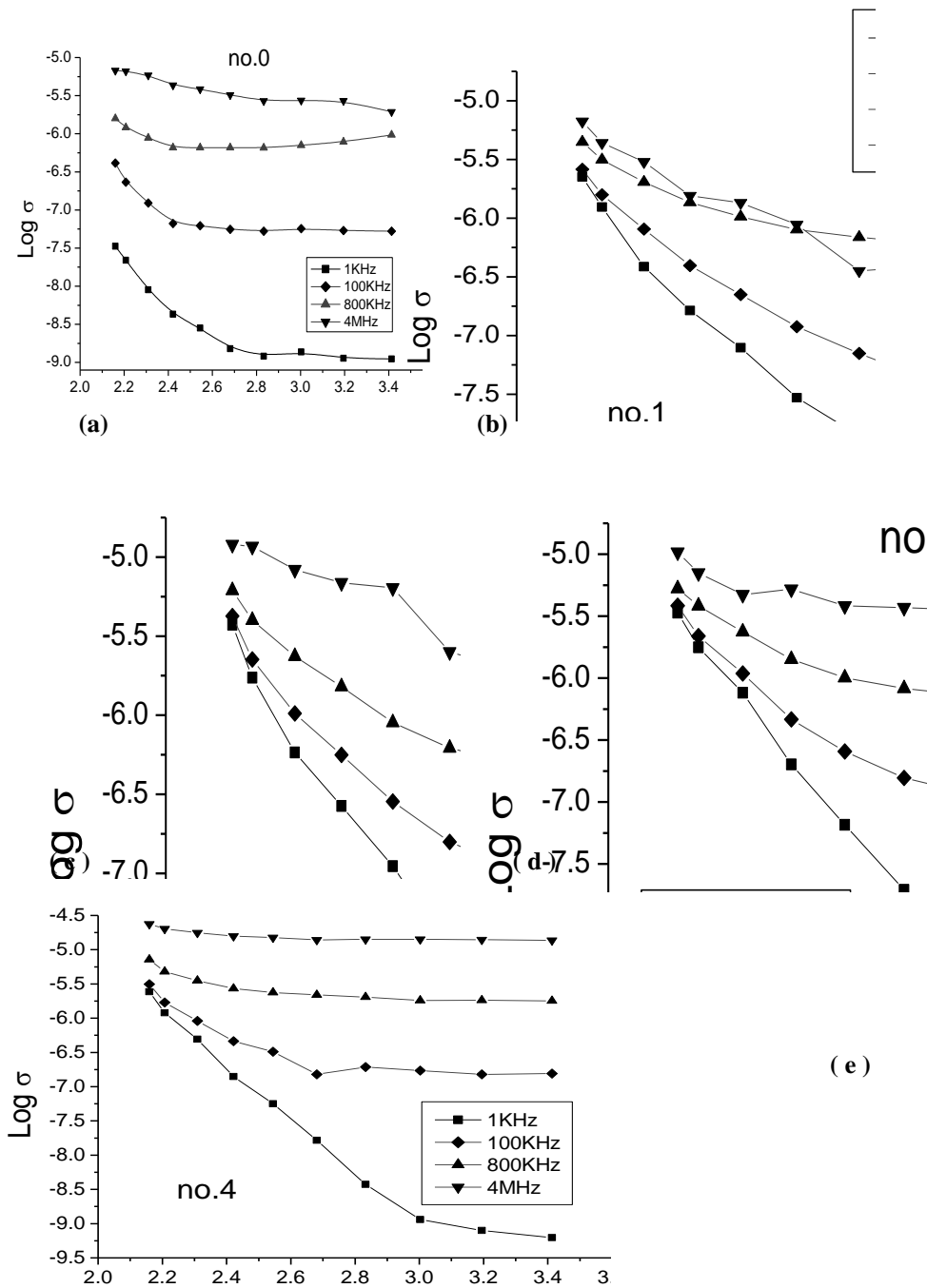| Glass No.	0	1	2	3	4
ΔE(eV) at low temperature	0.058	0.306	0.263	0.377	0.396
ΔE(eV) at high temperature	1.245	1.614	1.429	1.45	1.603

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Fig. 4. Log conductivity versus reciprocal of temperature at 1kHz to 5MHz for glasses containing different NiO concentrations (No.0= zero,1= 0.05,2=0.25, 3= 0.5, 4 = 7.5 (g NiO per 100 g glass) .

It is worthy to notice (Fig. 4e) that, for high Ni-content glasses the log σ becomes independent of temperature for high frequency measurements and with activation energy close to that of the Ni-free glass. This finding may indicate dominance of electronic conduction through hopping mechanism in such case.

The effect of NiO content on the conductivity was studied at different temperatures and frequencies. Figure 5 shows an example of such variation measured at 140°C. It can be seen that, the first additions of NiO cause an abrupt increase in the conductivity up to maximum value for glass with 0.25 g NiO per 100g glass. Further increase in the NiO - content seems to hardly affect the conductivity due to blocking of interstitial vacancies in the glass structure.

Figure 6 shows the variation of log conductivity (Ω^{-1}. cm^{-1}) with frequency at different temperatures for meta-phosphate glasses containing 0.25g NiO /100g glass. It can be noticed that, the conductivity increases as the frequency or temperature increases. The frequency dependence of ac conductivity of materials has been studied by several authors[20, 21]. Increasing conductivity with increasing frequency at the same temperature has been attributed to the dielectric behavior of the sample[20]. The increased conductivity with increasing temperature is more pronounced at lower frequencies than at the higher ones.

Dielectric properties

Figures 7 and 8 show the temperature dependence of dielectric constant $\varepsilon`$ and dielectric loss $\varepsilon``$, respectively, at spot frequency 1 KHz for phosphate glasses containing different NiO concentrations. A transition temperature can be seen in the range between 140 and 160 °C above which $\varepsilon`$ and $\varepsilon``$ abruptly increase. This observation has been attributed to the superposition of the two mechanisms, conduction and migration[20]. The effect of temperature below 100°C seems to be very small for $\varepsilon``$. In the case of $\varepsilon`$ this effect is also very small for samples with high NiO content (No.3 and 4), yet samples with low concentrations (No.1 and 2) may show dispersion effect similar to NiO-free glass (No. 0). The small effect of temperature below 100°C reflects that the migration mechanism plays a negligible role[20]. The observed increase of the dielectric constant (Fig.7) with increasing temperature is a typical behavior of the polar dielectrics in which the orientation of dipoles is facilitated with rising temperature and thereby the dielectric constant is increased. At low temperatures, the contribution of electronic and ionic components to the total polarizability will be small. As the temperature is increased the electronic and ionic polarizability sources start to increase [22].
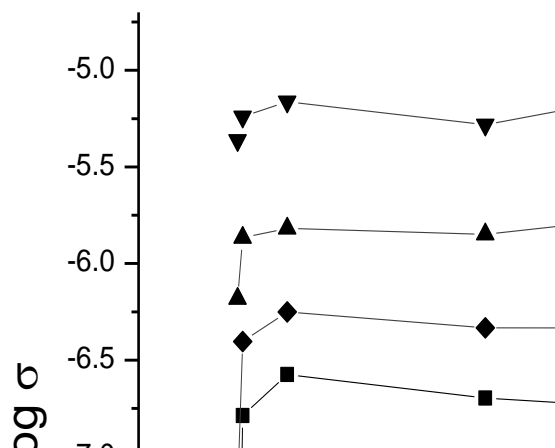
Fig. 5. Variation of conductivity with differenNiO - content (g per 100g glass) measured at 140°C for different frequencies.
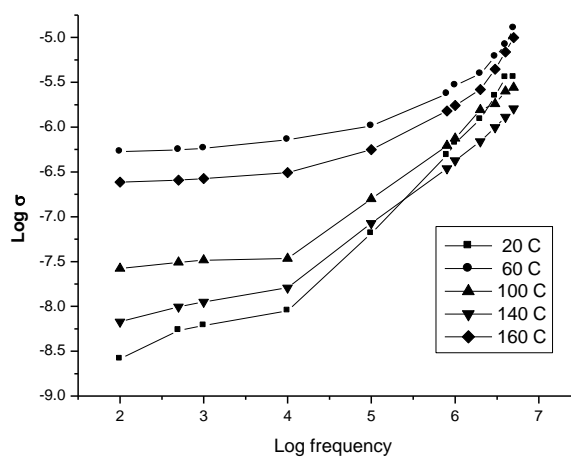

Fig. 6. Log conductivity vs. log frequency at different temperatures for glasses containing 0.25g/100g glass batch .
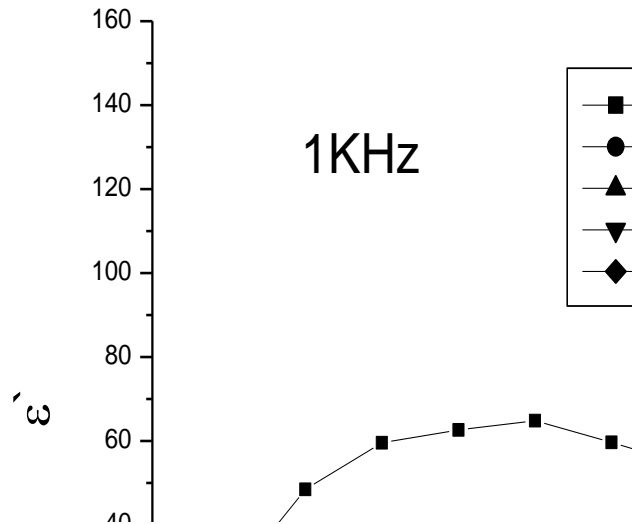
Fig. 7. Variation of dielectric constant with temperatures at 1kHz for glasses with different NiO concentrations (g /100g glass).
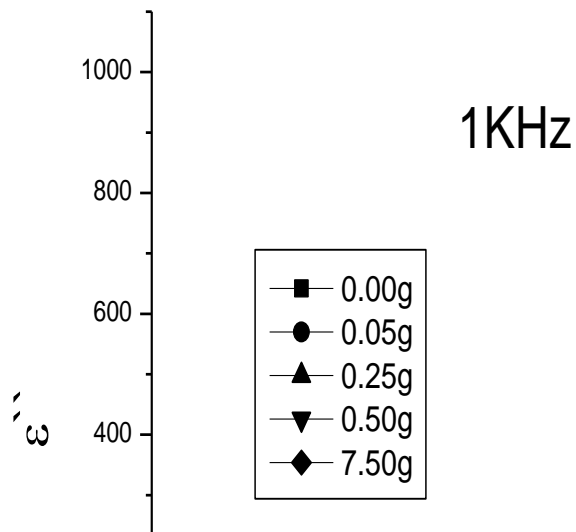

Fig. 8. Variation of dielectric loss ε`` with temperatures at 1kHz for glasses with different NiO concentrations (g /100g glass).

Figure 9 shows the variation of dielectric constant with frequency for meta-phosphate glasses containing 0.25g NiO /100g glass. It can be noticed that, the dielectric constant ε' decreases with increasing frequency. This may be due to the fact that as the frequency increases, the polarizability contribution from ionic and orientation sources decreases and finally disappears due to the inertia of the ions[22]. The noticed increase in the dielectric constant ε' at higher frequencies may indicate that the probability of new absorption may occur. The decrease in ε' with increasing frequency has also been attributed to the lag of the molecules behind the alternation of the electric field at higher frequency [23, 24].
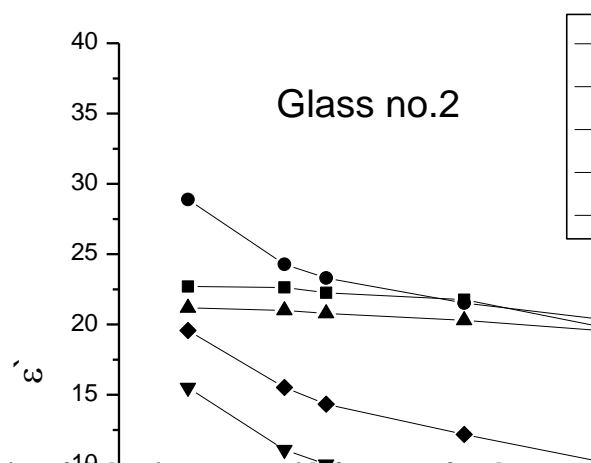

Fig. 9. Variation of dielectric constant with frequency for glasses containing 0.25g NiO /100g glass .

Conclusions

IR spectra revealed that the Ni^{2+} ions are located near P=O. Addition of Na_2O to metaphosphate glass causes a breakdown of the ring type structure in the studied glasses with the formation of more non-bridging oxygens and depolymerization of the network.

The electric conductivity of Ni-containing glasses is more than that of free phosphate glass, and attained maximum value in presence of 0.25 g NiO per 100g glass. Further increase of NiO content hardly affects the conductivity.

The relation between log σ and reciprocal of the temperature obeys the well known Arrhenius equation. The data suggest that, the mechanism predominating in the high temperature region is ionic in nature, while it is electronic in origin in the low temperature region for all glasses and for low frequencies (1KHz-100kHz) measurements. Introducing NiO extends the low temperature region to the high temperature one, indicating predominant of electronic conduction

through hopping mechanism for high Ni-content (7.5g/100g glass) metaphosphate glasses particularly at high frequency (4MHz).

The dielectric constant ε' and the dielectric loss $\varepsilon``$ increase with increasing temperature. This behavior is typical to the polar dielectrics. A transition temperature can be seen in the range between 140 and 160 °C above which $\varepsilon`$ and $\varepsilon``$ abruptly increase. This observation has been attributed to the superposition of the two mechanisms, conduction and migration.

The dielectric constant ε' decreases with increasing frequency due to decrease in the polarizability contribution from ionic and orientation sources and the lag of the molecules behind the alternation of the electric field at higher frequency.

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الخصائص الكهربية والبنائية لزجاج الفوسفات المحتوى على اكسيد النيكل

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تم تحضير بعض انواع من زجاج الفوسفات المحتوى على 7.5-0.5 جم/100جم زجاج باستخدام ملح فوسفات الصود ثنائى الهيدروجين وقياس طيف الاشعة فوق الحمراء ، والتوصيل الكهربى للتيار المتردد، وثوابت العزل (`ε) و(``ε). ودلت قياسات طيف الاشعة فوق الحمراء أن ايونات النيكل تتواجد بقرب الرابطة P=O، وأن اضافة اكسيد الصوديوم يسبب كسر الروبط الحلقية بالزجاج مع تكون كميات أكثر من الاكسجين غير ـ الرابط ومن الشبكية البنائية غير المتبلمرة. وأن التوصيل الكهربى للزجاج المحتوى على النيكل يكون أعلى من زجاج الفوسفات الخالى منه، وتصل أعلى قيمة له فى الزجاج المحتوى على 0.25 جم /100 جم زجاج. وتدلل العلاقة بين لوغاريتم التويصل الكهربى(σ log) ومقلوب درجة الحرارة أن ميكانيكية التوصيل تكون بالايونات فى منطقة الحرارة المرتفعة بينما يكون بالالكترونات فى منطقة الحرارة المنخفضة، ويمتد التوصيل بالالكترونات الى منطقة الحرارة المرتفعة عند اضافة اكسيد النيكل للزجاج وذلك من خلال التوصيل بميكانيكية وثب الالكترونات، وأن ثوابت العزل (`ε) و(``ε) تزداد مع ازدياد درجة الحرارة بينما يقل ثابت العزل (`ε) مع زيادة التردد المستخدم.