Optical Absorption Edge And IR Spectroscopy Of Phosphate Glasses

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Abstract

Sample of glass systems with compositions $(X\% BaO - (100-x)\% P_2O_5)$, $(X\% CaO - (100-x)\% P_2O_5)$, $(X\% MgO - (100-x)\% P_2O_5)$ where (x=20,30,40,50) were prepared .The result of investigations of density, optical absorption, refractive index, and infrared spectra are reported. The density measurements show that the density increases with BaO, CaO, and MgO content in the glass systems. The optical absorption measurements show that the fundamental absorption edge in the optical absorption is due

I. Introduction

Nowadays, glasses have a prominent role in electronics and have wide industry applications, space research, computer memories, etc. Phosphate as a glass host possesses many advantages, such as a high thermal expansion coefficient, which easily for the fiber preparation process [1]. Phosphate glasses have also emerged as a promising group of glasses for optical amplifiers, fibers, laser, etc. [2]. Phosphate glasses are an interesting vitreous system that has gained great attention in the past and recent applications [3].

The study of optical absorption has been one of the most productive methods in understanding the band structure and energy gap of both crystalline and amorphous materials. The energy gap of a number of amorphous oxide glasses [4-7] has been analyzed based on the theory suggested by Tauc et al. [8]. And Mott [9]. It has been reported [10-11] that the optical energy gap's value is affected by the addition of modifier ions in the glass structure.

In the present work, we prepared the glass samples. The composition dependence of the composition behavior, structural and optical properties of phosphate glasses containing BaO, CaO, and MgO has been investigated.

Optical absorption spectra of varying compositions were recorded in the visible region, and optical parameters such as optical energy gap $(E_{\rm opt})$ and Urbach energy $(E_{\rm o})$ were determined. It was found that the fundamental

to the forbidden indirect transition. The optical band gaps (E_{opt}) show a decreasing trend with an increasing amount of BaO, CaO, and MgO in the glass network. The glass exhibit a high refractive index and are useful for solid-state laser and optoelectronic devices. From the infrared absorption spectra, it is found that the addition of BaO, CaO, and MgO does not introduce any new absorption band in the infrared spectrum of the glass system.

Keywords: Optical properties, Absorption Edge, Structure properties, Oxide Glasses, phosphate oxide glasses,

absorption of the glasses is dependent upon compositions and arises from forbidden indirect transitions.

The infrared spectra showed that the addition of BaO, CaO, and MgO does not introduce any new absorption band in the infrared spectrum of the glass systems studied.

II. Experimental Details

For the present study , three groups of $X\% BaO - (100-x)\% P_2O_5$, $X\% CaO - (100-x)\% P_2O_5$ and $X\% MgO - (100-x)\% P_2O_5$ glasses of different compositions (x=20,30,40 and 50) were prepared . The starting materials, namely P_2O_5 , BaO, CaO, and MgO, each of purity 99.99% and in the powder form, were procured from Aldrich. All samples were fabricated in the air under the same conditions.

The appropriate amounts of oxides powder for each glass system were weighed and mixed in alumina crucibles using an alumina rod and then preheated at about (220°C) for one hour to minimize material volatilization. The crucible then transferred to a melting furnace for one hour. The melting temperature used was (950-1050)°C. The melts were stirred from time to time to get a homogenous melt. The glass discs (2-3) mm thick were made by raped quenching the melt by poured on the stainless steel plate and cast into a disc shape. It was believed that the glass preparation history, that is, melting temperature, melting time, annealing temperature, annealing time, rate of cooling, and melting atmosphere, could affect the properties of glasses, and it was decided to keep all these parameters the same for the samples of the one series.

X-ray diffraction (XRD) patterns of the samples were recorded using (X-ray 6000 diffractometer). The density of the samples was measured by the Archimedes principle using toluene as an immersion liquid. The FTIR spectra of the bulk samples in transmission mode were recorded in (8400S Perkin Elmer IR Spectrophotometer). For FTIR measurements, pellets of a thickness (1mm) were obtained by pressing a mixture of one part of the glass and 60 parts of kBr at a pressure of (200 kg/cm²). The absorbance mode samples' optical absorption spectra were recorded in the wavelength range (200-900) nm at room temperature (UV-VIS-SPECTROPHOTO METTER SP-8001).

III. Results and Discussion

A. X-Ray Diffraction (XRD).

X-ray is an electromagnetic wave of wavelength near about (1A°), which is the same order of magnitude as the lattice constant of crystals. Thus, X-rays are useful in the analyses of crystal structures [12].

In the present work, the measurements of diffraction patterns have been made using (X-ray 6000 diffractometer) model Expert PRD multipurpose diffraction (Voltage = 30 to 60 kV) the current of the Expert PRD is (10-66) mA.

All sample prepared was tested for the crystallinity. The patterns obtained from the X-ray diffraction (XRD) show broad peaks, which is a characteristic of an amorphous material [13]. Figure (1) shows the X-ray diffraction patterns for ($P_2O_5 - BaO$), ($P_2O_5 - CaO$), and ($P_2O_5 - MgO$) glass samples.



Fig. (1): X-ray diffraction patterns of three samples of phosphate glasses.

B. Density Measurements

The glass density (ρ_G) was measured by using the following relation [1]:

where (ρ_L) is the density of immersion liquid, which is toluene ($\rho_L=0.86$ g/cm³), (W_{air}) is the weighted sample in air, and (W_L) is the weighted sample when immersed in the immersion liquid. The molar volume (V_m) was calculated by using the obtained density (ρ_G) and the weight of one mole of the glass sample via the following relation [1]:

where (X_i) and (M_i) refer to the molar fraction and molecular weight of the (i^{th}) of the component, respectively. The obtained values of the density and molar volume have been presented in Table (1) and Figure (2) for $(P_2O_5 - BaO)$ glass system. It is observed that the density of the sample increases with an increase in BaO content, which may be due to replacement P_2O_5 with BaO molecules [14]. The result is in good agreement with Tahir [15]. The molar volume decreases with increases in BaO content. The slight decrease in the molar volume may be due to the lattice's rearrangement [16].

Table 1: Composition, Density, and Molar volume forP2O5 - BaO glasses.

| mol % BaO | ρ (gm/cm ³) | V _m (gm/mol) |
|-----------|------------------------------|-------------------------|
| content | | |
| 20 | 1.23 | 117.2 |
| 30 | 1.247 | 116.4 |
| 40 | 1.268 | 115.3 |
| 50 | 1.293 | 113.9 |



Fig. 2: Relative Density and Molar volume against BaO content in P₂O₅ - BaO glasses.

The density of P_2O_5 – CaO glass system increases from 1.257 g/cm³ to 1.30 g/cm³ while the molar volume decreases from 99.29 g/mol to 76.36 g/mol with the increment of the CaO content in the glass system as shown in Figure (3) and Table (2). The increases in density may be due to non-bridging oxygen, which essentially alters the glass structure [17].

| Table 2: | Composition, | Density, | and N | Aolar | volume | for |
|----------|--------------|----------|-------|-------|--------|-----|
| | P2O5 | -CaO gla | sses. | | | |

| mol % CaO | ρ (gm/cm ³) | $V m (cm^3)$ |
|-----------|------------------------------|--------------|
| content | | |
| 20 | 1.257 | 99.29 |
| 30 | 1.267 | 91.69 |
| 40 | 1.283 | 84.08 |
| 50 | 1.300 | 76.36 |



Fig. 3: Relative Density and Molar volume against CaO content in P₂O₅ - CaO glasses.

Figure (4) shows the variation of density and molar volume of $(P_2O_5 - MgO)$ glass system. As shown from the Figure, the density increases, and molar volume decreases with the increment of MgO content in the glass system. The values of density and molar volume for such a glass system are tabulated in Table (3).

Table 3: Composition, Density, and Molar volume forP2O5-MgO glasses.

| mol | % MgO content | ρ (gm/cm ³) | V m (gm/mol) |
|-----|---------------|-------------------------|--------------|
| 20 | | 1.317 | 92.34 |
| 30 | | 1.326 | 83.99 |
| 40 | | 1.342 | 75.41 |
| 50 | | 1.365 | 66.69 |



Fig. 4: Relative Density and Molar volume against MgO content in (P₂O₅-MgO) glasses.

C. Optical absorption

The optical absorption coefficient measurements, particularly near the fundamental absorption edge, provide a standard method for investigating optically induced electronic transitions and provide some ideas about the band structure and energy gap in both crystalline and noncrystalline material [18].

The absorption coefficient $\alpha(\omega)$ of the optical absorption near the band edge in many amorphous semiconductors show an exponential dependence on photon energy ($\hbar\omega$) and obey empirical relation due to Urbach [19],

$$\alpha(\omega) = \alpha_o \; e^{(\frac{\hbar\omega}{E_o})} \;(3)$$

where (α_o) is a constant and (E_o) related to the width of the tail of localized states in the bandgap. In the high absorption coefficient region, the absorption edge for non-direct transitions having fairly constant matrix elements and a relaxed <u>k</u>-conservation selection rule in most amorphous semiconductors [20] can be determined from the relation

$$\alpha(\omega) = \frac{B}{\hbar\omega} (\hbar\omega - E_{opt})^r \dots (4)$$

where (B) is a constant and (E_{opt}) the optical energy gap of the material.

The absorption coefficient $\alpha(\omega)$, which is a function of wavelength, can be calculated near the edge by using the following relation [21]:

where (d) is the thickness of the glass sample (I_o) and (I_t) are the intensities of the incident and transmitted beams, respectively.

Refractive index (n) is determined from the optical band energy (E_{opt}) using the relation proposed by Dimitrov and Sakka [22]:

The molar refractivity (R_m) has been calculated by using the relation [23] :

$$R_m = \left\{\frac{n^2 - 1}{n^2 + 2}\right\} V_m....(7)$$

a) optical absorption of $(P_2O_5 - BaO)$ glasses.

Optical absorption measurements were made as a function of photon energy at room temperature. Figure (5) shows the absorbance as a function of the wavelength for glass samples of different compositions. Figure (6) shows the plot of $(\alpha \hbar \omega)^{1/2}$ versus photon energy $(\hbar \omega)$ and the equivalent Urbach plot is presented in Figure (7) in which the absorption coefficient plot as a function of $(\hbar\omega)$ for different compositions of (P₂O₅ - BaO) glasses. The values of (E_{opt}) determined from Figure (6) by extrapolating the liner part of the curve to $(\alpha \hbar \omega)^{1/2} = 0$, and the values of (E_0) , which are determined from Figure (7) by using equation (3), are tabulated in Table (4). It is clear from Figure (8) that the optical band energy (Eopt) decreases as the proportion of BaO content in the glass network is increased. Also, it found that the width of the band tails of the localized state (E_0) increased with BaO content in the glass sample, as shown in Figure (9).



Fig. 5: Optical absorption spectra of P₂O₅-BaO glasses.



Fig. 6: $(\alpha \hbar \omega)^{1/2}$ is plotted against photon energy for P₂O₅-BaO glasses.



Fig.7: Variation of ln (α) with photon energy for P₂O₅-BaO glasses.



Fig.8: Variation of optical band energy (E_{opt}) with BaO content in P₂O₅-BaO glasses.



Fig.9: Variation of Urbach energy (E₀) with BaO content in P₂O₅-BaO glasses.

Refractive index (n) and molar refractivity can be evaluated from equation (6) and (7) for such a glass system. As shown in Figure (10), the refractive index increases with the increment of BaO content in the glass network while the molar refractivity decreases with BaO content. The value of (E_{opt} , E_o , n, and R_m) of the (P_2O_5 -BaO) glass network are tabulated in Table (4).

| able | e 4:Optical energy band, Orbach energy, refractive |
|------|---|
| | index, and molar refraction for P ₂ O ₅ -BaO. |
| | |

| Mol % | E _{opt} (eV) | $E_{o}(eV)$ | n | R _m |
|---------|-----------------------|-------------|--------|----------------|
| BaO | | | | |
| content | | | | |
| 20 | 3.98 | 0.181 | 2.1773 | 65.04 |
| 30 | 3.905 | 0.215 | 2.1884 | 64.96 |
| 40 | 3.83 | 0.251 | 2.2035 | 64.84 |
| 50 | 3.755 | 0.287 | 2.2189 | 64.55 |

b) Optical absorption of $(P_2O_5 - CaO)$ glasses.

The optical absorption spectra for $(P_2O_5 - CaO)$ glasses as a function of wavelength are shown in Figure (11). The values of optical band energy (Eopt) are obtained by extrapolation from the linear region of the plot of $(\alpha \hbar \omega)^{1/2}$ versus ($\hbar\omega$). Figure (12) represents ($\alpha\hbar\omega$)^{1/2} as a function of photon energy $(\hbar\omega)$ for such glasses. The graph shows straight lines with some deviation at low photon energy. Figure (13) shows (E_{opt}) as a function of CaO content in the glass network. The logarithm of the absorption coefficient is plotted against photon energy in accordance with equation (3) and shown in Figure (14). The appearance of Figure (14) shows that the exponential behavior of the absorption edge can be demonstrated. The values of (E_o) were calculated from Figure (14) using equation (3) and plotted as a function of CaO content, as shown in Figure (15) that (E_0) varies irregularly with increasing CaO content.



Fig.10: Variation of refractive index (n) and molar refraction (R_m) with BaO content in $(P_2O_5 - BaO)$ glasses.



Fig.11: Optical absorption spectra of (P₂O₅-CaO) glasses.



Fig.12: $(\alpha \hbar \omega)^{1/2}$ is plotted against photon energy for P₂O₅-CaO glasses.



Fig.13: Variation of optical band energy (E_{opt}) with CaO content in (P₂O₅-CaO) glasses.



Fig.14: Variation of ln(α) with photon energy for (P₂O₅-CaO) glasses.



Fig.15: Variation of Urbach energy (E₀) with CaO content in (P₂O₅-CaO).

The variation of refractive index (n) and molar refractivity (R_m) as a function of CaO content in the glass network are in Figure (16). Table (5) shows that the refractive index increased with the increment of CaO content in the glass network. The value of (E_{opt} , E_o , n, and R_m) of the (P_2O_5 -CaO) glass network are tabulated in Table (5).





Table 5: Optical energy band, Urbach energy, refractive index, and molar refraction for (P₂O₅-CaO) glasses.

| Mol % | E _{opt} (eV) | E _o (eV) | n | R _m |
|---------|-----------------------|---------------------|-------|----------------|
| CaO | | | | |
| Content | | | | |
| 20 | 3.82 | 0.468 | 2.206 | 55.91 |
| 30 | 3.78 | 0.586 | 2.214 | 51.99 |
| 40 | 3.74 | 0.631 | 2.222 | 47.59 |
| 50 | 3.70 | 1.030 | 2.23 | 43.4 |

c) Optical absorption of (P2O5-MgO) glasses.

The optical absorption measurements were carefully made as a function of photon energy at room temperature. Figure (17) shows the measurements of absorbance against wavelength for such glasses. Figure (18) shows the plot of $(\alpha\hbar\omega)^{1/2}$ versus the photon energy for various mentioned glasses' compositions.

The straight lines in Figure (18) suggest that the absorption follows the quadratic relation for the inter-band transition given by Davis and Mott [9]. The optical band gap was obtained by extrapolating the linear part of Figure (18) to the value of $(\alpha\hbar\omega)^{1/2}$ =0. As shown in Figure (19) and Table (6), there is a systematic decrease in the value of (E_{opt}) with increasing MgO content in the glass network, which suggests that the degree of disorder in these glasses is increased. The logarithm of the absorption coefficient is plotted versus the photon energy, as shown in Figure (20). The average value (E_o) was calculated from the slop of the straight lines in Figure (20) and shown in Figure (21) as a function of MgO content.



Fig.17: Optical absorption spectra of P2O5-MgO glasses.



Fig.18: $(\alpha\hbar\omega)^{1/2}$ is plotted against photon energy for (P₂O₅-MgO) glasses.



Fig.19: Variation of Urbach energy (E₀) with MgO content in P₂O₅-MgO glasses.



Fig.20: Variation of ln (α) with photon energy for (P₂O₅-MgO) glasses.



Fig.21: Variation of Urbach energy (E₀) with MgO content in P₂O₅-MgO.

Refractive index (n) and molar refractivity (R_m) was calculated and plotted as a function of MgO content. As shown in Figure (22), the Refractive index (n) increased with the increment MgO. Table (6) shows the values of (E_{opt} , E_o , n, and R_m) as a function of MgO content in the glass network.



Fig.22: variation of refractive index (n) and molar refraction (R_m) with BaO content inP₂O₅-PbO-BaO glasses.

| Mol % | E _{opt} (eV) | E _o (eV) | n | R _m |
|---------|-----------------------|---------------------|-------|----------------|
| MgO | | | | |
| content | | | | |
| 20 | 4.14 | 0.374 | 2.143 | 50.33 |
| 30 | 4.00 | 0.301 | 2.17 | 46.43 |
| 40 | 3.85 | 0.310 | 2.199 | 42.33 |
| 50 | 3.70 | 0.832 | 2.23 | 38.01 |

Table 6: Optical energy band, Urbach energy, refractive index, and molar refraction for P₂O₅-MgO glasses.

D. Infrared spectra

a) Infrared absorption spectra of $(P_2O_5 - BaO)$ glasses.

Infrared absorption spectra of P₂O₅- BaO) glasses of different compositions are shown in Figure (23). The band positions of such glasses are listed in Table (7). The absorption band in (472-493)cm⁻¹ appears in these glasses and corresponds to the absorption band at (500) cm⁻¹ in crystalline P₂O₅ which is known to be at the fundamental frequency of the $(PO_4)^3$ -group [24]. The absorption bands at (730-755)cm⁻¹ (1005-1080)cm⁻¹ and (1265-1278) cm⁻¹ could are due to the (P-O-P) range frequency (P-O) stretching frequency and stretching vibration of the (P=O) double band. The absorption band at (900-921)cm⁻¹ can be attributed to the (Ba-O-Ba) or (P-O-Ba) bands [25]. The absorption band at (1637-1645)cm⁻¹ is due to the moisture of (kBr) powder [26]. The absorption band at (3410-3665) cm⁻¹ was observed for these glasses. This was believed to be caused by a small amount of water in the glasses.



| mol% BaO | | Band position (cm ⁻¹) | | | | | |
|-------------|-----|-----------------------------------|-----|------|------|------|------|
| Content | 400 | 700 | 900 | 1000 | 1200 | 1600 | 3000 |
| 20 | 493 | 730 | 921 | 1010 | 1265 | 1645 | 3430 |
| 30 | 480 | 740 | 908 | 1010 | 1265 | | 3410 |
| 40 | 475 | 735 | | 1005 | 1278 | 1637 | 3425 |
| 50 | 472 | 755 | 900 | 1080 | 1273 | 1699 | 3665 |

b) Infrared absorption spectra of $(P_2O_5 - CaO)$ glasses.

Infrared spectra were obtained on glass with CaO concentration varying from (20 to 50) mol % with negligible shifts in band positions. The FTIR spectra of the P₂O₅- CaO glasses, as shown in Figure (24). Table (8) shows the position bands of the glasses. It has been shown that most of the absorption bands in the glasses are the same as for crystalline P₂O_{5.} The absorption band at (460-470)cm^{-1,} which appears in such glasses corresponding to the absorption at (500) cm⁻¹ in crystalline P₂O₅, is Known to be al the fundamental frequency of the $(PO_4)^{3-}$ group [24]. The absorption bands at (752-779)cm⁻¹, (1090)cm⁻¹, and (1295-1298)cm⁻¹ can be attributed to the (P-O-P) range frequency, (P-O) stretching frequency, and stretching vibration of the (P=O) double band. The absorption band at (900-904)cm⁻¹ could be due to the (Ca-O-Ca) or (P-O-Ca) bands [25]. An absorption band at (1687-1695)cm⁻¹ is due to kBr powder's moisture as assigned before [26]. The absorption band at (3620-3672)cm⁻¹ was believed to be due to the small amount of water trapped in the glass.



Fig.23: Infrared absorption spectra in (P₂O₅-BaO) glasses.



Fig.24: Infrared absorption spectra in (P₂O₅-CaO) glasses.

| mol% | Band position (cm ⁻¹) | | | | | | | | | |
|-------------|-----------------------------------|---|---------|----------|----------|-----|----------|--|--|--|
| Conten t | 40 0 | $\begin{array}{c ccccccccccccccccccccccccccccccccccc$ | | | | | | | | |
| 20 | 46 | 75 | 90 | 109 | 129 | 168 | 362 | | | |
| | 0 | 8 | 4 | 0 | 5 | 7 | 0 | | | |
| 30 | 46 | 77 | 90 | 109 | 129 | 169 | 366 | | | |
| | 0 | 9 | 4 | 0 | 8 | 5 | 6 | | | |
| 40 | 47 0 | 75 2 | 90 0 | 109 0 | 129 5 | | 367 2 | | | |
| 50 | 47 | 77 | 90 | 109 | 129 | 168 | 365 | | | |
| | 0 | 5 | 0 | 0 | 5 | 7 | 8 | | | |

Table.8: Band positions in (P₂O₅- CaO) glasses.

c) infrared absorption spectra of (P₂O₅- MgO) glasses.

Spectra were obtained for (P_2O_5-MgO) and presented in Figure (25). The band position is listed in Table (9). The absorption band at $(458-465)cm^{-1}$ appears in these glasses are corresponding to the $(PO_4)^{3-}$ as mentioned earlier [24]. The absorption bands at $(756-760)cm^{-1}$ and (1303- $1317)cm^{-1}$ could be possibly due to the (P-O-P) ring frequency and the stretching vibration of the (P=O) double band. The absorption band at $(920-925)cm^{-1}$ can be attributed to the (Mg-O-Mg) or (P-O-Mg) bonds [25]. The absorption band at $(1660-1686)cm^{-1}$ is caused by moisture in the kBr powder. As explained before, The absorption band at (3280- $3609)cm^{-1}$ was ascribed to trapped water in the glass.



Fig.25: Infrared absorption spectra in (P₂O₅-MgO) glasses.

Table. 9: Band positions in P2O5-MgO glasses .

| mol% | Band position (cm ⁻¹) | | | | | | | |
|---------|-----------------------------------|-----|-----|------|------|------|--|--|
| MgO | | | | | | | | |
| Content | 400 | 700 | 900 | 1200 | 1600 | 3000 | | |
| 20 | 464 | 758 | 920 | 1304 | 1686 | 3609 | | |
| 30 | 462 | 756 | 920 | 1317 | 1660 | 3378 | | |
| 40 | 458 | 760 | 925 | 1305 | 1680 | 3280 | | |
| 50 | 465 | 758 | 920 | 1303 | 1680 | 3574 | | |

Density is a useful parameter to measure when investigating changes in the structure of glasses. It is affected by structural softening / compactness, geometrical configuration changes, coordination number, and interstitial spaces.

The density measurements of (P_2O_5-BaO) , (P_2O_5-CaO) , and (P_2O_5-MgO) glasses increase with increasing BaO, CaO, and MgO content in the glass network, while the molar volume in all glass samples decreases. The increase in density could be due to non-bridging oxygen, which essentially alters the glass structure. As expected, BaO, CaO, and MgO to the phosphate network cause some Ba, Ca, and Mg atoms' structural rearrangement.

The increase in density in all glasses studies is associated with changes in the structures caused by the decrease of the inter-atomic spacing, resulting in a more compact and dense glass. In general, the density increase can be related to either the glass's constitutions or the structural variation in the type of phosphate structural units.

The (XRD) pattern of all glass samples was found to show no discrete or continuous sharp peaks but exhibit broad halo at around 2Θ =(23-28)°, which reflected the amorphous glass structure's character. This indicates the absence of long-range atomic arrangement and the threedimensional network's periodicity in the quenched material.

As can be seen from the infrared absorption spectra of phosphate glasses that these spectra are similar to the infrared absorption spectrum of crystalline P_2O_5 [24], which suggests that the phosphate tetrahedral are dementing the structure of these glasses. We need more results for such glasses in different compositions and different annealing temperatures in order to confirm our results.

The fundamental absorption edges for all the series of glasses were analyzed in terms of the theory of Davis and Mott [9], and the values of the so-called optical energy gap (E_{opt}) were evaluated. Data for all glasses seemed to fit the Davis and Mott theory [9] well, and this was taken as

evidence of the occurrence of non-direct inter-band transition in these glasses.

The optical energy gap (E_{opt}) was found to decrease with increasing BaO, CaO, and MgO content for all series of glasses in the present work. Such variation can be explained by suggesting that the non-bridging oxygen ion content increases with increasing BaO, CaO, and MgO contents shifting the band edge to higher energies and leading to a decrease in the optical energy value gap (E_{opt}) [16].

The increases in the refractive index with increasing BaO, CaO, and MgO contents related to the decrease in energy bandgap related to the glass's compactness [27], [28].

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