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Effect of gamma rays absorbed doses and heat treatment on the optical absorption spectra of silver ion-exchanged silicate glass

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ABSTRACT

Samples of a commercial silicate glass have been subjected to ion exchange at 320 °C in a molten mixture of $AgNO_3$ and $NaNO_3$ with molar ratio of 1:99 and 5:95 for 60 min. The ion exchange process was followed by gamma irradiation in the dose range of 1–250 kGy and heating at the temperature of 550 °C for different time periods ranging from 10 to 582 min. The spectral absorption in UV–Vis range of the Ag–Na ion exchanged glass was measured and used to determine the states of silver prevailing in the glass during the ion exchange, the gamma irradiation and the heat treatment. The gamma irradiation induced holes and electrons in the glass structure leading to the creation of a brown colour, and silver ions trapped electrons to form silver atoms. We observed the first stage of aggregation after irradiation, as well as after heating. The silver atoms diffused and then aggregated to form nanoclusters after heating at 550 °C. A characteristic band at about 430 nm was induced. The surface Plasmon absorption of silver nanoclusters in the glass indicated that the nanoclusters radius grew between 0.9 and 1.43 nm with increasing of annealing time from 10 to 242 min and then saturated. We also found that the size of aggregates depends on the value of gamma radiation absorbed dose. Contrary to what was expected, we found that 20 kGy is the optimal absorbed dose corresponding to the larger size of the aggregates which decreases for absorbed doses above 20 kGy.

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1. Introduction

Small silver particles embedded in glass matrix are widely studied because of their potential applications for glass colouration [1,2] for ionizing radiation dosimetry [3] and recently for fabricating optical devices [4]. The ion exchange has been considered one of the most important techniques to introduce noble metals in glass surface. This technique is simple, and does not require sophisticated equipments. Combined with a heat treatment, the ion exchange technique has received an increase attention as it can be used to introduce metallic nanosize particles such silver, gold and copper into glass matrix [5–7]. Recently ionizing radiation such as gamma rays, electrons, heavy ions, X-ray or laser beam, has been used, with or instead of the heat treatment, to produce silver colloids after the ion exchange in glass [8–11]. Ionizing radiation produces in insulating materials electron hole pairs (excitons) which lead to the production of stable defects and changes of the valence state of doping and impurities ions in the glasses. The production of irradiation defects cause preferential light absorption and consequently these defects are called "colour centres". These centres are of many types and depending on the glass composition and can be analyzed using optical absorption bands and EPR lines.

Gamma irradiation effects in silicate glass have been studied in our previous papers [12,13].

Only few works have been reported in the literature about the combined role of gamma irradiation and heat treatment on the formation of silver nanoparticles in ion exchanged glass.

Further investigations will be necessary to shed light on the relationship between the absorbed dose of gamma radiation and the properties of UV–visible spectra of silver ion-exchanged silicate glass. Our observations indicate that the first stage of aggregation can occur after irradiation before the heat treatment. We have

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observed that gamma irradiation followed by the heat treatment of an ion exchanged silicate glass can effectively promote silver migration and aggregation of metallic nanocluster. The effect of absorbed dose variation on the size of the aggregates has also investigated. In our knowledge, this effect had not been reported in previous work.

2. Experimental

2.1. Glass composition

The glass samples were obtained from the same glass sheets purchased from the local market and were cut into pieces of $11 \times 30 \times 1.5\ mm^3$ dimensions for optical measurements. The chemical composition of the glass samples were determined by the Prompt Gamma Activation Analysis technique [14] in the Budapest Neutron centre (constituents in wt%: 68.52 SiO_2, 13.77 Na_2O, 8.19 CaO, 4.34 MgO, 1.003 Al_2O_3, 0.588 K_2O, 0.105 Fe_2O_3 and about 3.5% of other components).

2.2. Procedure of ion exchange

Glass samples were dipped in a molten salt bath formed by a mixture of $AgNO_3$ and $NaNO_3$ with molar ratio of 1:99, 5:90 and 10:90 for 60 min in crucible of Al_20_3. The ion exchange has been carried out at a temperature of 320 °C for 1 h. During this step, the silver ions of the salt bath diffuse inside the glass matrix. The ion exchange samples are cleaned with distilled water and acetone to remove any silver nitrate adhering to their surface.

2.3. Gamma irradiation and heat treatment

Glass samples were irradiated at the Tunisian pilot plant ^{60}Co gamma irradiation facility [15] at a dose rate of 8.5 kGy/h and at room temperature with doses varying from 1 to 250 kGy.

Thermal annealing was performed in electrical furnace in air at the temperature of 550 °C for different time periods ranging from 10 to 582 min.

2.4. Optical absorption spectra

Optical absorption spectra of the glass samples were measured in the range of 300–700 nm with a Shimadzu UV–VIS spectrophotometer (model PharmaSpec UV-1700). The measurements were carried out against a glass sample subjected neither to ion exchange nor to gamma irradiation.

3. Results and discussion

3.1. Effect of ion-exchange

Glass samples were subjected to ion exchange procedure, with molar ratio of 5:95, at a temperature of 320 °C for 1 h. After ion exchange, the absorption spectrum showed significant difference from that before the ion exchange. As illustrated in Fig. 1, the spectrum is characterized by a sharp asymmetrical absorption band located at 305 nm. The asymmetric shape of this band may be due to the fact that the maximum measured does not match the maximum of the band but only to the loss of sensitivity of the spectrophotometer below 300 nm.

When samples of silicate glass are immersed in molten silver nitrate, sodium ions diffuse from the glass surfaces to the molten salt, and silver ions diffuse from the molten salt to the glass surfaces where they replace sodium ions. The exchanged glass is colourless or very faint yellow state. Accordingly, changes in the

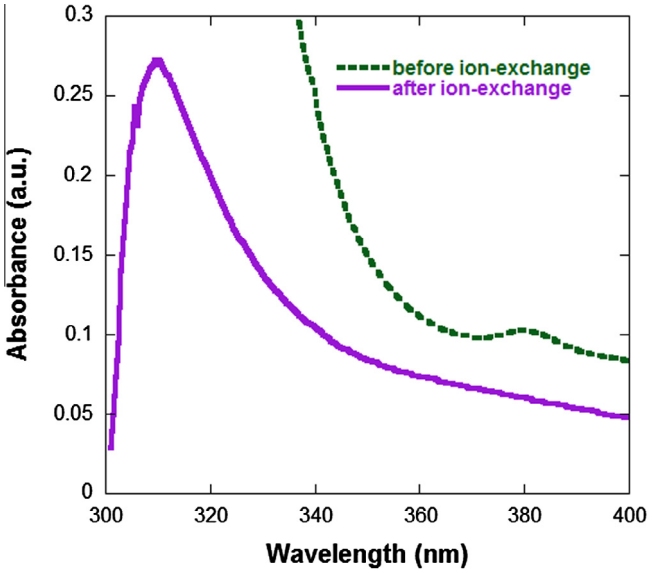


Fig. 1. Absorption optical spectra of silicate glass before and after ion-exchange.

spectrum of glass after ion exchange could be ascribed to modification of the composition of the glass surface by replacement of sodium ions by silver according to the reaction:

$$\equiv Si{-}O{-}Na^{+} + Ag^{+}NO_3^{-} \rightarrow \equiv Si{-}O{-}Ag^{+} + Na^{+}NO_3^{-} \quad (1)$$

The absorption band having a maximum at 305 nm was assigned to Ag^{+} ion. The information available in the literature about the origin of this band was reviewed in details by Ahmed et al. [16] and Paje et al. [17].

3.2. Effect of gamma irradiation

Silver exchanged as well as non-exchanged silicate glass samples were gamma-irradiated to 250 kGy. Optical absorption spectra were measured several days after irradiation are shown Fig. 2. The

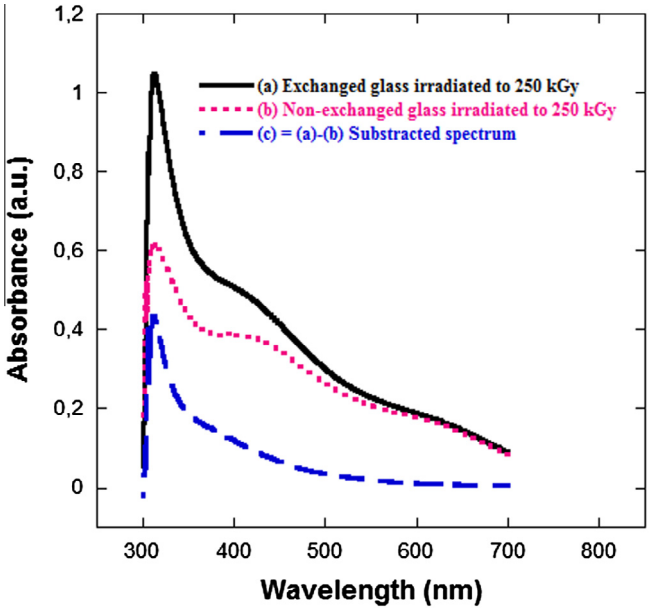


Fig. 2. Absorption spectra of the glass irradiated with γ rays at 250 kGy (a) spectrum of a 10% $AgNO_3$ exchanged glass; (b) spectrum of non-exchanged glass; (c) = (a) – (b).

Table 1
Gaussian fitting parameters of the optical spectrum (Fig. 2(b)) from non exchanged silicate glass after gamma irradiation at 250 kGy.

Peak position (nm)	FWHM[a] (nm)	Area under the band	Max. absorbance (u.a.)	Assignment
312	31.13	11.2	0.287	TE
400	190.7	88.4	0.37	HC_1
610	155	24.2	0.125	HC_2

[a] FWHM is the full width at half maximum.

optical absorption spectrum of the non-exchanged sample (b) display overlapping three characteristic bands with maxima at about 312, 400 and 610 nm respectively. Its spectrum was fitted by Gaussian curves whose parameters are given in Table 1. According to what reported by many authors [18,19] the absorption bands at around 400 and at 610 nm have been attributed to "non-bridging oxygen hole centres" (NBOHCs :≡ Si—O·), where the trapped hole correlated to the absorption at 400 nm denoted as HC_1 is a hole trapped in the 2p orbital of one and HC_2 is a hole trapped on two or three non-bridging oxygen bonded to the same silicon and correlating absorption band around 610 nm. The absorption band at about 312 nm have been attributed to trapped electrons (TE : " ≡ ") [20].

An additional absorption band was developed as a result of silver-exchange at about 354 nm. This band has been evidenced by comparing the optical absorption spectrum of the non-exchanged glass to that of gamma-irradiated silver-exchanged glass (subtraction spectrum (c)). The characteristics of the subtracted spectrum were determined by Lorentzian adjustment (Table 2). This band was attributed by many authors [10,21] to neutral silver atoms Ag^0. The observation of neutral silver means that a fraction of the photoelectron population has reduced silver ions directly by reactions:

$$Glass \xrightarrow{\gamma rays} h^+ + e^- \quad (2)$$

$$Ag^+ + e^- \rightarrow Ag^0 \quad (3)$$

where h^+ a hole is centre and e^- is an electron.

As shown in Fig. 3, irradiation of the silver exchanged sample at 1 kGy led to the significant decrease of the intensity of the Ag^+ band at 305 nm and the appearance of the new band at 354 nm assigned to Ag^0.

3.3. Effect of gamma irradiation and heat treatment

The only effect of γ irradiation at 250 kGy and annealing at 550 °C for 60 min or their combined effects on glass samples exchanged at 1% $AgNO_3$ were shown in Fig. 4. A new absorption band appears around 430 nm in the spectra of the exchanged samples (b and c). According to several authors [6,8], this absorption peak corresponds to the Surface-Plasmon Resonance (SPR) of the silver aggregates in glass. The formation of these aggregates is due to diffusion and agglomeration of neutral silver atoms in the glass matrix during gamma irradiation and/or heat treatment according to the reaction:

$$nAg^0 \rightarrow Ag_n \quad (4)$$

For spherical aggregates, the average aggregate radius R of is calculated from the FWHM, $\Delta\lambda$ of the optical absorption peak using the formula:

$$R = \frac{V_f.\lambda_m^2}{2\pi\Delta\lambda} \quad (5)$$

where V_f is the Fermi velocity of the electron in the bulk metal (silver = 1.39 × 108 cm/s), $\Delta\lambda$ is the full width at half maximum of the absorption band (FWHM) and λ_m is the characteristic wavelength at which SPR occurs.

We clearly observe that the spectrum (c) has a FWHM much narrower than that of the spectrum (b), the maximum absorbance is increased by a factor of 2 and the peak position is blueshifted from 430 to 421 nm (Table 3). It is also seen in Table 3 that

Table 2
Lorentzian fitting parameters of subtracted spectrum (Fig. 2(c)).

Peak position (nm)	FWHM (nm)	Area of the band	Max. absorbance (a.u.)	Assignment
315	19.4	9.4	0.31	Ag_2^+
354	144.3	37.4	0.165	Ag^0

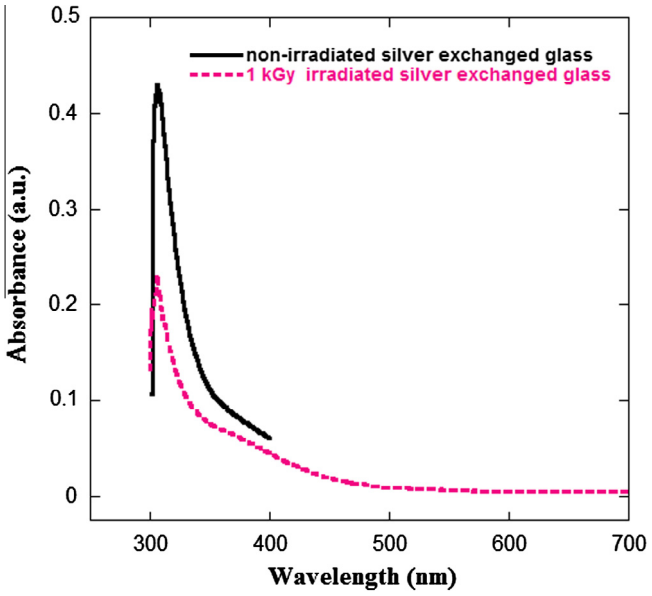


Fig. 3. Absorption spectra of ion-exchanged silicate glass (10% $AgNO_3$) and then irradiated at 1 kGy.

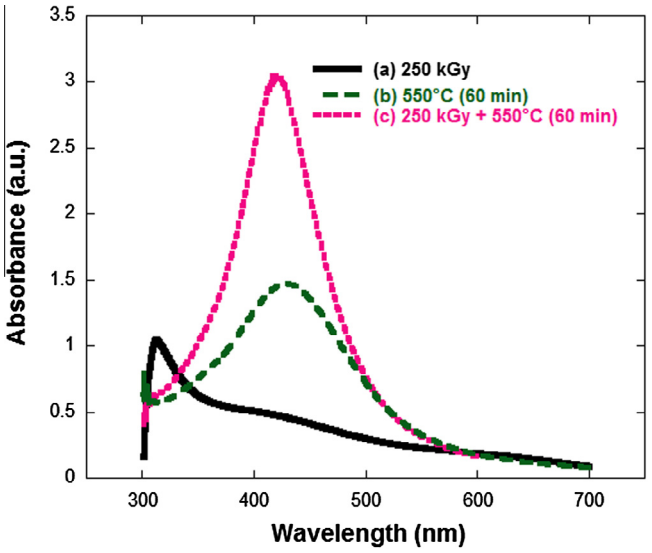


Fig. 4. Absorption spectra of ion-exchanged silicate glass, under the following conditions: (a) irradiated with γ rays at 250 kGy; (b) annealed at 550 °C for 60 min; and (c) irradiated at 250 kGy and annealed at 550 °C for 60 min.

Table 3
Characteristics of the plasmon resonance band, obtained by Lorentzian fitting, of two glass samples exchanged at 1% $AgNO_3$ and subjected to different treatments.

Treatment undergone by the glass sample	Peak position (nm)	FWHM (nm)	Max. absorbance (u.a.)	Average radius of the cluster (nm)
Annealed at 550 °C for 60 min	429.6	140.3	1.48	0.97
Irradiated at 250 kGy and annealed at 550 °C for 60 min	421.4	94.0	2.99	1.40

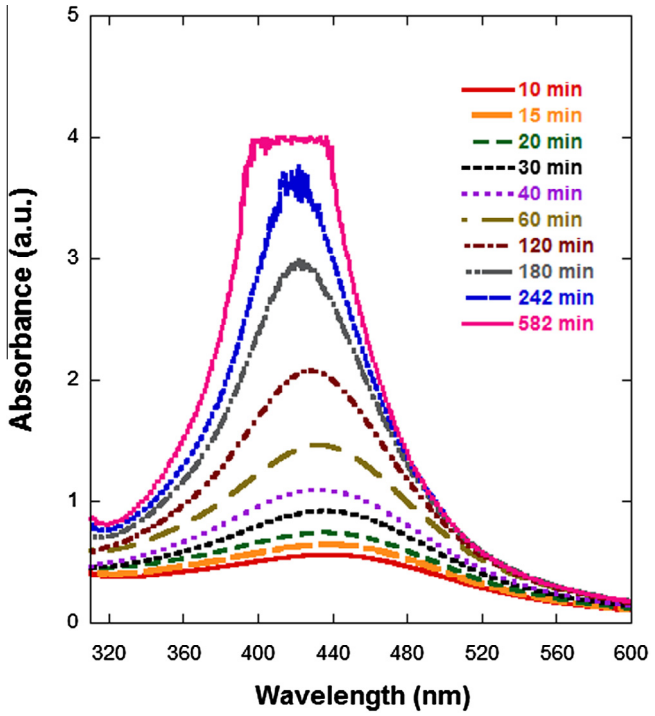


Fig. 5. Absorption spectra of ion-exchanged silicate glass, irradiated with γ rays at 250 kGy and annealed at 550 °C for the indicated times.

aggregate size increased significantly by about 43% in the irradiated sample. The increase in intensity of the absorption band in the irradiated sample is attributed to the increase in the total number of silver atoms constituting the aggregates. The conventional synthesis of silver nanoparticles in ion-exchanged glass requires the action of reducing agents present in the glass and enhanced diffusivity which is achieved only by thermal treatment at temperature well above 450 °C. By this procedure, only a few percent of the Ag^+ ions introduced are converted to silver atoms [8]. The improved formation process of silver nanoparticles by gamma irradiation is explained by the fact that the diffusion of silver in the glass increases by several orders of magnitude when the sample is irradiated by gamma rays before undergoing heat treatment.

Table 4
Characteristics of the plasmon resonance band as a function of annealing time at 550 °C, obtained by Lorentzian fitting, of glass samples exchanged to 5% $AgNO_3$ and irradiated to 250 kGy.

Annealing time (min)	Peak position (nm)	FWHM (nm)	R^a (nm)	Area under band (a.u.)
10	439.71	161.00	0.89	145.42
15	439.77	153.72	0.93	158.16
20	438.44	151.72	0.94	180.89
30	437.95	143.20	0.99	211.44
40	435.57	137.20	1.02	240.51
60	434.18	127.60	1.09	297.44
120	426.98	116.82	1.15	381.50
182	422.94	101.18	1.31	465.83
242	421.3	91.82	1.43	523.56
582	417.7	87.76	1.47	591.39

[a] The average aggregate radius *R*.

The gamma irradiation with 250 kGy of ion-exchange silicate glass was followed by annealing at the temperature of 550 °C for different time periods ranging from 10 to 582 min (Fig. 5). The intensity (or area under the band), shape and peak position of the absorption band at about 440 nm were found to be significantly by the increase of the annealing time.

The characteristics of this band as a function of increasing annealing time, obtained by Lorentzian fitting, are shown in Table 4. The area under the peak increases with increasing annealing time and its peak position is blueshifted from 440 to 418 nm. At the same time the band becomes more sharp, the full width at half maximum (FWHM) decrease from 161 to 88 nm.

Since this band has been associated with the formation of silver aggregates, it is expected that changes in the shape and size of theses aggregates induced by increasing of annealing time would affect the position, intensity or area under the band and FWHM of the induced absorption band. With increasing of annealing time, the number of neutral silver atoms increased leading to the increase in the number and dimensions of the aggregates. In Fig. 6 the proportionality between aggregates radius and area under the band is shown. This result is in good agreement with data published by Doremus [6]. Indeed, the total amount of silver constituting the aggregates was described by Doremus to be proportional to the area under the band. The proportionality between the reciprocal of the absorption band width and the radius of silver aggregates was also evidenced by Doremus, as long as the size distribution of the aggregates is narrow. Accordingly, Doremus attribute this absorption band to very small, spherical silver aggregates. If the aggregates were not spherical, the absorption peak position would be at longer wavelengths and would gradually shift to shorter wavelengths as the aggregates become more spherical with increasing time of annealing.

The dependence of the FWHM and the peak position of the absorption band on the size and the shape (eccentricity of the ellipsoid) of aggregate were also studied by Mennig and Berg [22]. They found that the FWHM is heavily dependent on the aggregate size but nearly independent of the (shape and the peak positions are significantly dependent on aggregate shape but nearly independent of the size.

The aggregates radius is plotted as a function of annealing time in Fig. 7. We observe that the aggregates radius grew between 0.9 and 1.43 nm with increasing of annealing time from 10 to 242 min and then saturated. Kowal et al. [8] suggest that the saturation of the absorption band for long annealing times may be due to the fact that all silver ions embedded in the samples have yet been reduced.

On the other hand, the precipitation of silver decreases the energy system, and therefore the silver atoms tend to precipitate to form aggregates [23]. The aggregation is accelerated at elevated temperature (550 °C) and continued growth by increasing of the annealed time up to critical size.

TEM images (Transmission Electron Microscopy) performed by Zhang et al. [11] on a silicate glass ion-exchanged to 2% $AgNO_3$ and annealed, show that the distances between neighboring aggregates remains high. Accordingly, once the precipitation completed,

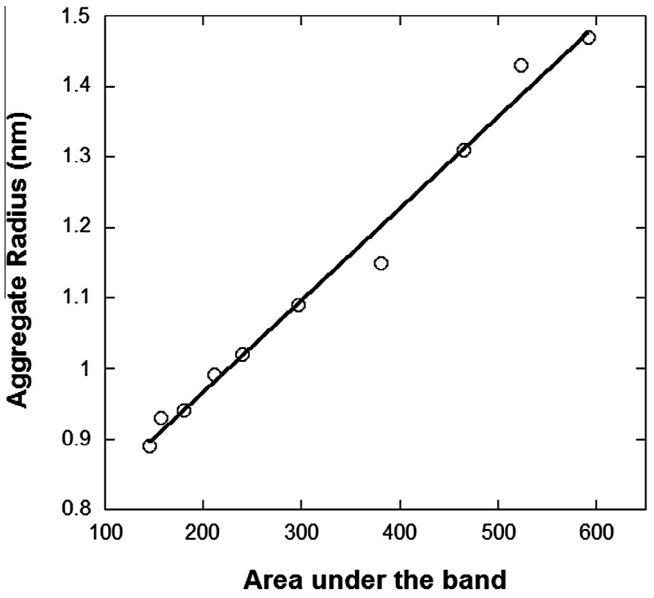


Fig. 6. Average aggregate radius as a function of area under the SPR absorption band.

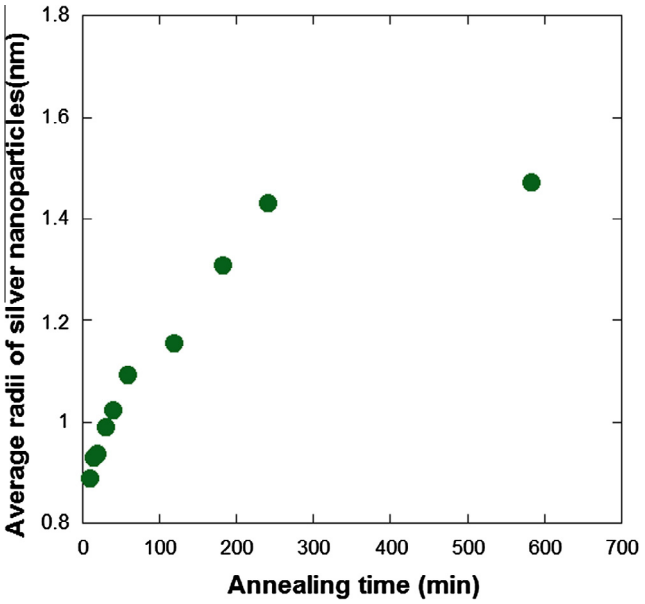


Fig. 7. Growth of silver aggregates as a function of annealed time (550 °C) in a glass exchanged to 5% $AgNO_3$ and irradiated by γ radiation to 250 kGy.

an aggregate of larger size cannot grow by intergranular diffusion process. These images show also that the aggregates have a spherical shape.

3.4. Effect of absorbed doses

In order to investigate the effect of absorbed dose variation on the characteristics of the absorption band attributed to the SPR, glasses samples exchanged at 1% $AgNO_3$ were irradiated in the dose range of 1–100 kGy. The samples were then annealed at 550 °C for 60 min. The results are shown in (Fig. 8). It shows that, for a dose of 1 kGy, the absorption spectrum appear to be due to the superposition of two absorption bands around 350 and 450 nm. However, when the dose exceeds 1 kGy, the band at 350 nm disappears and that at 450 nm has good resolution. The characteristics of this band as a function of increasing dose, obtained by Lorentzian fitting, are shown in Table 5.

For doses below 20 kGy, we observed a shift of the absorption peak to shorter wavelengths (from about 448 to 425 nm), a significant increase in its area under the absorption band (or intensity) and a reduction in its FWHM (from about 172 to 103 nm). It is also noted that these changes can be related to the increase in aggregate size as it has been shown previously. As well, no significant variation in the peak position was recorded for doses above 5 kGy, indicating that increasing absorbed doses above this limit has no effect on the shape of the aggregates. As mentioned previously [6,22], the peak position depends largely on the aggregate shape, while the FWHM depends mainly on the aggregate size. Therefore, perfectly spherical aggregates could be synthesized by irradiation at this dose level.

In other hand, based on spectroscopic calculations and data and electron microscopic studies, many authors [6,16,22] confirm that silver aggregates formed in glass treated by irradiation and/or heat treatment, which develop only a single absorption band, are spherical and very small size.

Moreover, it is noteworthy that for doses above 20 kGy, an important reduction in the area under the absorption band and

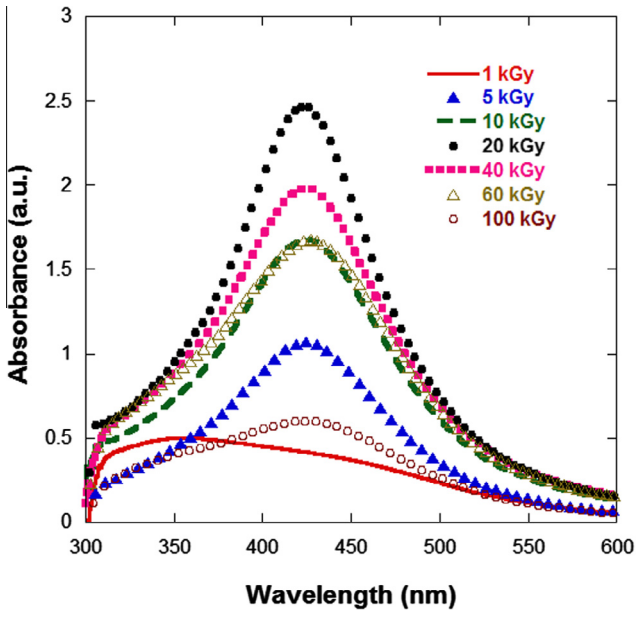


Fig. 8. Absorption spectra of ion-exchanged silicate glass, irradiated with γ rays in the dose range of 1–100 kGy and annealed at 550 °C for 60 min.

Table 5
Characteristics of the plasmon resonance band as a function of gamma irradiation dose, obtained by Lorentzian fitting, of glass samples exchanged to 1% $AgNO_3$.

Dose (kGy)	Peak position (nm)	FWHM (nm)	R^a (nm)	Area under band (a.u.)
1	448.03	171.86	0.86	101.50
5	425.5	122.64	1.09	205.93
10	427	121.46	1.11	319.38
20	425	103.46	1.29	401.41
40	426.5	118.54	1.14	368.49
60	428	132.22	1.03	347.67
100	422.5	155.40	0.85	146.95

[a] The average aggregates radius *R*.

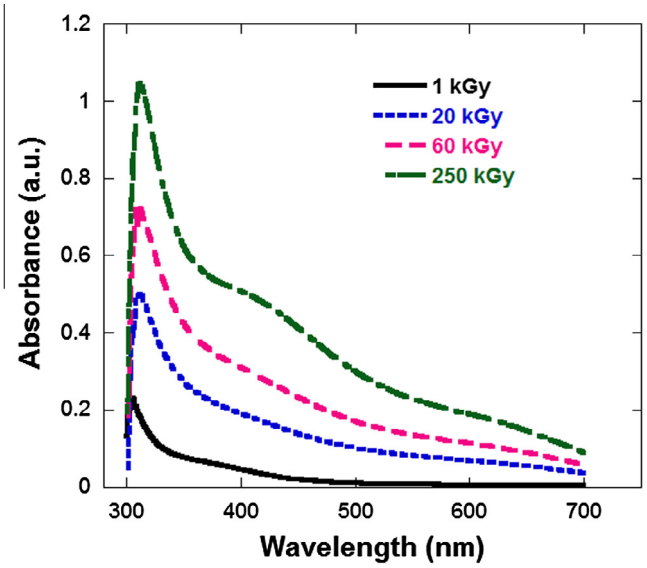


Fig. 9. Absorption spectra of ion-exchanged silicate glass (10% $AgNO_3$) and gamma irradiated at doses 1; 20; 60 and 250 kGy.

an increase in its FWHM, from about 103 to 155 nm, were observed. These observations can be explained by the partial neutralization of silver by γ radiation in a first step ($D \leqslant 20$ kGy). The increasing of the absorbed dose resulted in the observation of the first stage of silver aggregation, after irradiation and before the heat treatment, in silver ion exchanged silicate glass by converting Ag^+ ions into Ag_2^+ by the reaction:

$$Ag^0 + Ag^+ \rightarrow Ag_2^+ \quad (6)$$

The decrease in the area under the absorption band (or intensity) and the increase of its FWHM from 20 to 100 kGy can be explained by the decrease of Ag^0 concentration.

Because for doses $D > 20$ kGy, neutral silver atoms (Ag^0) formed by reduction of Ag^+ (reaction 2), convert to Ag_2^+ ions (reaction 6). It seems that for doses below 20 kGy the concentration of Ag^0 is not sufficient to activate the conversion reaction 6.

Evidence for Ag_2^+ ions was found in optical absorption spectra of gamma-irradiated silver ion-exchanged silicate glass samples between 1 and 250 kGy, as shown in Fig. 9, in which the absorption peak is red shifted from 305 nm (the characteristic wavelength of Ag^+) to 312 nm for absorbed doses above 1 kGy. We ascribed this 312 nm peak to Ag_2^+; its position is identical to that found for Ag_2^+ in radiolyzed aqueous solutions containing Ag^+ [21]. This peak was also identified by De Lamaëstre [9] at the same wavelength in a silicate glass doped with silver at a concentration of 118 ppm, irradiated to 21 kGy and annealed at 150 °C for 30 min.

4. Conclusion

In this work, we reported a study on the formation of nanoparticles in silicate glass using combined techniques. Silver-doped glass was obtained by Ag^+–Na^+ ion-exchange process. Glass samples were then subjected to gamma rays and/or to thermal annealing in air.

Silver nanoparticles have been characterized using UV–Visible absorption spectroscopy. The gamma irradiation induced hole centres and electrons in the glass matrix leading to creation of brown colour, and silver ions trapped electrons to form silver atoms. The silver atoms diffused and then aggregate to form nanoparticles after annealing at 550 °C for different time periods ranging from 10 to 582 min. A characteristic band at 430 nm was induced. The nanoparticles grew between 0.9 and 1.43 nm with increasing of annealing time between 10 and 242 min and then saturated.

The formation process of silver nanoparticles is improved if the samples were irradiated before undergoing thermal annealing because gamma irradiation increases the diffusivity of silver in the glass by several orders of magnitude.

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