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Vitrified municipal waste as a host form for high-level nuclear waste

N. A. El-Alaily · E. M. Abou-Hussein · Y. K. Abdel-Monem · T. D. Abd Elaziz · F. M. Ezz-Eldin

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Abstract Preparing glass to be used as a radioactive waste immobilizer from municipal waste is the aim of this paper. Up to 90 wt% of municipal waste was obtained by burning the raw waste at 700 °C for 5 h; this were successfully vitrified into borosilicate and sodium borate glasses at ~1,200 °C. The long term behavior of such glass is one of the most important factors, which is determined by their durability in aqueous solution. Experimental durability data of the prepared glass immersed in ground water together with γ-irradiation was found to be affected according to the different irradiation doses. In addition, thermal analysis and glass surface morphology were investigated. The evolution of the damage on the studied properties was correlated to the changes in the glass network depending on their composition and irradiation dose. The results showed that glass matrix containing higher amount of municipal waste possess high durability and low thermal expansion after being gamma irradiated. The results showed that glass containing higher amount of municipal waste possess high durability and low thermal expansion after irradiation.

Keywords Glasses · Recycling · Radiation · Corrosion

N. A. El-Alaily (✉) · E. M. Abou-Hussein · F. M. Ezz-Eldin
National Center for Radiation Research and Technology, P.O. Box 29, Nasr City, Cairo 11765, Egypt
e-mail: nalaily@hotmail.com

Y. K. Abdel-Monem
Faculty of Science, El-Mounofia University, El-Mounofia, Egypt

T. D. Abd Elaziz
October University for Modern Sciences and Arts (MSA), 6th October City, Egypt

Introduction

Municipal solid waste becomes recently a problem in Egypt. Nowadays one of the most important requirements for saving the environment is developing advanced air pollutant control system for municipal solid waste incinerators and the solidification or stabilization of the residues [1]. Cement solidification is a common method for stabilizing the solid municipal waste residue containing hazardous wastes. However the durability of the cement solidification decreases with time, especially after the fifth year of land filling [2], when leaching of heavy metals increase in the acidic medium of the landfill.

Currently, vitrification of solid waste has been demonstrated as one of the most successful methods, even though, a disadvantage of vitrification is its high cost of energy consumption required for melting. There are two options to decrease the production cost, the first is using glass forming materials having low melting temperature, while the second is making a valuable product of this vitrification.

Using glass as a safe and long term hosting matrix for hazardous wastes and for the immobilization of heavy metals and nuclear wastes has become an attractive method [3]. The most known glasses used as nuclear waste immobilizer are borosilicate named Pamela, SON68, and WAK [4] which mainly consists of SiO_2 in the range from 55 to 60 % and B_2O_5 in the range from 15 to 18 % of its composition. Durability of nuclear waste glass is a complex process which depends on many parameters; including the glass composition. Also ground water chemical composition, pH, time and temperature all these factors are affecting media [5]. The other important parameter controlling the use of glass as an immobilizer is the effect of irradiation in long term stability of glass constituents [6]. The best approach is to determine the basic parameters

governing the corrosion, so that a good evaluation can be made on the performance of the nominated glass.

The leaching processes of glass in water and aqueous solutions usually occur in two stages. The first stage of corrosion is ion exchange which controls the initial cation release, it is a selective method. The second stage is hydrolysis of the glass network (Si, B or Al, …) occurs only in the later stage and leads to glass congruent release of ions with time [7]. The basic mechanism is given by the following reaction [8]:

$$\equiv Si-O-M_{glass} + H_2O \rightarrow \equiv Si-O-H_{glass} + M-OH, \quad (1)$$

$$\equiv Si-O-Si\equiv + H_2O \rightarrow 2\equiv Si-OH. \quad (2)$$

The probable influence of irradiation in the glass corrosion stability is important for glass prepared for nuclear waste form. It is known that β and γ radiation are emitted in the nuclear waste glasses during the first hundreds of years [9]. The non-bridging hole centers (NBO) hole centers are formed by radiation chemical reaction:

$$(\equiv Si-O-M)_{glass} + \gamma \text{ irradiation} \rightarrow \equiv Si-O^{\bullet} + M^{+}. \quad (3)$$

It is assumed that the NBO hole centers resulting from irradiation are first trapped at ≡Si–O–M site and then the alkali ions are free to diffuse away to a site of a trapped electron to stabilize its charge [10]. This means that the association with NBO limits the local motion of alkali; whereas the absence of NBO allows the alkali ion to undergo long range migration through the glass structure. The main result of γ irradiation in glass is the formation of induced point defects [11].

For silicate glasses there are two types of point defects, oxygen deficiency and oxygen-excess defects [12]. In alkali silicate glasses, the most common point defect is the oxygen-excess centers, which are found in two types one of them is (NBO) and the other involves two NBOs on the same silicon [9]. The effect of radiation on borosilicate is more sophisticated than that of vitreous silica, possibly because of the combined double glass forming oxides and the two phase nature of many of borosilicate glass structure.

Thus we have planned to prepare glass containing large amount of municipal solid waste in its composition (70–90 %) with the addition of some glass former oxides. The origin of the used municipal waste is a collecting center of eastern Cairo zone at Khatamia. This municipal waste is the left over from house hold after separating the recycled part. It is mainly consists of food, construction and demolition wastes. After preparing the glass samples, this is followed by examining the most important factors affecting a glass candidate to be used for the immobilization of nuclear waste such as effect of irradiation on durability, hardness, density and the pH of the solution, which are the main parameters of the prepared glass.

Experimental procedure

Batches consists of 70 % waste + 10 % B_2O_3 + 10 % Na_2O + 10 % SiO_2 for G1 and 90 % waste + 10 % borax for G2 carefully weighed. The components were melted in platinum crucibles in an electric furnace at 1,300 ± 50 °C for about 2 h, after being thoroughly mixed. The homogeneity of the melt was achieved by swirling of the crucibles containing melt several times at about 30 min intervals and to make sure that the melt become free from air bubbles. The glass melt was poured in stainless steel hot mold with dimensions of 1 × 1 × 0.2 cm^3. The prepared samples were immediately annealed at 500 °C for 2 h and left in the annealing furnace over night with a cooling rate of 25 ± 2 °C/h to room temperature. The compositions of the prepared glasses were analyzed using dispersive X-ray analyzer as shown in Table 1.

The leaching experiments were carried out using cleaned glass slabs. Each sample was placed in a 200 ml polyethylene BD Falcon™ Conical Tube with Flip Top Cap, which allowed the leaching solution (150 ml) to completely cover the whole glass surfaces. The leaching solution is ground water obtained from a deep well in Siwa oasis with chemical composition 5.2 Ca, 17.0 K, 37 Na, 95.16 bicarbonates, 14 sulphates, 33 chlorides, 19 silicate, 0.4 fluorides, TDS 197 in mg/L, and then the tube was tightly closed. The ratio of glass surface area to leachant volume (*S*/*V*) was kept at 1.87 m^{-1} ± 0.1, and the leaching was carried out at different irradiation doses ranged from 1 to 100 kGy, tubes were exposed to gamma ray obtained from a ^{60}Co cell (2,000 Ci), with a dose rate of 1.182 Gy/s. Also leaching was carried out for different intervals of time

Table 1 Chemical analysis of the prepared glass

Elements	O	Na	Al	Si	P	K	Ca	Ti	Fe	Cu	Zn	Total
at.%												
G1	21.43	10.04	0.34	29.26	2.24	2.32	29.07	0.35	3.15	0.76	1.04	100.0
G2	21.50	4.32	2.50	25.34	2.31	2.78	35.34	0.39	3.27	0.76	1.59	100.0

either at room temperature 25 ± 5 °C or at 90 ± 5 °C. The weight loss was calculated by weighing the glass samples accurately three times before and after leaching to minimize the error to ±0.01 %.

The leachate solution was analyzed by identifying its pH after each irradiation dose and measuring the released sodium, boron and silicon ions at the final leaching stage using ICP and the data are shown in Table 2.

Density was measured at room temperature, using the suspended weight method based on Archimedes principle. Xylene was used as the immersion liquid. All the measurements were repeated three times, and the maximum error was found to be ±0.0002 g/cm^3. Density was calculated according to the following formula:

$$\rho = [a/(a-b)] \times 0.86,$$

where ρ is the density of the glass sample, a and b are the weight of the glass sample in the air and xylene, respectively, and 0.86 is the density of xylene at 20 °C.

The measurements were carried out by a Shimadzu microhardness tester. The diamond-shaped indentation was measured with an estimated accuracy of ±0.5 μm. Testing with a load of 100 g was used without causing visual micro cracks.

The crystallization process was investigated by differential thermal analysis technique using a Shimadzu-50 DTA apparatus using 10 mg glass powder heated from 30 to 600 °C at a rate 10 °C/min. DTA was carried out to determine the glass transition temperature (T_g) and crystallization peak temperature (T_c). Alumina was used as reference material during measurements.

Thermal expansion of the prepared glass was measured by a Shimadzu TMA-50 dilatometer system. The temperature was increased at rate of 10°/min. Data were obtained for each glass specimen from room temperature to the dilatometric softening temperature of the glass. Two measurements were made for each sample and the accuracy is ±2 %.

Scanning electron microscopy (SEM) model Jeol-JSM 5400 was utilized to study the surface topography of the specimen at high magnifications. Dispersive X-ray analyzer (EDX) model Oxford attached to SEM was used to analyze the composition of the municipal waste and of the prepared glass.

Table 2 Concentration of the released ions in the leachate in mg/L

Element in mg/L	Na	B	Si
G1 unirradiated	14.88	4. 44	0.386
G2 unirradiated	16.86	4.874	0.068
G1 irradiated	22.38	15.336	6.734
G2 irradiated	20.06	7.28	2.668
SON68 [6]	8.7	9.8	7.6

Results and discussion

The composition of municipal waste after being burned at 700 °C for 5 h the EDX analysis showed that it consists of the following elements 13.25 O_2, 1.5 Na, 7.8 Al, 5.3 Si, 6.2 P, 47.1 Ca, 5.1 Fe, 2.5 K, 1.1 Cu, 1.2 Zn, 0.3 Ti, 3.2 S, 5.5 Cl, all in at.%.

So the contents of the municipal wastes are mainly formed from oxides which can form glass with the addition of small amounts of glass former oxides.

Effect of irradiation on glass corrosion

Radiation is observed to affect the corrosion data and this can be explained to be due to two factors, the first is the alteration of glass structure; the second is the changing of the pH of the leaching solution by the formation of various products [10, 13]. From previous studies on the corrosion of γ irradiated glasses, it has been found that there was an incongruent dissolution of glass, especially for the in situ γ irradiated corrosion tests of waste glass [14].

According to Fig. 1a, it is observed that glass G2 is more durable than G1 and this can be understood by taking into consideration the chemical analysis of glass surface composition shown in Table 1. Glass G2 contains more percent of aluminum ions which are obviously more resistant to be corroded especially at the first stage of corrosion, and Ca ions which have large ionic radii causing blocking or retardation of the routes of diffusion of released sodium ions from ion exchange and inter-diffusion. Also, glass G1 contains more percent of sodium, and as mentioned before the release of the alkali ions is the first step in the corrosion process of silicate glasses depending on the ion exchange process. So, it is virtually expected that glass G2 will be more durable. For glass G1, there is a large deterioration in the glass durability with the increase of the irradiation dose until it reached 30 kGy followed by an enhancement in the glass durability as the irradiation dose further increases. For glass G2, it can be noticed that it has the same behavior as G1 but the deterioration in the glass durability takes place up to a dose of 50 kGy. The basic mechanism controlling the leaching of the glass with ground water at lower irradiation dose is the ion exchange process including counter diffusion between cations in glass and protons or hydronium ions (H_3O^+) from water which replace the cations released from the glass structure. The rate of release of cations into water during leaching is given by the magnitude of flux of cations through the surface of glass–water interface which depends on the cation concentration in the glass, the diffusion coefficient and temperature dependence [7]. While increasing the irradiation dose will give the chance for more free radicals to be formed, i.e. further ionization of glass modifiers will

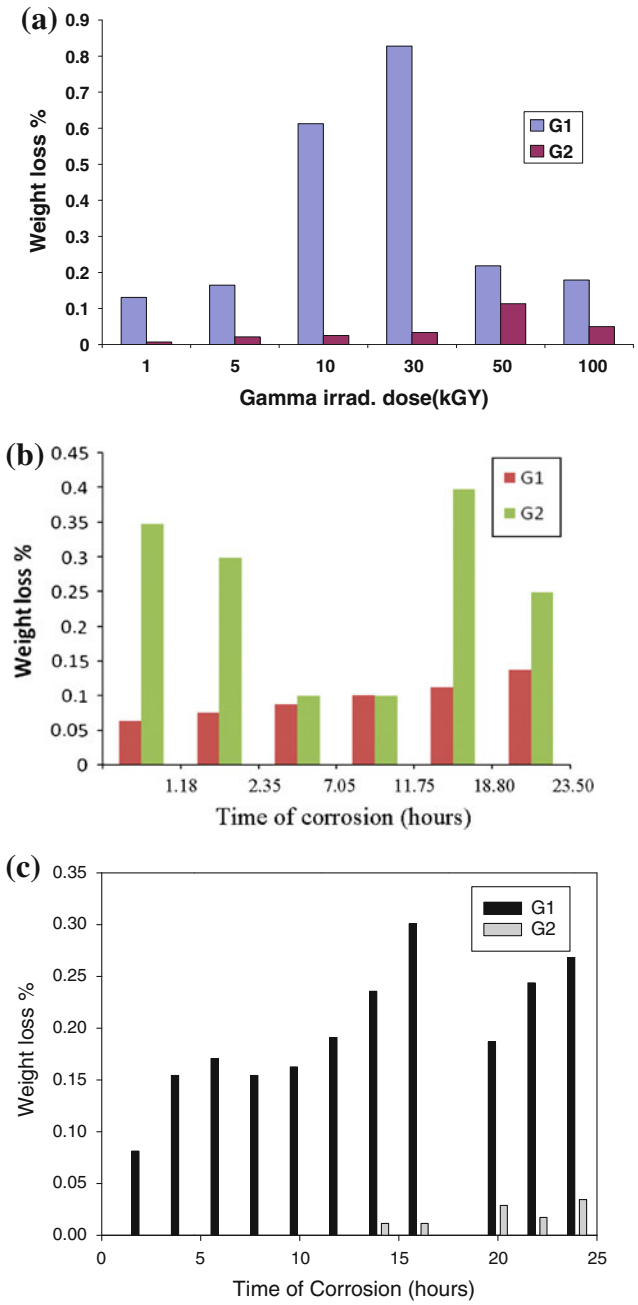

Fig. 1 **a** Effect of irradiation doses on glass corrosion. **b** Effect of time on glass corrosion. **c** Effect of time on glass corrosion at 90 °C

take place rather than alkali ions (which may be highly consumed), to form a defending layer precipitated in the glass surface formed from super-saturation and the possibility of forming immiscible compounds. This layer is responsible for the observed decrease in the corrosion at higher doses.

It is clear from Fig. 1a, that leaching is systematically larger when glass is leached during being irradiated. When leaching is carried out at room temperature without irradiation as shown in Fig. 1b, the contrary occurs where G1 is more durable than G2 This can be interpreted according to the assumption of Sheng et al. [15], where they have assumed that the controlling step in the leaching mechanism is the solubility of various elements that precipitate during the course of reaction. They also assumed that the rate limiting step in the reaction is the removal of $Si(OH)_4$ group from the glass surface. According to the last assumption it can be understood that glass G1, which contains 10 % SiO_2 is expected to be more durable than G2, where alkali ions are present inside the network structure of either Si or B lattice and their mobilities are limited without the process of irradiation. The process only depends on the amount of hydrogen or hydronium ions formed during the leaching process. Also it must be taken into consideration that Si ions show lower release rates, while the dissolution rate of B from borosilicate glass is usually much higher than Si ions although both of them are network former. This may be due to the presence of Si in a form of SiO_4 tetrahedra while B is found at least partly as BO_3 triangles, as well as due to the higher mobility and ease of solubility of B ions in aqueous media [7]. The observed deterioration in the durability of G1 upon irradiation can be referred to radiation ability to initiate ionization, which encourages the increase of ion exchange and since the alkali ions (i.e. most of Na and K ions shown in Table 1) are the highest released elements, besides it is well known that for the in situ γ irradiated corrosion tests large amounts of hydrogen and hydronium ions will be formed causing the fast release of alkali ions before the formation of any depleted layer, causing the observed increase in the weight loss.

To understand the effect of aging on the glass durability the leaching process were carried out for 24 h at 90 °C as shown in Fig. 1c, which may give a prediction of the corrosion process for 1 year [16]. The results show that the effect of leaching at high temperature is the same as that occurred when the glass is corroded during irradiation where G2 is the more durable and that the amount of weight loss is undetected up to 15 h followed by a small amount of weight loss. It can be also noticed that the weight loss at 90 °C for both glasses G1 and G2 is lower than that occurred at room temperature this was explained by the formation of complex aluminum species and insoluble hydrated silica deposited on the glass surface forming a protective layer [17].

Effect of leaching on the solution pH

The pH results as shown in Fig. 2 also show the same trend where the alkalinity of the solution is increased gradually to reach its maximum for G1 at dose of 30 kGy, while it reaches its maximum for glass G2 at the dose of 50 kGy, then the pH of the leaching solution decreases. The last

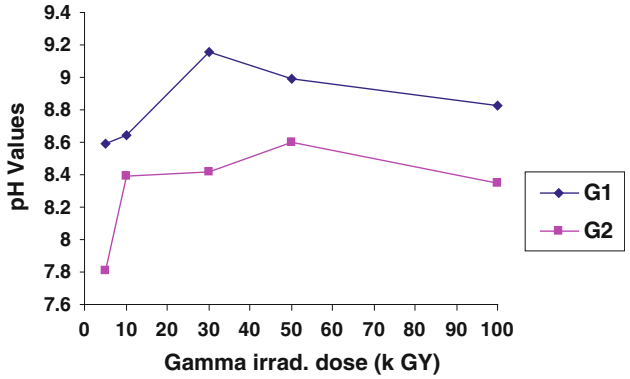

Fig. 2 Effect of irradiation doses in the leachate pH (the pH of underground water used = 7.64)

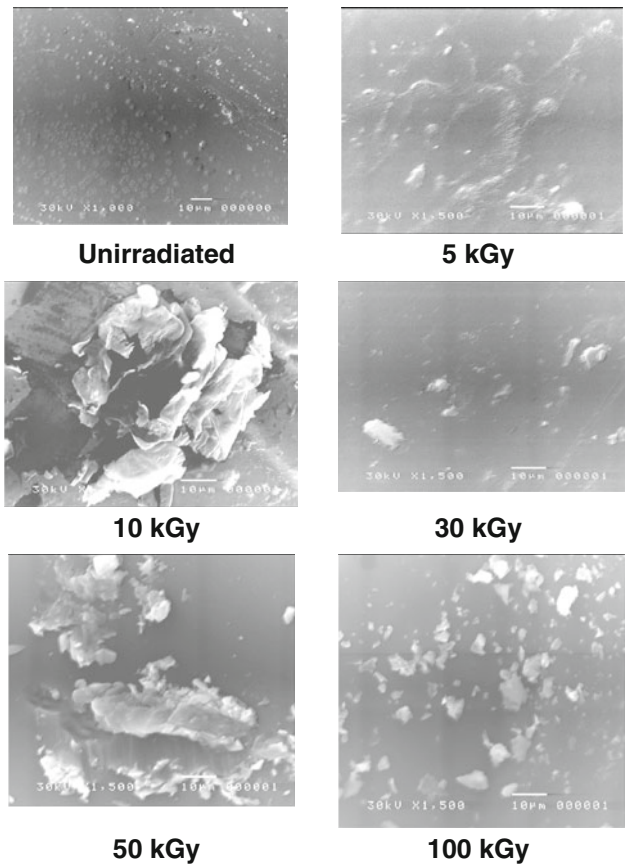

Fig. 3 Scanning electron microscope for G1

observations can be interpreted depending as mentioned before in two factors, the glass composition and the effect of irradiation. Where at the first irradiation dose, the alkali ions in the glass surface will be easily freed to release in the leaching solution. With further irradiation doses, the alkali ions inside the glass structure will become able to move through the glass matrix until it reaches the surface and released in the solution, besides the leaching solution will also has the ability for more penetration through the

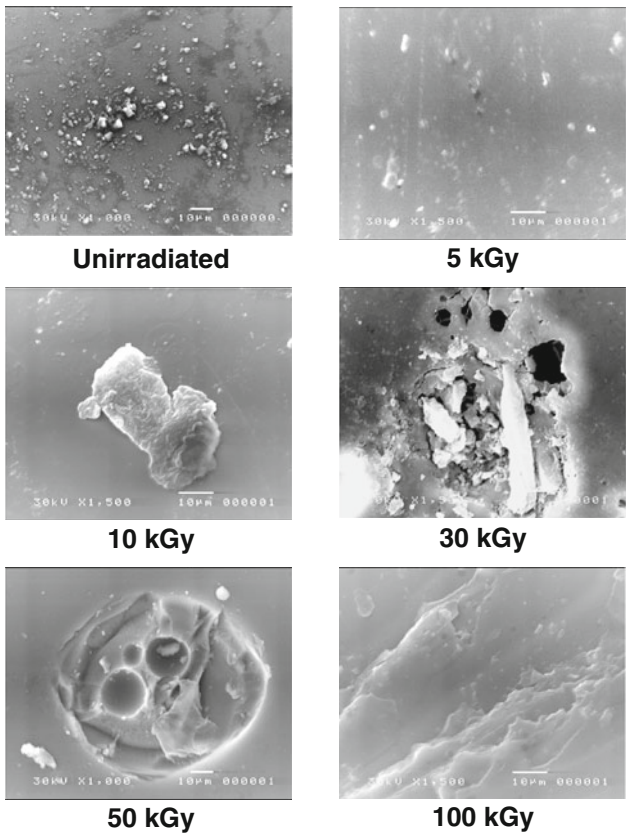

Fig. 4 Scanning electron microscope for G2

glass matrix until it reaches its maximum corrosion at 30, 50 kGy for G1 and G2, respectively.

The observed decrease of pH of leaching solution with further increase of irradiation dose may be due to one of the following assumptions: (a) either the hydrolysis of the glass network begins to take place in the leaching process and the presence of silicic or boric acid is responsible for the observed decrease in the solution pH, (b) or some of the dissolved alkali or alkaline ions released from the glass during the first leaching stages begin to precipitate on the glass surface, forming a protective layer [18]. According to the leaching results observed in Fig. 1, the second assumption is more reliable.

The SEM images shown in Figs. 3 and 4 also agreed with the leaching results, where the highest leaching occurred at doses 30 and 50 kGy for G1 and G2, respectively.

Density and microhardness

The density of glasses is controlled by the free volume of vitreous network and by the total masses of the ions present. In general the addition of alkali ions with small ionic size, which enter the interstices of the vitreous network tend to increase the density by shrinking the molar

volume of the parent glass. The addition of different ions to the glass will alter the density according to the housing of these ions in the network structure [19]. The results show that the density in g/cm^3 of G1 and G2 are 2.7321 and 2.7724, respectively, glass density of the prepared glass is slightly less than SON68 which is 2.8 [20]. After irradiating the glass samples with doses of 30, 50 and 100 kGy the density of G1 become 2.7389, 2.6786 and 2.5169, while for G2 it becomes 2.7774, 2.7811 and 2.7322. The results also show that glass density increases at the first irradiation dose for G1 and at 30, 50 kGy for G2. This can be understood, when taking into consideration the corrosion results, where as the corrosion increases in the first irradiation doses, the alkali ions are freed to immigrate to the solution and more of the non-bridging oxygen can be formed allowing trigonal BO_3 to become tetrahedral BO_4 which are strongly bonded, this causes more compaction of the glass molar volume producing the observed increase in density, also the recoiled oxygen ions from the sample may give an explanation for the volume changes [21]. At higher irradiation doses (50, 100 kGy for G1 and 100 kGy for G2 the density begins to decrease, this can be related to the beginning of hydrolysis which causes the breakage of the network and an expansion in the volume.

The microhardness results involve the creation of a compression when the indenter is pushed downwards into the glass with an applied load. After the removal of the indenter, it is obvious that in addition to the recoverable elastic compression experienced by the sample, some material in the neighborhood of the indenter could have actually been displaced [19]. The microhardness results show that the glass containing more municipal waste (G2) has higher density and lower microhardness 222.6 while G1 microhardness is 232.7 kg F/mm^2. This can be understood taking into consideration that the density changes depend on the ions housed in the network, according to

Table 3 Thermal expansion data of G1 and G2 before and after different irradiation doses

Glass no.	γ-IRRAD dose	T1–T2 (°C)	ΔL (μm)	α/K
(a) TMA of G1 and G2				
G1 (70 % waste + 10 % B_2O_3 + 10 % Na_2O + 10 % SiO_2)	Before irradiation	31.25–53.74	−1.74 (−0.07 %)	-29.23×10^{-6}
		80.30–421.75	14.68 (0.55 %)	16.24×10^{-6}
		441.14–496.89	−5.53 (−0.21 %)	-37.47×10^{-6}
	5 KGy	26.46–64.27	−3.23 (−0.14 %)	-36.11×10^{-6}
		84.85–454.30	20.26 (0.83 %)	22.55×10^{-6}
	10 KGy	30.05–46.80	−1.91 (−0.08 %)	-49.73×10^{-6}
		63.55–498.56	25.33 (1.10 %)	25.39×10^{-6}
	30 KGy	30.77–112.12	−15.29 (−0.57 %)	-70.08×10^{-6}
		142.99–372.70	12.12 (0.45 %)	19.67×10^{-6}
		393.28–495.93	−8.27 (−0.31 %)	-30.04×10^{-6}
	50 KGy	29.96–63.55	−3.89 (−0.14 %)	-40.88×10^{-6}
		96.09–499.51	21.22 (0.75 %)	18.57×10^{-6}
	100 KGy	26.94–65.70	−2.17 (−0.09 %)	-22.10×10^{-6}
		75.99–498.56	21.47 (0.85 %)	20.06×10^{-6}
(b) TMA of G2				
G2 (90 % waste + 10 % borax)	Before irradiation	29.28–94.90	−11.22 (−0.41 %)	-62.87×10^{-6}
		129.35–499.43	18.17 (0.69 %)	18.05×10^{-6}
	5 KGy	27.90–149.93	−21.36 (−0.81 %)	-66.20×10^{-6}
		175.77–498.09	12.20 (0.46 %)	14.32×10^{-6}
	10 KGy	25.26–141.32	−13.81 (−0.59 %)	-50.79×10^{-6}
		140.60–399.26	7.79 (0.33 %)	12.85×10^{-6}
	30 KGy	26.07–75.22	−2.26 (−0.09 %)	-18.08×10^{-6}
		79.85–593.35	20.36 (0.80 %)	15.59×10^{-6}
	50 KGy	27.52–102.40	−12.12 (−0.53 %)	-70.22×10^{-6}
		132.76–556.05	16.53 (0.72 %)	16.94×10^{-6}
	100 KGy	26.65–129.58	−12.52 (−0.41 %)	-39.63×10^{-6}
		129.58–270.10	5.82 (0.19 %)	13.50×10^{-6}
		328.50–521.07	8.4 (0.27 %)	14.21×10^{-6}

their size and the way they interconnect with the network. So it can be predicted that when small ions such as sodium ions are introduced to the glass network they will be housed in the interstices and causing an excess of oxygen forming nonbridging oxygen and the resultant will be an increase in the glass density. However, in our results glass G2, which contains fewer amounts of alkali and more amounts of Ca and Al ions has higher density; this can be due to the presence of ions with higher atomic weight, while the glass structure is less compacted, where the priority become to Al ions to consume some of the available oxygen to change to four fold coordination AlO_4 instead of changing BO_3 to BO_4.

Thermal expansion

To understand the change in thermal expansion of any glass, the internal structure and anharmonic spatial arrangements of all of the forming constituents of the glass structure, which reflects their response on the anharmonic vibrations of atoms, must be taken into consideration. The result of the presence of anharmonicity is that the amplitude of vibration increase, the inter-ionic distance increase and the ions vibrate independently to each other. The anharmonicity, however is not only functioning of the bond strength or amplitude of vibration but is also dependent upon the symmetry of the environment of oscillator [22].

Another postulation of the change of thermal expansion according to the change of glass composition is that, alkali ions are known to occupy solely network modifying position in glass network. In other words, it destroys the bridging oxygen bonds. Since those ions are the most weakly bonded constituents in the glass, so they would be able to move through the building units. As a result of such mobility, they will move from the nonbridging oxygen ions, and interact with neighboring bridging ions and contra-polarize them, which accordingly weaken the alkali–oxygen bond [23].

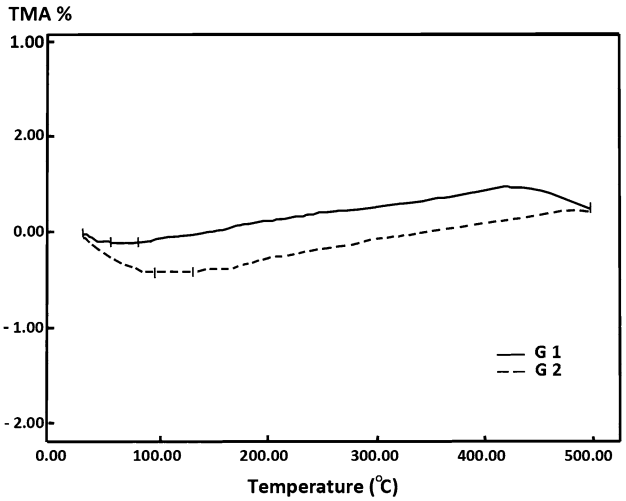

Fig. 5 TMA of G1 and G2 before irradiation

The results in Table 3a, b show that when the blank glass samples are exposed to low temperature a compaction of its length is observed, an assumption can be made that this is due to the evaporation of water, which may be absorbed by the glass samples from the atmospheric humidity. As the temperature increased (in the range from 80–421 °C for G1 and 129.35–499.43 °C for G2) the glass samples expand. Figure 5 represents thermal expansion diagram of the two samples, it is generally accepted that the values of thermal expansion depends on the magnitude of the binding energy between the cations and oxygen ions, ionic sizes and field strengths of the respective cations. These results can be understood by tracing the differential thermal analyses of the glass; it can be observed that G1 and G2 have low glass transition temperatures as shown in Fig. 6. Taking in consideration the X-ray diffraction pattern shown in Fig. 7 of the examined glasses, it can be

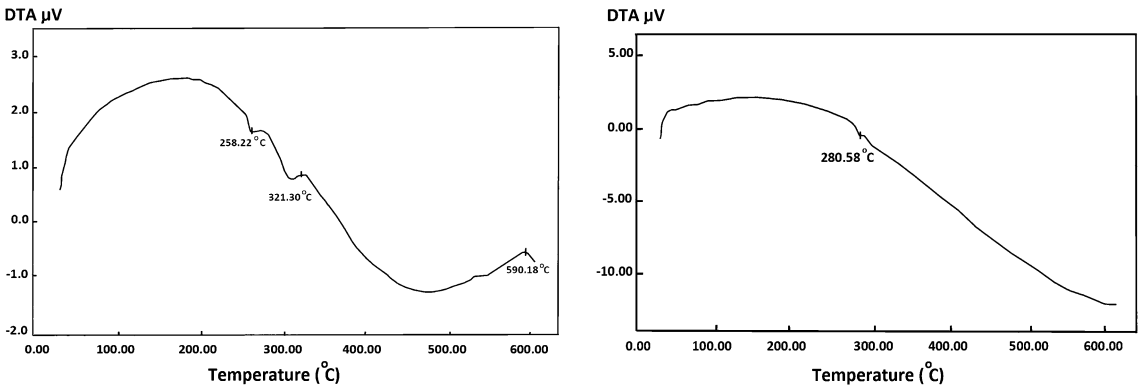

Fig. 6 a, b DTA for G1 and G2

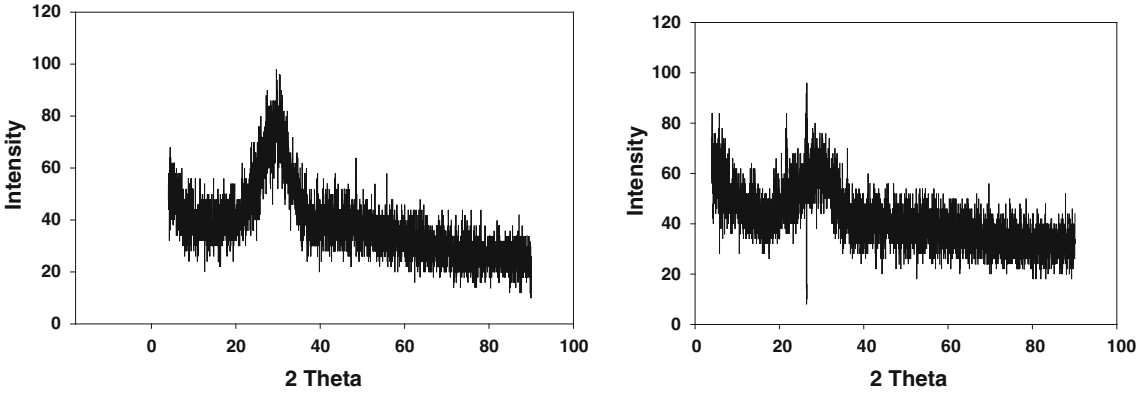

Fig. 7 **a**, **b** X-ray diffraction of G1 and G2

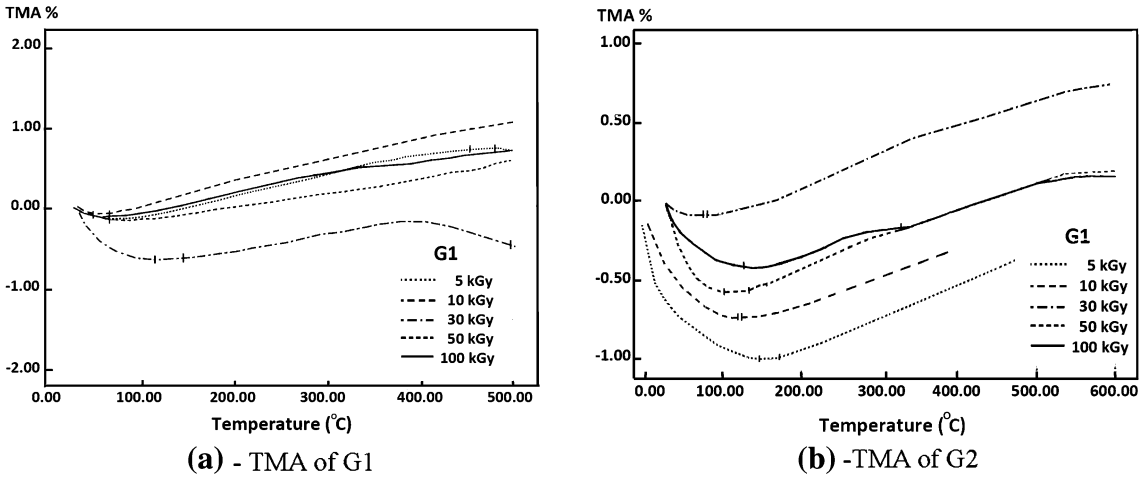

Fig. 8 **a**, **b** TMA of G1 and G2 at different irradiation doses

noticed that there is a start to form a separated phase observed at 2θ between 25 and 30. So one can expect easily breakage of the bonds resulting in the observed expansion, this is followed by the beginning of new rearrangement of the inter structure, which leads to the observed compaction for G1 between 441 and 496 °C.

When the glass samples exposed to different irradiation doses during the leaching process, the thermal expansion coefficient data are changed as shown in Fig. 8. For G1, the thermal expansion increases with the increase of irradiation dose until it reaches its maximum at 10 kGy (where it changed from 16.24×10^{-6} before irradiation to 25.39×10^{-6} at 10 kGy), then it is followed by a decrease with further increase in the irradiation dose. For G2 the thermal expansion coefficient decreases with the increase of the irradiation dose, although it increases at 30, 50 kGy but it still lower than the unirradiated specimen. The remarkable change in thermal expansion due to gamma irradiation of glass can be referred to the presence of a number of induced defects which may lead to a significant change in glass defect centers (vacancies, nonbridging oxygen, hole center) [21]. This effect causes the observed changes with prolonged irradiation.

Conclusions

The additions of high percentage of municipal solid wastes (70–90 %) as a source for different glasses with the addition of some percent of glass former oxides are observed to form promising glasses such glasses may be used for the immobilization of radioactive wastes, with low irradiation doses lower than 30 and 50 kg, where the results show that the highest corrosion data are reached at these doses for the

two prepared glasses G1 and G2, respectively. The results also show that glass G2 which contains higher amount of waste has higher chemical durability.

The results also show that the glass properties such as density, hardness and its thermal expansion are promising parameters which help to confirm the suitability for the postulation of using these glasses as immobilizer for radioactive wastes.

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