



## Characterization of lead, barium and strontium leachability from foam glasses elaborated using waste cathode ray-tube glasses

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### ABSTRACT

Foam glass manufacture is a promising mode for re-using cathode ray tube (CRT) glasses. Nevertheless, because CRTs employ glasses containing heavy metals such as lead, barium and strontium, the leaching behaviour of foam glasses fabricated from CRTs must be understood. Using the AFNOR X 31-210 leaching assessment procedure, the degree of element inertization in foam glasses synthesized from waste CRT glasses (funnel and panel glasses, containing lead and barium/strontium respectively) were determined. The amount of leached lead from foam glasses prepared from funnel glass depends on the nature and concentration of the reducing agent. The effects of the reducing agents on the generation of cellular structure in the fabrication of foam glass were studied. The fraction of lead released from foam glass was less than those extracted from funnel glass and was lower than the statutory limit. Leached concentrations of barium and strontium were found to be approximately constant in various tests and were also below regulatory limits.

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### 1. Introduction

Re-use of waste electrical and electronic equipment (WEEE) has raised a great practical interest in the last 10 years. Electric and electronic equipment (EEE) contains hazardous components and heavy elements. Taking into account the rapid proliferation of electronic devices and computer technologies, the amount of obsolete material to be discarded is growing dramatically. For these reasons, EEE represents a societal problem that needs to be solved by government and private industry. Currently, much EEE waste simply goes to landfill. A better solution is to re-use materials in electronic devices when their lifetime is attained [1]. One WEEE presenting disposal problems is the cathode ray tube (CRT) [2–5]. CRTs are the major components in older style television sets, computer monitors and feature heavy metal-containing glasses. Three types of glasses are usually found in recent colour CRTs, two of them containing lead (Pb) while the third is constituted by barium (Ba) and strontium (Sr). The Pb-containing glasses are found in the funnel and neck that are the hidden parts inside the monitor, whereas the glass based on Ba and Sr elements is present in the screen of the monitors or TV-sets [4]. Some previous studies have determined the compo-

sition of waste CRT glasses [4,6]. Lead oxide represents 21–24 wt% (Pb: 2.4–2.7 atomic weight %) of colour funnel glass whereas the concentration of Ba and Sr oxides in such glass reaches from 9 to 13 wt% (Ba: 1.4–1.6 at%) and 1–2 wt% (Sr: 1.5–2.1 at%) respectively [4,7]. It has been also established that Pb leachability from CRTs can cause critical environmental damage [3,8]. Several research groups have worked to solve the CRT disposal problem (see [9]). Various alternative solutions have been proposed, including (i) recycling of new CRT glasses with waste glasses, namely the closed-loop system [10,11]; (ii) manufacturing of other materials using CRT waste glasses, labelled as the open-loop system [6,4,12–26], (iii) admixturing the glasses with concrete to encapsulate lead containing glass particles [27]; or (iv) decontaminating the glass by removing Pb [28] or other heavy metals [29,30] contained therein. In regards of the large number of works carried out on CRTs re-use, only some of them deals with leaching of the elaborated materials [27,30]. The use of waste CRT glass in an open-loop system must be validated by normalized leaching tests. Foam glass is a useful material, with valuable thermal ( $\kappa = 0.08\text{--}0.43\text{ W/m/K}$ ) and electrical ( $\varepsilon' = 2.1\text{--}2.8\text{ F/m}$ ) insulating properties [25], but heavy element leachability must be operated under appropriate regulatory limits for the involved elements. While studies focusing on the leaching of lead silicate glasses are available in the literature [31–35], as far as we know, there is no study on the leaching behaviour of the very promising foam glass. These works were devoted to study lead silicate glasses changes upon leaching using different tests [35]. Bonnet et al. [32] and Cailleteau et al. [34] have demon-

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strated severe modifications of the glassy framework induced by leaching with a change of coordination for Pb. Leaching induce also strong defects on the glass surface with a resulting increase of the lead released [33,34].

The present paper reports the investigation of the Pb, Ba, and Sr leaching of foam glasses elaborated from waste CRT glasses, using the AFNOR X 31-210 leaching procedure. This procedure is admitted in France and widely used to determine the degree of inertization of either materials to be re-used or wastes to be disposed in landfill. In comparison with the commonly used US EPA 'Toxicity Characteristic Leaching Procedure TCLP' leaching procedure, AFNOR X 31-210 is less aggressive and comparable to other procedures used in Europe [36–38]. The major difference between European leaching tests and US-EPA 'TCLP' is the leachant used to digest the material: water, and acetic and buffered acetic solution respectively.

Foam glass properties have been found to depend on the nature and concentration of reducing agent used and the type of CRT glass. The aim of this work was at: (i) studying the leaching behaviour of Pb, Ba and Sr for the different synthesized foam glasses, (ii) determining if and which type of foam glasses meet regulatory criteria regarding limits for Pb, Ba, and Sr content and (iii) establishing the influence of the synthesis conditions on leaching. The local structure around Ba and Sr elements has been further probed by X-ray absorption spectroscopy (XAS) in order to bring some insights into the leaching mechanism.

## 2. Materials and methods

### 2.1. Sample preparation

Waste CRT glass powders and powdered reducing agents SiC, (Aldrich; 99.9%) or TiN (Alfa Aesar; 99.8%) by weight were mixed with a binder and cold-pressed under 5 tons to obtain 40 mm diameter pellets with a thickness of 6 mm. The average particle sizes of the different powders used were below 65  $\mu\text{m}$ , 63  $\mu\text{m}$ , and 10  $\mu\text{m}$  for waste CRT glass, SiC and TiN powders respectively.

Table 1 summarizes the samples studied, their compositions, and their synthesis conditions. Two types of elaboration for the foam glass can be distinguished; they involve either a single type of CRT glass (funnel or panel) or a mixture of funnel and panel glasses. The proportions of funnel and panel glasses (for the PF5S and PF4T compositions) are shown with respect to the initial composition of a CRT, where funnel and panel glasses represent 1/3 and 2/3 of the total mass of glass respectively. All pellets were treated in an electric furnace under air, removed from the furnace, and returned at room temperature in 'free fall'.

### 2.2. Leaching tests and analysis

Samples prepared as described above were ground to powder and sieved to a particle size smaller than 4 mm diameter. The leaching behaviour of the foam glasses was evaluated according to the well-established leaching procedure AFNOR X 31-210 [39]. Because of the extremely low density of the samples ( $\approx 0.5 \text{ g/cm}^3$ ), the leaching procedure was adapted to use conditions appropriate to a liquid (L)–solid (S) ratio (L/S) of 10 L/kg, as recommended [39]. Forty grams of foam glass were introduced into a 2 L cylindrical extraction vessel and 400 mL of pure water at  $\text{pH } 7.00 \pm 0.05$  was used as leachant after introduction into the vessel. The vessel was placed on a rotary extractor at  $60 \pm 2 \text{ rpm}$  for  $16 \pm 0.2 \text{ h}$ . Each leachate was filtered (Whatman no. 4) and centrifuged to eliminate all solid particles. The resulting solution was labelled leachate I. This operation was repeated again two times to get solutions named leachate II and III. Each of them was analysed by inductively coupled

plasma-optical emission spectrometry (ICP-OES) on a PU7000 Unicam spectrometer to determine the concentrations of Pb, Ba, and Sr using commercial solutions of each element with a concentration certified at 1.0 g/L as references. The leaching limit values applicable to inert waste acceptable for landfill are 50 and 300 mg/L for Pb and Ba respectively [40], whereas no minimum value has been specified for Sr.

### 2.3. X-ray absorption spectroscopy (XAS)

X-ray absorption spectroscopy (XAS) measurements were performed at the European Synchrotron Radiation Facility (Grenoble, France) at the Dutch–Belgian beam line "DUBBLE" (BM26a). The XAS data were measured in transmission mode at the Sr–K (16105 eV) edges and in fluorescence mode at the Ba–L<sub>III</sub> edge (5247 eV) at room temperature on powdered samples. The data were recorded with 0.04  $\text{\AA}^{-1}$  steps in *k*-space using at least two runs for each sample. X-ray absorption near-edge structure (XANES) was extracted using IFEFFIT package [41,42].

## 3. Results and discussion

Leaching tests were first carried out on waste CRT glasses following the procedure [39] described above. These results were compared to those obtained for the foam glass leaching tests. The mean concentrations of Pb, Ba, and Sr in the three leachate solutions (I, II, and III) are summarized in Tables 2–4. The reported values are averaged over 3 repeated experiments (see Section 2.2) for each type of investigated samples. The relative standard deviations estimated from 3 repeated experiments were found to be less than 0.2%. Displayed concentrations lower than 1.0 mg/L indicate that the concentration of the probed element was found below the detection limit.

### 3.1. Lead extraction

As shown in Table 2, the leachate from the original CRT funnel glass (F: the glass with the highest lead content) shows a total Pb concentration of 13 mg/L, which was below the acceptable leach limit for hazardous waste in landfill [7].

The Pb concentrations in leachates I, II, and III decrease progressively to attain a value lower than the detection limit for lead. Hence, with respect to the European legislation [1], such glasses may be disposed of as landfill. With panel glass (P), Pb concentrations in leachate solutions were all below the detection limit. These results are consistent with those of CRT glass analysis, where panel glasses were found to be free of Pb [24]. Moreover, foam glasses elaborated using such CRT glass exhibited similar behaviour. With the two synthesized samples (P5S and P4T), the Pb concentrations in leachates were close to the detection limits (Table 2). The analysis of the funnel glass-based foam glasses, showed a clear difference with respect to the two reducing agents employed. First, Pb concentrations were always higher in leachates, obtained from foam glasses prepared using TiN compared to the case of SiC, irrespective of the glass powder (funnel, panel, or mixture) used. Indeed, higher is the metallic Pb content in samples, greater is the Pb fraction extracted from these samples during leaching experiments. Although most samples yielded leaching rates below regulatory limits, those obtained using funnel glass and 4 wt% of TiN (F4T) were over the limit (at  $\approx 110 \text{ mg/L}$ ), consistent with previous observations. In earlier reports [22,24], we have pointed out that Pb(II) is reduced during the synthesis of the foam glass, and that both pore volume and specific surface of the so-obtained samples are strongly correlated with the concentration of the reducing agent used in glass synthesis. In addition, we have emphasized that a more significant fraction of Pb(0) is obtained by reduction inside

**Table 1**  
Manufacturing parameters, reducing agent content, type of glass used (in wt%) and reaction temperature (°C) and time (min), employed for synthesis of tested cellular materials. F, P, S and T represents funnel, panel glass, SiC and TiN respectively, number represents the reducing agent content in wt%: for example PF5S: foam glass prepared with panel and funnel glass with 5 wt% of SiC.

Sample	Funnel Glass (F)	Panel Glass (P)	SiC (S)	TiN (T)	Reaction temperature	Reaction time
F5S	95.0(1)	–	5.0(1)	–	850	60
F4T	96.0(1)	–	–	4.0(1)		
P5S	–	95.0(1)	5.0(1)	–		
P4T	–	96.0(1)	–	4.0(1)		
PF5S	31.7(1)	63.3(1)	5.0(1)	–		
PF4T	32.0(1)	64.0(1)	–	4.0(1)		

**Table 2**  
Amount of Pb released, in mg/L, after 16 h (leachate I), 32 h (leachate II), and 48 h (leachate III) of leaching and the sum of the Pb concentrations in leachates I to III.

Lead, Pb	Leachate I	Leachate II	Leachate III	Leachate I + II + III
F	13.00(1)	<1.0	<1.0	13.00(1)
F5S	<1.0	1.00(1)	1.10(1)	2.10(1)
F4T	2.30(1)	35.00(1)	74.00(1)	111.30(1)
P	<1.0	<1.0	<1.0	<1.0
P5S	<1.0	<1.0	1.00(1)	1.00(1)
P4T	1.00(1)	<1.0	<1.0	1.00(1)
PF5S	1.00(1)	1.00(1)	1.10(1)	3.10(1)
PF4T	1.50(1)	1.10(1)	2.40(1)	5.00(1)

**Table 3**  
Amount of Ba released, in mg/L, after 16 h (leachate I), 32 h (leachate II), and 48 h (leachate III) of leaching and the sum of the Ba concentrations in leachates I to III.

Barium, Ba	Leachate I	Leachate II	Leachate III	Leachate I + II + III
F	5.50(1)	5.50(1)	2.30(1)	13.30(1)
F5S	0.10(1)	0.30(1)	0.20(1)	0.60(1)
F4T	1.60(1)	0.90(1)	0.40(1)	2.90(1)
P	12.90(1)	5.10(1)	2.10(1)	20.10(1)
P5S	17.40(1)	11.80(1)	8.70(1)	37.90(1)
P4T	19.10(1)	11.50(1)	9.80(1)	40.40(1)
PF5S	1.40(1)	7.70(1)	10.00(1)	19.10(1)
PF4T	0.90(1)	3.20(1)	8.30(1)	12.40(1)

the glassy framework when TiN, rather than SiC, is employed as reducing agent [23,43,44]. Furthermore, we have evidenced that the Pb reduction is accompanied by an increase of the porosity, and, incidentally, by a modification of the accessible surface as attested by a slight evolution of the Langmuir surface areas measured by Kr adsorption at 77 K (0.6 m<sup>2</sup>/g and 1.0 m<sup>2</sup>/g for SiC and TiN respectively) [45]. Such experimental findings might suggest a larger amount of released Pb during leaching when one uses TiN as reducing agent [3]. The same trend was also evidenced for PF5S and PF4T (1/3 funnel–2/3 panel glass-based foam glasses). Further, the Pb leachability is revealed higher with the use of TiN following the sequence from leachate I to leachate III. The concentrations of Pb in leachates were found lower than those obtained for funnel-based foam glasses (F5S and F4T). By adding the panel glass to the funnel glass (2/3–1/3), the Pb concentration inside the original glass became lower than in funnel glass-based foam glasses. Ultimately, similar results were obtained for all samples, with Pb concentrations increasing from leachate I to leachate III. Higher is the level of Pb reduction, greater is the fraction of this element released

during the leaching. In all cases, the amount of Pb extracted from foam glass was lower than that from samples where Pb was initially encapsulated, after consideration of the leaching procedures employed [30].

In addition, if the current recycling practice recommends separating Pb- and Ba–Sr containing glasses, we have demonstrated that treating the mixture of these two glasses with SiC or TiN allows discarding these glasses in landfill, as the resulting lead concentration is lower than those obtained in case of the funnel glass.

### 3.2. Barium extraction

The concentrations of Barium in eluates are presented in Table 3. For original glasses, the amounts of Ba decreased from leachate I to III and were found lower than the statutory value ( $\approx 13.3$ – $\approx 20$  mg/L vs. 300 mg/L).

When the global behaviour of the foam glasses is examined, it can be seen that this property is distinct from that evidenced concerning leaching behaviour of Pb. For a material (F5S) elaborated

**Table 4**  
Amount of Sr released, in mg/L, after 16 h (leachate I), 32 h (leachate II), and 48 h (leachate III) of leaching and the sum of the Sr concentrations in leachates I to III.

Strontium, Sr	Leachate I	Leachate II	Leachate III	Leachate I + II + III
F	1.10(1)	0.40(1)	0.20(1)	1.70(1)
F5S	2.60(1)	1.70(1)	0.90(1)	5.20(1)
F4T	5.30(1)	1.10(1)	0.50(1)	6.90(1)
P	12.00(1)	3.40(1)	1.60(1)	17.00(1)
P5S	21.90(1)	12.40(1)	9.30(1)	43.60(1)
P4T	25.00(1)	13.70(1)	11.00(1)	49.70(1)
PF5S	24.40(1)	13.70(1)	13.20(1)	51.30(1)
PF4T	21.90(1)	9.10(1)	15.20(1)	46.20(1)

with funnel glass and containing a low concentration of Ba, the use of SiC, as reducing agent, led to a significant decrease of the Ba amounts in leachates. Moreover, the concentrations of Ba were close to 0.2 mg/L in the three leachate solutions, thus evidencing a total extraction of about 0.6 mg/L. In contrast, a material obtained using the same glass but employing TiN as reducing agent (F4T) exhibits a gradual decrease of the Ba content during the three consecutive leachates but the total amount of Ba released only attains about 3 mg/L. The differences can be explained by considering the modification of the specific surface properties; samples obtained using TiN contains more accessible porosity than those obtained using SiC. Turning to the panel glass-based samples (P5S and P4T in Table 3), increases in either SiC or TiN concentration yielded larger surface areas and thus increased the leachability of foam glass samples. Those values, compared to values seen in tests with the original glass, are then higher due to the increased of the leached surface. The amount of Ba in the foam glass leachates was twice higher ( $\approx 39$  mg/L) than those extracted from pure original glass ( $\approx 20$  mg/L). In contrast, materials (PF5S and PF4T) obtained by mixing panel and funnel glasses released a larger amount of Ba over time. Nevertheless, despite differences in cellular structure (pore size, surface area), the total concentration of Ba released was lower than those obtained from original glass. One possible explanation could rely on the considered funnel/panel glass ratio; the lower is the increase of the concentration of Ba due to the addition of 1/3 by wt of funnel glass, the lower is the concentration of leached Ba.

### 3.3. Strontium extraction

Strontium concentrations extracted from foam-glasses are summarized in Table 4. Trends concerning Sr for original panel glasses are similar to those found for Ba: the released concentration decreased again from leachate I to III.

Leachability of Sr was found to be even lower, with less than 2 mg/L released. As, only few data are available in the literature, it is difficult to draw useful conclusions from these observations. Some studies reported by Dondi's group (see, for example, [46]) have described certain trends and the Sr amounts released from glasses were found to be low. In the present work, using a different procedure and a distinct material (a glass), the Sr concentration in leachates was found to be close to 7 mg/L. Further, the amounts of Sr extracted from CRT and foam glasses were found to decrease with time. The largest amount of Sr leaching was seen with panel glass. It follows that the lowest released amounts of Sr were obtained with panel glass-based foam glasses (F5S and F4T, in Table 4). Whatever the choice of the reducing agent, the amount of leached Sr was higher than those obtained from original glass (6.5 mg/L vs. <2 mg/L). This difference can again be explained by considering the increase in porosity of foam glass. No significant changes were observed in any other sample. In all cases, and despite differences in the CRT glasses used, the Sr concentration in all leachates ranged from 44 to 51 mg/L. Compared to the value determined for panel glass, the increases in released Sr were obviously a consequence of the foaming process.

In addition, some trends can be explained by considering the porosity for the three studied elements. The obtained porosities of the samples are summarized in Table 5 [45].

In previous works it has been demonstrated that the larger pore diameters were obtained for the samples: (i) elaborated using TiN as reducing agent and (ii) containing higher contents of funnel glass [24,25]. Comparing porosity and the results of the leaching test for each type of samples, it is observed that higher is the porosity associated with larger pore sizes, higher is the metal concentration in the leachates. Another parameter that could be taken into account is the particles size. As it has been mentioned, the initial size of the particles is less than 4 mm diameter. Considering the porosity and

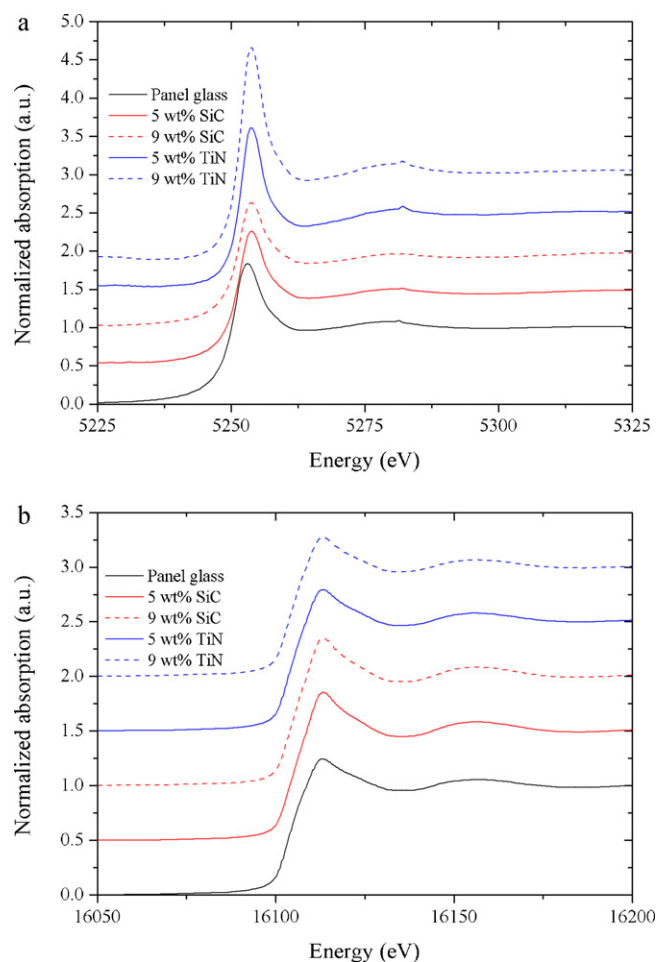


Fig. 1. Normalized Ba-L<sub>III</sub> (a), Sr-K (b) XANES spectra for funnel glass and foam glasses elaborated with SiC and TiN (C) compared to initial panel glass (for a clear presentation each profile was shifted by 0.5 from its lower-lying profile).

the mechanical behaviour of such porous materials we can deduce that the size should generally decrease during the leaching test for the most fragile samples (the one with the highest porosity). This leads to an increase of the surface available at the interface solid particles–water, giving rise to some differences from one leachate to another.

### 3.4. Local structure around Ba and Sr as a function of the reducing agent

Fig. 1 summarizes XANES (X-ray absorption near edge structure) spectra obtained using Ba-L<sub>III</sub> (a) and Sr-K (b) edges. The studied foam glasses have been elaborated using two contents: 5 and 9 wt% of SiC or TiN. All XANES spectra are compared with the spectra of the original panel glass.

As seen in Fig. 1, no differences were observed between panel glass and the foam glasses whatever the reducing agent content. The finger prints of the foams glass for the two studied elements are comparable to those of the initial glasses as well as those of crystalline references (BaO and SrO) [47]. It can be concluded that no change was induced on the local structure around barium and strontium treating the glasses by the reducing agents. In addition, extracted EXAFS (Extended X-ray absorption fine structure) oscillations, which after being Fourier transformed, are known to give Sr–O bond length and number of coordination (NC) around strontium, confirm the non-modification of its local structure. Bond lengths were found to remain constant for the two

**Table 5**  
Porosity of the studied samples as a function of the reducing agent content [45].

Sample	F5S	F4T	P5S	P4T	PF5S	PF4T
Porosity (%)	84 (1.7)	86.1 (1.7)	3.7 (0.1)	50.1 (1.0)	46.5 (0.9)	67.9 (1.4)

TiN and SiC contents: Sr–O = 2.52(1) Å with a number of coordination close to 5.70(2). Such findings are in contrast with the strong modification of the local structure around lead evidenced by XAFS combined with XPS in previous works [23,44,47]. These results give an explanation on the leaching behaviour observed for foam glasses compared to the one of the original glass. The enhancement in concentration of the two elements obtained in case of foam glasses, results only in the increase of the specific surface resulting of the foaming reaction.

#### 4. Conclusion

The results of leaching tests have shown variations in the behaviour of tested materials depending on the nature of the reducing agent employed and the type of CRT glass used to obtain the cellular material. The influence of the reducing agent was evidenced by a rise in leached Pb concentration, increasing in line with the accessible surface area of the sample. We showed that the amounts of Pb released from samples prepared with low concentrations of reducing agent, particularly those synthesized employing SiC, were under the regulatory limits. In contrast, when TiN was employed, the highest amounts of Pb released exceeded the legislative limits. The use of TiN for foam glass synthesis was appropriate when a mixture of the two types of glass–funnel and panel was employed. A porous material with lower lead leachability was obtained. The amounts of released Ba were under the regulatory concentration whatever the reducing agent content. Finally, the levels of released Sr depended on the accessible porosity of the material, and this was in turn influenced by the proportion of funnel glass and the concentration of the reducing agent.

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