Leaching behavior of CRT funnel glass

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ABSTRACT

The leaching behavior of cathode ray tube (CRT) funnel glass containing 23 mass percent of Pb in 0.001 N HCl, distilled water, and 0.001 N NaOH at 90 °C was investigated using a static method. The weight loss and leached amount of each component was measured and surface changes observed by SEM. The leaching mechanism is discussed. In acid solution, the leached amount of Pb showed $t^{1/2}$ dependence, that is, diffusion-controlled dependence, which is common in lead silicate glasses. In water and basic solutions, the leached amount showed saturation after higher initial dissolution than in acid and the deposition of many particles on the surface was observed. The amount leached was less for Pb than other components. The deposited particles formed a protective layer, which suppressed the dissolution of the glass. This dense layer must be formed as a result of a high initial dissolution rate.

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1. Introduction

Used cathode ray tube (CRT) glass is now recycled to produce new CRT glass, as required in Japan by the Home Appliance Recycling Law, which does not allow landfill disposal. However, since demand for CRTs is decreasing because of their replacement by flat-panel displays, used CRT glass will have to be managed differently in the near future. The use of CRT glass for other purposes is nowadays restricted to a considerable extent by its Pb content; since the RoHS directive came into effect, the production of Pb-containing materials has decreased and it is now more difficult to recycle lead glass. The typical composition of CRT funnel glass is $52.0SiO_2$–$22.0PbO$–$7.8K_2O$–$6.8Na_2O$–$4.0Al_2O_3$–$3.8CaO$–$1.8MgO$–$1.0BaO$–$0.5SrO$–$0.3Sb_2O_3$ (mass%) [1].

One method of dealing with this glass is to stock it in landfills as a future Pb resource. When the glass is stored in landfill, however, the leaching of Pb and other heavy metals into the groundwater may cause environmental problems. Musson et al. evaluated lead leachability from CRT glass using the toxicity characteristic leaching procedure [2] and concluded that CRT glass from color televisions is a hazardous waste material as it contains seal glass, which has a lead content of 80 mass% and is easily leached under acidic conditions. To avoid this problem, CRT glass should be separated according to composition into several types, each of which should then be tested for landfill safety and stored or treated separately. The risks from CRT glass landfill are twofold: Pb leaching into groundwater and diffusion of Pb into soil. In the present study, we investigated the former problem.

In Japan, a leaching test called JLT-13 [3] is used to evaluate whether a material can be disposed of in industrial waste landfills. The test was applied to a CRT glass by Nakamura et al. [4]. Since JLT-13 is a short-term test, longer-term leaching behavior should also be estimated to ensure safety. Environmental conditions must also be taken into account. The groundwater may be acidic due to acid rain or basic due to the dissolution of metals in fly ash disposed of in the landfill or the dissolution of Ca from concrete. The present research focused on CRT funnel glass in an examination of the leaching behavior of glass components into acid, neutral, and basic solutions. Surface changes after leaching were studied to determine the leaching mechanism and establish a corrosion model for CRT glass. For this purpose, the MCC-1 test [5], used for nuclear waste glass, was applied since it uses a glass block whose surface can be observed easily.

The corrosion of Pb-containing glass by acid has been extensively studied [2,6–17] since lead crystal glass is often used for tableware and the dissolution of Pb is harmful. The results of pre-
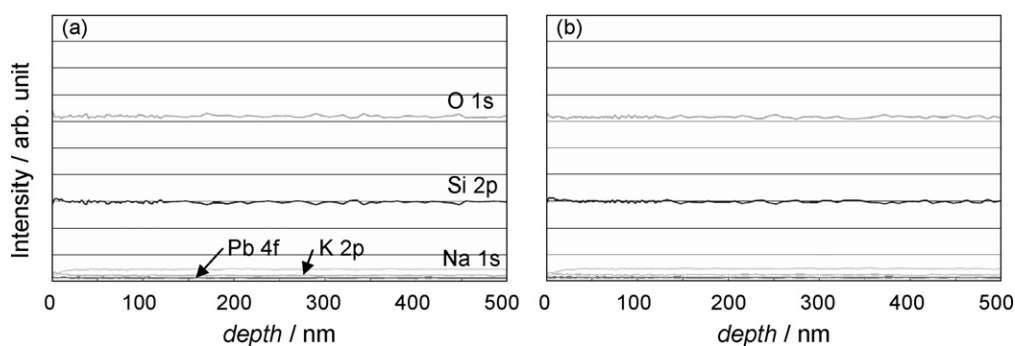
Fig. 1. Depth profile of elements measured by XPS; (a) fractured and (b) polished surface.

vious studies show that Pb is preferentially dissolved into acid solution from glass and that the time dependence of the dissolution indicates a diffusion-controlled mechanism in the first stage. The leaching rate becomes proportional to time after a certain period because of the formation and dissolution of a silica-gel layer. In contrast, there have been few studies on Pb glass corrosion under neutral [18] or basic conditions. It is known that conventional silicate glass is subject to significant corrosion by basic solutions as the Si–O–Si bond is attacked and broken by OH^- [19–21]. Hence the leaching of Pb caused by glass network dissolution under basic conditions also deserves to be studied.

Glass corrosion in neutral water has been extensively studied in the field of nuclear waste glasses [22–26]. Since this requires long-range prediction, many studies use models of thermodynamic stability. Recently, Casey showed the importance of surface structure dynamics in glass corrosion [27]. Cailleteau showed that the introduction of a thermodynamically stable element such as Zr into glass causes increase in the amount of corrosion since Zr prevents the formation of dense structures in the surface gel layer [28]. These studies reveal the importance of the surface structure of the protective layer formed on the glass surface during leaching.

2. Experimental

The CRT funnel was provided by Nippon Electric Glass (Malaysia) Sdn. Bhd. and produced from recycled cullet. The composition was $51.7SiO_2$–$23.2PbO$–$7.7K_2O$–$6.2Na_2O$–$4.3Al_2O_3$–$3.5CaO$–$1.9MgO$–$0.5SrO$–$0.3BaO$–$0.3B_2O_3$–$0.3Sb_2O_3$–$0.2Fe_2O_3$ (mass%), which was analyzed at NSG Techno-Research Co. Ltd. by wet analysis. This corresponded to $65.7SiO_2$–$7.9PbO$–$6.2K_2O$–$7.6Na_2O$–$3.2Al_2O_3$–$4.8CaO$–$3.7MgO$–$0.4SrO$–$0.2BaO$–$0.4B_2O_3$–$0.1Sb_2O_3$–$0.1Fe_2O_3$ (mol.%). The composition was similar to that of other funnel glass [1].

Samples for the leaching test were cut to approximately $16\,mm \times 7\,mm \times 4\,mm$ and ground to a flat surface. Since surface roughness or damage to the glass sample may affect the amount of leaching, samples prepared using a surface treatment process were studied and the most suitable process selected. A smooth surface was obtained after grinding with #2000 SiC paper on a wheeling machine or with #3000 SiC powder by hand. However, the weight loss of the #2000 ground sample was twice that of the #3000 ground sample in tests with 0.001 N HCl solution, which may have been due to residual stress on the samples. To reduce surface stress, we chose to carry out sample preparation by grinding the sample with #3000 SiC powder by hand. The grinding was performed in oil to avoid compositional change through leaching in water. The compositional difference between the ground surface and the fractured surface was measured by X-ray photoelectron spectroscopy (XPS) analysis with Ar etching at Sumika Chemical Analysis Service, Ltd.

The leaching test was carried out based on the MCC-1 test used for nuclear waste glass [5], a static leaching test method conducted at $90\,^{\circ}C$ at 7, 14, 28, 56, 91, and 182 days in a Teflon vessel. The present sample had a surface area (SA) of $400\,mm^2$ and the volume of the leaching solution (V) was 40 mL, giving a SA/V ratio of $10\,m^{-1}$. The glass sample was suspended in the leaching solution with Teflon thread. The leaching solutions were distilled water, 0.001 N HCl, and 0.001 N NaOH, with a pH in the range of 3–11. The tests at 1 and 3 days were also carried out at pH 11. The 182-day leaching test in distilled water has not yet been carried out. Three samples were tested for each set of conditions, except for the 56-day leaching test in NaOH solution. The weight of the sample was measured before and after the test and the concentration of each element in the solution after leaching was measured, using inductively coupled plasma (ICP) spectrometry (SPS7800; Seiko Instruments Inc.) for Si, Pb, Ca, Mg, and Al and atomic absorption spectrometry (AAS; AA-680, Shimadzu) for Na and K, with no dilution. Surface changes were observed by a scanning electron microscope equipped with an energy-dispersive X-ray spectrometer (SEM/EDS, JSM-5900LV JEOL). Surface particles were observed with a Raman microspectroscope (JRS-system 1000; JEOL). The X-ray diffraction pattern was measured with an X-ray diffractometer (RAD-II, Rigaku) with Cu-$K\alpha$, which was tested using a leached glass powder of 40–70 μm particle diameter.

3. Results

Fig. 1 presents a comparison of compositional difference near the surface in fractured and ground samples measured by XPS. Since there was no difference between the samples, the grinding method used was judged to be appropriate.

After 91 days of leaching, the weight loss reached 0.0014 g in 0.001 N HCl solution, but tended to remain constant at 0.0006 g and 0.0012 g in distilled water and 0.001 N NaOH, respectively. Since the corrosion rate depends on the SA of the sample, total mass loss (ML) was calculated from the following equation:

$$ML = \frac{\Delta W}{SA}$$

where ML = total mass loss (g m^{-2}); ΔW = weight change (g); and SA = surface area (m^2).

The graph plots of ML against the square root of leaching time are shown in Fig. 2. The error bars represent the dispersion of the three samples. Weight loss increased with the square root of leaching time in 0.001 N HCl, but remained almost constant in water and in 0.001 N NaOH. The leached solution was analyzed for its metal content using ICP techniques. The leached amount of each element was normalized with the mass fraction of the element from the
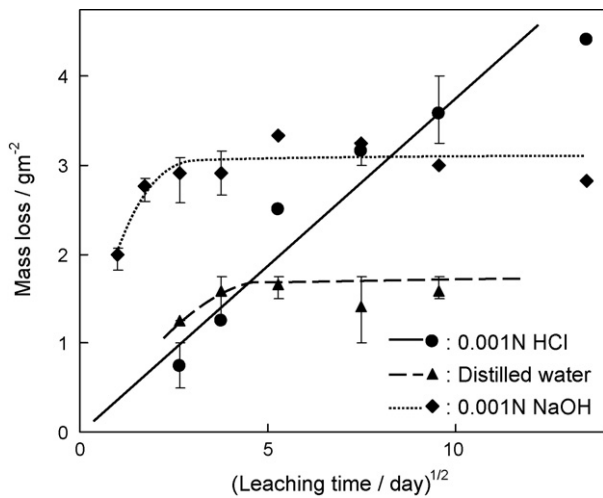
Fig. 2. Total mass loss measured by weight change against square root of time.
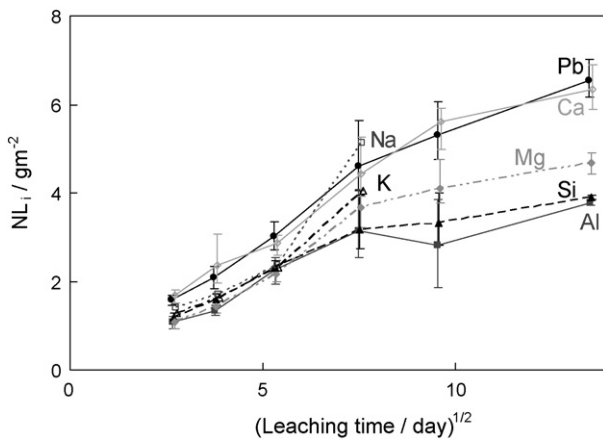

Fig. 3. Normalized mass loss of samples against square root of time after leaching in 0.001 N HCl solution.

equation:

$$NL_i = \frac{C_i}{f_i} \cdot \frac{V}{SA}$$

where NL_i = normalized elemental mass loss of element i (g m^{-2}); C_i = concentration of element i in leachant (g m^{-3}); f_i = original fraction of element i in glass; V = volume of leachant (m^3); and SA = surface area (m^2).
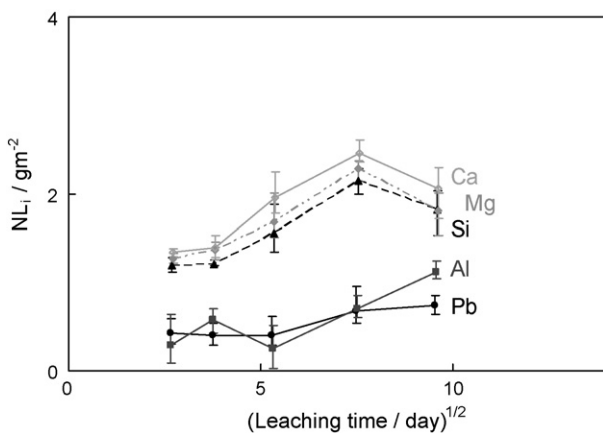

Fig. 4. Normalized mass loss of samples against square root of time after leaching in distilled water.
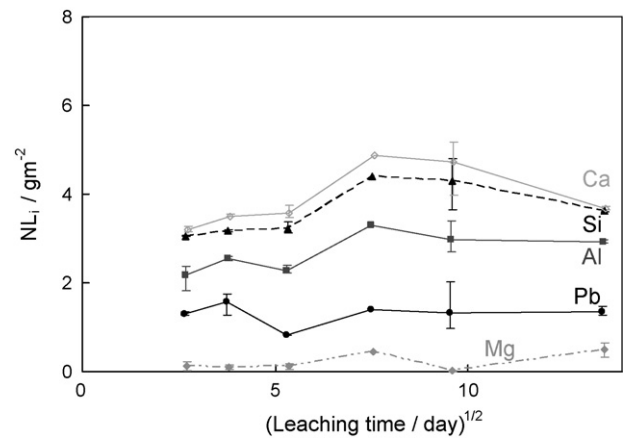

Fig. 5. Normalized mass loss of samples against square root of time after leaching in 0.001 N NaOH solution.

The normalized elemental mass loss (NL) for element i was calculated in each case and the graph plots of NL against the square root of leaching time are shown in Figs. 3–5. The lines on these figures were drawn as a visual aid. All elements except for Pb and Mg were below saturation in the NaOH solution because of the small SA/V ratio. The concentration of Si in the HCl solution after 182 days was 9.3 mg L^{-1}, which is about 1/20 of the solubility of amorphous silica at 100 °C [29].

NL_{Pb} content was high in the HCl solution but NL_{Si} and NL_{Al} content was low. The highest Pb release from sample was 0.56 mg in the HCl solution after 182 days, which corresponded to 6.5 g m^{-2} of NL_{Pb}. In the water and the NaOH solution, NL_{Pb} content was 0.69 and 1.3 g m^{-2}, respectively, much less than in the HCl solution. It is interesting that NL_{Mg} content was low only in the NaOH solution. The time dependence of the leached amount showed a similar trend to the time dependence of weight loss shown in Fig. 2. The weight losses estimated from the NL_i values were compared with the mass losses in Fig. 2 and shown in Table 1. The mass losses were slightly smaller than those calculated from the leached ionic concentration. This may be caused by uptake of water in altered glass. In NaOH solution, although the concentration of alkalis was not measured, the mass losses were about 80% of the value calculated from ionic concentration except for alkalis. If we consider NL_{Na} and NL_K to be the same as NL_{Si}, the value estimated from the ionic concentration slightly exceeds the mass loss in a few samples. This is caused by precipitation of Pb onto the Teflon vessel. A slight amount of Pb was detected by ICP measurement in acid washing of the vessel.

SEM/EDS was used to observe surface changes in the sample after leaching. In the HCl solution, the CRT glass was corroded with many hollows and a number of small glass fragments were found on the surface, as shown in Fig. 6(b)–(f). These hollows may have formed due to residual stress during grinding. Corrosion increased with increasing leaching time. EDS results show a slight loss of all metal components except Al with leaching time in the acid solution,

Table 1
Comparison of mass loss measured by weight change and calculated from concentration of elements in leached solution measured by ICP for samples leached in 0.001 N HCl. Each value has an error of approximately ±0.1 mg.

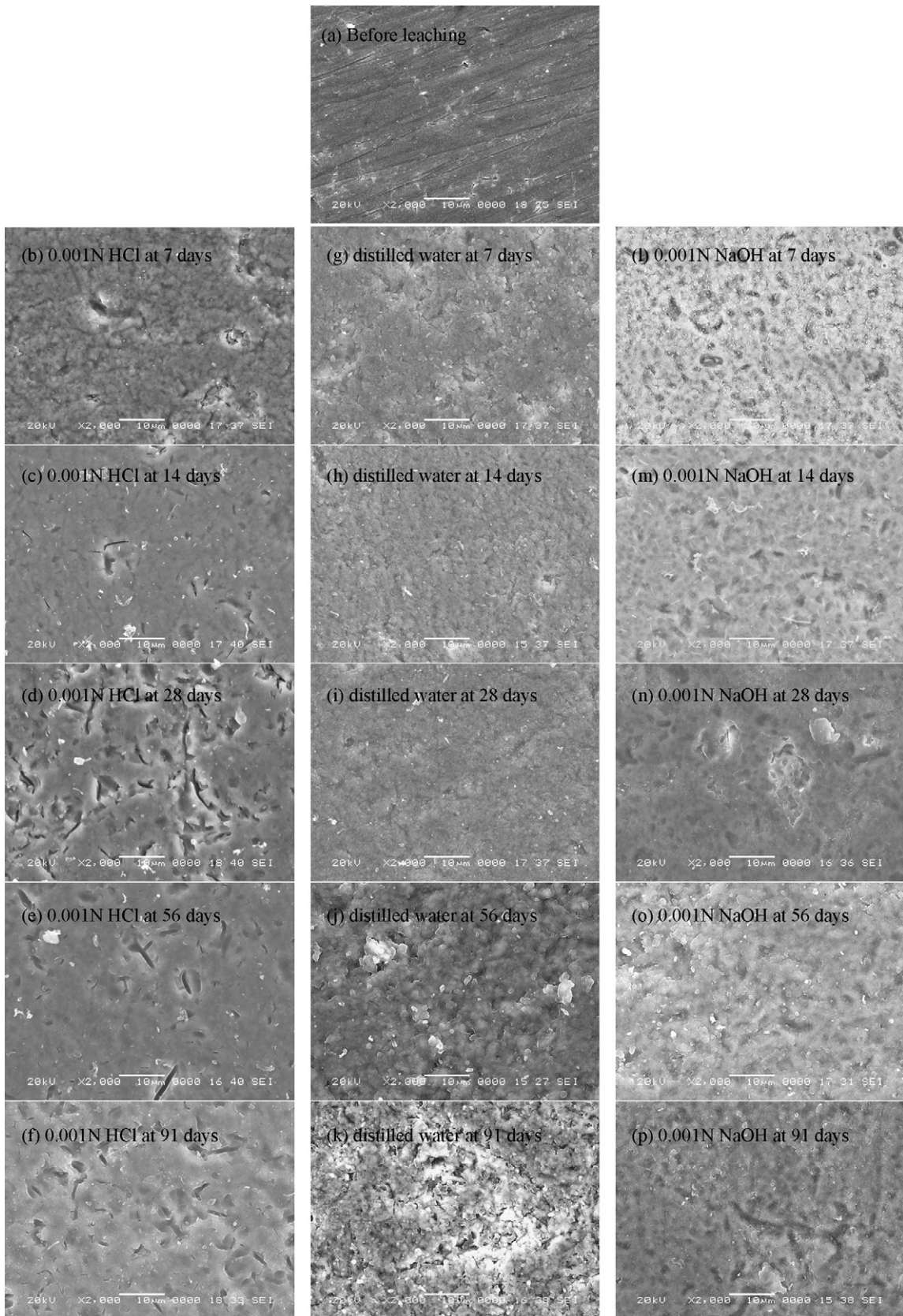| Leaching period/day	Mass loss/mg	
	Weight	ICP
7	0.3	0.51
14	0.5	0.67
28	1.0	0.97
56	1.27	1.46

Fig. 6. SEM micrographs of samples before and after leaching in 0.001 N HCl, distilled water and 0.001 N NaOH solutions at 90 °C for 7, 14, 28, 56, and 91 days.
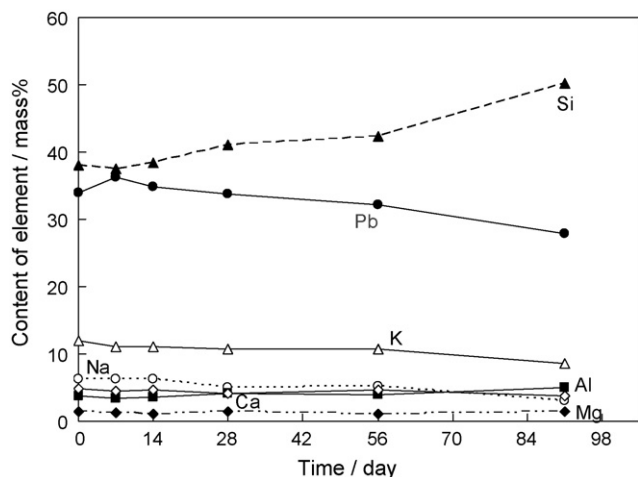
Fig. 7. Element analysis by EDS at sample surface after leaching at 90 °C in 0.001 N HCl solution.

Fig. 9. Element analysis by EDS at sample surface after leaching at 90 °C in 0.001 N NaOH solution.

as shown in Fig. 7. The Si and O elements remained near the surface. This tendency is consistent with the results for *NL*.

In water, the surface of CRT glass corroded slowly, and a rough and porous surface was seen after leaching for about 4 weeks. Particles were embedded on the surface to form layer after 8 weeks as shown in Fig. 6(j). Fig. 8 shows the EDS results, indicating that, except for Pb and Ca, the amount of metal components at the surface decreased after leaching. Unlike in the results observed on the leached surface in acidic solution, Pb concentration was high at the surface.

In NaOH solution, the surface of the CRT glass was severely corroded by the end of the first week. A thin altered layer was observed on the surface, as shown in Fig. 6(l). A deposit layer of many particles was clearly observed on the surface during days 7–28 of leaching. EDS results showed that Si was present in lesser quantities both at the surface and in particles on the surface, as shown in Fig. 9. It is interesting that the Pb and Mg contents of the leaching surface were higher than on the original surface. It remains unclear whether these elements remained bonded in a glass network or appeared as a precipitate on the surface. EDS analysis of the particles showed slightly higher Pb and Mg content than in the results shown in Fig. 9 and also indicated the presence of other elements. This means that the particles did not consist of pure hydroxide precipitation.
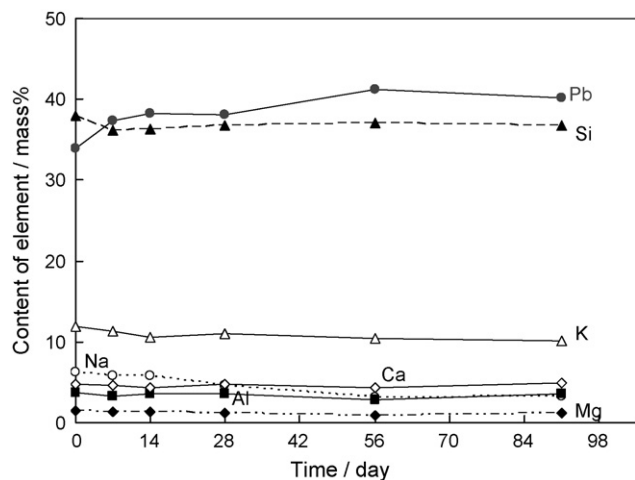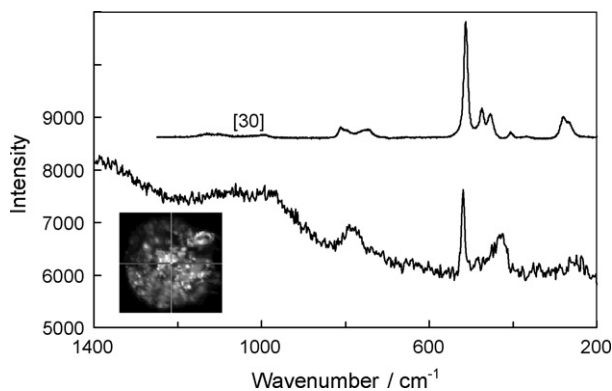

Fig. 10. Micro-Raman spectrum of a particle found on a sample leached in 0.001 N NaOH solution at 90 °C for 56 days. The spectrum of $KAlSi_3O_8$ feldspar [30] is also shown for comparison.

There were some particles on the deposit particle layer and their micro-Raman spectra showed many different types of spectra with sharper peaks than the spectrum of deposit layer. This suggests that the particles consisted of crystals, most of them however unknown. For a particle on the surface on day 56 of leaching in water, the spectrum resembled that of $KAlSi_3O_8$-orthoclase feldspar [30] as shown in Fig. 10. Since no X-ray diffract pattern was found on any leached glass block, powder glass was leached in 0.001 N NaOH solution at 90 °C for 35 days. However, no peak was found in the spectrum, as shown in Fig. 11.
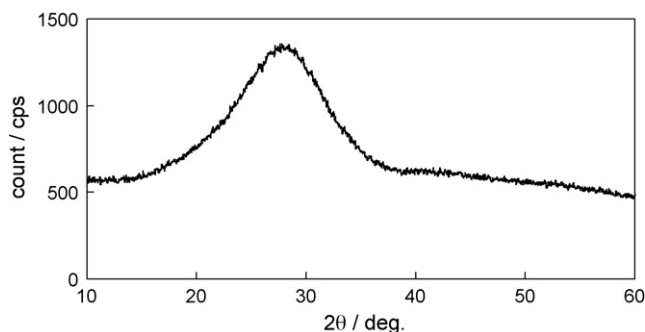

Fig. 8. Element analysis by EDS at sample surface after leaching at 90 °C in distilled water.

Fig. 11. X-ray diffract pattern of powder glass leached in 0.001 N NaOH solution at 90 °C for 35 days. No peak was found.

4. Discussion

Over the 91-day experimental period, corrosion in the acidic solution was higher than in the other leaching solutions and ML reached 4.4 g m^{-2} at day 182. The surface after leaching was smooth but contained many pores. EDS results showed that the Si content remained high at the surface while content of Pb and other metals was lower than before leaching. ICP results also showed that the NL of alkaline metals was high, with NL_{Pb} the highest and NL_{Si} the lowest. Raman results showed no change in the Si structure of the glass surface until the 28th day, from when a number of particles were found embedded in the surface.

The primary reaction expected in acidic solution is glass network hydrolysis/dissolution, for which it has been suggested by Hench and Clark [20] that the leaching mechanism includes the following two reactions: dealkalization by H$^+$ exchange, which is a diffusion-controlled $t^{1/2}$-dependent reaction; and network dissolution by OH$^-$, which is an interface-controlled t^1-dependent reaction. These reactions also apply in the case of lead silicate glasses. The formation of a silica-rich gel layer and its dissolution has been studied previously [6–8]. Pb and alkalis are preferentially leached from these lead glasses. Since the dependence of ML and NL on leaching period in CRT funnel glass is similar to that in these glasses, the corrosion mechanism should also be similar. The enrichment of silica shown in Fig. 7 supports the gel layer formation.

With a longer leaching period, the leaching rate should show linear time dependence [6] when the dissolution becomes steady state. In the present study, however, no constant leaching rate was apparent within the maximum experimental period of 182 days. Since usually the dissolution rate slows down with time until it shows linear time dependence, the maximum rate after a long period will be less than the rate at 182 days. A longer experimental period is needed to detect the final dissolution rate.

In contrast, leaching behavior under neutral and basic conditions was very different. ML values were less time-dependent and almost constant, regardless of the experimental period, for 3 days under basic conditions or 14 days under neutral conditions, as shown in Fig. 2. This means that the dissolution of glass virtually ceased during this period. Many particles were found embedded on the surface. The ML value in alkaline solution was twice that in distilled water. Since the elements leached from the glass and those remaining at the surface differed between these two conditions, the structure of the protective layer may be slightly different.

In basic solution, the formation of a protective layer by low-solubility compounds is important [19]. SEM photographs show the surface changes with advance of the leaching period. At 7 days' leaching, many small particles were found on the surface as shown in Fig. 6(l). At 56 days, the surface was covered with larger particles bonded together as shown in Fig. 6(o). Since there was no change in ML in this period, the particles may grow with time through a dissolution–precipitation mechanism. Since the initial dissolution rate was much higher than in acid solution, the surface appears to become porous on the molecular scale at first, which eases the formation of dense protective layer [28]. In such a scenario, the growing particles may not directly relate to the protective layer since the ML ceased only at 3 days. In the present case, since Si is preferentially dissolved, other oxide will form the layer instead of silica. In CRT funnel glass, Pb(OH)$_2$ and Mg(OH)$_2$ have very low-solubility product constants and may form a protective layer. The solubility of Pb^{2+} at pH 11 was calculated to be much less than 1 mg L^{-1} at room temperature. Since the measured Pb concentration after leaching in alkaline solution was approximately 3 mg L^{-1}, Pb must be present not as a bare cation, but in some other form, such as a colloid in solution. When we assess the diffusion of Pb into soil, we might consider the existence of the colloid. The dense structure may not be stable over long periods of time because of

strain from temperature change and may detach from the surface [18]. Under such conditions, the release of Pb from the glass will increase. Hence, the stability of the protective layer is important for the estimation of leaching over long periods of time. In the present study, no such detachment or crack on the leached surface was observed within the experimental period. Again, longer leaching tests are important to determine whether such detachment occurs after a long period.

The surface changes under neutral conditions are clearly apparent in the SEM photograph in Fig. 6, which shows precipitation-like dense structures, and, as shown by a comparison of Fig. 6(g) and (l), the layer formation with particles is more apparent than in basic solution. Since the tendency of time dependence of ML is similar to that in basic solution, the layer formation mechanism should be similar: a high initial dissolution rate produces a dense protective layer [28]. The EDS results in Fig. 8 indicate that Pb must participate in the protective layer formation. Although silica is not as soluble in neutral solution as in basic solution, much Si was detected in leached solution, as shown in Fig. 4. Compared to the findings of Ahmed et al. [18], NL_{Si}/ML in the present study was higher. One possible reason for the silica dissolution is a rise in pH [18]. However, although the pH values were measured in several samples after leaching, the values were unstable and did not exceed 8, probably because of the low concentration of dissolved elements caused by the low SA/V ratio. The difference between the present study and Ahmed et al.'s study is in glass composition and experimental conditions: CRT glass contains alumina and more alkalis than the crystal glass in their study, and they conducted experiments at room temperature for a long period and at high temperature for a short period. These differences may cause different leaching behavior.

The temperature difference between the testing conditions and landfill may also cause different behavior. Sheng et al. [25] studied the leaching behavior of borosilicate glass in water and discussed the properties of the surface layer, concluding that the layer became protective at 90 °C, but was not so at 70 °C. If a protective layer does not form, the silica network will successively dissolve and the less soluble elements will be released into the solution as colloids. Although this result may not apply directly to CRT glass because of the difference in composition, leaching tests at lower temperatures should be carried out to confirm the applicability of the present study.

5. Conclusion

Leaching tests of CRT funnel glass were performed at 90 °C using the MCC-1 method for 7, 14, 28, 56, 91, and 182 days in 0.001 N HCl, distilled water, and 0.001 N NaOH solutions.

In HCl solution, ML increased with the square root of leaching time within the leaching period of 91 days and reached 4.4 g m^{-2} at 182 days. NL calculated from ICP results showed that the amount leached of Pb and alkaline metals such as Na (NL_{Pb}, NL_{Na}) was high while that of Si (NL_{Si}) was the lowest, and that the time dependence of NL was almost the same as that of ML. SEM showed clear corrosion on the sample surface. The leaching mechanism is assumed to be the same as that in other lead–silicate glasses, whose soluble elements such as alkalis and Pb are preferentially dissolved and on which a silica gel surface layer is formed. Estimation of the dissolution rate of the gel layer is important for the evaluation of leaching over long periods of time.

In water and NaOH, ML reached a peak in 14 and 3 days, respectively, but then tended to remain constant at around 1.5 and 3.0 g m^{-2}, respectively. NL_{Ca} and NL_{Si} showed higher values than for other elements. It was noted that, in NaOH solution, NL_{Pb} and NL_{Mg} values were low. Particles were observed by SEM after leaching for

only 7 days. EDS results showed that Si content at the surface was low while Pb remained high in NaOH solution, strongly suggesting that some sort of protective layer was formed on the surface. A solid protective layer would be formed by a high dissolution rate at the beginning and following densification. Pb must play an important role in the formation of the layer. The stability of this layer needs to be clarified to allow estimation of leaching behavior over long periods of time.

Acknowledgment

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