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DEVELOPMENT OF GLASS COMPOSITIONS FOR IMMOBILIZATION OF SRP WASTE

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ABSTRACT

An improved composition of glass frit, termed "411", was developed for the immobilization of SRP high-level radioactive waste. In comparison with frit compositions previously studied, the new composition offers the following advantages:

- The amount of ferrite-spinel crystals formed is greatly decreased because the TiO_2 content is much lower.

- A higher lithium-to-sodium ratio decreases the viscosity of glass mixtures containing high-aluminum waste.

The compositional studies leading to the development of the improved glass are reported.

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INTRODUCTION

Radioactive waste produced from reprocessing nuclear fuel for defense programs at Savannah River Plant (SRP) is stored in large underground tanks on the plant site. This alkaline waste is made up of two parts. Most of the waste actinides and fission products are contained in an insoluble sludge, consisting of mainly hydroxides and hydrous oxides of Fe, Al, and Mn. The rest of the waste is either in the form of a soluble crystalline salt cake or a nearly-saturated supernatant salt solution.[1,2] This fraction contains ∿95% of the fission products cesium and traces of other radionuclides.

Methods to immobilize SRP waste for long-term storage are being developed at Savannah River Laboratory. The current reference process begins by pumping off the supernatant liquid for decontamination by ion exchange.[3] The sludge is slurried with water, removed from the tank, and washed with hot caustic to remove ∿75 wt % of the Al. The radioactive sludge and radionuclides from ion exchange are dried by spray calcination, mixed with glassforming chemicals and melted to form a borosilicate glass (Figure 1).

Initial glass composition development was directed toward demonstrating the feasibility of immobilizing SRP waste in glass.[4,5,6] A large amount of experimental data was obtained on ~~the properties of simulated and actual waste immobilized in~~ a glassformer composition called Frit 18 (Table 1). On a laboratory scale, this composition could not successfully immobilize more than 25 wt % of sludges high in aluminum. A new composition, Frit 21 (Table 1), in which 4 wt % Li_2O was substituted for 4 wt % Na_2O could immobilize up to 35 wt % of these same sludges and was adopted as the reference glass composition. Waste forms made from Frit 21 have exhibited good leach resistance and adequate solubility for SRP waste, on a laboratory scale.[7]

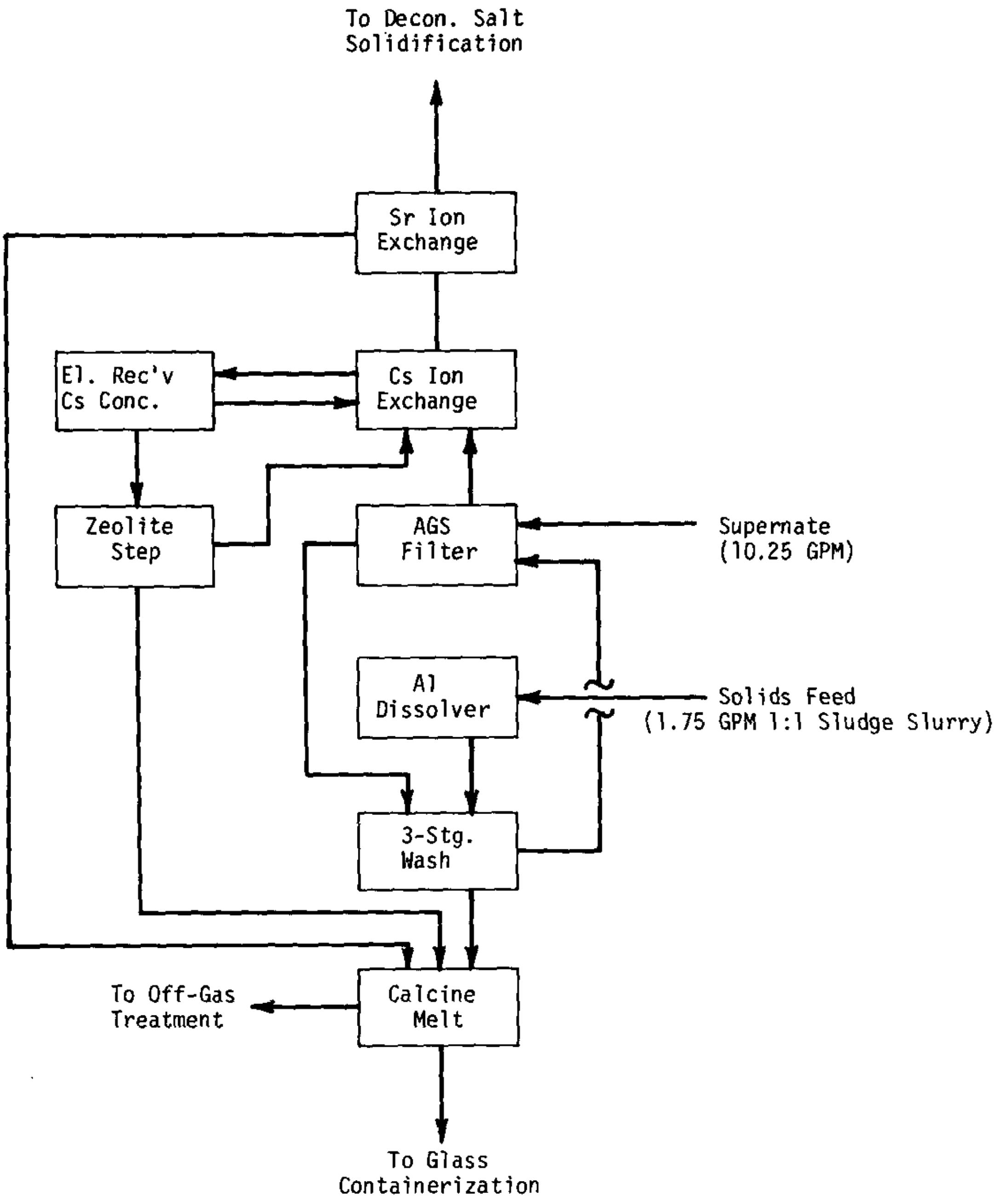

FIGURE 1. Waste Solidification Process

However, subsequent studies[7,8] and tests in large-scale equipment at Battelle-Pacific Northwest Laboratories have indicated three processing problems involving Frit 21-sludge glasses.

(1) The viscosity of melts rich in Al_2O_3 is quite high, and therefore melting rates are very low.

(2) Sludges containing large amounts of Fe_2O_3 precipitate ferrite-spinel crystals during melting. These can settle to the bottom of the melt, forming slags, and can contribute to foaming problems.

(3) Melts high in alumina also readily devitrify, forming refractory aluminosilicates.

Thus, this study was undertaken to improve the processing characteristics of SRP waste glasses, without greatly altering glass quality.

Three properties relating to processing were evaluated.

- *Viscosity.* This is the most important property since it determines the melting rate.[9]
- *Solubility of solid phases.* As already noted, buildup of ferrite-spinel slags could make a melter inoperable. There are several materials, such as Ru salts, or PuO_2, which are known to be of limited solubility in borosilicate glasses and could also form insoluble slags.
- *Devitrification.* If a glass devitrifies too readily, it could form crystalline plugs in a glass melter in areas where some cooling occurs, such as the pour spout.

These properties were evaluated as function of glass former composition. Previous studies have examined the effects of sludge composition of these same properties,[7,8] and have found that 1) Al increases the viscosity, and tendency to rapidly devitrify; 2) Fe reduces the viscosity, but can form insoluble phases; and 3) the other sludge components have little effect on viscosity, but Ni and U retard devitrification.

TABLE 1

Glass Frits

Component	*Amount (wt %)* *Frit 18*	 *Frit 21*
SiO_2	52.5	52.5
Na_2O	22.5	18.5
B_2O_3	10.0	10.0
TiO_2	10.0	10.0
CaO	5.0	5.0
Li_2O	-	4.0

Effects of Frit Components

Silica, alkali, and titanium dioxide had the largest effect on glass processing. Increasing silica content greatly increased the viscosity, but had little effect on devitrification. Increasing alkali content greatly reduced the viscosity, but increased devitrification. TiO_2 reduced the viscosity somewhat, but greatly increased devitrification and formation of insoluble phases.

Other frit modifications investigated included variations in the amount, concentration, and kind of alkaline earth oxide, substitution of Al_2O_3 for B_2O_3, and use of copper oxides. None of these reduced the viscosity significantly.

Procedures

The composition of the reference glass frit (Frit 21) and of the simulated sludges used are listed in Tables 1 and 2. All glasses contained 25 wt % simulated sludge with each candidate frit. The procedures reported previously were used to measure viscosities.[8] Viscosities of Newtonian melts were used in Fulcher's equation:[10,11]

$$\log \eta = A + B/(T-T_0)$$

All composition changes were first tested on glasses that contained composite sludge, and melted within 3 hours. Promising compositions were then tested with other sludges.

Effect of SiO_2

Silica has relatively little effect on devitrification or solubility of solid phases. However, it strongly affects the viscosity (Figure 2). For the range of 50-55 wt % SiO_2 in the frit, the viscosity was increased by increasing the silica content. As the SiO_2 content of the glass former varied from 50-55 wt %, the viscosity at all temperatures increased by about a factor of 2.

TABLE 2

Composition of Simulated Calcined Sludges

Component	*Composition (wt %)* *Average*	*Composite*	*High Iron*	*High Aluminum*
Fe_2O_3	43.5	31.6	61.4	6.0
Al_2O_3	25.8	46.4	5.6	86.3
MnO_2	11.7	10.3	4.1	4.9
U_3O_8	11.0	6.1	14.2	1.5
CaO	3.0	3.3	4.2	0.4
NiO	5.0	2.3	10.5	0.9

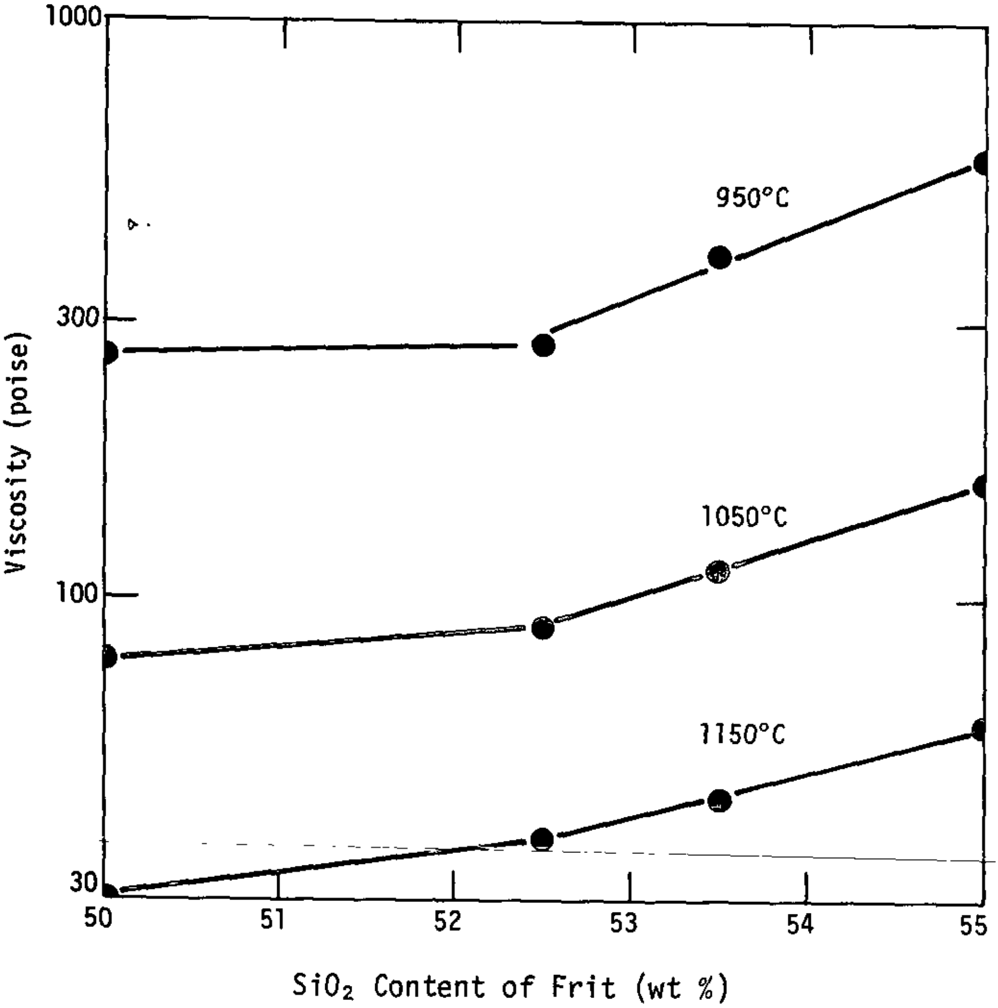

FIGURE 2. Effect of Silica Content of Frit on Viscosity of Composite Sludge-Glass. All Samples Contain 25 wt % Sludge.

Role of Alkali Metal Oxide

For glasses similar to Frit 21, the viscosity at 1150°C of melts containing composite sludge is determined by:

- Moles of alkali metal oxide per mole of silica.

- Moles of Li_2O per mole total alkali metal oxide.

The effects of both the type and concentration of alkali oxide were investigated. Some melts had potassium substituted for some of the sodium to try to reduce separation of the ferrite phase, and devitrification resulting from a high aluminum phase. These efforts did not succeed, and produced much more viscous melts (Table 3). Substituting Li_2O for Na_2O in other melts (while maintaining the alkali metal oxide content at 22.5 wt % of the frit) was very successful at reducing the viscosity.

Figure 3 shows the viscosity of several melts at 1150°C as a function of the moles of alkali metal oxide/mole SiO_2. The composition of each melt is listed in Table 3.

The variation of log viscosity at 1150°C with alkali metal oxide/silica ratio (R) is almost linear (Figure 3). However, Li_2O had been included in Frit 21 because of the mixed alkali effect.[11,12] Many glass properties have maxima, minima, or points of inflection at certain ratios of Li_2O to total alkali content. Melts were prepared with the same value of R as Frit 21 (0.494), but different values of the Li_2O/total alkali ratio (R_a). Figure 4 shows that for constant R, R_a controls the viscosity.

One other important observation was that for values of R_a greater than 0.6, the glasses readily devitrified to form a $LiAlSiO_4$-type phase, similar but not always identical to β-eucryptite. As R_a increased, this phase appeared at higher temperatures. Thus, for glasses similar to Frit 21, $R_a<0.6$ should be maintained.

TABLE 3

Composition[a] and Viscosity of Frits in Figures 1 and 2

Viscosity (poise) at 1150°C	*Alkali to Silica Molar Ratio*	*Composition (relative wt units)*						
		SiO_2	*B_2O_3*	*TiO_2*	*Na_2O*	*Li_2O*	*K_2O*	*CaO*
293.0	0.330	52.5	10.0	10.0	8.9	-	13.6	5.0
229.0	0.345	52.5	10.0	10.0	11.25	-	11.25	5.0
102.0	0.415	52.5	10.0	10.0	22.5	-	-	5.0
79.0	0.362	52.5	10.0	10.0	-	5.4	17.1	5.0
79.0	0.416	52.5	10.0	10.0	3.6	7.4	11.5	5.0
60.2	0.447	55.0	9.5	9.5	17.5	3.8	-	4.7
45.0	0.475	53.5	9.8	9.8	18.1	3.9	-	4.9
38.3	0.494	52.5	10.0	10.0	18.5	4.0	-	5.0
34.8	0.513	51.8	9.9	9.9	19.5	4.0	-	5.0
31.1	0.534	52.0	9.9	9.9	18.3	5.0	-	4.9
29.0	0.547	50.0	10.5	10.5	19.5	4.2	-	5.3
28.0	0.517	52.5	10.0	10.0	7.5	7.5	7.5	5.0
24.2	0.560	52.5	10.0	10.0	15.2	7.3	-	5.0
11.1	0.638	52.5	10.0	10.0	11.25	11.25	-	5.0
5.1	0.862	52.5	10.0	10.0	-	22.5	-	5.0
57.6	0.494	52.5	10.0	10.0	26.8	-	-	5.0
29.2	0.494	52.5	10.0	10.0	13.4	6.4	-	5.0
19.6	0.494	52.5	10.0	10.0	6.7	9.7	-	5.0
15.7	0.494	52.5	10.0	10.0	-	12.9	-	5.0

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[a] All melts contained 75 wt % of each frit, 25 wt % composite sludge. Values listed are relative weight units.

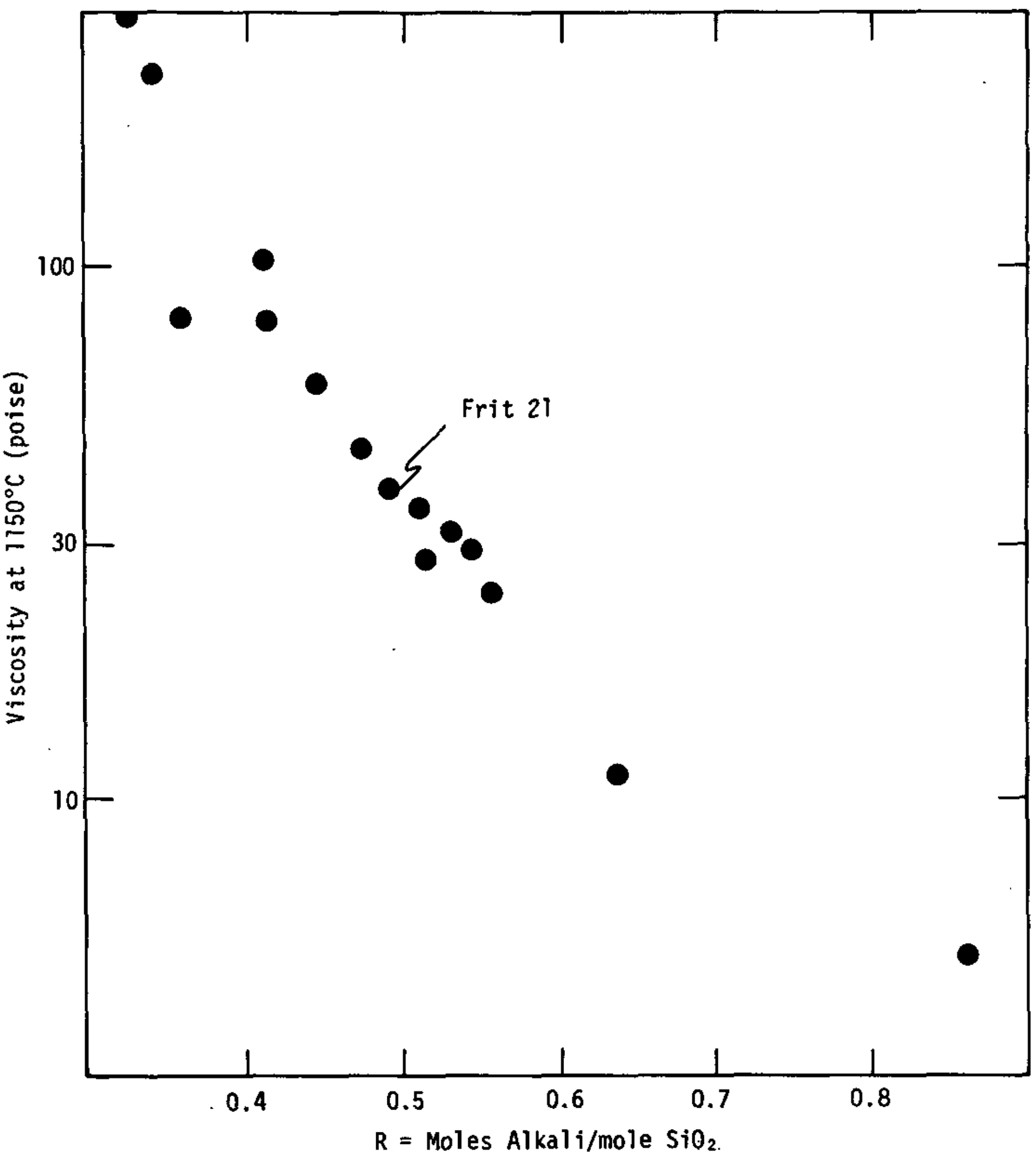

FIGURE 3. Effect of Alkali/silica Ration on Viscosity at 1150°C. All Glasses Contain 25 wt % Composite Sludge.

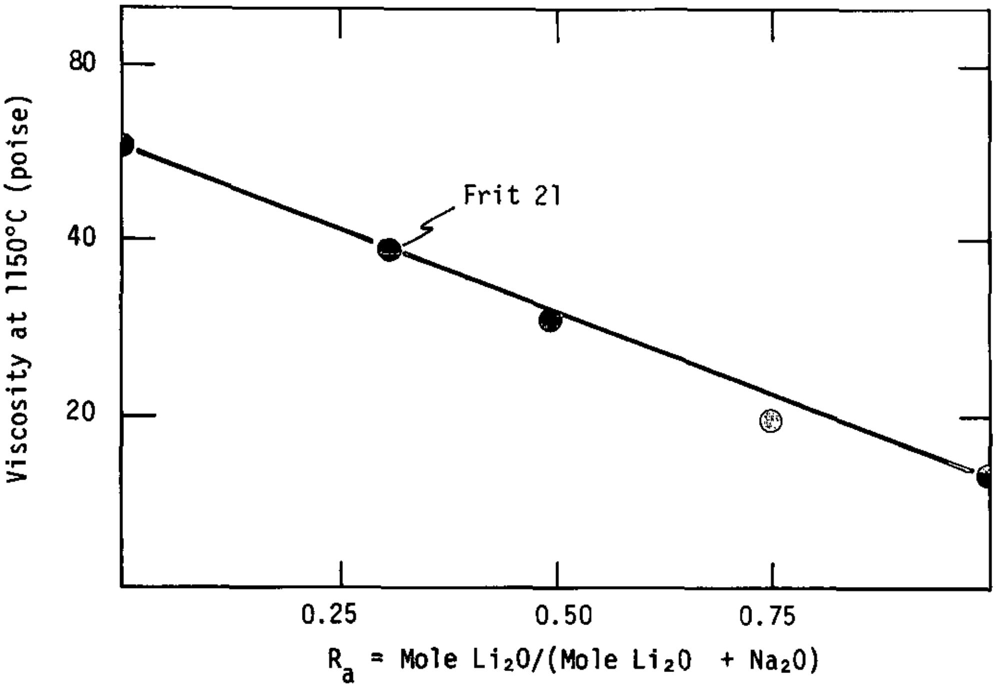

FIGURE 4. Effect of $Li_2O/(Li_2O + Na_2O)$ Ratio on Viscosity at 1150°C. For All Glasses, R = 0.494.

Effects of TiO_2

Melts were prepared containing 0-11 wt % TiO_2. In general, TiO_2 slightly decreased the viscosity in the melting range (∿1150°C). The amount of ferrite observed was reduced by lower TiO_2 content, as was the extent of devitrification to the $LiAlSiO_4$ phase. Eliminating TiO_2 from the frit allows frits with higher values of Ra to be used.

At 1150°C, the viscosities of melts containing all frit components with the same weight proportions as Frit 21 is plotted as a function of TiO_2 content in Figure 5. The composition of the melts is in Table 4. Although there is fairly extensive scatter in the data, there is a significant decrease in viscosity with increasing TiO_2 content. Microscopic examination showed that the glasses became more homogeneous, as the TiO_2 content decreased.

As shown previously,[8] the presence of large amounts of crystalline material can cause glass melts to become non-Newtonian. Glasses which devitrify become non-Newtonian when the amount of crystalline material exceeds ∿3 volume %. If two glasses have essentially the same glassy phase and devitrify to the same crystalline phase, the temperature at which the melts first display non-Newtonian behavior indicates the devitrification rate. The higher this temperature, the greater is the tendency of the glass to devitrify. Melts were made from frits containing only Li_2O, SiO_2, B_2O_3, and CaO. Composite sludge and 0, 5, or 10 wt % TiO_2 (see Table 4) were incorporated into the melts. Melt viscosities were measured as a function of temperature and shear rate. Table 5 lists the temperature at which the measured viscosity began to depend on the rate of shear, i.e. where significant non-Newtonian behavior was first observed. The melt/viscosity at 1150°C is also listed.

All three glasses devitrified significantly. Thus, a glass containing only Li_2O (no Na_2O) probably could not be used as a frit for solidifying SRP waste. However, the data also show that as the TiO_2 content decreases, the devitrification rate decreases. Examination of the glasses after cooling confirmed that the extent of devitrification depended directly on the TiO_2 concentration. This indicated that glasses with higher proportions of Li_2O to total alkali (larger R_a) than Frit 21 might be used for waste solidification if TiO_2 were omitted.

TABLE 4

Composition[a] and Viscosity of Frits with Varying TiO_2 Content

Composition (relative weight units)						*Viscosity (poise) at 1150°C*
SiO_2	*B_2O_3*	*TiO_2*	*Na_2O*	*Li_2O*	*CaO*	
51.9	9.9	11.0	18.3	4.0	4.9	36.6
52.5	10.0	10.0	18.5	4.0	5.0	38.3
54.0	10.3	7.5	19.0	4.1	5.1	44.0
55.4	10.6	5.0	19.5	4.2	5.3	37.6
56.9	10.8	2.5	20.0	4.3	5.4	46.2
58.3	11.1	-	20.6	4.4	5.6	42.9
52.5	10.0	10.0	-	12.9	5.0	15.7
52.5	10.0	5.0	-	12.9	5.0	22.1
52.5	10.0	-	-	12.9	5.0	20.1
52.5	10.0	10.0	11.25	11.25	5.0	11.1
58.3	11.1	-	12.5	12.5	5.6	15.2

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[a] Values listed are in relative weight units. All melts contained 75 wt % frit and 25 wt % composite sludge.

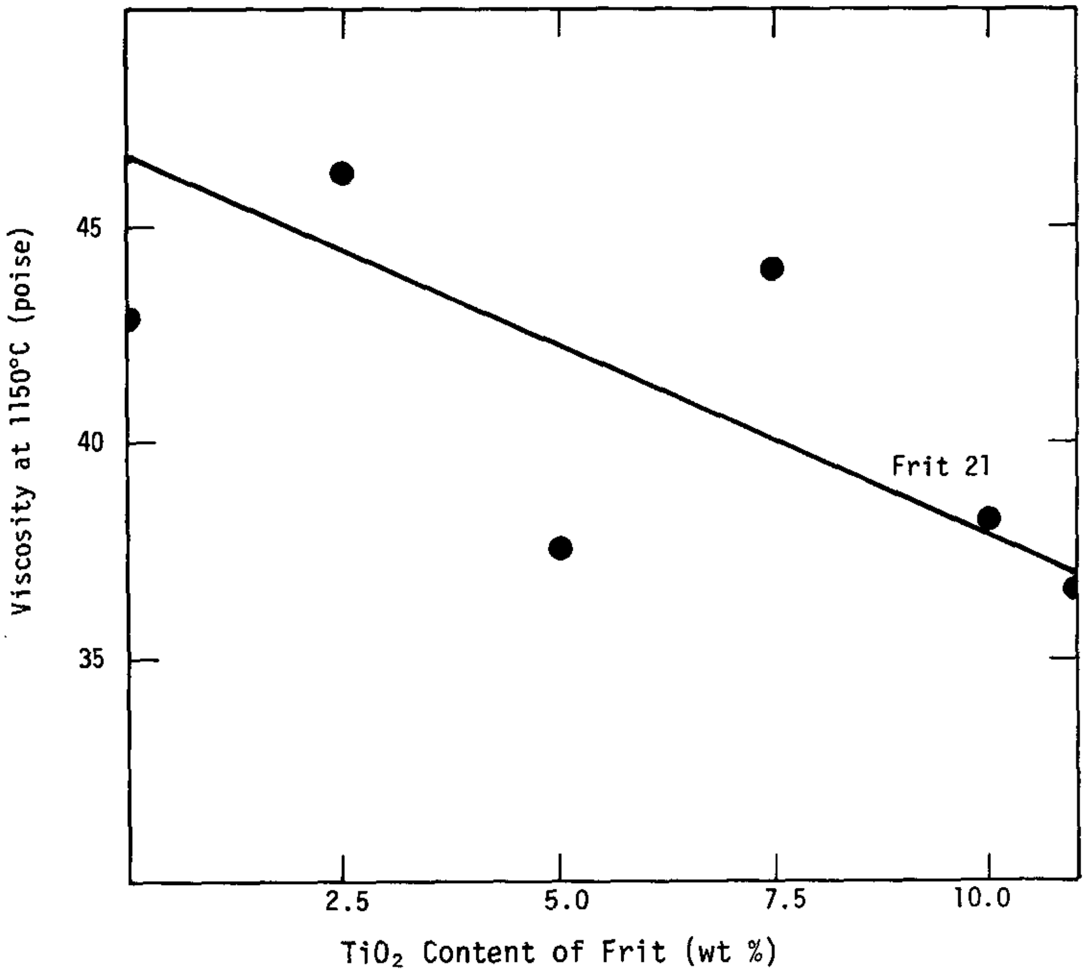

FIGURE 5. Effect of TiO_2 on Viscosity at 1150°C. All Glasses Contain 25 wt % Composite Sludge.

Wirkus and Wilder have demonstrated that TiO_2 increases the solubility of uranium oxides in glass.[13] Experiments were performed to evaluate the effect of TiO_2 on uranium and plutonium solubilities in SRP waste glasses. Small amounts of TiO_2 increased actinide solubility while larger amounts reduced it. For glasses similar to the reference glass, as the TiO_2 content was varied from 0 to 5 to 10 wt %, the amount of uranium which could be dissolved in the glass was 15, 30, and 20 wt % respectively. These solubilities are at least twice that needed for SRP wastes. The situation is similar for plutonium. At least 4 wt % PuO_2 can be dissolved in Frit 21, while only about 2 wt % can be dissolved if TiO_2 is omitted. However, this is at least three times the solubility needed for immobilization of SRP waste.

Effects of Alkaline Earth Oxide

Melts that substituted 5 wt % MgO or BaO for the 5 wt % CaO in Frit 21 were prepared. A melt that contained 2.5 wt % MgO and 2.5 wt % CaO was also prepared. At any temperature above ∿925°C, the viscosity decreased in the order, MgO + CaO melt > CaO melt $\gtrsim$ MgO ≥ BaO. Below 925°C, the BaO melt became non-Newtonian. Subsequent microscopic examination suggested devitrification as the probable cause. Data for these melts are plotted in Figure 6.

Melts in which the CaO content was varied between 0 and 6 wt % were also prepared while changing the other frit components proportionately (by weight). Figure 7 shows, that the glass containing 2.5 wt % CaO was much more viscous than Frit 21, while the other melts were not significantly different from the reference material.

Effects of Boric Oxide

Varying the B_2O_3 content from 5 to 15 wt % of the frit did not seriously affect the viscosity (Figure 8). However, substituting Al_2O_3 for B_2O_3 increased viscosity by up to a factor of 4 at 1150°C. Also, the glasses containing Al_2O_3 were of poorer quality due to extensive devitrification.

TABLE 5

Effect of TiO_2 on $LiAlSiO_4$ Formation

Composition (relative weight units)							*Temp.*	*Amount[a] of*
SiO_2	*B_2O_3*	*TiO_2*	*Na_2O*	*Li_2O*	*CaO*	*η(1150°C)*	*Non-Newtonian(°C)*	*Devitrification After Cooling*
52.5	10.0	10.0	-	12.9	5.0	15.7	1030	Nearly complete (∿75-90 vol %)
52.5	10.0	5.0	-	12.9	5.0	22.1	1000	30-50 vol %
52.5	10.0	-	-	12.9	5.0	20.1	930	5-10 vol %

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[a] Determined by microscopic examination.

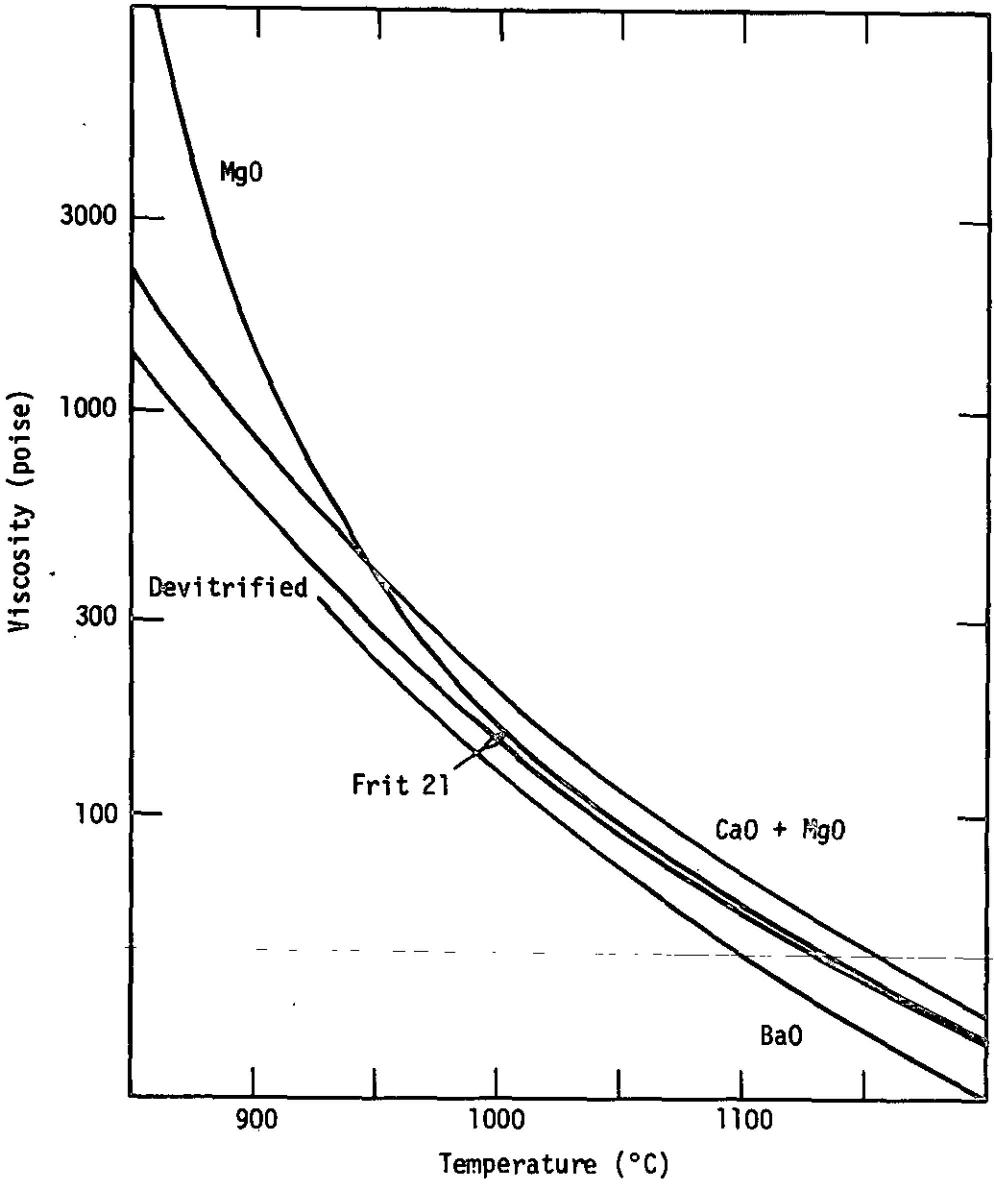

FIGURE 6. Effect of Varying Alkaline Earth Oxide-Viscosity of Composite Sludge-Gass

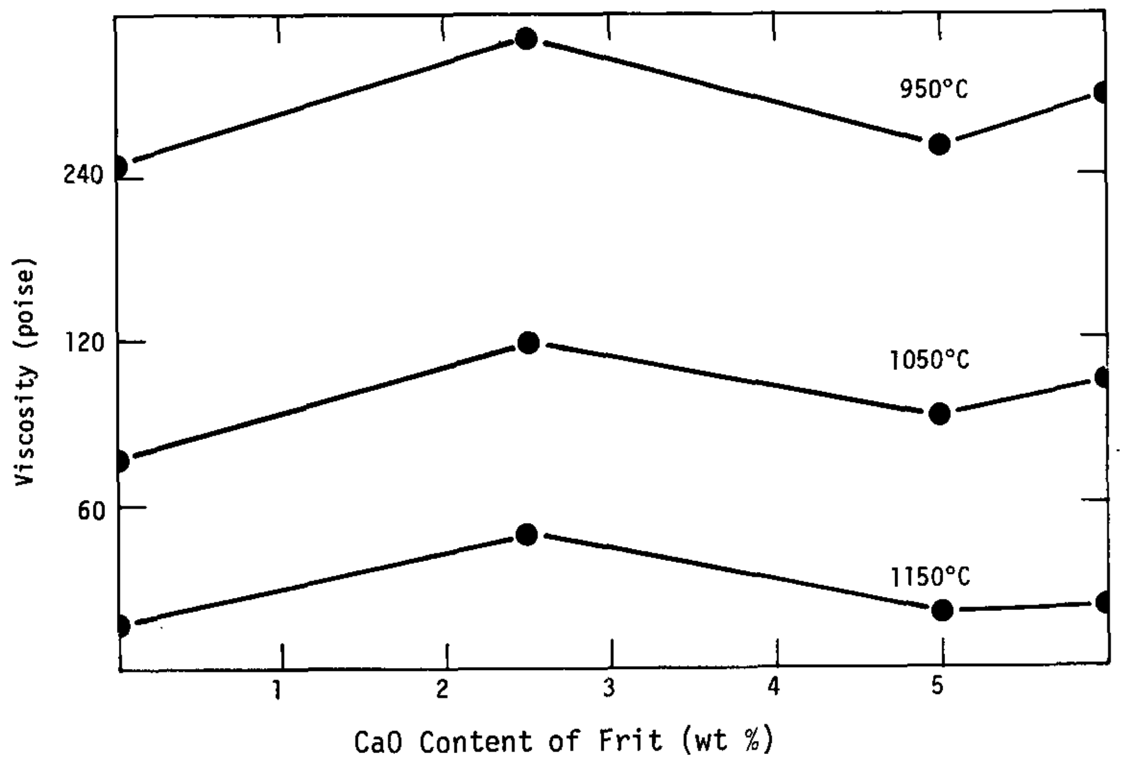

FIGURE 7. Effect of Varying CaO Content of Frit on Viscosity

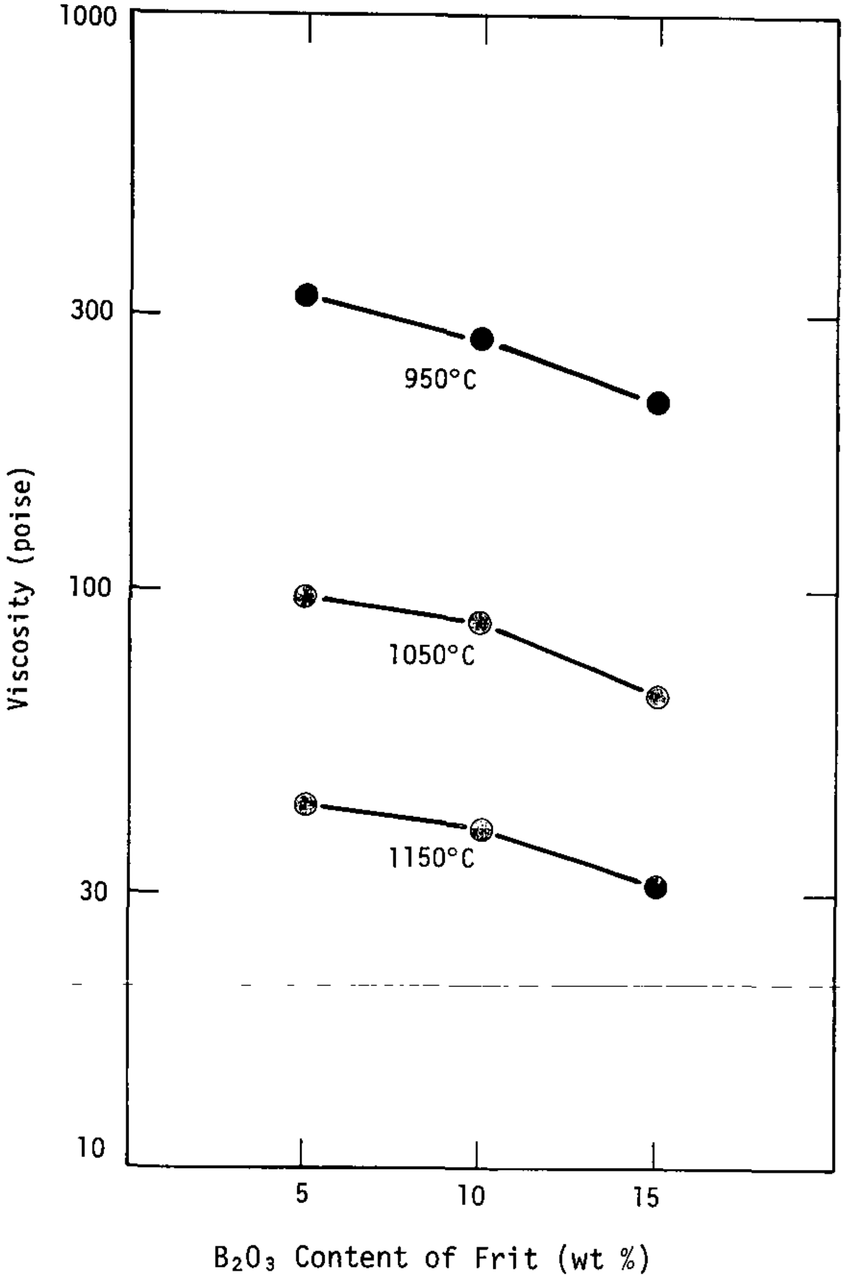

FIGURE 8. Effect of Boric Oxide Content on Viscosity

Effects of Other Additives

Three other additives were tested for possible inclusion in the glass composition - copper, lead, and vanadium oxides. The viscosities of melts containing each were compared to Frit 18. Since each was added principally to reduce viscosity, this also allowed comparison of each with lithium oxide. None was effective as lithium; of the three, V_2O_5 was most effective.

Copper Oxides and Lead Oxides

The form in which copper oxide was added had no effect on the viscosity. Copper oxide addition decreased the viscosity of these melts by less than 20% (Figure 9). Thus, copper oxide is not as effective as lithium oxide. When the equivalent of 4 wt % lead oxide was added to Frit 18 as either $Pb(NO_3)_2$ or PbO_2, viscosities were unaffected (Figure 10). There was virtually no difference in the effects of copper oxide and lead oxides on viscosity or crystalline content.

Vanadium Pentoxide

The viscosity of a melt of Frit 18 was compared to one that included 4 wt % V_2O_5 (Figure 10). V_2O_5 was twice as effective as the other additives in reducing the viscosity, especially at low temperatures. However, the viscosity of the vanadium glass was still ∿2X that of the reference frit (Frit 21) melt, indicating that vanadium is not as effective as lithium oxide.

Improved Frit Composition

Based on the data in the preceding section, an improved frit, designated Frit 411, was selected for more extensive comparison to Frit 21. The composition is listed in Table 4. Frit 411 has several processing advantages over Frit 21:

- Frit 411 sludge melts are much less viscous.
- Frit 411 can dissolve more ferrite-spinel than Frit 21.
- Frit 411 can dissolve more U_3O_8 or PuO_2 than Frit 21.

Its long-term durability and thermal stability must still be tested.

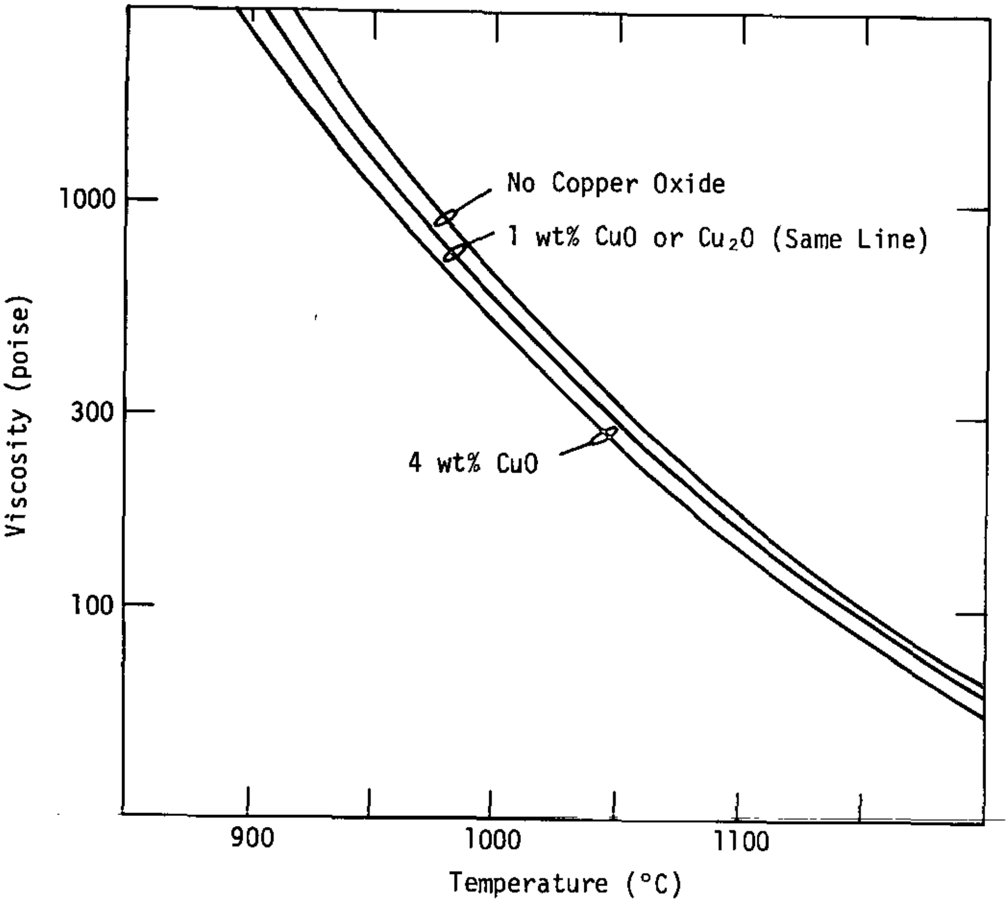

FIGURE 9. Effect of Copper Oxide on Viscosity of Composite Sludge-Glasses

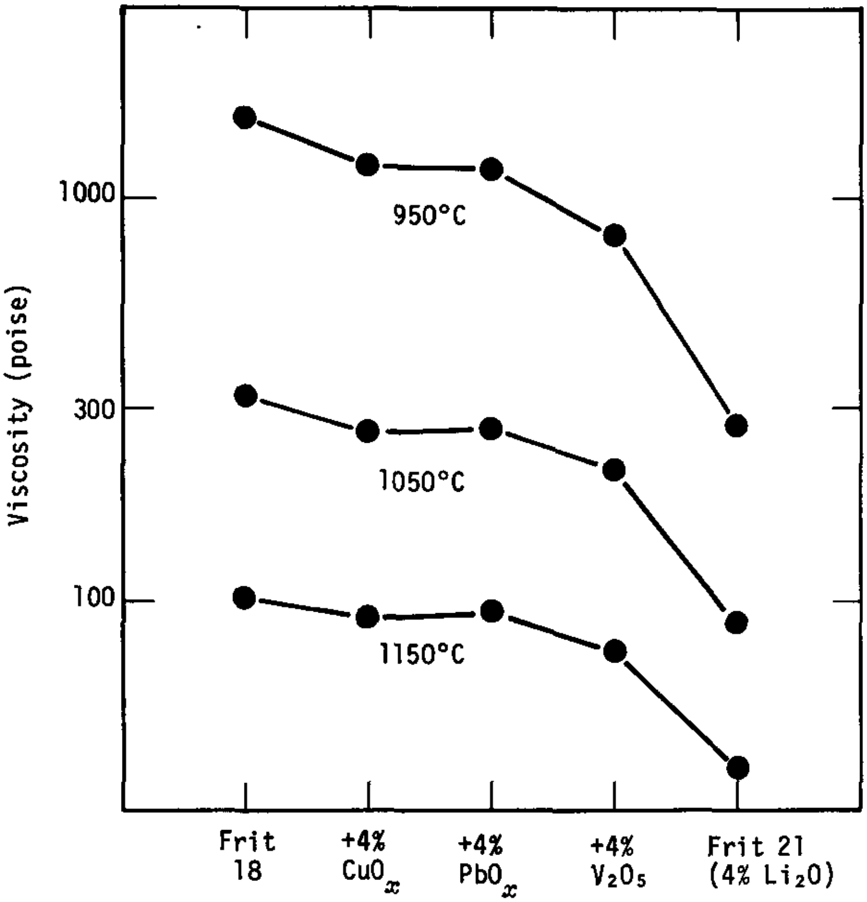

FIGURE 10. Effects of Other Additives on Viscosity of Melts Containing 25 wt % Composite Sludge

Selection of Composition

The most significant factors identified in the previous section were alkali to silica ratio (R), Li_2O to total alkali ratio (R_a), and the level of TiO_2. TiO_2 was originally included in Frit 18 to suppress cesium volatility.[4] Recent work has shown that for full-scale equipment, the effect is marginal.[14] Thus, based on its role in ferrite solubility and promotion of devitrification, TiO_2 was not included in Frit 411.

In a new composition, the ratios R and R_a should be maximized to achieve minimum viscosity. However, devitrification to aluminosilicate practically would limit the alkali concentration. In the presence of TiO_2, this limit was $R_a < 0.6$. Because TiO_2 was not to be included in the frit, higher values of R_a were usable. Figure 3 shows that after R is increased to about 0.64 further increases are less effective in decreasing the viscosity. Thus Frit 411, a modification of composition 41, eliminating TiO_2, was selected for further testing.

Comparison of Frit 411 and Frit 21

Viscosity

The viscosities of melts containing 25 wt % of each sludge type in either frit are shown in Table 6. Values of A, B, and T_0 for the Fulcher equation are listed in Table 7. For a given sludge, the Frit 411 melt will reach the same viscosity ∿100°C lower than the Frit 21 melt. This indicates that Frit 411 melts could be processed at lower temperatures than Frit 21 melts.

Resistivity

The resistivities for Frit 411 melts are lower than for Frit 21 melts (Figure 11). This would require increased current levels to maintain the same power generation at a given temperature. The amount of increase necessary must be determined in large-scale equipment. However, since electrical resistivities are largely controlled by alkali content, further increases in alkali content to reduce viscosity will probably not be possible in frits designed for joule-heated melting.

TABLE 6

Comparison of Viscosities of Frit 21 and Frit 411 Glasses

Sludge[a]	*Viscosity*	*Frit 21*	*Frit 411*
Composite	950°C	273.0	98.6
	1050°C	87.4	33.6
	1150°C	38.1	15.1
High Aluminum	950°C	1240.0	340.0
	1050°C	307.0	117.0
	1150°C	107.0	50.9
High Iron	950°C	108.0	29.6
	1050°C	28.1	12.6
	1150°C	11.1	7.0
Average	950°C	227.0	44.2
	1050°C	58.8	18.5
	1150°C	21.9	9.5

---

[a] All melts contain 25 wt % of the indicated sludge.

TABLE 7

Constants for Fulcher Equation

$\log \eta = A + B/(T-To)$ (η in poise, T in °C)

Frit	*Sludge*[a]	*A*	*B*	*To*
21	Composite	-0.722	1702	411
	High Aluminum	-1.261	2695	331
	High Iron	-1.157	1420	505
	Average	-1.445	2083	402
411	Composite	-1.182	1842	370
	High Aluminum	-1.119	2501	265
	High Iron	-0.496	846	520
	Average	-1.194	1843	301

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[a] All melts contain 25 wt % of the indicated sludge.

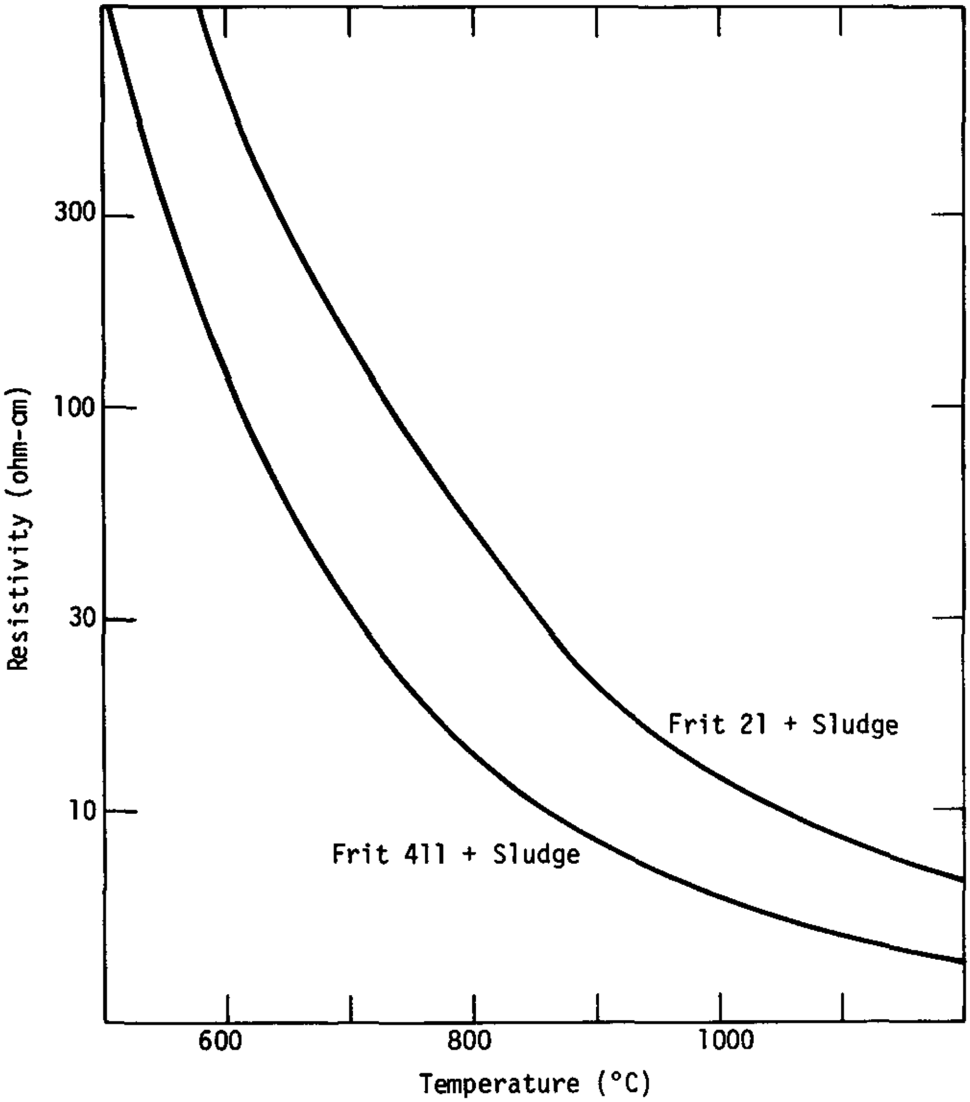

FIGURE 11. Comparison of Resistivities of Glasses Made with Frits 21 or 411. Both Glasses Contain 30 wt % Composite Sludge

Solubility of Ferrite-Spinels

Glasses containing 25 wt % of each sludge type were melted for 3 hrs at 1150°C, then rapidly quenched to room temperature. The glasses and the crucibles used for melting were examined microscopically. Frit 21-sludge melts containing high iron, average and composite sludge all contained large numbers of ferrite-spinel crystals. The only Frit 411 melt to exhibit any crystallinity was that containing high iron sludge which had just two relatively large (∿0.1 mm) crystals adhering to the crucible wall. These were probably nucleated by the crucible itself. To further test solubilities for ferrite-spinel material, the amount of sludge which caused the glass to become non-Newtonian was determined, for high iron and average sludge. For both, ∿5% more sludge was required than for Frit 21 (35 wt % waste oxides in Frit 411, 30 wt % in Frit 21).

Actinide and Ruthenium Solubility

As already noted, Frit 21 can dissolve up to 4 wt % PuO_2. Similar tests with Frit 411 showed that up to 7 wt % PuO_2 could be dissolved. This is ∿8X the amount expected in SRP waste glasses. Ruthenium solubilities in both frits were ∿0.02 wt %. Thus, there is no practical difference between the two compositions for accepting these solutes.

Conclusion

Melts made with Frit 411 are less viscous, have greater solubility for ferrite-spinels and actinides, but have lower ~~resistivities than comparable melts made with Frit 21. However,~~ further testing is needed to determine the long-term thermal stability and leach rate of these glasses. Frit 411 will be tested in large-scale tests at Battelle-Pacific Northwest Laboratories.

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