

Barium borosilicate glass – a potential matrix for immobilization of sulfate bearing high-level radioactive liquid waste

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Abstract

Borosilicate glass formulations adopted worldwide for immobilization of high-level radioactive liquid waste (HLW) is not suitable for sulphate bearing HLW, because of its low solubility in such glass. A suitable glass matrix based on barium borosilicate has been developed for immobilization of sulphate bearing HLW. Various compositions based on different glass formulations were made to examine compatibility with waste oxide with around 10 wt% sulfate content. The vitrified waste product obtained from barium borosilicate glass matrix was extensively evaluated for its characteristic properties like homogeneity, chemical durability, glass transition temperature, thermal conductivity, impact strength, etc. using appropriate techniques. Process parameters like melt viscosity and pour temperature were also determined. It is found that SB-44 glass composition (SiO₂: 30.5 wt%, B₂O₃: 20.0 wt%, Na₂O: 9.5 wt% and BaO: 19.0 wt%) can be safely loaded with 21 wt% waste oxide without any phase separation. The other product qualities of SB-44 waste glass are also found to be on a par with internationally adopted waste glass matrices. This formulation has been successfully implemented in plant scale. © 2006 Elsevier B.V. All rights reserved.

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1. Introduction

Since the advent of nuclear power, the search for suitable technology for treatment of different types

of radioactive wastes generated in the entire nuclear fuel cycle has been continued. Unlike the waste encountered in other industries, radioactive waste assumes special importance and different dimensions, owing to the associated radiation hazard. Many alternatives for their treatment have been investigated and some of them have been applied successfully in the waste treatment plants. However, no method is universally applicable and the

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adoption of any treatment technique for a given type of waste will depend on efficacy of the method, characteristics of the waste, availability of the material and of course economical consideration.

The majority of the radioactivity in the entire nuclear fuel cycle is concentrated in high-level radioactive liquid waste (HLW), which is generated during reprocessing of irradiated nuclear fuels. Borosilicate glass matrices have found wide acceptance in the containment of high-level nuclear wastes [1]. Although the basic network is of silicon and boron oxides, other modifiers are necessary to take into account site specific variations in HLW composition. The presently stored HLW at Bhabha Atomic Research Centre (BARC), Trombay, is characterized by significant concentration of uranium, sodium and sulfate ions in addition to fission products, corrosion products and small amount of actinides. Sulfate in the waste is derived from ferrous sulfamate $[\text{Fe}(\text{NH}_2\text{SO}_3)_2]$ used as reducing agent for conversion of Pu^{4+} to Pu^{3+} at partitioning stage of the actinides during reprocessing and is one of the troublesome constituents with respect to vitrification. Vitrification of sulfate bearing waste is associated with several difficulties. First of all it has been suggested that sulfur could occur in multiple valence state, from -2 to $+6$ within glass depending upon preparation route [2]. However, in borosilicate glass prepared in oxidizing atmosphere, sulfur mostly occurs in $+6$ valence state as SO_4^{2-} [3]. Earlier studies indicated that solubility of sulphate in borosilicate matrix is very poor (≈ 1 wt%) [4]. At higher sulfate concentration, a separate phase of alkali sulphate known as 'gall' is formed. It is a yellow coloured mass predominantly comprising sodium sulfate commonly known as thenardite (Na_2SO_4). Yellow phase being lighter in density than the glass melt floats on the surface of molten vitreous mass in the process pot. This acts as a barrier to the release of the gas bubbles formed during the vitrification process and thus results in swelling of the vitreous mass. The difference in viscosity also creates problem during pouring of molten borosilicate material in canister. In addition to these, yellow phase acts as sink for ^{137}Cs and ^{90}Sr , and high solubility in water may provide easy release of these isotopes to human environment. To prevent formation of yellow phase in the glass matrix, researchers have suggested addition of charcoal, sucrose, etc. in the vitreous melts wherein carbon reduces sulphate to sulfite and other reduced state of sulfur [5,6]. However, such addition to

HLW in concentrated nitric acid medium is undesirable because of the vigor nitrate reduction involved and requirement for excessive amounts of reductant.

An alternative route to this is to identify suitable glass matrix having higher solubility for sulfate anion. Sodium borosilicate glass is not of much use in this case as addition of sulfate rich waste in such matrices results in formation of yellow phase [4]. To identify physically and chemically durable sulfate phase for immobilization of HLW, reference was made to the chemical contents of sulphate deposits in nature. Thermodynamically barium sulfate is indeed one of the most stable and chemically durable phases. References are also made to the reported phase diagrams of alkaline earth borosilicate systems. It is noted that among the various $\text{RO}-\text{B}_2\text{O}_3-\text{SiO}_2$ ($\text{R} =$ alkaline earth elements) ternary systems, $\text{BaO}-\text{B}_2\text{O}_3-\text{SiO}_2$ shows the minimum immiscibility region [7]. The detailed literature survey on natural sulphate deposits indicates that barite (BaSO_4) is one of the most chemically durable and thermally stable phases. Barite bearing litho units as old as ~ 3500 million years have been reported [8]. Consideration of these reported facts prompted present authors to investigate into quaternary $\text{BaO}-\text{Na}_2\text{O}-\text{B}_2\text{O}_3-\text{SiO}_2$ system. Na_2O component was chosen to decrease the pouring temperature of the glass. As a part of this work, maximum waste (sulfate rich HLW) loading capacity of barium borosilicate glass matrix was assessed. The investigation was carried out in two main stages: (a) identification of suitable matrix composition and (b) detailed physico-chemical characterization of the promising compositions. Simulated waste used in this investigation represents composition close to the HLW to be processed. Physical and chemical characterizations of vitrified waste product samples have been done using various analytical tools like optical microscope, X-ray diffractometer (XRD), Electron Probe Micro-Analyzer (EPMA), Differential Thermal Analyzer (DTA), Thermal Conductivity Meter, Flame Photometer (FM), Inductively Coupled Plasma-Atomic Emission Spectrometer (ICP-AES) and viscometer.

2. Selection of glass compositions

Homogeneity, chemical durability and pouring temperature are the three most important criteria used for the matrix composition selection. The first

two can be considered as product durability factors whereas the last one is processing parameter related to the plant application. The type of furnace and material of construction used for the furnace advocates the glass pouring temperature. For induction heated metallic melters, the safe operating temperature is limited up to $\sim 1100^\circ\text{C}$ beyond which temperature dependant mechanical deformation becomes significant. In the present case, 950°C was selected as the maximum allowable pouring temperature for base glass and 925°C for waste glass to improve the usable life of the process pot in view of enhanced corrosion with temperature.

As indicated earlier, $\text{BaO-Na}_2\text{O-B}_2\text{O}_3\text{-SiO}_2$ quaternary was used as the basic system for the matrix composition selection. Glass made up from this system is referred here as base glass. Before selecting the base glass compositions, relevant ternary phase diagrams i.e. $\text{BaO-B}_2\text{O}_3\text{-SiO}_2$, $\text{Na}_2\text{O-B}_2\text{O}_3\text{-SiO}_2$, $\text{BaO-Na}_2\text{O-SiO}_2$ have been studied [9–11]. Data related to the glass forming regions were collected for each of these systems. These data were compared and a small glass-forming region, which was common to all these diagrams, was outlined. High SiO_2 and Na_2O compositions were avoided as SiO_2 increases pouring temperature and Na_2O decreases leach resistance. Hence compositions falling within central part of the quaternary volume were selected. Thus base glass compositions in the range of $\sim 35\text{--}40$ wt% of SiO_2 , $\sim 20\text{--}25$ wt% B_2O_3 , $2\text{--}15$ wt% Na_2O and $2\text{--}25$ wt% BaO were selected in accordance with glass forming region. These base glasses were loaded with $15\text{--}30$ wt% waste oxides to obtain waste glass. Representative compositions of the various glasses prepared are given in Table 1.

3. Selection of simulated waste compositions

High radioactivity associated with actual HLW is the main barrier for its laboratory scale usage. However, for any meaningful study on waste matrix development, chemical complexity of HLW has to be retained. Simulated waste is a compromise between the two, it lacks radioactivity but contain similar chemical composition as that of HLW. In the present case, certain elements, which are present within HLW in trace amounts, such as transuranic elements (Pu, Am, etc.), platinum group of elements (Pd, Rh, etc.), process chemicals (DBP, TBP, etc.) are not added in simulated waste. A chemical composition of the simulated waste is given in Table 2. It is essentially a concentrated acid solution of 1.2 M HNO_3 . Sulfur was added in the form of sodium sulfate and other elements like Ca, Al, Fe, Na, Ni, Cs, Sr, Ce and U were mixed as nitrate salts. Ru was the sole representative of 'platinum group of elements' and

Table 2
Composition of the simulated waste

Additive compounds	Concentration (gm/l)
NaNO_3	105.7900
U-solution	22.3000
$\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	88.9200
$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	42.4200
$\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$	22.4200
$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	3.6472
CrO_3	1.1100
CsNO_3	0.1813
$\text{Sr}(\text{NO}_3)_2$	0.0390
$\text{Ce}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	0.0177
Nd_2O_3	0.0066
RuO_2	0.0024
Na_2SO_4	14.7200

Table 1
Representative chemical compositions (in wt%) of waste glass samples and their corresponding pouring temperatures ($^\circ\text{C}$)

Code	Glass formers $\text{SiO}_2 + \text{B}_2\text{O}_3$	Glass modifiers $\text{Na}_2\text{O} + \text{BaO} + \text{CaO}$	Waste oxide	Pouring temperature ($^\circ\text{C}$)	Swelling index ^a
SB01	48.5	31.5	20.0	925	2.62
SB14	50.0	30.0	20.0	925	2.60
SB15	47.5	32.5	20.0	925	2.72
SB22	54.0	24.0	22.0	925	2.22
SB23	51.0	25.0	24.0	940	2.42
SB44	50.5	28.5	21.0	925	2.56
SB45	50.5	28.0	21.5	925	2.54
SB46	45.0	30.0	25.0	Not pourable even at 975	2.65

^a Swelling index is the ratio of final height to which fused mass rose during the process of calcinations/fusion with respect to initial height of the raw mix.

was included in its oxide form. Cr and Nd were also incorporated as oxides.

4. Experimental

4.1. Preparation of the barium borosilicate matrix

In laboratory scale experiments, glasses are generally prepared in very small amount (~5 g) using Pt–Au crucibles. Extrapolation of data based on this minute amount of glass to plant scale is often found to be difficult. For this reason, in the present case glasses were made in batches of 100 g using fire-clay crucibles. Analytical reagent grade chemicals were used at all stages of experimentation. Preparation routes for base and waste glasses were same, latter one involved only few more steps. The weighed constituents of base glasses (SiO_2 in the form of crystalline (Quartz) silica powder, Na_2O as sodium nitrate, BaO as barium nitrate, B_2O_3 as boric acid) were mixed and slurry was formed. For waste glass, a known amount of simulated waste was mixed with required amount of base glass constituents. The slurry was mixed well and dried under an infrared lamp. Subsequently the mixture was crushed to fine powders using an agate mortar. The powdered mass was then put in fireclay crucibles. The crucibles were heated gradually under static air using a resistance furnace (with a temperature controller to maintain the temperature within $\pm 5^\circ\text{C}$) till the mass attained pourable molten state. At this stage the molten mass was soaked for two hours and then poured directly on clean stainless steel plate maintained at room temperature. Pouring temperatures for all the samples are reported in Table 1 and are found to vary within 925–960 °C. Swelling behavior of the samples was assessed from the difference in heights of the vitrified and calcined mass. Extent of swelling has been expressed as swelling index in Table 1.

4.2. Microstructural characterization

Microstructural characterizations of base glass and waste glass samples were done at various stages. The samples were observed under reflected optical microscope. Crystallinity of the samples was examined using Philips Diffractometer PW 1710. X-ray diffraction (XRD) patterns of the powdered glass samples were recorded from 10° to 70° (2θ) with monochromatized Cu K_α radiation at 40 kV excitation voltage and 30 mA tube current ($K_{\alpha 1} =$

1.5406 \AA and $K_{\alpha 2} = 1.5444 \text{ \AA}$). Electron Probe Micro Analyzer (CAMECA SX 100) was used for assessing chemical homogeneity of the glass samples. An acceleration voltage of 20 kV and 4–100 nA stabilized beam current was used for secondary and back scattered electron imaging and X-ray analysis.

4.3. Physical properties

Besides usual microstructural characterizations, certain physical properties, e.g., glass transition temperature, density, thermal conductivity, impact strength and viscosity were also measured. Differential thermal analyses of the waste glass samples were carried out using SETARAM instrument under inert atmosphere. The heating rate was kept at $10^\circ\text{C}/\text{min}$ and powdered Al_2O_3 was used as the reference material. Density of the glass samples was measured following ‘weight loss method’ using distilled water as the immersing medium. Thermal conductivity was measured by a steady-state axial heat flow comparative apparatus (Thermal Conductivity Instrument model TCFCM, Dynatech R/D Company, Cambridge). In this, the sample was inserted between two identical reference materials having the same diameter as that of sample. An axial heat flow was established through this three element stack by sandwiching it between a heat source and a heat sink and inserting the stack in a guard furnace consisting of four separate heaters. The thermal conductivity of the sample was determined from the thermal conductivity of the reference material and the measured temperature gradients along the sample and reference when steady state was achieved. Impact strength was determined by allowing a known weight to fall on the sample of particular geometry from a fixed height and increase in surface area was measured. Viscosity of the molten glass was measured by Brookfield viscometer.

4.4. Chemical durability assessment

Conventional leach test under total reflux method was used for the assessment of chemical durability of waste glass. The leaching was carried out using a conventional boiling water unit wherein powdered and screened glass samples of $-16 + 25$ BSS ($850 \mu\text{m}$) grain size were exposed to the attack of boiling distilled water. The total reflux conditions avoided the loss of leachant during the tests; water was replaced at different interval of time. The

amount of sodium leached out during each interval was determined using flame photometry. Leach rates are calculated on the basis of sodium released at each interval using the formula given below:

$$\begin{aligned} \text{Leach rate (g cm}^{-2} \text{ day}^{-1}) &= [(\text{Na leached/Na present in sample}) \\ &\quad \times \text{wt of sample}]/\text{surface area (cm}^2) \times \text{time (days)} \end{aligned}$$

Samples were also analyzed for barium, uranium and sulfate to check the leachability of the conditioned product for the above constituents.

5. Results and discussion

5.1. Microstructural characterization

In general it was observed that all base glass formulations with 25 wt% or more waste oxide loading gave rise to yellow phase separation. Most of the waste glass samples (with 15–20 wt% waste oxide), showed homogeneous microstructure. It was noted that addition of PbO as modifier in the borosilicate

matrix did not improve the solubility of sulphate in the matrix. However incorporation of alkaline earth oxides such as BaO and CaO as modifier in borosilicate matrix showed marked improvement in enhancing the solubility of sulphate in the glass thereby yielding a chemically homogenous glass without having any phase separation. The effect of BaO on sulphate solubility in glass matrix was more prominent than CaO. This observation is in line with the finding of other researches also [12]. In the present study, detailed microstructural characterizations of both homogeneous and heterogeneous glass samples have been done to understand the cause of phase separation in the waste glass and to determine the solubility limit of the HLW.

5.1.1. Glass samples

A typical backscattered electron (BSE) image (with atomic number contrast) of the SB-44 glass (Fig. 1(a)) shows that the sample is homogeneous in composition. Si K_α, B K_α and Ba K_α X-ray images taken over 1600 μm² area (Fig. 1(b)–(d)) also indicates that the glass network formers and

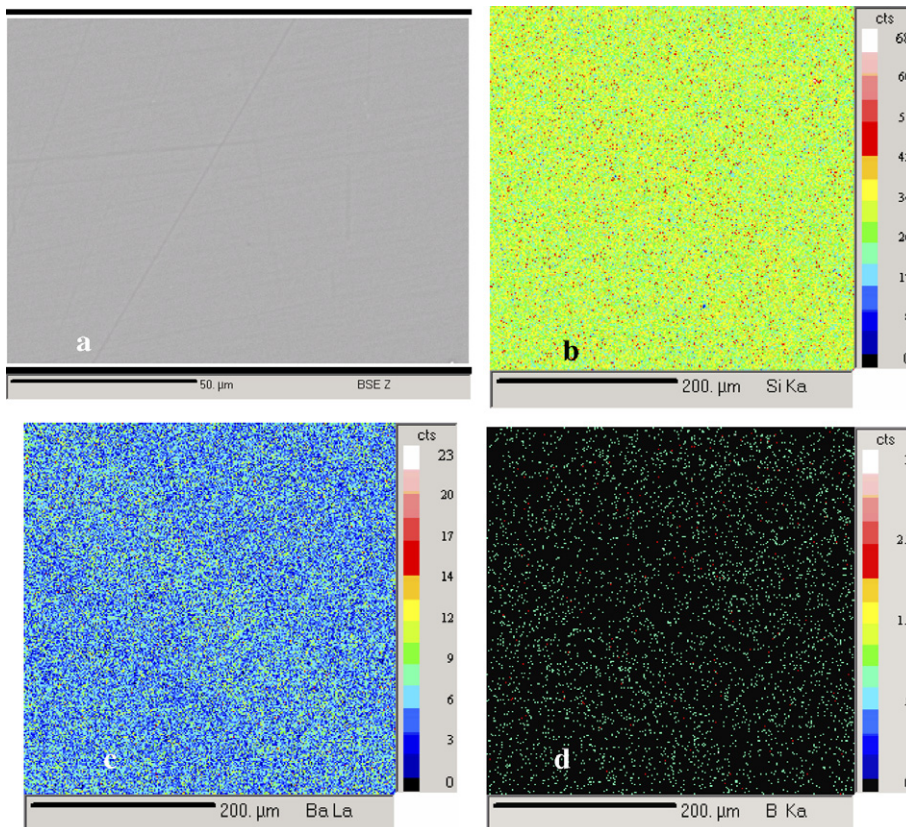


Fig. 1. (a) BSE and X-ray images for (b) Si K_α, (c) B K_α and (d) Ba K_α of SB44 glass.

Table 3
SB-44 glass compositions (wt%) with various waste loading

Code	SiO ₂	B ₂ O ₃	Na ₂ O	BaO	Waste oxide	Pouring temperature (°C)	Homogeneity
SB-44	30.50	20.0	9.50	19.0	21.00	925	Homogeneous
SB-44A	29.73	19.49	9.26	18.52	23.00	925	Homogeneous
SB-44B	28.96	18.98	9.02	18.04	25.00	925	Heterogeneous
SB-44C	28.18	18.48	8.78	17.56	27.00	925	Heterogeneous

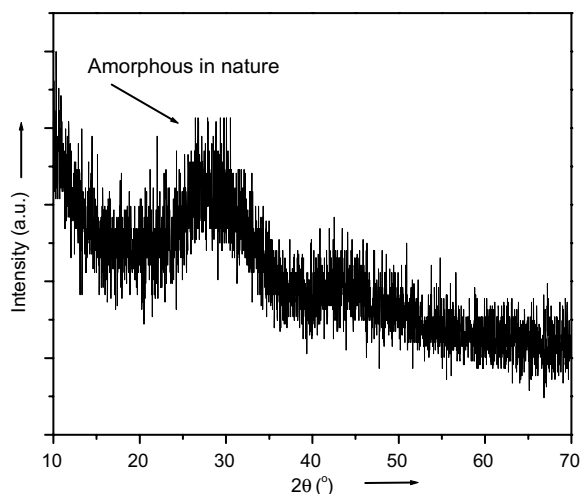


Fig. 2. XRD spectrum of SB44 glass.

modifiers are homogeneously distributed in the matrix. The waste loading of this glass was gradually increased in steps of 1 wt% beginning with 15 wt%, to assess the maximum waste loading possible. The homogeneity and pouring temperature of the glass with different waste loading are given in Table 3. It is seen from the table that up to 23 wt% waste oxide loading, no phase separation takes place in the matrix (Fig. 2). Homogeneity of the glass was checked along the transverse as well as longitudinal sections of the sample and in neither case phase separation could be identified.

5.1.2. Yellow phase

Beyond 23 wt% waste oxide loading, the barium borosilicate glass matrix became heterogeneous owing to separation of yellow phase. A representative part of such yellow phase was scooped out and dissolved in aqueous solution for ICP-AES analysis. Typical elemental composition of the yellow phase is given in Table 4a, which indicates it is essentially a Na-based compound with a trace amount of Ca, Cr, Fe and Ba. Optical (Fig. 3(a)) and BSE (Fig. 3(b)) images of the yellow phase

Table 4a

A typical composition of yellow phase obtained through ICP-AES analysis

Element	Composition (mg/g)
Na	290.00
Ca	4.94
Cr	3.78
Fe	3.57
Al	0.46
U	0.63
Ba	23.76

reveal that it is an aggregate of different phases. Elemental constituents of each of these phases were identified using Wavelength Dispersive X-ray Spectroscopy (WDS) scans in EPMA and are tabulated in Table 4b. Phases 1 and 2 basically contain Ba, Cr, S and O whereas phases 3 and 4 are enriched in Na, Cr, S and O. To understand the elemental distributions across these different phases, X-ray images for Na K_α, S K_α, Cr K_α and Ba L_α X-ray lines were taken. Overlaid X-ray images were prepared by superimposing these X-ray images one above another and are shown in Fig. 4(1)–(4). These images identify four different phases, namely correspond to Na and S bearing, Na and Cr bearing, Ba and Cr bearing and Ba and S bearing compounds. XRD spectrum obtained from the same yellow phase sample is shown in Fig. 5. The XRD spectrum indicates that yellow phase is crystalline in nature. The peaks observed in the XRD spectrum were matched with available standard data files and the presence of Na₂SO₄, BaSO₄, Na₂CrO₄ and BaCrO₄ were confirmed [13–15], however some of the peaks could not be identified. These data corroborated well with those obtained from EPMA study. In an earlier experiment, Schiewer et al. [16] reported the presence of sodium sulfates, sodium chromates, sodium molybdate, calcium molybdate and barium chromate within the yellow phase cluster developed with barium borosilicate glass matrix. Such variations in the yellow phase assemblages

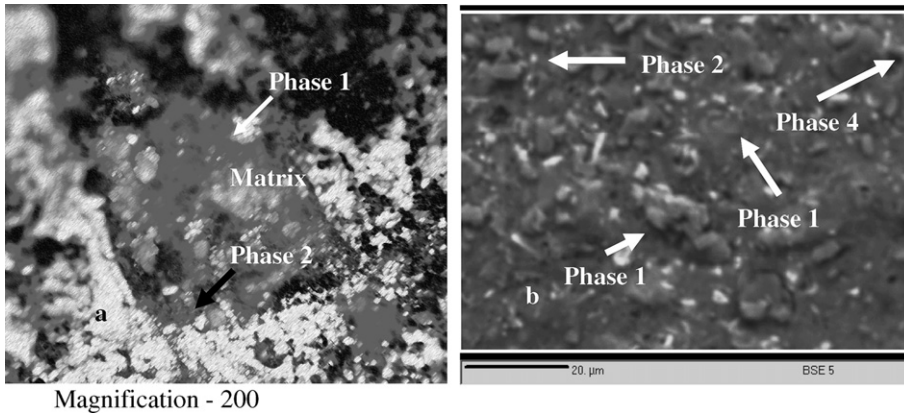


Fig. 3. (a) Optical micrograph and (b) BSE image of yellow phase.

Table 4b
Elemental composition of constituent phases of yellow phase obtained through EPMA analysis

Elements/phases	Phase 1	Phase 2	Phase 3	Phase 4
Major elements	Ba, Cr, O	Ba, S, O	Na, Cr, O	Na, S, O
Minor elements	S	Cr	S	Cr

may be due to the higher content of Mo in their [16] fused mass compared to our glass.

5.2. Physical properties

Physical parameters such as thermal stability, mechanical integrity and radiation stability are

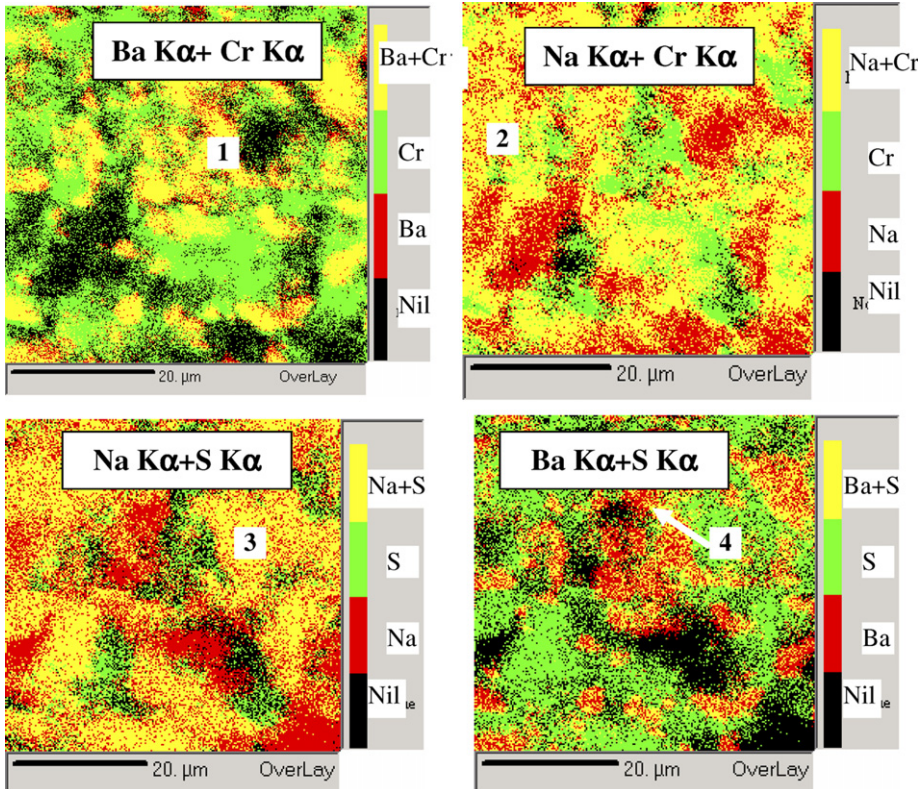


Fig. 4. Overlaid X-ray images showing presence of BaCrO₄ (1), Na₂CrO₄ (2), Na₂SO₄ (3) and BaSO₄ (4).

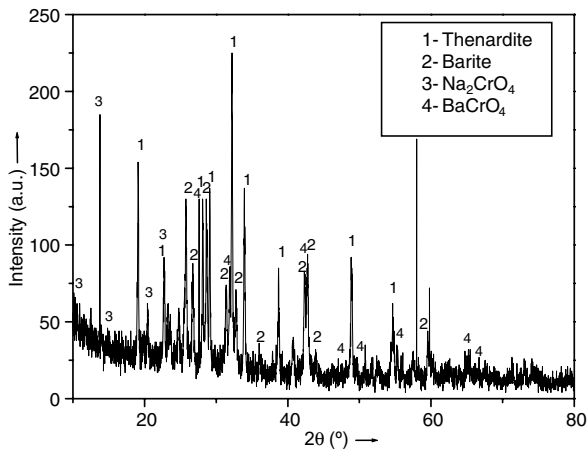


Fig. 5. XRD pattern of separated phase in barium glass (35% W.O.).

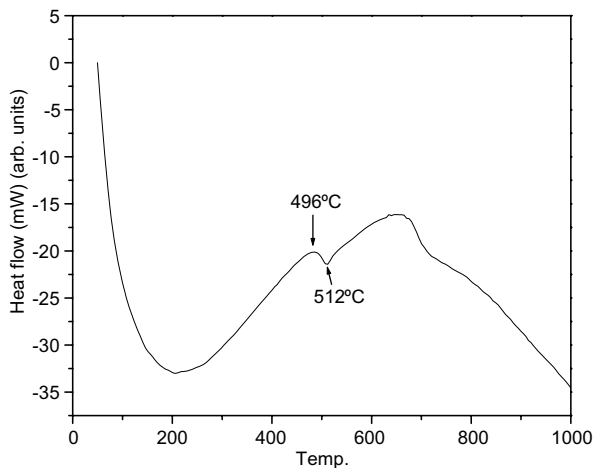


Fig. 6. DTA pattern of SB- 44 glass.

important aspects of waste glass. Thermal stability of the homogenous matrix was studied by DTA technique. DTA pattern of the glass sample is shown in Fig. 6, which is characterized by a broad endothermic peak centered around 512 °C with an onset around 496 °C. The broad endothermic peak has been attributed to the structural relaxation, taking place within the glass matrix and the onset of temperature of this peak (496 °C) corresponds to the glass transition temperature (T_g) of this glass. T_g signifies the temperature below which glassy phase remains intact. This temperature range is safe from the devitrification of glass under repository conditions point of view as the ambient temper-

ature will be less than 496 °C. Estimations show that the centerline temperature of the glass–canister assembly due to decay heat is not expected to reach such a high temperature even if long-term storage is considered and hence the vitrified waste product is safe under repository condition against devitrification.

Density of the present waste glass samples was found to vary within a narrow limit of 3.0–3.2 g/cm³, which is similar to other glasses used for waste disposal [17]. High density of the matrix is probably due to high uranium oxide content within the waste oxides (Table 2). In general, high density is a coveted property for waste glass as it gives flexibility to load higher amount of waste oxides within a given glass matrix volume.

The viscosity of the vitrified waste product at the pouring temperature (925 °C) was found to be 50 dPa s (deci-Pascal second) that is closer to other reported values [17]. Such low viscosity of glass at pouring temperature helps in smooth draining of vitrified waste product in canister.

5.3. Chemical durability

For chemical durability assessment, conventional leach test was carried out with powder sample of SB-44 glass for 700 days. Leaching pattern of the sample is shown in Fig. 7 and the leach rate was found to be 2.32×10^{-6} g cm⁻² day⁻¹ at the end of 700th day. Further leaching experiment with SB-44 pellet is presently going on and the detail experimental results will be discussed elsewhere.

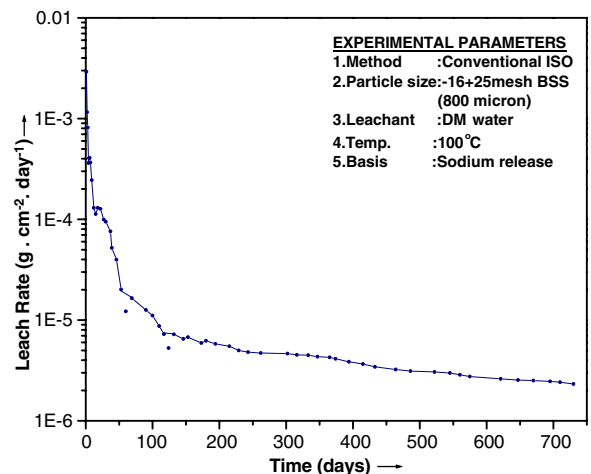


Fig. 7. Leaching pattern of barium borosilicate glass (SB-44).

5.4. Plant scale operation

Based on the encouraging laboratory studies, the composition has been adopted in the plant scale for vitrification of sulfate bearing HLW. About 11 000 kg of vitrified waste products has been successfully made immobilizing about 10^7 GBq of radioactivity.

6. Summary and conclusion

The main aim of the present study was to identify a suitable matrix for sulfate bearing high-level radioactive liquid waste whose stability (physical, chemical, mechanical) is independent of the fluctuations of waste composition. The elaborate studies carried out with barium borosilicate matrix in laboratory scale yielded promising results and these are summarized below:

- (i) Incorporation of alkaline earth oxide like BaO and CaO improve the solubility of sulfate in borosilicate glass matrix. The present study shows that barium borosilicate glass matrix can host up to 23 wt% of waste oxide (of the present composition) homogeneously.
- (ii) Salient features of the SB-44 glass are tabulated in Table 5, which suggests that its prod-

uct quality is reasonably well from nuclear waste disposal point of view and is comparable to internationally adopted glass composition employed for vitrification of HLW [18].

- (iii) Above 23 wt% of waste oxide, yellow phase cluster constituted of four crystalline phases, namely sodium sulfate, sodium chromate, barium sulfate and barium chromate develops within the present glass matrix.
- (iv) Plant scale operation shows sulfate bearing active waste can be loaded within barium borosilicate matrix without yellow phase formation. Hence it can be said that 'barium borosilicate glass' is a potential matrix for the immobilisation of sulfur rich high-level waste.

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Table 5
Salient features of SB-44

Features	Data
Composition (wt%)	SiO ₂ + B ₂ O ₃ 50.5%, Na ₂ O + BaO 28.5% Waste oxides: 21.0
Pouring temperatures (°C)	Base glass: 950, waste glass: 925
Homogeneity	Amorphous and homogeneous
Leach rate (after 700 days leaching)	$\sim 2.32 \times 10^{-6}$ g cm ⁻² day ⁻¹
Glass transition temperature (°C)	496
Viscosity (dPa s)	50 at pouring temperature
Density (g/cm ³)	~ 3.0
Thermal conductivity at 100 °C (W m ⁻¹ °C ⁻¹)	0.92
Impact strength (RIAJ) ^a	0.84
Plant scale operation	<ul style="list-style-type: none"> • Pouring temperature 925 °C • No excessive foaming • No accumulation of yellow phase • No unusual release of waste and glass components in off-gas system • 11 000 kg of VWP prepared safely and successfully

^a RIAJ – Relative increase in area per joule of impacted energy.

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