

## Plasma treatment of air pollution control residues

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## **Abstract**

Air pollution control (APC) residues from waste incineration have been blended with silica and alumina and the mix melted using DC plasma arc technology. The chemical composition of the fully amorphous homogeneous glass formed has been determined. Waste acceptance criteria compliance leach testing demonstrates that the APC residue derived glass releases only trace levels of heavy metals (Pb (0.007 mg/kg) and Zn (0.02 mg/kg)) and Cl<sup>-</sup> (0.2 mg/kg). These are significantly below the limit values for disposal to inert landfill. It is concluded that plasma treatment of APC residues can produce an inert glass that may have potential to be used either in bulk civil engineering applications or in the production of higher value glass-ceramic products.

**Keywords:** APC residues; incineration; DC plasma technology, vitrification, glass

## **1. Introduction**

The incineration of municipal solid waste (MSW) is expected to increase as landfill disposal becomes more problematic. Modern energy from waste (EfW) plants control the release of atmospheric pollutants to below regulatory levels by removing flue gas pollutants using air pollution abatement systems. Pollutants are captured and contained in a fine granular waste known as air pollution control (APC) residues which are produced from dry and semi-dry scrubber systems. These involve the injection of an alkaline powder or slurry to remove acid gases, particulates and flue gas condensation/reaction products. Fabric filters in bag houses are used downstream from the scrubber systems to remove the fine particulates. APC residues also include the solid phase generated by wet scrubber systems (scrubber sludge) and the production could be minimised through this method. APC residues are regulated in the UK by the Environment Agency under the hazardous waste regulation act as hazardous waste, as they have an absolute entry in the European Waste Catalogue (19 01 07\*). This is primarily because of high alkalinity (> pH 12) due to the use of excess lime, although they also contain volatile heavy metals, soluble chlorides and sulphate salts, and organic contaminants including dioxins and furans ((EC, 1999; DEFRA, 2005a; ESA, 2005; WRc Ref : 11873-0, 2004)

Waste acceptance criteria (WAC) for hazardous and inert landfill sites came into force in the EU in July 2005, with the end of co-disposal, and set leaching limit values that must be achieved if a particular pretreated waste is to be acceptable for landfill disposal. These are very difficult for APC residues with current compositions to achieve, primarily because of high levels of soluble and therefore leachable chloride salts that primarily originate from polyvinyl chloride (PVC) found in MSW.

The safe and effective management of APC residues from waste incineration has become an problematic issue for the EfW industry. APC residues require some form of physico-chemical or thermal treatment before either disposal or reuse/recycling and a variety of technologies have been developed that may provide sustainable APC residue treatment (Amutha Rani et al., 2006).

In thermal vitrification using plasma, hazardous wastes are melted at temperatures above 1400°C, and through vitrification and solidification, leachable compounds are stabilised in the amorphous microstructure of the product. Vitrification of waste materials and the production of glass-based products have previously been reported (Colombo et al., 2003; Rawlings et al., 2006). Studies on the vitrification of mixed medical wastes, incinerator fly ash and radioactive wastes have shown that plasma is a promising technology for hazardous waste treatment (Chu et al., 1998; 2002; Tzeng et al., 1998; Sakai and Hiraoka, 2000). Although plasma technology and other thermal treatments have been used to vitrify incinerator bottom ash (Barbieri et al., 1998; 2000a; Bethani et al., 2002) and incinerator fly ash (Boccaccini et al 1996; 2000; Rincon et al., 1999; Cheng et al., 2002; 2004), there has been no reported work on plasma treatment of APC residues. Therefore the objective of this study was to investigate the viability of using DC plasma arc technology for the thermal treatment of APC residues and to characterise the glassy material produced in terms of composition and leaching behaviour.

## **2. Materials and methods**

### *2.1 APC residue characterisation*

APC residues used in this work were from a major EfW plant in London UK, which processes approximately 420,000 tonnes of MSW per year. These are produced

from cleaning the gaseous emissions generated during combustion of wastes at EfW plants. Typical composition data for this material obtained by aqua regia total metals/water soluble ions is shown in Table 1 (4). The physical properties and chemical compositions vary depending on waste composition, type of plant and the air pollution control system. The high CaO content is due to excess lime used in the scrubbing process, while the high levels of chloride originate primarily from the significant volumes of polyvinyl chloride (PVC) found in MSW.

Scanning electron microscopy (JEOL-JSM-840A) of the as-received APC residues is shown in Figure 1. This shows agglomerated spherical particles with a mean particle size determined by laser diffraction particle size analysis of approximately 25 $\mu$ m (Malvern Instruments, Mastersizer). Crystalline phase analysis of as-received APC residues was carried out by X-ray diffraction (XRD, Philips PW1700 series) using Cu K $\alpha$  radiation with a secondary graphite crystal as mono-chromator. Thermal analysis was completed on as-received APC residues (Stanton Redcroft Thermal Analyser STA-780 series) in an argon atmosphere at a heating rate of 10 $^{\circ}$ C/min up to 1200 $^{\circ}$ C in an alumina crucible with alumina powder as reference.

## 2.2 *Batch composition*

Silica and alumina were added because the as-received APC residues do not contain sufficient glass formers to produce a single-phase amorphous glass. The material fed to the plasma furnace consisted of 69.8 wt% APC residues, 21.9 wt% SiO<sub>2</sub> (supplied by TJ Sansum, UK, 99% purity, < 1 mm grain size) and 8.3 wt% Al<sub>2</sub>O<sub>3</sub> (added as Chinese bauxite, supplied by Ryder Point Processing, UK, 3-5 mm grain size). The bulk oxide input composition has been chosen to be within the anorthite region of the Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>-CaO phase diagram.

## 2.3 *Plasma furnace operation*

The plasma system consists of a DC hollow graphite cathode installed through the roof of a furnace and supported by a vertical manipulator column. Argon is injected down to the centre of the cathode to produce a stable plasma arc that is transferred to the furnace melt. The anode consists of conductive elements built into the furnace hearth, and the process is capable of handling a wide range of ashes of varying particle size and chemical composition. The furnace operates under controlled argon rich conditions and this experiment was run at approximately 1600°C. The electrical power supply to the plasma furnace is dependant on capacity, but is usually of the 100's kW order and is controlled independently of other process variables. Remote water cooled elements are employed at the melt line to form a protective frozen layer, ensuring good refractory performance (Tetronics).

A flow diagram of the prototype plasma treatment system used in this work is shown schematically in Figure 3. After preheating for 2.5 hours, the feed was introduced into the furnace at a rate of approximately 50 kg/hour for 4.5 hours. Pig iron was added to the furnace to form a melt pool as a return electrode and initial reaction zone. The APC residues blended with silica and alumina were metered at a controlled rate to the furnace and the plasma power was modulated to maintain the specific energy input, and therefore temperature at around 1600°C. A layer of untreated feed is maintained on top of the molten slag, where gasification reactions occur. The feed is rapidly melted and the molten slag phase is periodically tapped, where it is cast into APC residue derived glass. Exhaust gases exiting the unit were treated in a thermal oxidiser unit to fully oxidise residual combustible gas species (CO<sub>2</sub>, H<sub>2</sub>). The particulates are removed in a bag house filter and acid gases are removed by the wet scrubber prior to venting to atmosphere.

#### *2.4 Characterisation of the APC residue derived glass*

XRD was used to determine the bulk phase analysis of the APC residue derived glass. The glass was crushed to powder and both the bulk and powder were thermally analysed (STA-780 thermal analyser). Scanning electron microscopy (JEOL-JSM-840A) combined with energy dispersive x-ray spectrometry (EDS) was used to analyse the chemical composition of the glass.

Leaching from APC residue derived glass was evaluated using the EU compliance leaching test for granular waste with a particle size less than 10 mm using a liquid to solid ratio (L/S) of 10 l kg<sup>-1</sup> and water as the leachant (BS EN 12457-4). The APC residue derived glass was crushed to give the required particle size. Elemental analysis of the leachates was completed using inductively coupled plasma atomic emission spectroscopy (ICP-AES) and the Cl<sup>-</sup> in leachates was determined by AgNO<sub>3</sub> titration method standardised against the blank sample (AWWA, 2005).

### *2.5 Characterisation of secondary residues*

In addition to characterising the plasma treated APC residues derived glass, phase analysis was also completed by XRD on the secondary residues collected in the bag house filter (secondary residues in Figure 3) (Philips PW 1700 X-ray diffractometer by varying the scan angle ( $2\theta$ ) between 5 and 60°).

## **3. Results**

### *3.1 APC residue characterisation*

XRD data for the as-received APC residues is shown in Figure 4. The major Ca containing phases are Ca(OH)<sub>2</sub>, CaCO<sub>3</sub> and CaClOH. The major soluble salts identified were NaCl and KCl. Table 1 shows that approximately 16 wt% of the APC residues is present as chloride salts. These compounds are non-hazardous but are highly volatile at temperatures above 1200°C as experienced during plasma treatment.

DTA data of the as-received APC residues is shown in Figure 5. Minor peaks observed at temperatures between 100 and 140°C are due to loss of H<sub>2</sub>O. The endothermic peak at 420°C is due to decomposition of Ca(OH)<sub>2</sub> and the sharp endothermic peak at 500°C is attributed to eutectic melting of an equimolar mixture of CaCl<sub>2</sub> and NaCl and the destruction of CaOHCl (Geysen et al.,2006; Castrillejo et al., 2003). The third endothermic peak at 675°C is caused by decomposition of CaCO<sub>3</sub>. The broad endothermic peak around 1000°C is associated with a weight loss of 30% and is believed to be due to the melting and evaporation of soluble Na, K and Ca containing chloride salts.

### 3.2 Plasma treatment

Figure 6 shows the plasma power during the APC residue vitrification trial. After preheating, the plasma furnace achieves steady state and the total heat loss was calculated to be 90 kW. Thermodynamic modelling predicted a required power input of 145 kW to maintain the slag at 1600°C for the feed rate of 50 kg/hr. This is in good agreement with recorded mean input power of 135 kW which correlates to a mean input current of 1070 A and a mean voltage of 126 V. A total of 220 kg of blended feed was fed over a period of 4.5 hours with a flow of 30 l/min of Ar gas. Additionally, 120 l/min of O<sub>2</sub> was introduced to ensure complete oxidation of residual carbon. The theoretical energy requirement to vitrify APC at 1600°C is typically 700 kWh/tonne.

Table 2 summarises the air emissions data recorded during the prototype scale APC residue vitrification trial. The measured emissions of pollutants, except VOC (volatile organic compounds) and CO (carbon monoxide) that fractionally exceeded the emission limit values (ELVs), were below the ELVs specified in the waste incineration directive (WID) (DEFRA, 2005b). The high levels of VOC and CO occur due to the limitation of the off-gas abatement system. Average values are presented, and transient



operational difficulties were experienced, which compromised plant performance. The burner of the combustion chamber also operated intermittently, resulting in the thermal oxidiser only operating at a maximum temperature of 400-500°C. It is impossible for the emission reported to result from waste, due to the low level of TC and TOC present within the source APC.

### 3.3 Characterisation of plasma treated APC residue derived glassy slag

Plasma treatment of APC residues blended with SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> resulted in significant volume reduction of about 70-75%. The resulting glass is shown in Figure 7. XRD data given in Figure 8 shows a broad hump around 30° indicating that the APC derived glass is amorphous and that crystalline phases have not been formed.

Figure 9 shows DTA data for the APC residue derived glass and a powdered sample of the same material. Both curves exhibit a similar glass transition temperature T<sub>g</sub> at around 760°C. The curve for the glass powder shows an exothermic peak at 1040°C (T<sub>x</sub>; crystallisation temperature) followed by an endothermic peak at 1160°C (T<sub>m</sub>; melting point). The bulk sample does not exhibit distinct crystallisation peaks.

In a multicomponent system, the glass forming ability (GFA) depends on certain parameters such as T<sub>g</sub>, T<sub>x</sub> and T<sub>m</sub> which are correlated to the precursor composition. The GFA can be calculated from the following equation (Saad and Poulain. 1987).

$$GFA = \frac{(T_m - T_x)(T_x - T_g)}{T_g} \quad (1)$$

The GFA obtained in the present study through DTA analysis was 44.20. This is comparable to the value of 42.66 obtained for a 70 wt% fly ash, 30 wt% SiO<sub>2</sub> mix and confirmed the high ability of glass formation (Park and Heo, 2002). The GFA calculations will be extended in future studies to compare the ability of glass formation from samples with different initial compositions. The glass obtained from plasma

processing is believed to be highly stable as the crystallisation exotherm ( $T_x$ ) and melting peak ( $T_m$ ) are close together and exhibit a broad exotherm (Park and Heo, 2002).

Elemental analysis of the glass by EDS analysis is presented in Figure 10. The major elemental components are O (40.0 wt %), Ca (24.0 wt %), Si (19.2 wt %), Al (10.7 wt %), Fe (2.6 wt %), Mg (1.1 wt %), Cl (2.0 wt %) and Ti (0.7 wt %). These results were obtained from calculating the average from approximately 10 analyses carried out on different parts of the APC derived glass.

Compliance leaching test results for the APC derived glass is given in Table 3. The concentrations of heavy metals in leachates were low, with Pb at 0.007 and Zn at 0.020 mg/kg. While the original APC residues typically leach 140,000 to 170,000 mg/kg Cl<sup>-</sup> the APC derived glass leached only 0.2 mg/kg. Table 3 includes the inert landfill WAC and demonstrated that plasma treated APC residues are suitable for disposal to inert waste landfill.

#### *3.4 Characterisation of the secondary residues*

Figure 11 shows XRD data of the secondary residues collected by the baghouse filter during plasma treatment of APC residues. This material contains crystalline phases of silica, calcium silicate, calcium aluminate and Fe<sub>3</sub>Si. Cl<sup>-</sup> containing phases were not detected in the secondary residues. This suggests that low density free flowing Ca containing APC residue particles present in the plasma furnace chamber are collected in the bag house filter due to the process of physical carryover.

## **4. Discussion**

DC plasma arc technology is an innovative thermal waste treatment technique, which is very effective for vitrification of APC residues. Rapid cooling and

solidification of the APC residue melt results in a solidified product of amorphous glass consisting primarily of Ca, Si and Al compounds. The properties of the APC derived glass depend on the input composition and operation mode of the plasma rig. As-received APC residues will not form a glass without the addition of other glass forming additives and therefore SiO<sub>2</sub> (21.9 wt%) and Al<sub>2</sub>O<sub>3</sub> (8.3 wt%) were added to the plasma input feed in order to control the properties of the glass product.

The composition of APC residues with the glass forming additives and the composition of the glass product are shown in Table 4. Heavy metals and soluble salts in the vitrified matrix were below the detection limit of EDS analysis. Therefore it is concluded that a significant amount of heavy metals (Pb, Zn, and Cd), soluble salts and Cl<sup>-</sup> in the APC residues volatilised during plasma treatment. The bulk calcium-aluminosilicate glass only shows the presence of low amounts of transition metals such as Fe (2.6 wt%), Ti (0.7 wt%) and 2.0 wt% Cl<sup>-</sup>. The increased amounts of Fe, Ti and Mg are possibly arise from the Fe molten bath which acts as the initial reaction zone during plasma processing. The APC residue treatment exploits the ability of plasma to rapidly initiate a variety of chemical reactions including reduction, decomposition, evaporation and pyrolysis, with oxidation reactions occurring during cooling (Katou et al., 2001).

Compliance leach test results for the APC derived glass demonstrated that only trace amounts of Pb (<0.007 mg/kg) and Zn (0.02 mg/kg) were leached and that these were well below the inert waste landfill WAC leach limits. Analysis of the secondary residues indicates the presence of calcium and iron rich silicate phases. APC residues are thoroughly melted at the high temperatures experienced during plasma treatment and the levels of hazardous elements present in the vitrified product and the secondary residues are below EDS detection limits. Further plasma vitrification trials including

analysis of the secondary wet scrubber residues are being completed to further understand the fate of heavy metals and Cl<sup>-</sup> during the process.

The low emissions resulting during the plasma vitrification process indicate that the present technique can be used as an alternative to other waste treatment technologies. The glass produced from the plasma vitrification process has the potential to be converted into glass-ceramics. The most appropriate methods for achieving glass ceramics is by either sintering or controlled cooling and therefore the crystallisation behaviour of the APC derived glass was studied by thermal analysis for both the powdered and bulk glass samples. The powdered glass exhibited distinct crystallisation peaks whereas the bulk glass showed only a shallow broad endothermic peak. This indicated that surface crystallisation is the predominant mechanism for the devitrification of the vitrified glass (Hernandez-crespo et al., 2006). From thermal analysis, the  $T_g/T_m$  ratio was 0.65, and this lies in the range 0.62–0.66, indicating a heterogeneous nucleation and surface crystallisation mechanism (Zanotto and Muller, 1991). The most frequent crystallisation mechanism, the one at the surface, has proved to be quite an effective method for enhancing the strength of glass (Barbieri et al., 2000b). It is concluded that plasma treatment of APC residues produces a highly inert and stable glass which is suitable for disposal in inert landfill or use in bulk civil engineering applications. Alternatively, the glass has potential to be converted into a glass-ceramic for use in a range of higher grade applications.

## **5. Conclusions**

DC plasma arc technology is an effective and versatile method for treating APC residues from waste incineration. This produces a stable, non-leachable and potentially recyclable glass product.

Heavy metals volatilise readily when APC residues are melted in the plasma furnace under reducing conditions. There is a significant reduction in the levels of heavy metals and soluble salts present in the APC derived glass. The high temperature and inert argon atmosphere present during plasma processing inhibits the generation of NO<sub>x</sub>, HCl and SO<sub>x</sub>. Organic contaminants including dioxins and furans are expected to be completely decomposed during the melting process.

Compliance leach tests show that leaching of heavy metals (Pb: 0.007 mg/kg and Zn: 0.02 mg/kg) and Cl<sup>-</sup> (0.2 mg/kg) are well below the limit WAC values for inert landfill disposal.

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**Table**

Table 1  
 APC residue composition data and range of concentrations typically leached using the L/S=10  
 EU compliance leaching test for granular wastes BS EN 12457-3 (WRc Ref: /11873-0, 2004)

APC composition data	Aqua regia total metals/water soluble ions mg/kg
Al	10000-24000
As	10-210
Ba	70-400
Ca	30-35%w/w
Cd	100-150
Co	9-14
Cr	12-200
Cu	350-600
Fe	3000-5200
Hg	<1-16
K	9000-24000
Mg	4000-6000
Mn	350-500
Mo	2-13
Na	13500-20500
Ni	15-35
P	1500-3000
Pb	2500-3500
Sb	200-500
Se	0.1-6
<sup>(1)</sup> Si	Nd
Sn	200-800
Ti	900-4000
Tl	0.5-0.8
V	<30
Zn	4000-8500
<b>water extractions</b>	
Br	1000-2000
F	100-1500
Cl	16 wt%
<sup>(2)</sup> water soluble SO <sub>4</sub>	0.8-3 wt%
CO <sub>3</sub> as CaCO <sub>3</sub>	10000 - 45000
water soluble alkalinity as CaCO <sub>3</sub>	-
water soluble OH as CaCO <sub>3</sub>	-
NH <sub>3</sub> -N	<5
NO <sub>3</sub> -N	-
total N	-
pH	12.0-12.6
sulphite	200 - 600
free lime %w/w CaO	150000 - 200000
Insoluble matter %w/w %w/w Ca(OH) <sub>2</sub>	-
Total phenols (ng/g)	< 0.03
Total PAHs (ng/g)	-
PCDD/DF (ITEQ ng/g)	0.5 - 1.3
Total carbon	10000 - 250000
Total organic carbon	10000 - 250000
VM (%) (LOI)	1.5 - 2.7

<sup>(1)</sup> Nd – not determined

<sup>(2)</sup> NB - high SO<sub>4</sub> values may have been calculated from S determinations on digests (i.e, include other S forms)



Table 2  
Summary of Air Emissions during the APC residue vitrification

Pollutant	Concentration normalised to 273K, 101.3 kPa, 11% O <sub>2</sub> , dry gas [mg/Nm <sup>3</sup> ]	Operating Condition	Oxygen concentration [%]	Measured concentration (not normalized) [mg/Nm <sup>3</sup> ]	Release Limit specified by the Waste Incineration Directive (WID) [mg/Nm <sup>3</sup> ]
TPM (Total Particulate Matter)	2.9	Steady feeding	10	3.18	10
VOC (Volatile Organic Compounds)	9.9	Steady feeding	10	10.5	10
HCl (Hydrogen Chloride)	1.11	Steady feeding	10	1.23	10
HCN (Hydrogen Cyanide)	0.03	Steady feeding	10		-
HF (Hydrogen Fluoride)	0.12	Steady feeding	10	0.13	1
SO <sub>2</sub> (Sulphur Dioxide)	0.56	Steady feeding	10	0.62	50
NO <sub>x</sub> (as Nitrogen Dioxide)	236	Steady feeding	10	260	400
CO (Carbon Monoxide)	117	Steady feeding	10	129	50
CO	395	Cool down (no feeding, burner on)	17	157	-
CO	4369	Cool down (no feeding, burner off)	19.7	552	-

Table 3  
Compliance leaching test (L/S=10) for granular wastes (BS EN 12457-3)

Elements	APC derived glass	Leachable metals/ions in APC residues	Inert landfill WAC
As	<0.007	<0.0005-4	0.5
Ba	0.053	10-45	20
Cd	<0.0025	<0.5	0.04
Cr	<0.016	0.5-2.5	0.50
Cu	0.076	1.3-3	2
Hg	0.0031	0.04-0.7	0.01
Mo	0.012	<1-4	0.5
Ni	0.023	0.2-45	0.4
Pb	<0.007	300-700	0.5
Sb	0.063	<0.0001-0.02	0.06
Zn	0.02	40-85	4
Cl	0.2	1400000-170000	800
SO <sub>4</sub>	<50	7000-1200	1000
TDS*	592	-	4000
DOC <sup>+</sup>	<15	-	500

Leachate concentration in mg/kg

\*TDS\* - Total dissolved solids

<sup>+</sup>DOC<sup>+</sup> - Dissolved organic carbon

Table 4  
Elemental analysis during different stages of processing

Elements	As-received APC residue*		Plasma input feed@		APC derived glass <sup>+</sup>	
	mg/kg	mmol/kg	mg/kg	mmol/kg	mg/kg	mmol/kg
Ca	330,000	8234	209,400	5225	236,500	5900
Na	17,000	742	11,866	516	< DL	< DL
K	16,500	423	11,517	295	< DL	< DL
Al	17,000	630	60,900	2257	106,700	3955
Pb	3,000	15	2,094	10	< DL	< DL
Zn	6,250	96	4,363	67	< DL	< DL
Mg	5,000	208	3,490	144	11,300	465
Fe	4,100	73	2,862	51	26,300	471
Ti	2,450	51	1,710	36	7,200	150
Si	nd	nd	102,100	3635	192,400	6849
Sb	350	3	244	2	< DL	< DL
Cu	475	7	332	5	< DL	< DL
Cd	125	1	87	0.8	< DL	< DL
Cl	160,000	4513	111,680	3150	20,000	564
O	nd	nd	nd	nd	399,700	24981

- \* - Representative data for as-received APC residues  
 @ - Calculated plasma input feed  
 + - Obtained from EDS analysis  
 nd - Not determined  
 < DL - Below detection limit of EDS analysis

Figure

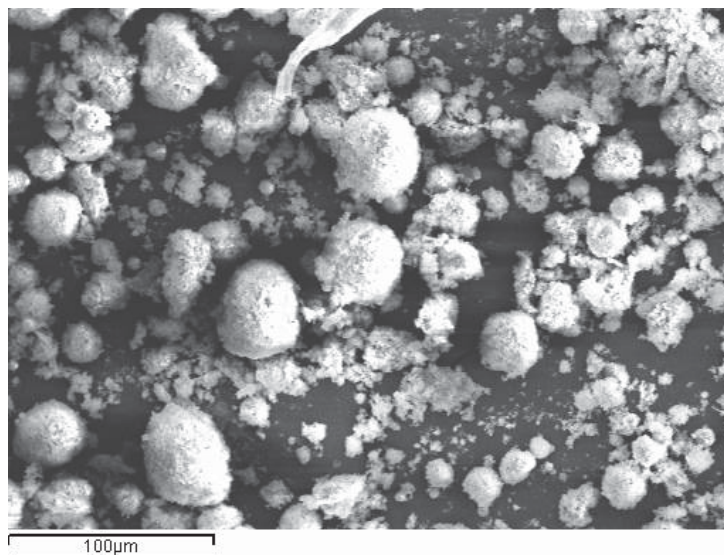


Fig. 1. SEM image of as-received APC residues.

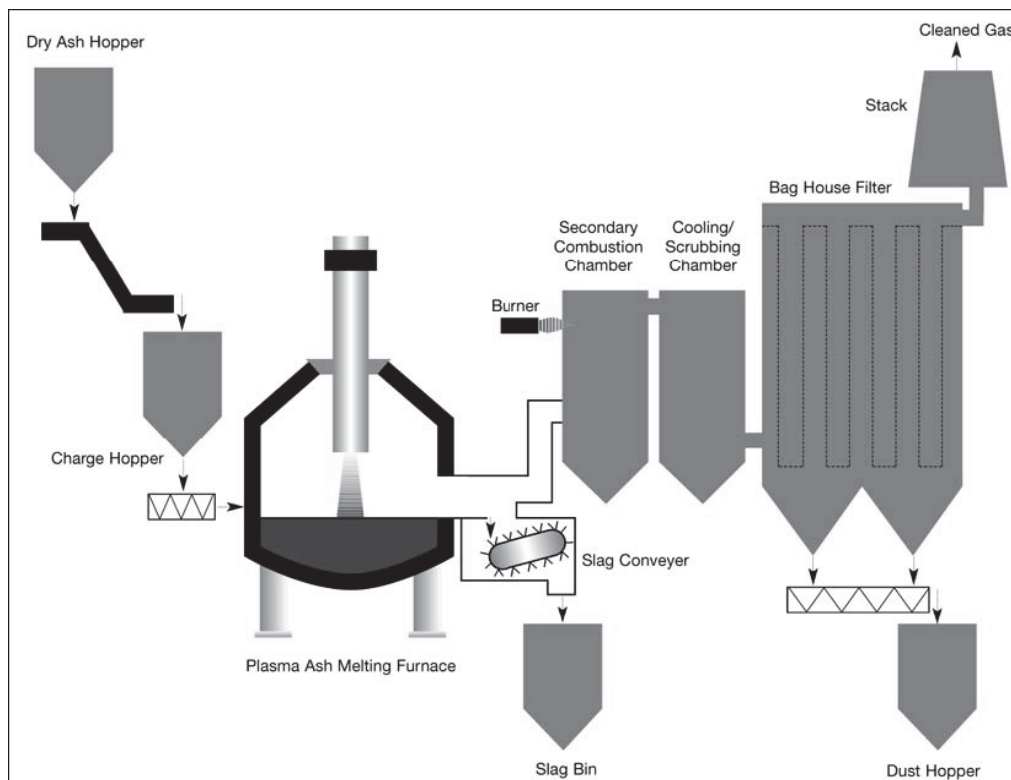


Fig. 2. Process flow diagram for the plasma vitrification of incinerator ashes.

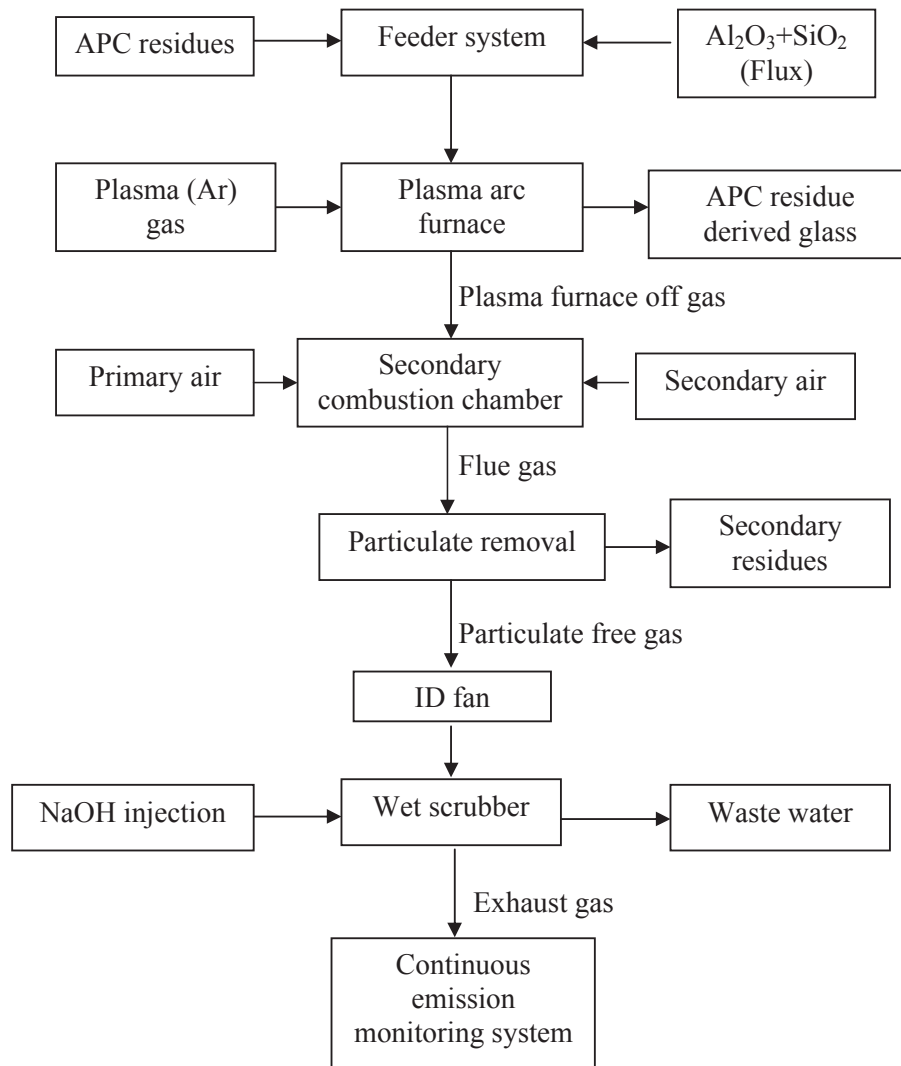


Fig. 3. Flow diagram of plasma operation.

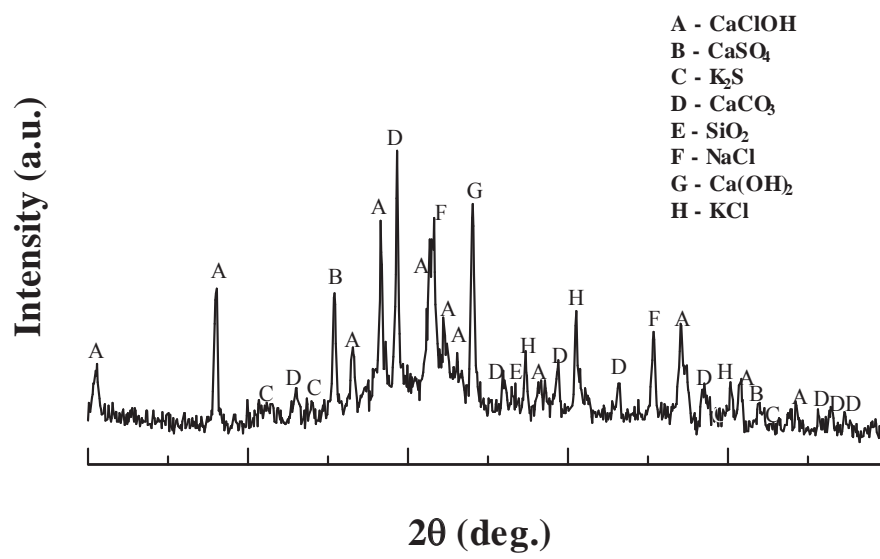


Fig. 4. XRD data for as-received APC residues.

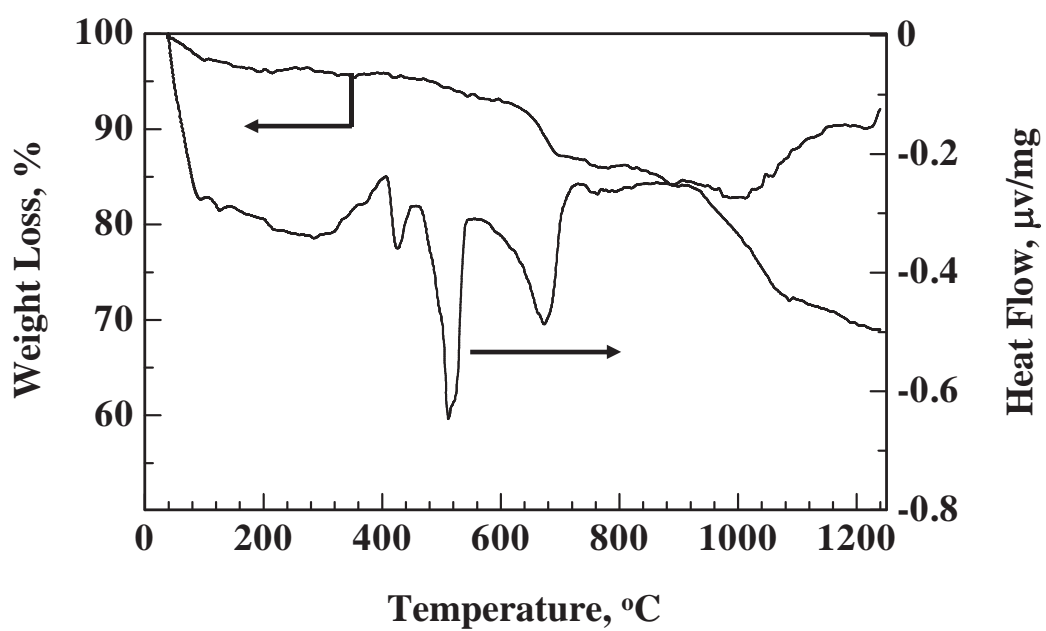


Fig. 5. DTA and TGA data for as-received APC residues.



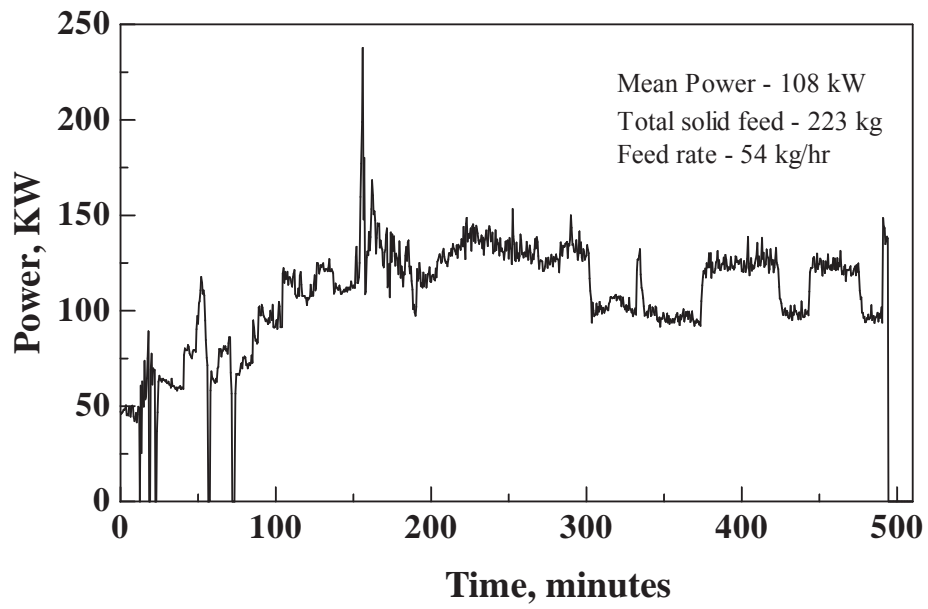


Fig. 6. Plasma power characteristics.



Fig. 7. APC derived glass from treatment.

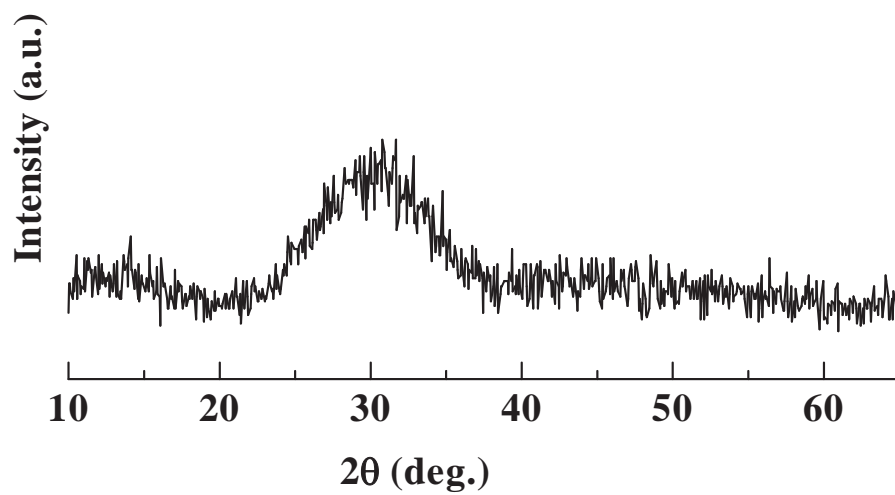


Fig. 8. XRD data for the APC derived glass.

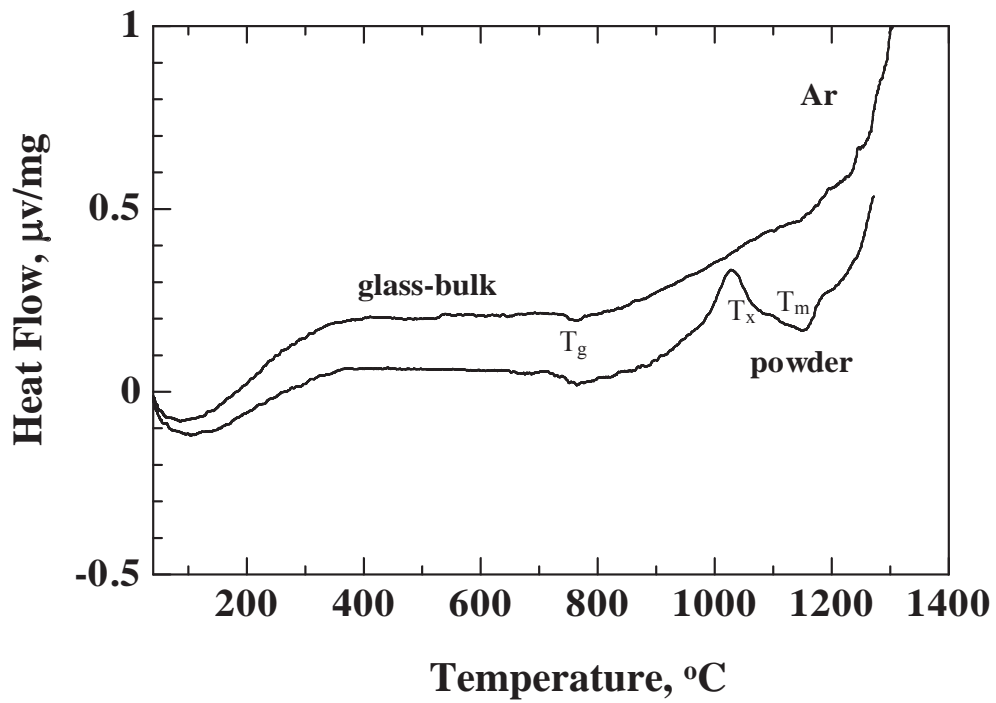
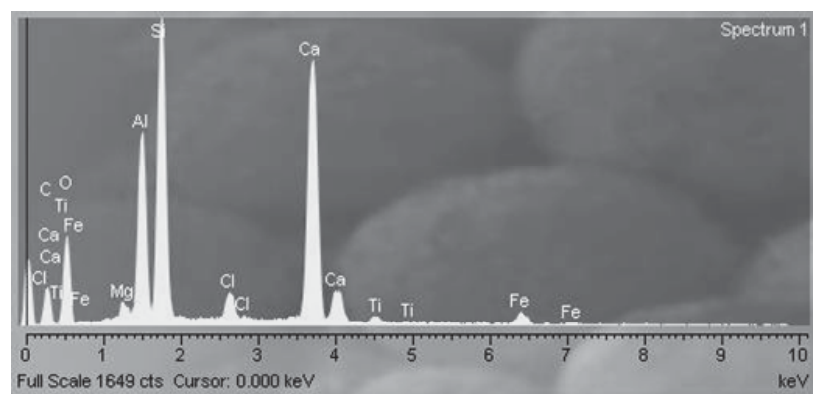


Fig. 9. DTA curves of bulk and powdered APC residue derived glass.



Elements (wt%)	O	Ca	Si	Al	Fe	Mg	Cl	Ti
	40.0	24.0	19.2	10.7	2.6	1.1	2.0	0.7

Elemental compositions are obtained from an average of 10 analyses carried out on different sites of the APC derived glass

Fig. 10. Chemical analysis of the APC derived glass by EDS-SEM.

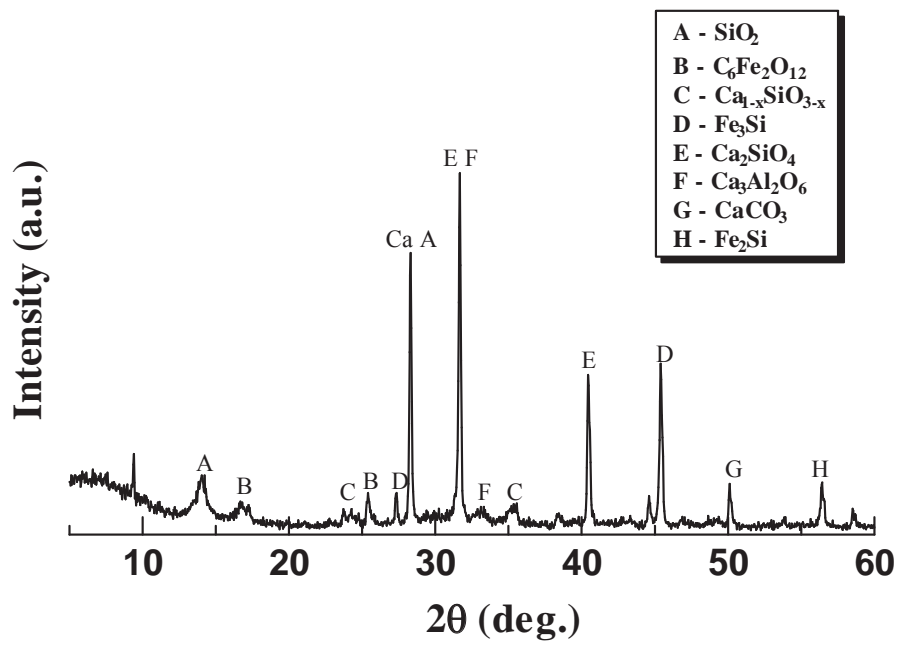


Fig. 11. XRD data for the secondary residues generated during plasma treatment of APC residues.