Katanax®
A SPEX SamplePrep Company

Borate Fusion

APPLICATION NOTE

Preparation and XRF Analysis of Fly Ash Fused Beads

ABSTRACT

Fly ash is a waste product from the combustion of coal and is comprised of the mineral particles that rise with the flue gases. Recovered fly ash is used as a component in certain cement mixes and improves the durability and strength of concrete.

In this study, a sample of fly ash was pulverized and blended, and then fused into two sets of glass beads using a Katanax K1 electric fusion fluxer. Two different lithium borate fusion fluxes from SPEX CertiPrep were used to prepare five beads with each flux. The beads were then analyzed by energy dispersive X-ray flourescence spectroscopy (ED-XRF) and the results were compared both within a set and between the two sets to evaulate consistency of beads produced using the K1 and effect of flux composition on the XRF measurements.

Fusion Method

The fly ash was roasted in a porcelain crucible for 2 hr. at 750° C to eliminate residual carbon (~0.2%). 6.40 g of flux and 0.80 g of fly ash (a flux/sample ratio of 8:1) were weighed into a Pt/Au crucible, gently mixed by hand with a plastic laboratory spatula, and fused into a 32 mm glass bead using the K1. The fusion program used was a modified version of the K1's fixed OXIDE program, with the main fusion stage (Step 4) set for 15 minutes at 1035° C.

Five beads were prepared in this manner for each of two fluxes: SPEX CertiPrep FFB-5005-02 (1:1 lithium tetraborate:lithium metaborate with 0.5% lithium bromide as a non-wetting agent) and FFB-6705-02 (2:1 lithium tetraborate:lithium metaborate with 0.5% lithium bromide).

Analysis

The two sets of glass beads were analyzed by ED-XRF. Each bead was measured ten times, with the results averaged for each bead. The results for the five beads within a set were then averaged again to obtain overall results for the set. The results are shown in Tables 1 & 2 as percentage of composition for each analyte, with the major components being silica and alumina. Oxides of iron, potassium, calcium, magnesium, phosphorus, titanium, sodium, and sulfur were present in lesser amounts and comprised the remainder of the fly ash.

XRF results show excellent consistency within a set, indicating that the fly ash sample was homogenous and that the K1 prepares fused beads with a high level of reproducibility. In addition, the overall averages from Table 1 and Table 2 are nearly identical. Thus, it is apparent that the choice of flux composition had no effect on the XRF results. Since both fluxes produced clear glass beads with the fly ash sample, either is appropriate to use with this fly ash.

Measured results were also consistent for the ten scans of each bead. An example for one bead is shown in Appendix 1 and is indicative of results obtained from repetitive scans of the other nine beads. Complete data for this study are on file at SPEX SamplePrep, LLC, Metuchen, NJ, USA.

- APPLICATION NOTE SP026:
  Preparation and XRF Analysis of Fly Ash Fused Beads
- APPARATUS:
  Katanax®
- APPLICATION:
  Cement, Slag & Fly Ash

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Table 1 – Fly Ash Composition (%) using FFB-5005-02

Analyte	Average	Bead 1	Bead 2	Bead 3	Bead 4	Bead 5
SiO_2	52.08	52.11	52.07	52.11	52.08	52.06
Al_2O_3	26.75	26.74	26.76	26.74	26.74	26.76
Fe_2O_3	6.52	6.52	6.52	6.52	6.52	6.52
K_2O	4.14	4.14	4.13	4.14	4.14	4.15
CaO	3.46	3.46	3.45	3.46	3.46	3.46
MgO	2.04	2.04	2.04	2.04	2.04	2.05
P_2O_5	1.42	1.42	1.43	1.42	1.43	1.43
TiO_2	1.26	1.26	1.25	1.26	1.26	1.26
Na_2O	1.23	1.22	1.25	1.22	1.24	1.23
SO_3	0.16	0.16	0.16	0.16	0.16	0.16

Table 2 – Fly Ash Composition (%) using FFB-6705-02

Analyte	Average	Bead 1	Bead 2	Bead 3	Bead 4	Bead 5
SiO_2	52.07	52.05	52.12	52.09	52.09	52.02
Al_2O_3	26.75	26.75	26.79	26.75	26.69	26.76
Fe_2O_3	6.49	6.50	6.47	6.50	6.50	6.50
K_2O	4.15	4.15	4.15	4.15	4.16	4.16
CaO	3.47	3.48	3.45	3.48	3.48	3.47
MgO	2.05	2.06	2.02	2.06	2.06	2.06
P_2O_5	1.42	1.41	1.41	1.41	1.42	1.42
TiO_2	1.26	1.26	1.25	1.26	1.26	1.26
Na_2O	1.24	1.24	1.23	1.24	1.24	1.25
SO_3	0.16	0.16	0.16	0.16	0.16	0.16

Conclusion

The Katanax K1 electric fusion fluxer is easy to operate and produces high quality, reproducible glass beads. The two fluxes used in this study gave XRF results that indicate that flux choice did not influence the analysis. In addition, the high degree of bead to bead consistency within a set indicates that the elemental composition of the flux is uniform.

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Table 3

Fly Ash results for 10 successive XRF scans of Bead 4 prepared using FFB-5005-02.

Analyte	SiO_2	Al_2O_3	Fe_2O_3	K_2O	CaO
Scan 1	52.09	26.81	6.52	4.14	3.47
Scan 2	52.05	26.81	6.51	4.13	3.46
Scan 3	52.14	26.71	6.52	4.15	3.45
Scan 4	52.04	26.71	6.51	4.15	3.47
Scan 5	52.07	26.80	6.51	4.14	3.48
Scan 6	52.11	26.76	6.53	4.12	3.46
Scan 7	52.04	26.71	6.53	4.15	3.47
Scan 8	52.05	26.75	6.51	4.16	3.42
Scan 9	52.14	26.67	6.52	4.15	3.48
Scan 10	52.07	26.67	6.52	4.15	3.46
Average	**52.08**	**26.74**	**6.52**	**4.14**	**3.46**

Analyte	MgO	P_2O_5	TiO_2	Na_2O	SO_3
Scan 1	2.04	1.42	1.26	1.18	0.16
Scan 2	2.02	1.42	1.26	1.24	0.16
Scan 3	2.04	1.42	1.27	1.21	0.16
Scan 4	2.05	1.45	1.26	1.29	0.16
Scan 5	2.02	1.42	1.26	1.23	0.16
Scan 6	2.03	1.42	1.26	1.20	0.16
Scan 7	2.07	1.46	1.26	1.24	0.16
Scan 8	2.05	1.46	1.26	1.27	0.16
Scan 9	2.07	1.42	1.27	1.21	0.16
Scan 10	2.03	1.42	1.25	1.31	0.16
Average	**2.04**	**1.43**	**1.26**	**1.24**	**0.16**

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