Application of Fusion sample preparation X Ray Fluorescence Spectrometry in the Identification of Solid Waste Characteristic of Imported Coated Fibre Cloth

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Abstract: The composition of imported coated fibre cloth was quickly identified by fusion sample preparation X-ray fluorescence spectrometry. Na₂O, MgO, Al₂O₃, SiO₂, P₂O₅, SO₃, K₂O, CaO, TiO₂, Cr₂O₃, MnO, Fe₂O₃, NiO, CuO, ZnO, As₂O₃, SrO, ZrO, CdO, BaO, PbO in the matrix of the coated fibre cloth were determined. In this paper, the technology of direct melting of flake sample is studied. The method of pre-oxidation is that the sample piece is obliquely placed on the inner wall of crucible to increase the air circulation space and remove the organic matter and halide in a short time. The 66.5:33.5 mixed flux was used as the flux, and LiBr was used as the release agent. The temperature was about 1050 °C, and the time was about 17 min. The problem of melting of inorganic fibre and flux melt was found and solved. The calibration curve is constructed by silicate rock, bauxite, cement and other reference materials and high-purity reagents. The matrix effect correction adopts the variable theoretical α influence coefficient method, and B₂O₃ represents the unmeasured components as the elimination item. The maximum Al₂O₃, SiO₂, CaO curves of this method are 84.4%, 91.5%, 62.6%, respectively. The Cr, Ni, Cu, As, Cd, Pb curve range of harmful elements is greater than 0.005%-0.33%; 21 curves have good linearity. R² divided by BaO equals 0.997 and the rest are greater than 0.9993; the detection limit of harmful elements is less than 15 mg·g⁻¹; precision relative standard deviation (n=11) principal component is less than 0.5%, and trace component is less than 15%; after comparison of standard substances and standard methods, the results are consistent. Accuracy of methods is good, and it can quickly identify the content of the main elements of the coated fibre cloth and provide a strong support for the subsequent identification of solid waste characteristic.

1. Introduction
The coated fibre cloth refers to the multilayer composite material coated with organic polymer on the inorganic fibre cloth, which can provide insulation, heat insulation, flame retardant, noise reduction, sealing and other functions for the product, such as circuit board substrate [1]. Immersion fibre cloth is a semi-finished product in circuit board production. It contains many additives such as flame retardants. Improper disposal is easy to cause serious environmental problems. It is an important object of customs solid waste supervision [2]. However, some enterprises collect the leached fibre
cloth scraps and leftovers in the circuit board production process and then import them in the name of sub brand materials or stock materials. The purpose of the action plan is to intercept such solid waste outside the country. The composition of coated fibre cloth produced by different production processes and different production purposes is different, and its production source can be judged by composition analysis, which provides data support for further solid waste attribute identification. Reference [3] introduces that inorganic fibre cloth can be divided into two categories: glass fibre and asbestos, the main components of glass fibre are SiO$_2$, B$_2$O$_3$, Na$_2$O, MgO, Al$_2$O$_3$, CaO, ZrO, and secondary components are Li$_2$O, F, K$_2$O, TiO$_2$, Fe$_2$O$_3$. The content range is less than 30%. Common asbestos is slag cotton, basalt cotton, refractory fibre and so on, and the range of SiO$_2$ is more than 60%. According to the catalogue notes of the 2017 edition of the Explanatory Notes to the Harmonized Commodity Description and Coding System, mineral fibre cloth is divided into two categories: glass fibre cloth and asbestos fibre cloth.

A complete detection method has not been found for the composition analysis of coated fibre cloth, and general analysis needs to be combined with several methods. The process is generally to determine proportion of impregnate (ashing), grinding and crushing, and judge the type of fibre cloth, and finally use the corresponding method to analyze the composition. The analysis method can be divided into wet chemical method and X-ray fluorescence spectrometry (XRF). Wet chemical method has fibre glass standard GB/T 1549[4] and GB/T 33999[5], mineral cotton standard GB/T 14506.30[6], GB/T 6901[7], GB/T 34333[8]. Microwave digestion-inductively coupled plasma emission spectroscopy (ICP-OES) and inductively coupled plasma mass spectrometry (ICP-MS) were used to determine the quantity and trace elements, but the principal components still need to be determined by titration, which requires a variety of methods to complete the component detection, which is time-consuming and inefficient. XRF method has silicate rock standard GB/T 14506[9] and refractory standard GB/T 21114[10]. It is only for refractory materials composed of inorganic salts, and it is not for composite materials, and no specific method is provided. In addition to the standard, the paper [11-12] introduces the glass fibre and soil analysis method (the main component is silicate) of tableting sample XRF, which is simple but it cannot eliminate the particle effect and it is not suitable for inorganic fibre cloth; reference [13-14] introduces the method of melting sample and analysis by XRF for the determination of 10 primary and secondary elements in different types of rocks, such as silicate and kaolin. Reference [15] introduces the method of XRF for the determination of 15 components in siliceous, aluminous, magnesia and chromium refractories. The above method or literature is either not operable or only for rocks and refractory materials detection, and it cannot be fully applied to the composition detection of coated fibre.

The purpose of above methods is detecting the main elements, and no harmful trace elements such as As, Cd, Pb are detected. In this paper, modified melting sample preparation technology and ultra-light element treatment method based on full element analysis [16] are used to establish calibration curves with various standard samples to deal with inorganic fibre cloth with complex components, and add high purity reagents to the standard samples to expand the analysis range of harmful heavy metal elements and Zr. Totally the method detects burning loss and Na, Mg, Al, Si, S, P, K, Ca, Ti, Cr, Mn, Fe, Ni, Cu, Zn, As, Sr, Zr, Cd, Ba, Pb and other 21 elements, semi-quantitative Br and B elements, one method multi-use, and it provides effective and rapid detection technology for customs law enforcement work.

2. Experimental part

2.1. Instruments and reagents

S8 sequential X-ray fluorescence spectrometer (Bruker AXS, Germany); ISP 4×4 semi-automatic sample melting furnace (ISP, Australia); 30mL platinum gold crucible and 40mm mold (Changhong Precious Metal Co., Ltd., China); CWF 12/23 Muffle furnace (Carbolite, UK); XP205 analytical balance (Mettler Toledo, Switzerland).
Standard materials: Glass GBW03132 (National Center for Testing of Building Materials), Soil GBW07401-GBW07406 (Institute of Geophysical and Geochemical Exploration), Rock GBW07110 (Institute of Rock and Mineral Testing Technology, Ministry of Geology and Mineral Resources), Copper Ore GBW07170 (Central Laboratory of Geological and Mineral Exploration and Development Bureau of Tibet Autonomous Region), Iron Ore ECRM610-1 (French Iron and Steel Institute), GQB series total 12 (German Bruker Corporation).

Reagents: Flux (Li$_2$B$_4$O$_7$:LiBO$_2$=66.5:33.5), High Purity, German Fluxana, Inc., before using, 600°C cauterized 2 h; release agent, LiBr solution, 20 mg·mL$^{-1}$; ZrO$_2$, Spectral Purity, Tianjin Institute of Photosynthetic Fine Chemical Engineering; Cr$_2$O$_3$, CdO, PbO, Spectral Purity, Shanghai Reagent I Plant.

2.2. Sample preparation

Weigh the mass of the dry and constant weight crucible, and weigh the 0.8 g-1.2 g sheet sample whose size is less than 1cm×2cm. Lean against the crucible (Fig 1-a), and put it into the muffle furnace for pre-oxidation. Keep it at 350 ℃ for 30min, and raise it to 700 ℃ at the speed of 30 ℃/min. Keep it for 20min (the holding time can be extended according to the amount of Br), and take out the crucible and cool it to room temperature in the desiccant, and weigh the mass; then weigh 6.0000g dissolution and put into the same crucible (without stirring, Fig 1-b). Add 0.5mL mold release agent, and directly put into the sample melting furnace with the temperature of 1050±25 ℃ for 15min ~ 20min, in which the crucible is taken out by hand and rotated and vibrated every other time for 3 times in total; finally, the melt is cast into the mold and air cooled for 10min, and the test piece is taken out of the mold for testing. The mass weighed in the process is used to calculate the loss on ignition and the mass of inorganic substances. For calculation, please refer to literature [7].

![Fig 1. Sample weighing diagram, (a) weigh the sample, (b) weigh the flux](image)

2.3. Preparation of calibration samples

33 test sheets were prepared with 22 standard materials and 4 spectral pure reagents, of which 13 mixed standard samples. The nominal weighing sample of standard material and flux is 0.6000 g and 6.0000 g, mold release agent 0.5 mL, melting temperature 1050℃, time 15 min. The synthesis standard sample is called flux, standard substance, added substance in turn, and the sum of the latter two masses is about 0.6 g (accurate to 0.01 mg). The reagent synthesis is as follows: ZrO$_2$ (0.05 g -0.1 g) were added to GBW07110 and GQB20, and GBW07170 (0.03 g -0.08 g) were added to GQB6 and GQB20, Cr$_2$O$_3$, CdO, PbO (0.001 g-0.01 g) were added to the GBW07110, GQB8 and blank.

2.4. Instrument measurement

According to the content of the measured elements and the mutual influence, the analytical spectral line and the measurement conditions (see Table 1) are selected. The position of the analytical spectral line and the background is determined according to the actual scanning. The flow gas counter is used for the element detector for Ti and before Ti, and the scintillation counter is used for the element after Ti. The measurement time adopts the optimization mode provided by the software. The maximum measurement time of Pb and As peak is 100s, and that of other elements is 30s. The matrix effect (Cd does not correct the matrix effect) is corrected by the variable theoretical α influence coefficient.
method, and B₂O₃ is used as the elimination term (margin). The slope and intercept of the calibration curve are obtained by regression of equation 1. In addition, the output Br Kα intensity is used to monitor the effect of Br on the principal components.

\[ C_i = s \times I_i \times (1 + \sum \alpha_{ij} \times C_j) + b \]  

(1)

In the formula, \( C_i \) and \( C_j \) represents the content of the measured element and the influencing element; \( s \) and \( b \) represents the slope and intercept of the calibration curve; \( I_i \) represents the X ray fluorescence intensity of the measured element; \( \alpha_{ij} \) represents the influence coefficient of theory or experience \( \alpha \).

| Elements | Analyse Line | Voltage /kV | Currents/mA | Crystal     | Collimator/° |
|----------|--------------|-------------|-------------|-------------|--------------|
| Na、Mg   | Kα           | 30          | 120         | XS55        | 0.46         |
| Al、Si   | Kα           | 30          | 120         | PET         | 0.23         |
| P、S     | Kα           | 30          | 120         | XS-Ge-C     | 0.23         |
| K、Ca、Ti| Kα           | 50          | 72          | LiF200      | 0.23         |
| Cr、Mn、Ni、Cu、Zn、Br | Kα | 60          | 50          | LiF200      | 0.46         |
| Fe       | Kα           | 50          | 60          | LiF200      | 0.23         |
| As、Sr、Cd| Kα           | 60          | 10          | LiF220      | 0.23         |
| Zr       | Kα           | 60          | 72          | LiF200      | 0.23         |
| Ba       | Lα           | 50          | 10          | LiF200      | 0.23         |
| Pb       | Lβ           | 60          | 10          | LiF200      | 0.23         |

3. Results and discussion

3.1. Preliminary heat treatment in oxidizing

To investigate the degree of decomposition of carbon and bromides, the thermal cracking test temperature 350℃-450℃(epoxy resin cracking temperature is generally greater than 300℃) and the ashing test temperature is 700℃-850℃(glass fibre softening temperature is up to 750℃), four samples with large differences in shape and composition (see Fig 2) were selected for the study of independent and mixed pre-oxidation (sample, flux, oxidant mixing) according to the IEC62321-5[16] polymer ashing method. The heating rate is about 30℃/min, and the sample loss is 25%, 61%, 22%, 41%, respectively. The sample size is 0.6 g-1.2g.

![Sample shape and properties](image)

(a) single-layer glass fibre bisphenol A epoxy resin sheet with flame retardant  
(b) single-layer mineral wool bisphenol A epoxy resin sheet with flame retardant  
(c) single-layer mineral wool resin sheet  
(d) multilayer glass fibre epoxy resin board with flame retardant

Fig 2. Sample shape and properties

The test results are as follows: a). The most stable reaction of the samples is pyrolysis at 350℃ and ashing at 700℃, and the sample does not burn and soften; b) The mixing ashing process has low efficiency, and carbon can be decomposed within 20 min after sample a and b are added oxidant. The bromide needs 60 min (Br content is about 1%) to reach the controllable range, so the mixing method is only suitable for the bromine-free sample; c) The independent method needs to lean the sample to the inner wall of the crucible and increase the air contact area. If it is stacked at the bottom, the ashing time will be much longer; d) The pre-oxidation time is related to the polymer quality in the sample,
and the pre-oxidation condition of 2.2 is formulated with reference to the b sample with the largest amount of burning loss.

3.2. Melt
The inorganic fibre sample will melt and agglomerate alone at high temperature, which makes the sample exist as a large independent body in the flux, such as the larger particles in the powder sample, which increases the melting difficulty. Even increasing the unidirectional rocking speed, increasing temperature and prolonging time cannot effectively improve the uniformity of the test sheet. It was found that the two melts can be mixed by manual rapid rotation oscillation. From the tested samples, it is necessary to rotate the oscillation three times to ensure the uniformity of the test sheet. Table 2 shows the test sheet parallelism before and after the improvement.

| Sample-component | Before improvement, mass% | After improvement, mass% |
|------------------|--------------------------|--------------------------|
| e-SiO2           | 54.30/55.18/54.06        | 54.61/54.55/54.54        |
| e-CaO            | 24.00/24.25/24.06        | 24.15/24.17/24.16        |
| c-SiO2           | 74.08/62.96/63.64        | 66.24/66.35/66.29        |
| c-CaO            | 17.57/15.08/15.10        | 16.01/16.07/16.05        |

3.3. Influence of Br
The Br in melt sample preparation exists on the surface of the melt sheet in the form of release agent. Different Br amount will cause fluorescence intensity deviation. The bromide in coated fibre cloth is generally used as flame retardant, and the content is uncertain. If the pre-oxidation decomposition is not complete, the Br in the sample sheet and the standard sheet will be inconsistent. Fig 3 shows its effect on the primary and secondary components. As shown in the Fig 3 that the LiBr addition amount has a nonlinear relationship with the measured results, and the less interval LiBr of the same size causes the smaller deviation. Therefore, this method takes 10 mg as the reference addition quantity under the premise of guaranteed remoulding and takes 8 mg-12 mg as the reliable interval, and the relative deviation in the interval is less than 0.3%. It extends the pre-oxidation time if it is more than 12 mg.

Fig 3. Changes in measurement results caused by different amounts of bromine added.

3.4. Calibration curve
There is a total of 21 calibration curves. Their range, standard deviation (SD) and detection limit are shown in table 3. Detection limit (LLD) is calculated according to the formula (LLD = 3√Tb/m):

Where, m is the measurement sensitivity (cps.%⁻¹), that is, the change of X-ray fluorescence intensity
caused by each 1% change in content; X-ray fluorescence intensity (cps) with I_b as the background; T_b is the background measurement time (s). The correlation coefficient B_aO is about 0.997, TiO_2 and Cr_2O_3, ZnO, SrO is about 0.9995, and the others are greater than 0.9999, which meet the composition range of domestic and foreign fibreglass and asbestos and the limit range of solid waste provided in literature [3]. Br curves were constructed with the addition amount (mg) and fluorescence intensity to determine whether to extend the pre-oxidation time.

### Table 3. Calibration curves information

| Component | Range (mass %) | S.D. (mass %) | LLD (mg·kg^{-1}) | Component | Range (mass %) | S.D. (mass %) | LLD (mg·kg^{-1}) |
|-----------|----------------|---------------|-----------------|-----------|----------------|---------------|-----------------|
| Na_2O     | 0.06-10.6      | 0.022         | 19              | Fe_2O_3   | 0.02-57.5      | 0.064          | /               |
| MgO       | 0.26-18.8      | 0.053         | 22              | NiO       | 0.005-1.59     | 0.001          | 6               |
| Al_2O_3   | 1.93-84.4      | 0.14          | /               | CuO       | 0.003-2.08     | 0.008          | 6               |
| SiO_2     | 2.00-91.5      | 0.19          | /               | ZnO       | 0.001-0.19     | 0.001          | 7               |
| P_2O_5    | 0.01-6.11      | 0.010         | 12              | As_2O_3   | 0.001-0.44     | 0.002          | 6               |
| SO_3      | 0.05-17.8      | 0.042         | 11              | SrO       | 0.001-0.26     | 0.002          | 3               |
| K_2O      | 0.01-5.14      | 0.015         | 25              | ZrO_2     | 0.01-16.7      | 0.052          | 13              |
| CaO       | 0.01-62.6      | 0.087         | 10              | CdO       | 0.005-1.24     | 0.004          | 15              |
| TiO_2     | 0.01-3.73      | 0.015         | 21              | BaO       | 0.012-0.14     | 0.002          | 42              |
| Cr_2O_3   | 0.005-2.28     | 0.008         | 11              | PbO       | 0.003-1.69     | 0.001          | 5               |
| MnO       | 0.002-0.64     | 0.002         | 8               | Br        | 6mg-22mg       | /              | /               |

3.5. Precision

The precision was obtained by statistics of the measurement data of 9 test pieces of the same sample melted at different times. Table 4 shows the precision of 3 different types of samples and lists the data greater than 3 times the detection limit.

### Table 4. Precision

| Component | S1, mass% | S2, mass% | S3, mass% |
|-----------|-----------|-----------|-----------|
| Na_2O     | 0.23±0.007| 0.34±0.009| 0.49±0.006|
| MgO       | 0.67±0.012| 0.56±0.018| 0.50±0.016|
| Al_2O_3   | 14.23±0.06| 75.37±0.11| 10.36±0.049|
| SiO_2     | 54.54±0.084| 12.42±0.17| 66.11±0.16|
| P_2O_5    | 0.026±0.001| 0.052±0.001| 0.61±0.01|
| SO_3      | 0.11±0.005| 0.17±0.006| 0.090±0.003|
| K_2O      | 0.083±0.004| /          | 0.037±0.004|
| CaO       | 24.12±0.055| 5.48±0.089| 16.04±0.065|
| TiO_2     | 0.50±0.003| 0.078±0.008| 0.38±0.003|
| Cr_2O_3   | 0.012±0.002| <0.005     | 0.014±0.002|
| MnO       | 0.006±0.001| 0.006±0.001| 0.004±0.001|
| Fe_2O_3   | 0.40±0.008| 0.091±0.022| 0.19±0.013|
| NiO       | /         | /         | 0.004±0.001|
| CuO       | /         | 0.006±0.001| 0.014±0.001|
| ZnO       | 0.005±0.0004| 0.004±0.0005| 0.007±0.0005|
| As_2O_3   | 54±6      | /         | /         |
| SrO       | 0.20±0.001| 0.030±0.001| 0.14±0.003|
| ZrO_2     | 0.044±0.001| 0.027±0.002| 0.031±0.003|
| CdO       | /         | /         | 63±7     |
| BaO       | /         | 0.043±0.003| /         |
| PbO       | 28±3      | 50±6       | /         |

Note: *Unit: mg·kg^{-1}
3.6. Method comparison
GB/T 21114 only provides the guiding direction, lack of practical operation, and this method is obviously different from the GB/T 21114 in the sample preparation step. After comparison, it can be seen that the results of sample non-crushing melting and comminuting melting are consistent (see S2 and S3 data in table 5). The results of glass standard sample GBW0312 are consistent with the standard value, which indicates that this method can determine the sample containing ultra-light elements such as constant B2O3, and obtain the approximate content of ultra-light elements at the same time. As, Cd and Pb compared with GB/T 33999 microwave digestion IPC-OES method, the results were consistent (Table 6).

Table 5. Comparison of results (1)

| component      | GBW 03132, mass% | S2, mass% | S3, mass% |
|----------------|------------------|-----------|-----------|
|                | This method      | Standard value | This method | GB/T 21114 | This method | GB/T 21114 |
| Na2O           | 0.089            | 0.096     | 0.34      | 0.34       | 0.49       | 0.47       |
| MgO            | 4.39             | 4.40      | 0.56      | 0.60       | 0.50       | 0.49       |
| Al2O3          | 14.45            | 14.50     | 75.37     | 75.26      | 10.36      | 10.58      |
| SiO2           | 53.66            | 53.98     | 12.42     | 12.49      | 66.11      | 66.30      |
| P2O5           | /                | /         | 0.052     | 0.052      | 0.61       | 0.59       |
| SO3            | /                | /         | 0.17      | 0.17       | 0.090      | 0.093      |
| K2O            | 0.58             | 0.59      | /         | /          | 0.037      | 0.039      |
| CaO            | 16.70            | 16.54     | 5.48      | 5.383      | 16.04      | 15.92      |
| TiO2           | 0.18             | 0.19      | 0.078     | 0.068      | 0.38       | 0.37       |
| Cr2O3          | /                | /         | /         | /          | 0.014      | 0.012      |
| MnO            | /                | /         | 0.006     | 0.005      | 0.004      | 0.004      |
| Fe2O3          | 0.36             | 0.34      | 0.091     | 0.085      | 0.19       | 0.20       |
| NiO            | /                | /         | /         | /          | 0.004      | 0.005      |
| CuO            | /                | /         | 0.006     | 0.006      | 0.014      | 0.014      |
| ZnO            | /                | /         | 0.004     | 0.004      | 0.007      | 0.008      |
| SrO            | /                | /         | 0.030     | 0.028      | 0.14       | 0.14       |
| ZrO2           | /                | /         | 0.027     | 0.024      | 0.031      | 0.033      |
| BaO            | /                | /         | 0.043     | 0.042      | /          | /          |
| Ultra-light components | 9.41 | 9.02 | / | / | / | / |

Table 6. Comparison of results (2)

| components | S4, mg·kg⁻¹ | S5, mg·kg⁻¹ | S6, mg·kg⁻¹ |
|------------|-------------|-------------|-------------|
|            | This method | GB/T 33999  | This method | GB/T 33999  | This method | GB/T 33999  |
| As2O3      | 2933        | 3081        | 416         | 445         | 82          | 77          |
| CdO        | 88          | 95          | 363         | 384         | <50         | 0.8         |
| PbO        | 407         | 397         | 357         | 364         | 62          | 57          |

3.7. Identification of actual sample
In the middle of 2019, a batch of coated fibre cloth imported by a company from Taiwan, China, was declared as coated fibreglass cloth (residual material). Field inspection found that the specifications and packaging were inconsistent, and some mixed and rewinding traces existed. The samples were sent to the laboratory for identification. The laboratory analyzed the composition of the samples according to the above method. The results are shown in the following Table 7:
Table 7. The result of actual sample

| Sample | Components, mass% | Categorization |
|--------|------------------|----------------|
|        | SiO₂  | K₂O | Na₂O | Other harmful elements |         |
| A1     | 63.2  | 1.2 | 2.3  | /                 | glass   |
| A2     | 58.7  | 0.4 | 0.6  | /                 | asbestos|
| A3     | 65.3  | 1.7 | 2.2  | /                 | glass   |
| A4     | 55.3  | 0.5 | 0.7  | Pb(1.2 mass%)      | asbestos|

Combined with the above data, it is judged that the composition of the matrix material of the sample is different, because the mixture of coated fibre cloth produced by different specifications and different production purposes is different, and combined with other test data, the goods are judged as solid waste.

4. Conclusions
This method improves the melt sample preparation and solves the problem of melt sample preparation of fibre cloth rich in organic matter by simple method and establishes the calibration curve suitable for glass fibre and asbestos with various standard materials and high purity reagents. Instead of several limited standard methods, it can effectively reduce the detection period of coated fibre cloth and measure the trace harmful heavy metal elements while determining the main components. Determination makes up for the deficiency of similar methods and provides technical support for the identification of solid waste characteristic of such products.

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