Application of X-Ray Diffraction Analysis on characterization of municipal solid waste incinerator residues

Zhang Ruina Student Member Institute of Environmental Systems, Kyushu University

Takayuki Shimaoka Member Institute of Environmental Systems, Kyushu University

1. Introduction

As a powerful detection method, XRD can detect the crystal species, however because of the instrument detection limit, the low concentration of the important heavy metal species which are commonly accepted as the main problem of MSWI residues can be hardly detected. In order to know the XRD detection limit, a series of artificial mineralogy samples which contains different concentrations of $PbCO_3$, PbO and PbS within glass matrix has been studied, the result indicated although the XRD instrument used in this study can detect the weak signals even if the concentration is lower than 1 %, the data can't be processed by JADE after measurement.

Water washing -based Mineralogy XRD analyses of MSWI residues indicated that the heavy metals could be detected after short time water washing.

Particle size distribution-Based mineralogy XRD analyses of MSWI residues indicated that for the heavy metals especially lead compounds which is difficult to be detected by XRD in the whole particle size range can be detected in the fine particles, although the strongest peaks of the different particle size have no big difference, as the small peaks and the interaction peaks was concerned, this method is more efficient compare with the bulk concentration measurement.

2. Experiments

2.1 Instruments XRD

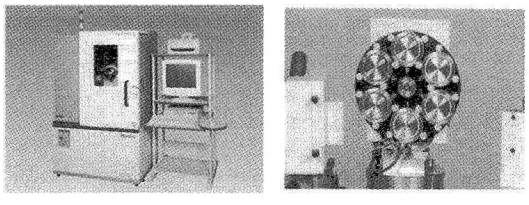

Fig.1. Rigaku-multiflex XRD machine and Automatic SampleChanger for 6 Samples (ASC-6) applied in this study

A Rigaku-multiflex machine was used, shown in figure 1; a copper tube (44 kV, 34 mA, 1500W) was employed as an X-ray source. Powders were run in triplicate; Automatic Sample Changer for 6 Samples (ASC-6) was used to hold the samples. Scans were conducted from 6.00 to 90.00° at a rate of 0.6° 2θ/min. Tungsten external standards were employed. JADE and Rigaku software was used to identify possible crystalline phases in the residues. And calculation of figure of merit (FOM) based on weightings of 90% for peak location and 10% for peak intensity. An FOM of 20 is considered a good fit, and an FOM of 10 is considered an excellent fit for these complex polycrystalline powders.

XRPD analyses were conducted on the artificial samples and the MSWI residues.

2.2 Materials

Artificial mineralogy samples was made by glass which has been crushed to 30 μ m and different percentage of analytical purity lead compounds, the compounds have been crushed and mixed for 2 hours before go to XRD analysis. The lead species and concentration of sample 1# to 6# is shown as figure 2.

BA1 were from a Japanese mass burn MSW waste-to energy facility. The production condition was described. [1] **Sample FA1** were from a Chinese mass burn MSW waste-to energy facility. The facility has a rating of 1000 t/day. The combustor is comprised of 2 parallel units consisting of a vibratory feed hopper, vibratory grates, waterwall boiler/ economizer, two field ESP, dry lime scrubber, and fabric filters. ESP ash was collected on August 25,2003, from screw conveyors exiting the ESP ash hoppers. Samples were collected every 0.5 h over a 4-h period. The samples were composited, mixed, and subsampled 3-kg working sample for this study. **Sample B-0** was taken from the okagachi landfill site through bore sampling. The sample height is 20cm from the surface. An l-kg subsample was created from the 3-kg working sample using a cone and quartering procedure. Powders were stored under vacuum desiccation before using.

2.3 processes

A series of sieves was used to analyze the particle size distribution and separate the samples for the XRD analysis. XRD analysis were applied to the three parts 0-106 μ m, >2mm surface and particle size>2mm particles from the obtained sieved fractions.

Water washing was done at room temperature at a liquid to solid (L/S) ratio of 2 (L/kg). A total of 10.0 g of unfractionated ESP ash was added to 20 mL of Milli-Q type I1 water and stirred in an closed bottle, washing time is 30min and 60 min, the solid part was frozen dried after separated from the liquid through 0.1 μ m filter.

Detective Sensitivity

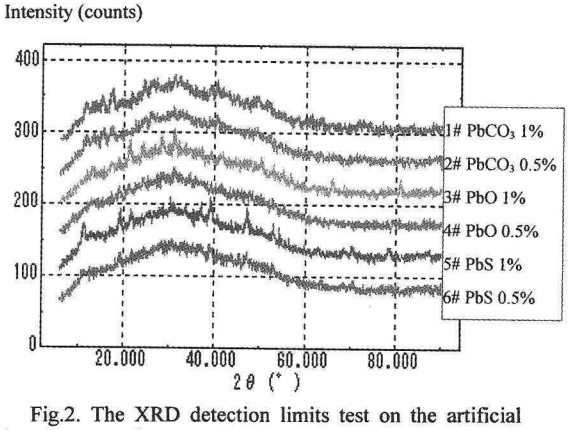

Fig.2. The XRD detection limits test on the artificial mineralogy samples

XRD instrument is sensitive to the concentration, concentration of 1% based on weight can be detected as shown in figure 2, however such weak signals are difficult for softwares to analyze. The only way to analyze this data is to appoint the element composition, but when it comes to environmental samples, this is almost impossible for the complex composition. For $PbCO_3$, the detection limit for software analysis is around 3%.

3. Water washing -based Mineralogy XRD analyses

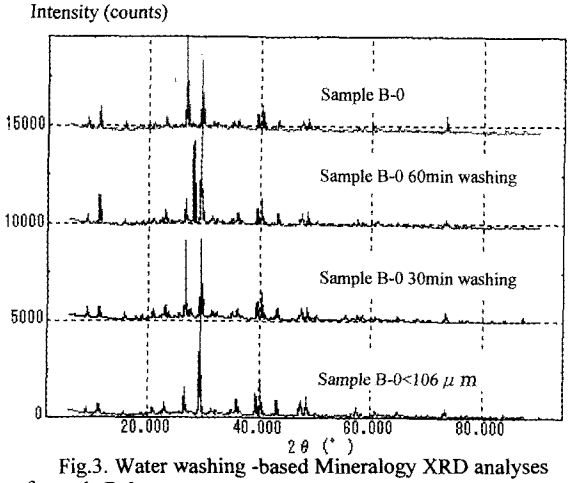

Fig.3. Water washing -based Mineralogy XRD analyses of sample B-0

After short time water washing, the concentration of soluble salts has decrease obviously as shown in Figure 3, so compare with sample B-0, the samples after washing shows more peaks of trace element, as a result, $PbTiO_3$ has been detected by JADE as a FOM value 10 to 12. And the composition of the ash has no big chemical change after washing which is important for the characterization of the sample, however in order to prove the detection of $PbTiO_3$, still need some additional analysis such as XAFS and so on.

4. Influence of particle size distribution on XRD analyses

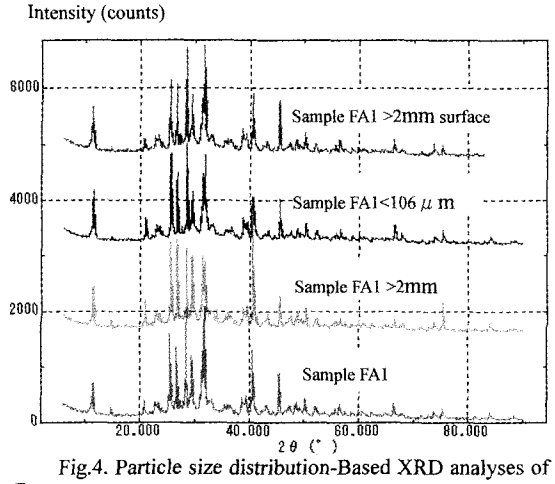

Fig.4. Particle size distribution-Based XRD analyses of FA1

Particle size distribution-Based mineralogy XRD analyses has no big influence to detect the chemical species of fly ash, which indicated fly ash is much more homogeneous than bottom ash, but the concentration of major species in the different particle range has a big difference.

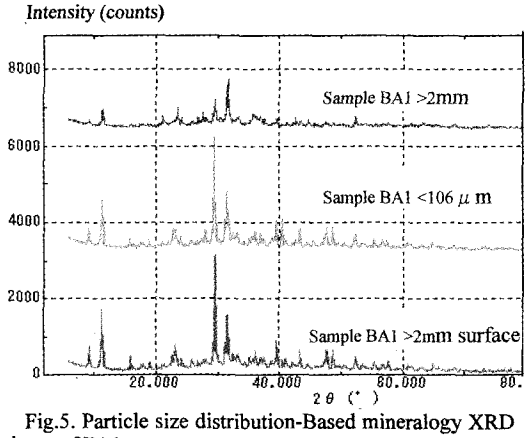

Fig.5. Particle size distribution-Based mineralogy XRD analyses of FA1

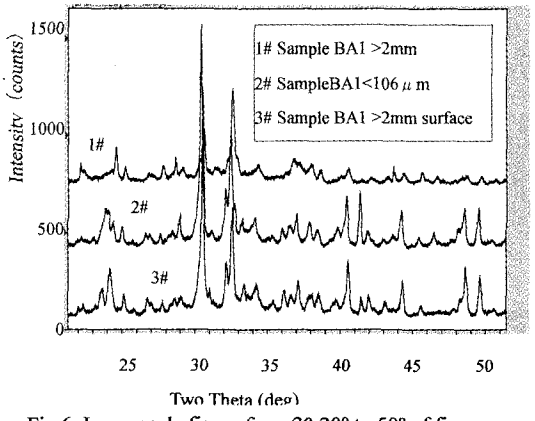

Fig.6. Large scale figure from 2θ 20° to 50° of figure

Particle size distribution-Based mineralogy XRD analyses is a reasonable method for the analysis of bottom ash because of the special particle structure[2]. As shown in figure 4, fine particles and the surface of the bottom ash contains more minor and trace components compare with particles diameter>2 mm. Which will provide much more important information for the identification of heavy metals species.

5. Conclusions

The advantages of water washing based and Particle size distribution-Based XRD analyses are important to analysis minor and trace components of MSWI residues when XRD is applied to analyze the environmental samples.

References

1. Chimenos, J.M., Segarra, M., Ferna ´ ndez, M.A., Espiell, F., 1998.Characterization of the bottom ash in municipal solid waste incinerator. Journal of Hazardous Materials 64, 211-222.

2. Kersten, M., Moor, H.Ch., Johnson, C.A., 1997. speciation of trace metals in leachate from MSWI bottom ash land.ll. Applied Geochemistry 12, 675-683.