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Single-particle characterization of municipal solid waste (MSW) ash particles using low-*Z* particle electron probe X-ray microanalysis

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Abstract

Environmentally benign treatment of municipal solid waste (MSW) ashes has been a worldwide issue since more countries are implementing incineration to reduce waste volume. A single-particle analytical technique, named low-*Z* particle electron probe X-ray microanalysis (low-*Z* particle EPMA) was applied to characterize MSW fly- and bottom-ash particle samples collected from two municipal incinerators in Korea. According to their chemical composition, many distinctive particle types were identified. For fly ash sample collected in one incinerator (sample S1), where lime slurry injection is used for acid–gas treatment, $CaCO_3$-containing particles (28.4%) are the most abundantly encountered, followed by carbonaceous (23.6%), SiO_2-containing (13.8%), NaCl-containing (13.1%), and iron-containing (10.5%) particles. For fly ash sample collected at the other incinerator (sample S2), NaCl-containing particles (40.4%) are the most abundantly encountered, followed by iron-containing (29.1%), carbonaceous (11.8%), $CaCO_3$-containing (2.2%), and SiO_2-containing (7.0%) particles.

For bottom ash sample collected at one incinerator (sample S3), iron-containing particles (46.6%) are the most abundantly encountered, followed by $CaCO_3$-containing (17.3%), carbonaceous (16.6%), and Si and/or Al oxide-containing (15.8%) particles. For bottom ash sample collected in the other incinerator (sample S4), iron-containing particles (63.4%) are also the most abundantly encountered, followed by carbonaceous (14.0%), $CaCO_3$-containing (10.0%), and Si and/or Al oxide-containing (6.1%) particles. Chemical compositions of the two bottom ash samples are not much different compared to those of the two fly ash samples.

It was demonstrated that the single-particle characterization using this low-*Z* particle EPMA technique provided detailed information on various types of chemical species in the MSW ash samples. In addition, the technique has advantage over conventional analytical techniques in the point that both crystalline- and glass-like ash particles can be analyzed at the same time.
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1. Introduction

Municipal solid wastes (MSW) are usually treated by means of burial, incineration, or recycling.

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Nowadays, incineration and recycling for MSW treatment are getting more attention since they can be useful in reducing volume of the wastes. Advantage of burning away MSW is its minimum requirement of landfills and its feasibility for good use of generated heat. However, incineration can produce exhaust gas and fly ash from stack, so that the prevention of those air pollutants should be ensured in incinerator plants. In general, generated particulates from incineration process are removed either by an electrostatic precipitator or a bag filter, and acidic gases such as HCl, SO_x, and NO_x are treated by basic solutions before being discharged into the air.

Effective and safe management of bottom and fly ashes resulting from the incineration of MSW essentially relies on our knowledge for their chemical composition. Therefore, many studies have been performed on chemical composition of MSW ashes and it is known that MSW ashes are mainly composed of metal oxides such as Al_2O_3, SiO_2, Na_2O, K_2O, MgO, CaO, and Fe_2O_3 and contain some heavy metals and dioxins (Hinton and Lane, 1991; Rausch et al., 1993; Kirby and Rimstldt, 1993; Taylor Eighmy et al., 1995). The analysis of MSW ashes originated from two municipal incinerators in Charlotte, USA, showed that MSW ash samples obtained by mixing bottom and flyashes are composed of 30% crystalline and 70% glass-like ash particles (Kirby and Rimstldt, 1993). By the use of various analytical methods, such as inductively coupled plasma-atomic emission spectrometry (ICP-AES), atomic absorption spectrophotometry (AAS), neutron activation analysis (NAA), and titration analysis, they found that the glass-like ash particles were mainly composed of SiO_2, Al_2O_3, CaO, Fe_2O_3, Na_2O, SO_4, K_2O, TiO_2, MgO, ZnO, P_2O_5, PbO, and Cl^-. X-ray diffraction (XRD) analysis was used for the characterization of crystalline ash particles, and were identified various chemical species such as Fe_2O_3 (hematite), $CaCO_3$ (calcite), NaCl (halite), SiO_2 (quartz), TiO_2 (rutile), $CaSO_4 \cdot 2H_2O$ (gypsum), $CaSO_4$ (anhydrite), FeO (wustite), KCl (sylvite), CaO (lime), $CaTiO_3$ (perovskite), Na_2HPO_4, Na_2SO_4, Al_2O_3, K_2ZnCl_4, and $(Fe,Mn,Ca)_3(PO_4)_2$. Fermo et al. (1999, 2000) analyzed fly ash samples using ICP-AES and they reported that major elements observed in the samples are Cl, Si, S, Ca, Na, Al, K, Zn, P, and Pb, and minor elements are Fe, Ti, Sn, Cu, Ba, and Sb. They also identified many different types of crystalline chemical species, such as NaCl (halite), KCl (sylvite), CaSO4 (anhydrite), $CaSO_4 \cdot 0.5H_2O$ (bassanite), $CaSO_4 \cdot 2H_2O$ (gypsum), $K_2Ca(SO_4)_2 \cdot H_2O$ (syngenite), Fe_2O_3 (hematite), $CaCO_3$ (calcite), TiO_2 (rutile), $(NH_4)_2SO_4$, NH_4NO_3, and various silicates. Speiser et al. (2000) analyzed some MSW bottom ashes using XRD and scanning electron microscopy/energy dispersion X-ray analysis (SEM/EDX) to identify $Ca_2Al_2SiO_7$, $(Ca,Mg,Fe^{2+},Al)_2(Si,Al)_2O_6$, $Ca(Mg,Fe)Si_2O_6$, Ca CO_3, SiO_2, $Fe^{2+}Fe_2^{3+}O_4$, Fe_2O_3, FeS_2, Fe_7S_8, NaCl, and KCl. Indeed, these studies clearly reveal their complexity of chemical compositions of MSW ashes. However, the characterization of chemical compositions of MSW ashes is not simple to carry out; XRD analysis can be useful to identify chemical species of crystalline particles, and yet just 30% of MSW ash particles are in the crystalline form and it is difficult to obtain quantitative information using XRD. When the other analytical techniques such as ICP-AES and AAS are used for the characterization of MSW ash particles, chemical elemental concentrations can be obtained, and yet it is difficult to identify chemical forms of those chemical elements.

In this study, a single-particle analytical technique, named low-*Z* particle electron probe X-ray microanalysis (low-*Z* particle EPMA), is applied to characterize chemical compositions of MSW ash particles. The low-*Z* particle EPMA allows the determination of the concentration of low-*Z* elements such as carbon, nitrogen and oxygen, as well as the elements which are observed using conventional EPMA, in individual particles of micrometer size. By the application of a quantification method, which employs Monte Carlo simulation combined with successive approximations, quantitative specification of the chemical compositions can be done (Ro et al., 1999, 2000; Szaloki et al., 2000; Osán et al., 2000). Up to now, the low-*Z* particle EPMA has been successfully applied to characterize various types of airborne particles (Ro et al., 2001a,b, 2002, 2005; Hwang and Ro, 2005). And it is expected that the single-particle characterization using this low-*Z* particle EPMA technique can provide detailed information on various types of chemical species in MSW ash samples. In addition, this technique has advantage over conventional analytical techniques in the point that both crystalline and glass-like ash particles can be analyzed at the same time.

2. Experimental section

2.1. Samples

Fly- and bottom-ash samples were collected in two incinerators located in Bucheon, Korea. The

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