Evaluation of Thermal Treatments for Elutriated Mixed Incinerator Ashes. Part 1: Co-Incineration with Laboratory Waste

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ABSTRACT

This study describes polychlorinated dibenzo-p-dioxin and dibenzofuran (PCDD/F) behavior during the co-incineration of elutriated mixed incinerator ashes with laboratory waste. The input and output materials during elutriation and incineration processes were sampled and analyzed using a high-resolution gas chromatography/high-resolution mass spectrometry assay. The elutriation process mainly washed out soluble salts and thus resulted in an increase in the level of PCDD/F in the mixed incinerator ashes. After co-incineration with laboratory waste, the PCDD/F concentration of the flue gas met the regulated standard in Taiwan, and the PCDD/F mainly existed as a particulate phase. However, the PCDD/F levels in fly ashes that included elutriated ash in the incinerator were higher than those of fly ashes without it. The co-incineration output-mass/input-mass ratio of elutriated ash with laboratory waste was 0.34. According to the X-ray diffraction analysis results and scanning electron microscopy images, the main crystalline phase of the fly ashes was NaCl. The NaCl came from the reaction of HCl in the flue gas and the NaOH injected in the quenching tower after treatment in the co-incineration system.

Keywords: Air pollution control devices; Fly ash; Co-incineration; Elutriation.

INTRODUCTION

Laboratory waste (LW) from universities and high schools is a problem due to the variety of chemicals and trace amounts left in the environment. The Ministry of Education of Taiwan has thus subsidized the Sustainable Environment Research Center (SERC) at National Cheng Kung University, Tainan, Taiwan, to build a treatment plant to dispose of LW. This LW treatment plant includes three systems: a physicochemical treatment system, an incineration system, and a plasma melting system. The physicochemical treatment system focuses on the treatment of liquid waste, which might contain heavy metals, acids, alkali compounds, cyanide, etc. The incineration system aims to treat organic solid/liquid LW. This kind of LW includes combustible waste from medical and biochemical experiments, which usually has Cl levels > 10% (Wu et al., 2014). Polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDD/Fs), which are unintentional by-products in the incineration process, are generated through a series of complex reactions that involve Cl, especially in the incinerator start-up period (Lin et al., 2014). Therefore, the PCDD/F content in the ashes generated in an LW incinerator are five to ten times higher than those in municipal solid waste (MSW) incinerators (Wang et al., 2010; Liao et al., 2014). Similar levels have been found in hazardous waste incinerators due to the complicated composition of the input materials (Coutinho et al., 2006; Wang et al., 2014). High PCDD/F exposure is a health risk (Tsai et al., 2014).

The ash and sludge from the physicochemical treatment system and incineration system are sent to the plasma melting system for vitrification with cullet and silica gel (Kuo et al., 2010). The hazardous materials are transformed via vitrification into stable slag, which can be made into a composite with polyester resin (Kuo et al., 2011). Vitrification can be used to stabilize hazardous metals,
destroy organic pollutants, and recover valuable metals (Kuo, 2012; Huang et al., 2013). However, its operational cost is much higher than that of other technology for disposing of hazardous materials. Therefore, reducing the cost of the final disposal of hazardous materials is an important goal of the SERC. The objective of the present study is to evaluate the treatment effect of LW incineration of fly ashes. The fly ashes were first elutriated to wash out NaCl in order to avoid the formation of HCl during the sequential thermal processes. The elutriated ash was then treated using two thermal approaches: co-incineration with original laboratory waste (OLW) and plasma melting.

For the plasma melting process, the effect of decomposition of PCDD/F is undoubtedly better than that of co-incineration, but the operational cost is also much higher in comparison with co-incineration. Therefore, determining which of these two methods is most suitable to treat fly ashes is worthy of investigation. This research investigates the operational characteristics of the two approaches and is divided into two parts. Part 1 investigates the emission characteristics of PCDD/Fs when elutriated ash (ELA) is incinerated with OLW. Part 2 focuses on the emission characteristics of PCDD/Fs in the plasma melting system and compares the performance of the two technologies.

Here, either the PCDD/F and Cl content or the concentration of the input and output materials in the elutriation and incineration processes are measured. The distribution behavior of PCDD/Fs during the two processes is investigated. In addition, the PCDD/F profiles and the surface characteristics of ashes are also analyzed.

METHODS

**Incineration System Processes**

The LW treatment plant set up by the SERC is located in Annan District, Tainan City, Taiwan. The incineration system in this plant has a capacity of 120 kg h⁻¹. Its flow diagram is shown in Fig. 1. In this system, solid LW is fed through a feeding unit into an incinerator with two chambers. In the primary incineration chamber, the combustion temperature is 900°C, and the residue is emitted from the bottom of the incineration chamber as bottom ash. The flue gas is then heated to 1150°C by a diesel combustor, which is installed in the secondary incineration chamber to destroy PCDD/Fs, CO, and other persistent organics. After being emitted from the incineration chambers, the flue gas passes through a series of air pollution control devices (APCDs), namely the primary quenching tower, the secondary quenching tower, a fabric filter with injection of activated carbon, and a wet scrubber, and then is emitted to the ambient air.

First, a solution of 10 wt.% NaOH is injected into the primary and secondary quenching towers to reduce the temperature of the flue gas from 1050°C to 450°C and 150°C, respectively, to suppress the formation of PCDD/Fs (Tuan et al., 2012). Activated carbon is injected within the flue gas to adsorb PCDD/Fs and other gaseous organic pollutants. After passing through the fabric filters, the activated carbon and particulates are trapped as fabric filter ash (FFA). The wet scrubber removes the residual acid gas.
HxCDD, and 13C12-1,2,3,4,7,8,s9-HpCDF. After a 4-h metals and PCDD/Fs effectively (Chang et al., 2012). The ashes of these APCD units are collected and stored together as raw mixed ash (RMA). It is not suitable to solidify the RMA with cement because of its high levels of Cl and PCDD/Fs. It is therefore sent to the plasma melting system for final disposal (Kuo et al., 2011).

**Elutriation, Mixing, and Re-Incineration Processes**

To reduce the high operational cost associated with the plasma melting system, RMA is elutriated using water with a liquid to solid ratio of 2:1 to dissolve soluble salts, which are mainly NaCl (Chang et al., 2012). The wastewater is pumped into the physicochemical treatment system for further treatment. The ELA still has a high level of Cl even after being elutriated, and thus a large amount of PCDD/Fs will be formed if excess ELA is added. Therefore, the mass mixing ratio of the ELA to OLW was set as 1:20 in this study. The mixed input material was fed into the incineration system at a rate of 120 kg h⁻¹ to evaluate the feasibility of treating ELA using incineration.

**Sampling of Flue Gas and Solid Specimens**

The sampling of the flue gas for the PCDD/F analysis followed the standard procedure given in NIEA A807.74C, issued by the Environmental Analysis Laboratory of the Taiwan Environmental Protection Administration (Environmental Analysis Laboratory, 2010). The flue gas was collected isokinetically with a sampling probe, which was cleaned with acetone, dichloromethane, and toluene sequentially before sampling. During the sampling procedure, no sealing grease was added to the components of the sampling train in order to meet the modified method 5 requirement of the US Environmental Protection Agency (EPA). Before the gas-phase sampling module, a quartz filter was used to collect the particulate phase. The quartz filter was conditioned for 24 h to ensure consistent moisture content, and it was weighed both before and after the sampling procedure. The gas-phase sampling train cartridge was filled with about 30 g of adsorbent (Amberlite XAD-2®) in advance and was spiked with PCDD/F surrogate standards pre-labeled with isotopes, namely 37Cl-2,3,7,8-TCDD, 13C17-1,2,3,4,7,8-HxCDD, 13C17-2,3,4,7,8-PoCDF, 13C17-1,2,3,4,7,8-HxCDF, and 13C17-1,2,3,4,7,8,9-HpCDF. After a 4-h sampling procedure, all samples were preserved at 10°C during transportation to the laboratory for further analysis. The flue gas and solid and liquid specimens, RMA, ELA, OLW, bottom ash (BTA), primary quenching tower ash (PQA), secondary quenching tower ash (SQAA), FFA, wet scrubber ash (WSA), and elutriated wastewater (ELW), were sampled in triplicate to determine the relative standard deviation (RSD). The specimens were then extracted using the procedure discussed below for further PCDD/F analysis.

**PCDD/F Pretreatment and Analysis**

The pretreatment and analysis of PCDD/Fs followed the procedure given in US EPA Modified Methods 23 and 1613. The specimens were all spiked with a 13C12-labeled internal standard in advance. In the pretreatment procedure, Soxhlet extraction with n-hexane, nitrogen-blowing concentration, the cleanup and fractionation procedure, and nitrogen-blowing re-concentration were sequentially conducted before the PCDD/F analysis. To determine the PCDD/F concentration, high-resolution gas chromatography (HRGC; Hewlett Packard 6970 Series gas, CA, USA) and high-resolution mass spectrometry (HRMS; Micromass Autospec Ultima, Manchester, UK) were conducted to analyze the final eluate (about 1 mL). More procedural details can be found in a previous study (Wang and Chang-Chien, 2007).

**Examination of Surface and Crystalline Characteristics**

Scanning electron microscopy (SEM, JEOL JSM-7000) with energy-dispersive X-ray spectroscopy (EDS) was used to examine the ashes to evaluate their surface characteristics. Initially, the solid specimens were pulverized and screened using a sieve with a mesh size smaller than 74 μm. The screened, powdery specimens were adhered to a metallic plate using silver adhesive tape and then were coated with Au film via metal ion sputtering. The Au-coated specimens were scanned using electron beams accelerated at a voltage of 25 kV. The magnification factor of the observation was set at 10,000×. The screened powder was then analyzed using X-ray powder diffraction (XRD; Geigerflex 3063) to determine the crystalline phases. The powder was measured in the 20 range of 10–60° using Ni-filtered Cu-Kα radiation with an angular speed of 4° min⁻¹.

**RESULTS AND DISCUSSION**

**PCDD/F Characteristics of Input Materials**

Table 1 shows the content and distribution of PCDD/Fs and Cl in the materials during the elutriation process. The PCDD/F content levels in the RMA were 157 in total ng g⁻¹ and 13.3 ng I-TEQ g⁻¹. After the elutriation process, the PCDD/F levels in the ELA were elevated to 234 in total ng g⁻¹ and 20.3 ng I-TEQ g⁻¹, nearly double those for the RMA. According to the PCDD/F mass distribution behavior, < 0.001% of the PCDD/Fs was discharged from the ELW and > 99.999% of the PCDD/Fs stayed in the ELA. The Cl level in the RMA went up to 18.1% and decreased to 2.36% after the elutriation process. The ELW had a Cl concentration of 84,600 mg L⁻¹, and the elutriation process washed out 93.5% of the Cl from the RMA. The dissolution of salts during the elutriation process resulted in a great mass reduction (40.5%) in the RMA and an elevation of PCDD/F content (49%).

Table 2 shows the PCDD/F and Cl content in the materials during the mixing process. The ELA and OLW were mixed at a mass ratio of 1:20 to serve as the input material. The OLW had a PCDD/F content of 2.41 ng I-TEQ g⁻¹; the PCDD/F level in the input materials was notably elevated to 3.26 ng I-TEQ g⁻¹ due to the contribution of the ELA. The Cl level in the ELA was only 2.36%, which was much lower than that in the OLW (11.4%), and thus, the level in the feed materials was slightly reduced to 11.0%.

(HCl) and particulates sequentially (Lee et al., 2004). The flue gas is heated to 120°C to avoid the formation of white steam before being emitted into the ambient air. The APCDs in the incineration systems have been proven to remove heavy metals and PCDD/Fs effectively (Chang et al., 2012).
PCDD/F Emission Behavior of Output Materials

Table 3 shows the pollutant concentration of the flue gas. The emission of gas pollutants, SOx, NOx, CO, and HCl, were 1.32, 43.0, 4.04, and 1.67 ppm, respectively. The heavy metals in the flue gas were Pb (0.375 mg Nm⁻³), Cd (0.0272 mg Nm⁻³), and Hg (0.0820 mg Nm⁻³). The gas and particulate PCDD/F phases were 0.004 and 0.410 ng I-TEQ Nm⁻³, respectively, accounting for 0.414 ng I-TEQ Nm⁻³. The particulate phase played the major role (99.0%) in the flue gas. The flue gas emission of the pollutants met all the regulated standards in Taiwan (TEPA, 2005).

Table 4 shows PCDD/F content of all of the ashes. The PCDD/F content in the BTA was 0.401 ng I-TEQ g⁻¹, which is an order of magnitude higher than the value reported for municipal solid waste (MSW) incinerators (0.0138–0.0787 ng I-TEQ g⁻¹) in a previous study (Wang et al., 2010). The annual sampling and analysis data for MSW in Taiwan show that the Cl content ranged from 0.05% to 0.17% from 2003 to 2014 (TEPA, 2015). The Cl content in the feed materials (11.0%) was nearly two orders of magnitude higher than that for MSW. Previous studies have reported that high Cl levels in the input materials enhance PCDD/F formation during the incineration process (Aurell et al., 2009; Liao et al., 2014). The Cl in the input materials has been shown to transform into chlorinated organic compounds or metal chlorides and to serve as a catalyst for PCDD/F formation (Hatanaka et al., 2005; Chin et al., 2012).

The PCDD/F content levels in the fly ashes were even higher than those in the BTA. The PCDD/F levels in the PQA, SQA, FFA, and WSA were 10.9, 25.3, 8.75, and 3.24 ng I-TEQ g⁻¹, respectively, which was much higher than the regulated standard for general solid waste and even higher than those in the bottom ash of medical waste incinerators (Gidarakos et al., 2009; TEPA 2012). The flue gas was quenched from 1050 to 450°C in the primary quenching tower, and the FQA PCDD/Fs were formed via a homogeneous pyrogenic route that took place in the gas phase between 500 to 800°C (Zhang et al., 2016). The flue gas was then quenched to 150°C through the use of the secondary quenching tower and the PCDD/Fs were generated via a low temperature catalytic (de novo synthesis), which was catalyzed with metal chloride (Addink and Olie, 1995). Both routes to the formation of PCDD/Fs were enhanced due to the high Cl levels of FQA (8.69%) and SQA (9.70%). The de novo synthesis was regarded as the major source of PCDD/Fs, and, thus, the SQA had the highest PCDD/F level (25.3 ng I-TEQ g⁻¹) among the ashes under investigation. In the fabric filter, the flue gas temperature was about 150°C, and de novo synthesis did not govern the formation of PCDD/F. However, the memory effect caused by aged bag filters would still allow generation of PCDD/Fs, and the PCDD/F level of the FFA was 8.75 ng I-TEQ g⁻¹ (Li et al., 2011; Zimmermann et al., 2001). After passing through the fabric filter, the residual particulate was washed with the wet scrubber and the PCDD/F level (3.24 ng I-TEQ g⁻¹) was only half that in the FFA due the lack of the memory effect. The results show that fly ashes cannot be buried in landfills as general industrial waste even after solidification with cementation. In addition, the PCDD/F levels in fly
Table 2. PCDD/F and Cl content in input materials.

<table>
<thead>
<tr>
<th></th>
<th>ELA</th>
<th>OLW</th>
<th>Feed materials</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCDD/F content (ng I-TEQ g⁻¹), RSD (%)</td>
<td>20.3, 12.4</td>
<td>2.41, 8.91</td>
<td>3.26, 17.3</td>
</tr>
<tr>
<td>Cl content (%), RSD (%)</td>
<td>2.36, 12.7</td>
<td>11.4, 12.4</td>
<td>11.0, 12.4</td>
</tr>
</tbody>
</table>

Table 3. Pollutant flue gas concentration.

<table>
<thead>
<tr>
<th>Item</th>
<th>Concentration (mg Nm⁻³)</th>
<th>RSD (%)</th>
<th>Regulated standard</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particulate phase</td>
<td>2.00</td>
<td>12.1</td>
<td>180</td>
</tr>
<tr>
<td>SO₅ (ppm)</td>
<td>1.32</td>
<td>15.6</td>
<td>180</td>
</tr>
<tr>
<td>NOₓ (ppm)</td>
<td>43.0</td>
<td>19.4</td>
<td>180</td>
</tr>
<tr>
<td>CO (ppm)</td>
<td>4.04</td>
<td>10.5</td>
<td>220</td>
</tr>
<tr>
<td>HCl (ppm)</td>
<td>1.67</td>
<td>5.1</td>
<td>60</td>
</tr>
<tr>
<td>Pb (mg Nm⁻³)</td>
<td>0.375</td>
<td>16.4</td>
<td>0.5</td>
</tr>
<tr>
<td>Cd (mg Nm⁻³)</td>
<td>0.0272</td>
<td>18.5</td>
<td>0.04</td>
</tr>
<tr>
<td>Hg (mg Nm⁻³)</td>
<td>0.0820</td>
<td>13.1</td>
<td>0.1</td>
</tr>
<tr>
<td>Gas phase PCDD/Fs</td>
<td>0.058</td>
<td>24.3</td>
<td>--</td>
</tr>
<tr>
<td></td>
<td>0.004</td>
<td>31.4</td>
<td>--</td>
</tr>
<tr>
<td>Particulate phase</td>
<td>7.10</td>
<td>17.7</td>
<td>--</td>
</tr>
<tr>
<td></td>
<td>0.410</td>
<td>18.5</td>
<td>0.5</td>
</tr>
</tbody>
</table>

1. Gas phase + Particulate phase = 0.5 I-TEQ ng Nm⁻³

Table 4. PCDD/F and Cl content in the ashes.

<table>
<thead>
<tr>
<th>Item</th>
<th>Bottom ash</th>
<th>PQA</th>
<th>SQA</th>
<th>FFA</th>
<th>WSA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Generating mass of ash (kg hr⁻¹)</td>
<td>24.2</td>
<td>6.13</td>
<td>1.21</td>
<td>3.62</td>
<td>0.617</td>
</tr>
<tr>
<td>PCDD/Fs in total mass (ng g⁻¹), RSD (%)</td>
<td>27.5, 20.3</td>
<td>135, 18.6</td>
<td>438, 24.4</td>
<td>108, 30.7</td>
<td>37.5, 8.97</td>
</tr>
<tr>
<td>PCDD/Fs in I-TEQ (ng g⁻¹), RSD (%)</td>
<td>0.401, 15.4</td>
<td>10.9, 14.8</td>
<td>25.3, 16.5</td>
<td>8.75, 28.9</td>
<td>3.24, 25.9</td>
</tr>
<tr>
<td>Cl (%), RSD (%)</td>
<td>2.63, 14.8</td>
<td>8.69, 12.7</td>
<td>9.70, 13.5</td>
<td>18.1, 9.17</td>
<td>7.73, 18.4</td>
</tr>
</tbody>
</table>

Table 5. PCDD/F distribution of output materials.

<table>
<thead>
<tr>
<th>Output materials</th>
<th>Bottom ash</th>
<th>Fly ashes</th>
<th>Flue gas</th>
<th>O/I ratio of whole system</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass distribution</td>
<td>BTA</td>
<td>PQA</td>
<td>SQA</td>
<td>FFA</td>
</tr>
<tr>
<td>PCDD/Fs (%)</td>
<td>27.2</td>
<td>33.4</td>
<td>21.6</td>
<td>16.0</td>
</tr>
<tr>
<td>TEQ(I-TEQ) (%)</td>
<td>6.9</td>
<td>46.6</td>
<td>21.7</td>
<td>22.5</td>
</tr>
</tbody>
</table>

ashes that included the ELA in the incinerator were higher than those in the fly ashes without it, indicating that re-incineration of ELW will increase the PCDD/F levels in fly ashes (Liao et al., 2014).

PCDD/F Distribution in Output Materials

Table 5 shows the PCDD/F distribution in the output materials, namely BTA, PQA, SQA, FFA, WSA, and flue gas. The PCDD/F total mass and I-TEQ show different distribution patterns. PCDD/Fs mainly were present in the PQA (33.4%), BTA (27.2%), SQA (21.6%), and FFA (16.0%). The fraction of PCDD/Fs discharged from the flue gas was only 0.8%. For PCDD/Fs in terms of I-TEQ, the mass fraction in BTA (6.9%) was much smaller than that in total mass (27.2%). The distribution patterns show that the PCDD/F species in the BTA were less toxic than those in the fly ashes. According to the PCDD/F distribution in terms of I-TEQ, the three major fractions were FFA (22.5%), PQA (46.6%), and SQA (21.7%), accounting for 90.9% of the total. The output-mass/input-mass (O/I) ratio was 0.34 in both the total mass and I-TEQ, indicating that co-incineration of ELA and a 665 reduction in PCDD/Fs (including the amount generated in the incineration and quenching process and originally exiting in the incinerator with the input-materials).

PCDD/F Patterns in Output Materials

Fig. 2 shows the PCDD/F profiles in terms of the I-TEQ of the ashes. BTA, 1,2,3,4,6,7,8-HpCDF (33.2%), OCDF (28.5%), and OCDD (25.7%) were the major components of the PCDD/F mass. The fractions of other PCDD/F species in the BTA were all smaller than 5%. The PCDD/F profiles of fly ashes were somewhat similar. 1,2,3,4,6,7,8-HpCDF (25 to 30%) was the most abundant. The fractions of other PCDD/F species, namely 1,2,3,4,7,8-HxCDF, 1,2,3,6,7,8-HxCDF, 2,3,4,6,7,8-HxCDF, 1,2,3,4,6,7,8-HpCDD, and OCDD, ranged from 5 to 10%. For the flue gas (see Fig. 3), the PCDD/F profile for the particulate phase was similar to
Fig. 2. PCDD/F profile in (a) BTA, (b) PQA, (c) SQA, (d) FFA, and (e) WSA.
that of fly ashes because the same source was used. However, the gas phase showed a completely different pattern. The major species was OCDD (37.7%). The fractions of 2,3,7,8-TeCDF, 1,2,3,4,6,7,8-HpCDF, OCDF, and 1,2,3,4,6,7,8-HpCDD all ranged from 5 to 10%.

**Crystalline and Surface Characteristics of Output Materials**

Fig. 4 shows the XRD patterns of the incinerator ashes. The crystalline phases in the BTA were mainly NaCl and Na$_2$SO$_4$. Cations and anions are commonly used reagents in experiments, and thus it was reasonable to find them in the BTA. The crystalline patterns of the fly ashes were identical. The XRD pattern of the fabric filter ash was thus taken as an example. The main crystalline phase of the NaCl in all of the ashes was only NaCl, which was formed via the following equation:

\[
\text{Org-Cl + H}^- \rightarrow \text{HCl (in the incinerator)} \quad (1)
\]

\[
\text{HCl + NaOH} \rightarrow \text{NaCl} + \text{H}_2\text{O (in the primary and secondary quenching towers)} \quad (2)
\]

Fig. 5 shows the SEM images of the ashes. Fig. 5(a) shows that the BTA had bulk crystals and some powder on the surface, which might be NaSO$_4$ and NaCl, respectively. The ashes in Figs. 5(b), 5(c), and 5(e) show similar surface characteristics; the powdery, porous structure and powdery crystals should be NaCl, according to the XRD patterns. The FFA structure was bulky with powdery NaCl (see Fig. 5(d)), which was completely different from that of the other ashes. However, the XRD pattern of the FFA shows NaCl as the only crystalline phase. According to the SEM images and XRD analysis, the bulky structure was amorphous and may be activated carbon, which was injected before the flue gas passing through the fabric filter.
CONCLUSION

Part 1 of this study was an evaluation of the treatment of RMA with high PCDD/F and Cl content using elutriation and co-incineration processes. The elutriation process washed out 93.5% of the Cl from the RMA and reduced the Cl level from 18.1% to 2.36%. However, most of the PCDD/Fs stayed in the RMA and became further concentrated due to mass reduction. After co-incineration with OLW, the PCDD/F emission of the flue gas was 0.414 ng I-TEQ Nm⁻³, which met the regulated standard in Taiwan. The PCDD/F content levels in the bottom ash and fly ashes were much higher than those for MSW incinerators due to the high Cl level in the input materials (11.0%). Even after solidification with cementation, these ashes should be regarded as hazardous materials.

The PCDD/F distribution in the output materials shows that PCDD/Fs are mainly discharged from fly ashes and that the PCDD/F species in fly ashes are more toxic than those in BTA. The O/I ratio (0.34) indicated that co-incineration with ELA reduced PCDD/Fs by 66%. The main crystalline phase of all ashes was NaCl, which formed in the primary and secondary quenching towers. Consistent results were observed in the SEM and XRD analysis.

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Fig. 5. SEM images of (a) BTA, (b) PQA, (c) SQA, (d) FFA, and (e) WSA.


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