Phosphorus recovery and reuse potential from smouldered sewage sludge ash

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Supplementary Materials

Phosphorus Recovery and Reuse Potential from Smouldered Sewage Sludge Ash

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\textsuperscript{*}Corresponding author
S1. Supplementary Information on Drum Reactor Experiments

Figure S1-1: Schematic of smouldering reactor set-up.

Loading Procedure

The sludge/sand tests consisted of sludge as the fuel embedded in coarse silica sand as the porous media. The co-smouldering tests had a fuel mixture of sludge and woodchips, where the woodchips dually acted as the porous media. The experimental set-ups followed the same procedures for both tests, except for two minor differences for the co-smouldering test. An additional layer of coarse silica sand was added to the base of the reactor, 0.6-2.5 cm thick, to provide insulation between the hot smouldering mixture and the supporting screen. A subsequent layer of woodchips, 1-2 cm thick, was added above the clean sand layer to assist with ignition. Moreover, a clean sand cap was added on top of the sludge/sand pack to lower the exiting temperature for safety purposes.
The sludge mixture was loaded in small batches that were gently lowered to the base of the reactor. Furthermore, to achieve a more uniform density of mixture, while still ensuring material homogeneity, the surface was leveled instead of tamped.

![Temperature profile for the sludge-sand experiment](image)

Figure S1-2: Temperature profile for the sludge-sand experiment, a self-sustaining smouldering experiment with a 3.81% moisture content sludge in a fixed bed with 25.5 g/g sand/sludge mass ratio. Plenum, centreline, and wall thermocouples are presented. Note the air flux was changed at 190, 238, 288, 290, and 296 minutes.
Figure S1-3: Temperature profile for the sludge-woodchips experiment, a self-sustaining smouldering experiment with a 75% moisture content sludge in a fixed bed with 0.4/0.3/1 g/g/g woodchips/extra water/sludge mass ratio. Plenum, centreline, and wall thermocouples are presented. Note the air flux was changed at 112 minutes.
S2. Supplementary Information on Material Characterization and Mass Balances

Table S2-1: Total elemental concentrations

<table>
<thead>
<tr>
<th>Element</th>
<th>Concentration (mg/kg-dry virgin material) ± SE&lt;sup&gt;a&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Sludge</td>
</tr>
<tr>
<td>Al</td>
<td>5400 ± 300</td>
</tr>
<tr>
<td>Cd</td>
<td>2.6 ± 0.2</td>
</tr>
<tr>
<td>Co</td>
<td>3.8 ± 0.3</td>
</tr>
<tr>
<td>Cr</td>
<td>120 ± 10</td>
</tr>
<tr>
<td>Cu</td>
<td>480 ± 40</td>
</tr>
<tr>
<td>Fe</td>
<td>53000 ± 5000</td>
</tr>
<tr>
<td>Mg</td>
<td>4200 ± 400</td>
</tr>
<tr>
<td>Mn</td>
<td>260 ± 30</td>
</tr>
<tr>
<td>Mo</td>
<td>22 ± 2</td>
</tr>
<tr>
<td>Ni</td>
<td>47 ± 6</td>
</tr>
<tr>
<td>P</td>
<td>26000 ± 3000</td>
</tr>
<tr>
<td>Pb</td>
<td>110 ± 10</td>
</tr>
<tr>
<td>Zn</td>
<td>630 ± 90</td>
</tr>
</tbody>
</table>

<sup>a</sup> Standard error calculated as \( \frac{\sigma}{\sqrt{n}} \)

<sup>b</sup> The sludge ash is considered all materials from smouldering experiments of sand mixed with sludge finer than 0.250 mm (< #60 sieve)

<sup>c</sup> The post-treatment ash from smouldering experiments consisting of sludge mixed with woodchips

<sup>d</sup> Woodchips ash generated in the lab according to ASTM-D2866-11

<sup>e</sup> The sand is considered all materials from smouldering experiments of sand mixed with sludge coarser than 0.250 mm (> #60 sieve)
Table S2-2: Mass balance of sludge and sand experiment.

<table>
<thead>
<tr>
<th>Element</th>
<th>Total Elemental Contents $^a$ ± SE $^*$ (mg)</th>
<th>Total Mass in Fraction $^b$ ± SE $^*$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Sludge</td>
<td>Sand</td>
</tr>
<tr>
<td>Al</td>
<td>45000 ± 2500</td>
<td>67000 ± 22000</td>
</tr>
<tr>
<td>Cd</td>
<td>21 ± 1.7</td>
<td>13 ± 2.2</td>
</tr>
<tr>
<td>Co</td>
<td>31 ± 2.5</td>
<td>34 ± 2.2</td>
</tr>
<tr>
<td>Cr</td>
<td>1000 ± 83</td>
<td>930 ± 150</td>
</tr>
<tr>
<td>Cu</td>
<td>4000 ± 330</td>
<td>780 ± 450</td>
</tr>
<tr>
<td>Fe</td>
<td>440000 ± 42000</td>
<td>140000 ± 44000</td>
</tr>
<tr>
<td>Mg</td>
<td>35000 ± 3300</td>
<td>8100 ± 2900</td>
</tr>
<tr>
<td>Mn</td>
<td>2200 ± 250</td>
<td>910 ± 220</td>
</tr>
<tr>
<td>Mo</td>
<td>180 ± 17</td>
<td>41 ± 66</td>
</tr>
<tr>
<td>Ni</td>
<td>390 ± 50</td>
<td>370 ± 44</td>
</tr>
<tr>
<td>P</td>
<td>210000 ± 25000</td>
<td>63000 ± 11000</td>
</tr>
<tr>
<td>Pb</td>
<td>920 ± 83</td>
<td>1900 ± 48</td>
</tr>
<tr>
<td>Zn</td>
<td>5200 ± 740</td>
<td>1700 ± 660</td>
</tr>
</tbody>
</table>

$^a$ Calculated as $(\text{elemental concentration} \frac{mg}{kg} - \text{dry matter}) \times (\text{mass of fraction in reactor} [kg - \text{dry matter}])$

$^b$ Calculated as $(\text{elemental content} [mg]) \div (\text{elemental content in virgin material} [mg]) \times 100\%$

$^c$ The sand is considered all materials from smouldering experiments of sand mixed with sludge coarser than 0.250 mm (> #60 sieve)

$^d$ The sludge ash is considered all materials from smouldering experiments of sand mixed with sludge finer than 0.250 mm (< #60 sieve)

$^e$ Calculated as $(\text{elemental content} [mg])_{\text{sand}} + (\text{elemental content} [mg])_{\text{ash}}$

$^*$ The standard error was calculated from the uncertainties from each calculation added in quadrature
<table>
<thead>
<tr>
<th>Element</th>
<th>Total Elemental Contents ± SE (mg)</th>
<th>Virgin Materials</th>
<th>Mixed Ashes</th>
<th>Elemental Retention (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Sludge</td>
<td>Woodchips</td>
<td>Theoretical Maximum</td>
</tr>
<tr>
<td>Al</td>
<td>53600 ± 3000</td>
<td>16300 ± 6200</td>
<td>70000 ± 6900</td>
<td>10600 ± 400</td>
</tr>
<tr>
<td>Cd</td>
<td>26 ± 2</td>
<td>4.0 ± 3</td>
<td>30 ± 5</td>
<td>4.4 ± 0.5</td>
</tr>
<tr>
<td>Co</td>
<td>38 ± 3</td>
<td>5.8 ± 5</td>
<td>44 ± 6</td>
<td>12 ± 2</td>
</tr>
<tr>
<td>Cr</td>
<td>1200 ± 100</td>
<td>480 ± 30</td>
<td>1700 ± 100</td>
<td>240 ± 8</td>
</tr>
<tr>
<td>Cu</td>
<td>4800 ± 400</td>
<td>220 ± 60</td>
<td>5000 ± 400</td>
<td>1300 ± 30</td>
</tr>
<tr>
<td>Fe</td>
<td>527000 ± 50000</td>
<td>30600 ± 8300</td>
<td>557000 ± 50000</td>
<td>80200 ± 4200</td>
</tr>
<tr>
<td>Mg</td>
<td>420000 ± 4000</td>
<td>23300 ± 4200</td>
<td>65000 ± 6000</td>
<td>18700 ± 400</td>
</tr>
<tr>
<td>Mn</td>
<td>2600 ± 300</td>
<td>2900 ± 500</td>
<td>5400 ± 600</td>
<td>1400 ± 40</td>
</tr>
<tr>
<td>Mo</td>
<td>220 ± 20</td>
<td>20 ± 20</td>
<td>240 ± 50</td>
<td>27 ± 1</td>
</tr>
<tr>
<td>Ni</td>
<td>470 ± 60</td>
<td>180 ± 30</td>
<td>650 ± 70</td>
<td>110 ± 3</td>
</tr>
<tr>
<td>P</td>
<td>2580000 ± 30000</td>
<td>6900 ± 1300</td>
<td>265000 ± 30000</td>
<td>57300 ± 100</td>
</tr>
<tr>
<td>Pb</td>
<td>1100 ± 100</td>
<td>990 ± 140</td>
<td>2100 ± 200</td>
<td>140 ± 18</td>
</tr>
<tr>
<td>Zn</td>
<td>6300 ± 900</td>
<td>910 ± 340</td>
<td>7200 ± 1000</td>
<td>1600 ± 60</td>
</tr>
</tbody>
</table>

\(^a\) Calculated as \(\left(\text{elemental concentration} \left[ \frac{mg}{kg}\right] - \text{dry matter}\right) \times (\text{mass added to reactor [kg – dry matter]})\)

\(^b\) Combined ashes from smouldering treatment of sludge mixed with woodchips

\(^c\) Calculated as \(\left(\frac{\text{(actual elemental content [mg element]}}}{}\right) \div (\text{theoretical maximum elemental content [mg element]}) \times 100\%\)

\(^d\) Assuming no losses, calculated as the sum of the contents in the virgin sludge and woodchips

\(^e\) Calculated as \(\left(\text{elemental concentration} \left[ \frac{mg}{kg}\right] - \text{ash}\right) \times (\text{mass of ash remaining in reactor [kg – ash]})\)

\(^f\) Elemental retention considering only contribution of the sludge to the post-treatment ash

\(^g\) Elemental retention considering both the contribution of the woodchips and the sludge

* The standard error was calculated from the uncertainties from each calculation added in quadrature.
S3. Supplementary Information on Leaching Tests and Extraction Potentials

Figure S3-2: pH changes observed during the column percolation experimental (following USEPA Method 1314). The results are presented for sludge and post-treatment ash and sand as a function of the liquid-to-solids ratio.
Figure S3-3: column percolation experimental results (following USEPA Method 1314) for 6 common potentially toxic elements from the virgin sludge and post-treatment ash and sand. The elemental release is shown as cumulative release as a function of the liquid-to-solid ratio. The values have been normalized to mg of element per kg of dry sludge. The available content of the materials from USEPA Method 1313 at native pH has been plotted at an L/S of 10 mL/g – dry. A dotted line with a slope of 1 has been added to each plot.
Figure S3-4: pH-dependent leaching (following USEPA Method 1313) of 4 other elements of interest from the virgin sludge compared to the post-treatment ash and sand. All values have been normalized to mg of P per kg of dry material.

Figure S3-5: Available and total phosphorus contents within the pre- and post-treatment materials from sludge-sand and mixed sludge-woodchips smouldering experiments. Virgin woodchips are denoted as ‘WC’, ‘S/S’ is sieved ash from sludge-sand smouldering experiments, and ‘S/WC’ is ash from mixed sludge-woodchips smouldering.
Figure S3-6: Available and total contents of 8 potentially toxic elements within the pre- and post-treatment materials from sludge-sand and mixed sludge-woodchips smouldering experiments. Virgin woodchips are denoted as ‘WC’, ‘S/S’ is sieved ash from sludge-sand smouldering experiments, and ‘S/WC’ is ash from mixed sludge-woodchips smouldering.
Figure S3-7: Available and total contents of aluminum, iron, magnesium, and manganese within the pre- and post-treatment materials from sludge-sand and mixed sludge-woodchips smouldering experiments. Virgin woodchips are denoted as 'WC', 'S/S' is sieved ash from sludge-sand smouldering experiments, and 'S/WC' is ash from mixed sludge-woodchips smouldering.
<table>
<thead>
<tr>
<th>Element</th>
<th>Water (^{b})</th>
<th>pH 2 (^{c})</th>
<th>pH 13 (^{c})</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Maximum Recoverable Content (mg of element/kg of dry sludge) ± SE(^{a})</td>
<td>Sludge</td>
<td>Sand + Ash (^{d})</td>
</tr>
<tr>
<td>Al</td>
<td>3 ± 0.3</td>
<td>16 ± 7</td>
<td>320 ± 32</td>
</tr>
<tr>
<td>Cd</td>
<td>0.03 ± 0.004</td>
<td>0.1 ± 0.04</td>
<td>0.3 ± 0.04</td>
</tr>
<tr>
<td>Co</td>
<td>0.6 ± 0.08</td>
<td>0.04 ± 0.03</td>
<td>2 ± 0.3</td>
</tr>
<tr>
<td>Cr</td>
<td>0.3 ± 0.04</td>
<td>2 ± 0.6</td>
<td>1.6 ± 0.2</td>
</tr>
<tr>
<td>Cu</td>
<td>10 ± 1</td>
<td>12 ± 9</td>
<td>21 ± 2</td>
</tr>
<tr>
<td>Fe</td>
<td>21 ± 3</td>
<td>66 ± 28</td>
<td>900 ± 130</td>
</tr>
<tr>
<td>Mg</td>
<td>210 ± 30</td>
<td>870 ± 290</td>
<td>920 ± 130</td>
</tr>
<tr>
<td>Mn</td>
<td>5 ± 1</td>
<td>2.6 ± 0.8</td>
<td>71 ± 10</td>
</tr>
<tr>
<td>Mo</td>
<td>0.2 ± 0.04</td>
<td>1.4 ± 0.8</td>
<td>0.09 ± 0.02</td>
</tr>
<tr>
<td>Ni</td>
<td>4 ± 1</td>
<td>0.5 ± 0.1</td>
<td>6 ± 1</td>
</tr>
<tr>
<td>P</td>
<td>160 ± 25</td>
<td>500 ± 97</td>
<td>1400 ± 210</td>
</tr>
<tr>
<td>Pb</td>
<td>0.2 ± 0.07</td>
<td>0.8 ± 0.4</td>
<td>0.8 ± 0.2</td>
</tr>
<tr>
<td>Zn</td>
<td>13 ± 3</td>
<td>2.7 ± 0.7</td>
<td>330 ± 63</td>
</tr>
</tbody>
</table>

\(^{a}\) Standard error calculated as \(\sigma / \sqrt{n}\)

\(^{b}\) Recovery at native pH where samples were mixed with only deionized water (pH 6 for sludge, 7 for sand, and 8 for post-treatment ash)

\(^{c}\) The actual sample pH values are within ± 0.5 pH units of the specified value

\(^{d}\) Combined post-treatment materials (i.e., coarse-grained quartz sand and smouldered ash)
<table>
<thead>
<tr>
<th>Element</th>
<th>Water (^b)</th>
<th>pH 2 (^c)</th>
<th>pH 13 (^c)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>0.2 ± 0.1%</td>
<td>29 ± 11%</td>
<td>18 ± 7%</td>
</tr>
<tr>
<td>Cd</td>
<td>6.9 ± 2.1%</td>
<td>18 ± 5%</td>
<td>2.7 ± 0.7%</td>
</tr>
<tr>
<td>Co</td>
<td>1.1 ± 0.9%</td>
<td>34 ± 9%</td>
<td>1.7 ± 1.3%</td>
</tr>
<tr>
<td>Cr</td>
<td>1.8 ± 0.6%</td>
<td>13 ± 4%</td>
<td>2.6 ± 0.8%</td>
</tr>
<tr>
<td>Cu</td>
<td>2.4 ± 1.8%</td>
<td>54 ± 25%</td>
<td>10 ± 7%</td>
</tr>
<tr>
<td>Fe</td>
<td>0.2 ± 0.1%</td>
<td>35 ± 11%</td>
<td>0.2 ± 0.1%</td>
</tr>
<tr>
<td>Mg</td>
<td>22 ± 8%</td>
<td>71 ± 28%</td>
<td>0.4 ± 0.2%</td>
</tr>
<tr>
<td>Mn</td>
<td>1.1 ± 0.4%</td>
<td>72 ± 33%</td>
<td>0.4 ± 0.1%</td>
</tr>
<tr>
<td>Mo</td>
<td>13 ± 7%</td>
<td>7.9 ± 4%</td>
<td>34 ± 21%</td>
</tr>
<tr>
<td>Ni</td>
<td>1.3 ± 0.3%</td>
<td>27 ± 11%</td>
<td>3.5 ± 1.4%</td>
</tr>
<tr>
<td>P</td>
<td>0.1 ± 0.01%</td>
<td>55 ± 14%</td>
<td>25 ± 6%</td>
</tr>
<tr>
<td>Pb</td>
<td>1.3 ± 0.5%</td>
<td>35 ± 16%</td>
<td>4.1 ± 0.5%</td>
</tr>
<tr>
<td>Zn</td>
<td>0.4 ± 0.1%</td>
<td>20 ± 7%</td>
<td>0.6 ± 0.2%</td>
</tr>
</tbody>
</table>

\(^a\) Standard error calculated as \(\frac{\sigma}{\sqrt{n}}\)
\(^b\) Recovery at native pH where samples were mixed with only deionized water (pH 6 for sludge, 7 for sand, and 8 for post-treatment ash)
\(^c\) The actual sample pH values are within ±0.5 pH units of the specified value
<table>
<thead>
<tr>
<th>Element</th>
<th>Elemental Retention $^a \pm SE^*$(%)</th>
<th>Maximum Recoverable Content $^b \pm SE^*$(mg/kg – dry starting material)</th>
<th>Recovery Potential $^c \pm SE^*$(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mixed Ash $^d$</td>
<td>Emissions $^e$</td>
<td>pH 2</td>
</tr>
<tr>
<td>Al</td>
<td>15 ± 1.6</td>
<td>85 ± 1.6</td>
<td>82 ± 7.4</td>
</tr>
<tr>
<td>Cd</td>
<td>15 ± 3.1</td>
<td>85 ± 3.1</td>
<td>0.2 ± 0.04</td>
</tr>
<tr>
<td>Co</td>
<td>28 ± 5.1</td>
<td>72 ± 5.1</td>
<td>0.3 ± 0.02</td>
</tr>
<tr>
<td>Cr</td>
<td>14 ± 0.9</td>
<td>86 ± 0.9</td>
<td>1.1 ± 0.82</td>
</tr>
<tr>
<td>Cu</td>
<td>25 ± 2.1</td>
<td>75 ± 2.1</td>
<td>28 ± 9.8</td>
</tr>
<tr>
<td>Fe</td>
<td>14 ± 1.5</td>
<td>86 ± 1.5</td>
<td>220 ± 220</td>
</tr>
<tr>
<td>Mg</td>
<td>29 ± 2.6</td>
<td>71 ± 2.6</td>
<td>980 ± 170</td>
</tr>
<tr>
<td>Mn</td>
<td>27 ± 3.0</td>
<td>73 ± 3.0</td>
<td>79 ± 19</td>
</tr>
<tr>
<td>Mo</td>
<td>11 ± 2.7</td>
<td>89 ± 2.7</td>
<td>0.4 ± 0.34</td>
</tr>
<tr>
<td>Ni</td>
<td>17 ± 1.8</td>
<td>83 ± 1.8</td>
<td>1.7 ± 0.36</td>
</tr>
<tr>
<td>P</td>
<td>22 ± 2.4</td>
<td>78 ± 2.4</td>
<td>2700 ± 1800</td>
</tr>
<tr>
<td>Pb</td>
<td>6 ± 1.0</td>
<td>94 ± 1.0</td>
<td>6.6 ± 1.1</td>
</tr>
<tr>
<td>Zn</td>
<td>22 ± 3.0</td>
<td>78 ± 3.0</td>
<td>83 ± 34</td>
</tr>
</tbody>
</table>

$^a$ Elemental retention of the mixed sludge and woodchips ash determined in Table S2-3

$^b$ Available elemental content determined from USEPA Method 1313

$^c$ Calculated as $\frac{((elemental\ content[mg\ element])}{(total\ elemental\ content\ in\ starting\ material\ [mg\ element])}\times\ 100\%$

$^d$ Combined ashes from smouldering treatment of sludge mixed with woodchips

$^e$ Calculated as $100\% - (elemental\ retention\ in\ mixed\ ash[\%])$

* The standard error was calculated from the uncertainties from each calculation added in quadrature