Investigation of potential alkali getters for gasification using a new high temperature pressurized simultaneous thermal analyzer (P-STA)

Franz Hauk, Stephan Gleis, Hartmut Spliethoff

Institute of Energy Systems
Technische Universität München
Motivation: Alkali Removal at highest temperatures

Goal for IGCC power plant:
Prevention of hot gas (alkali) corrosion at minimum loss of efficiency

“State of the art”
Massive cooling of crude syngas + subsequent cleaning

Solution:
Hot Gas Cleaning - Usage of ceramic high temperature getter materials
(kaolin, bauxite, sands, … aluminosilicates with large-scale availability)
“Hot” - Temperatures up to 1800 °C
Approach

Tools:
High-temperature Pressurized simultaneous thermal analyzer (P-STA)
Allows experimental conditions close to power plants
FactSage – Thermochemical equilibrium calculations

Experiments:
Investigation of Alkali chemisorption capacity of getter
Experimental + theoretical methods
What is thermal analysis (TA)?

Techniques where a physical property of a substance is measured as a function of temperature (or time) while being exposed to a controlled temperature program.

e.g. Thermogravimetry (TG)

- Continuous recording of sample mass during controlled heating program

Other representatives of TA:
- DTA  Differential Thermal Analysis
- DSC  Differential Scanning Calorimetry

Combination of TG and DTA/DSC

Simultaneous Thermal Analysis (STA)
Overview of Pressurized Thermogravimetry

![Graph showing pressure vs. temperature with marked Commercial and Research facilities at 6 bar and 1750 °C]
P-STA at Institute of Energy Systems, TUM

High density + purity alumina tube, pressure loaded; Finite creep strength!

Sample

Volumetrically tared high precision beam balance

Pressure vessel

Source: Linseis Messgeräte GmbH, Selb, Germany
P-STA at Institute of Energy Systems, TUM

**High temperature Furnace (1750 °C)**

**P-STA PT-1750, 5 bar**

**TGA**

**DTA / H-DSC**

**Gas box** (Pressure control, reaction gas supply)
Experiments – Chemisorption capacity of Kaolin Suprex

How much NaCl can be sorbed by Suprex depending on temperature and pressure?

Chemical Analysis of Suprex, a hard kaolin (aluminosilicate):

<table>
<thead>
<tr>
<th></th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>Na₂O</th>
<th>K₂O</th>
<th>CaO</th>
<th>MgO</th>
<th>Fe₂O₃</th>
<th>TiO₂</th>
<th>P₂O₅</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass-%</td>
<td>45.1</td>
<td>38.1</td>
<td>Trace</td>
<td>0.2</td>
<td>0.02</td>
<td>Trace</td>
<td>1.6</td>
<td>1.5</td>
<td>0.1</td>
</tr>
</tbody>
</table>

Method: 2 subsequent experiments

1000 mg Suprex + 100 mg NaCl

1000 mg Suprex (pure)

Sorbed Alkali Mass
Results – Development of mass release curves

Continuous trend of temperature dependent sorption capacity and release velocity

Reducing atmosphere (5 vol.-% H₂ in N₂), reaction gas flow 2.0 l/h, heating rate 10 K/min, dense Al₂O₃ crucible, Suprex
Results – Mass release and release rate

Reducing atmosphere (5 vol.-% H₂ in N₂), reaction gas flow 2.0 l/h, heating rate 10 K/min, dense Al₂O₃ crucible, Suprex

- 550 °C: < 2 % released
- 590/570 °C: first local release speed max.
- 801 °C: melting point NaCl
- 970 °C: absolut (amb.)/local (0.5 MPa) maximum
- 1060 °C: local max. (amb.)
- 1110 °C: absolut max.(0.5 MPa)
- 1600 °C: 72 % (amb.)/ 68 % (0.5 MPa) of NaCl released
Results – Experiments vs. Thermochemical calculations

Ambient pressure
At 0.5 MPa: For 1400 °C max. sodium release: 20 mass-% of added Na

Na release (calc.)
0.5 MPa

Reducing atmosphere (5 vol.-% H₂ in N₂), reaction gas flow 2.0 l/h, heating rate 10 K/min, dense Al₂O₃ crucible, Suprex;
Thermochemical calculations: FactSage 5.5
Conclusions

- Pressurized STA with outstanding parameters was developed:
  1750 °C / 0.5 MPa overpressure

- Chemisorption capacity experiments are a good tool for classification of potential getter materials

- Thermochemical calculations reflect experiments in wide areas

- Pressure has significant effect on alkali (Na) release…

Outlook

- Experiments at higher pressures!
- In-situ optical measurements

Thank you for your Attention!

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