In-situ spectroscopy for the gas analysis during coal gasification

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Introduction

• Measurement of gas composition and temperature in temporal and spatial resolution inside a demonstration reactor to improve the measurement techniques
• Variation of gas composition, temperature and pressure
• Analyze CO-formation around a C-particle in a CO$_2$-stream for a better understanding of the coal gasification process
• Compare with simulation/theory

• Challenging task:
  – High temperatures and pressures during the measurement
    ➢ Raman spectroscopy and Laser Induced Fluorescence (LIF)
  – High laser power needed
1. Introduction

2. Theory

3. Setup

4. Experimental Results
   - Raman spectroscopy
   - Laser Induced Fluorescence (LIF)

5. Summary & Outlook
Raman spectroscopy

- Rayleigh scattering: elastic
- Raman scattering: inelastic
- Some scattered electrons can loose (or gain) energy due to simultaneous vibrational transitions (Stokes and Anti-Stokes)
- Inelastic scattering 10000 times less likely than elastic scattering (special equipment required)
- Frequency shift depends on the gas component
- Intensity of Raman scattering is proportional to the concentration of the gas component
Laser Induced Fluorescence (LIF)

- Technique for species-specific imaging of gas temperature, pressure, or gas concentration along a laser beam
- LIF is performed at ultraviolet or visible wavelengths using high-power lasers to image combustion molecules (e.g. CO)
- LIF occurs when a molecule absorbs laser light and then emits a photon(s) as it relaxes back to another state ($E_1$)
- Fluorescence is imaged by a camera or detector to acquire spatially resolved information about the local gas conditions
- LIF method is very sensitive, concentrations down to the ppm range are detectable
Setup

Dye laser → Nd:YAG laser → Reactor

Spectrometer + camera

Detector

Beam dump

Laser

Port 1/1 → Port 2/1 → Port 3 (optional) → Port 1/2
Raman signals at room temperature and an absolute pressure of 20 bar
Experimental Results – Raman spectroscopy

Variation of pressure at room temperature

1.2 bar

5 bar

10 bar

15 bar

20 bar
Experimental Results – Raman spectroscopy

Variation of pressure at room temperature

<table>
<thead>
<tr>
<th>Gas components</th>
<th>1.2 bar</th>
<th>5 bar</th>
<th>15 bar</th>
<th>20 bar</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>GC</td>
<td>Raman</td>
<td>GC</td>
<td>Raman</td>
</tr>
<tr>
<td>CO</td>
<td>5.6%</td>
<td>(6.2±1.8)%</td>
<td>5.5%</td>
<td>(5.9±1.4)%</td>
</tr>
<tr>
<td>CO₂</td>
<td>44.9%</td>
<td>(46.1±3.8)%</td>
<td>45.7%</td>
<td>(42.9±4.8)%</td>
</tr>
<tr>
<td>N₂</td>
<td>49.5%</td>
<td>(47.8±2.5)%</td>
<td>48.8%</td>
<td>(51.1±4.4)%</td>
</tr>
</tbody>
</table>

The 10 bar Raman measurement served as calibration image
Experimental Results – Raman spectroscopy

Temperature calculation

<table>
<thead>
<tr>
<th>Thermal element</th>
<th>293 K</th>
<th>928 K</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermal element</td>
<td>(481±5) K</td>
<td>(600±10) K</td>
</tr>
<tr>
<td>Extrapolation from Rayleigh signal</td>
<td>487±11 K</td>
<td>594±7 K</td>
</tr>
</tbody>
</table>
Variation of temperature at 10 bar

296 K
483 K
668 K
993 K
1238 K
Experimental Results – Raman spectroscopy

Variation of temperature at 10 bar

<table>
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<tr>
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<th>668 K</th>
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<tbody>
<tr>
<td></td>
<td>GC</td>
<td>Raman</td>
<td>GC</td>
<td>Raman</td>
</tr>
<tr>
<td>CO</td>
<td>4.9%</td>
<td>(5.1±1.0)%</td>
<td>5.0%</td>
<td>(5.0±0.8)%</td>
</tr>
<tr>
<td>CO₂</td>
<td>50.1%</td>
<td>(48.3±5.5)%</td>
<td>49.1%</td>
<td>(48.0±2.9)%</td>
</tr>
<tr>
<td>N₂</td>
<td>45.0%</td>
<td>(46.7±5.0)%</td>
<td>46.0%</td>
<td>(47.0±3.0)%</td>
</tr>
</tbody>
</table>

The 296 K Raman measurement served as calibration image
Measurements in N\textsubscript{2} and CO\textsubscript{2} atmosphere

- 15% CO, 15% CO\textsubscript{2} and 70% N\textsubscript{2}
- 15% CO, 85% CO\textsubscript{2}
Experimental Results – Laser Induced Fluorescence

CO concentrations in pure CO\(_2\) atmosphere

15\% CO, 85\% CO\(_2\)

7.6\% CO, 92.4\% CO\(_2\)

2\% CO, 98\% CO\(_2\)
Measurements in CO$_2$/Ar atmosphere

- 2% CO, 98% CO$_2$
- 2% CO, 49% CO$_2$, 49% Ar
- 2% CO, 98% Ar
Lowest CO concentration $c_{CO} = 0.6\%$

0.6% CO, 49.7% CO$_2$, 49.7% Ar

Simulation of a single coal particle (by A. Richter, 06.03.2014)
Summary

- Measurement of gas concentrations under various conditions of temperature (300-1000K), pressure (1-20 bar) and carrier gas (CO$_2$, CO$_2$/N$_2$, CO$_2$/Ar) relevant for coal gasification in the HITECOM reactor
- Raman: values down to 5% CO could be measured
- LIF: CO concentrations down to 0.6% detectable, but until now only qualitative measurements

Outlook

- Increase laser power to detect lower concentrations
- Implement method to counteract the convection streams at high temperature
- Combine Raman spectroscopy and LIF for quantitative LIF measurements

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