Research of high temperature CO$_2$ sorption from flue gas using carbonate loop

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Acronym: hitecarlo

Tests realized on the apparatus with fixed bed adsorber

Presentation of initial phase of experimental activities
Tests in fixed bed reactor

Initial proposal of the experimental laboratory apparatus

Fig. 1  Schematic drawing of changes in apparatus design (liquid chiller disassembled due to high pressure drop)

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Actual state of the experimental apparatus

Fig. 2

Overall view at the apparatus
Detail of the upper fittings (joining quartz/steel)

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Actual state of the experimental apparatus

Fig. 3

Reactor with quartz wool and the sample

Cooling down of the oven

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Actual problems with the apparatus to be solved

1) Big dead volume of the gas phase above the sample – ground glass joint as well as the connection quartz/steel must be outside the oven, while the sample must be placed in the lower third of the oven shaft;

2) Necessity to use low flow rates only (in the range of $1 - 3 \text{ dm}^3\text{.min}^{-1}$);

3) Pressure limitation due to reactor made of fused quartz – only atmospheric pressure or minimum overpressure allowed for the experiments;

4) Mechanical limitations due to reactor made of fused quartz – fragile joining quartz/steel with different thermal dilatation (risk of cracking, gas leakage etc.);

5) Mechanical limitations due to reactor made of fused quartz – permanent stuck of the ground glass connection (for this reason the filling and removal of the sample realized through the upper tee-piece Superlok with 12 mm diameter)

6) Insufficient length of the thermocouple probe due to inserting new tee-piece between fused quartz and steel pipes (see above the point no. 5)
## Experimental conditions

### Samples
- **Particle sizes:** fraction in the range of 1 – 2 mm
- **Batch size:** bulk material with volume 70 ml ⇒ weight 90 – 95 g

### Temperature program
- **Calcination:** 1 000 °C (ramp 10 °C.min⁻¹)
  - atmosphere N₂
  - flow rate 2 dm³.min⁻¹
- **Carbonation:** 400, 650, 750 °C (isotherm)
  - atmosphere N₂+CO₂ 14 % vol.
- Repeating of the cycle calcination/carbonation

### Measured param.
- **CO₂ in the outlet** IR analyzer ASEKO AIR-LF
- **Temperature:** thermocouple probe Ni-CrNi
- **Gas volume:** wet gas meter
Contemporary results of experiments

Tests in fixed bed reactor

Sample „Branžovy“ – content of CO₂ in gas outlet at isothermal conditions

Course of carbonation

CO₂ content in the gas outlet ; [% vol.]

Time ; [s]

Cycle 1 (200 °C)  Cycle 2 (400 °C)  Cycle 3 (650 °C)
Tests in fixed bed reactor

Contemporary results of experiments

Sample „Libotín“ – content of CO₂ in gas outlet at isothermal conditions (650 °C)
Tests in fixed bed reactor

Contemporary results of experiments

Fig. 6 Sample „Libotín“ – content of CO$_2$ in gas outlet during calcination up to 1 000 °C
### Contemporary results of experiments

<table>
<thead>
<tr>
<th>Process</th>
<th>Information</th>
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<tbody>
<tr>
<td><strong>Calcination</strong></td>
<td>Start of process: 800 – 830 °C</td>
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<tr>
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<td>Released CO₂: 96 – 98 % compared with theoretical amount</td>
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<td></td>
<td>(calculated from content of calcite analyzed using XRF method)</td>
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<tr>
<td><strong>Carbonation</strong></td>
<td>Captured CO₂: ca. 75 % at 650 °C compared with theoretical amount</td>
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<td>(calculated from content of calcite analyzed using XRF method)</td>
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<td>5 – 6 % at 400 °C</td>
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<td>(note: reaction does not reach the equilibrium state)</td>
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<td></td>
<td>0,2 – 0,5 % at 200 °C</td>
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<td>(note: reaction does not reach the equilibrium state)</td>
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<td><strong>Cyclic measurement</strong></td>
<td>Periodical repeating of calcination/carbonation cycles will be the subject</td>
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<td>of subsequent experiments.</td>
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