Research Report nr D332
Helsinki 2015

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Laboratory scale oxygen carrier characterisation - test rig design, construction and test procedure

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ccsp
Carbon Capture and Storage Program

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Key words: CLC, chemical cycling, oxidation-reduction, oxygen carriers

Abstract

Small scale fluidized bed tests facility was planned and constructed. Chemical looping combustion CLC normally consist of two fluidized bed reactors where oxidation of the oxygen carrier powder occurs in the other and reduction of it by the fuel in the other reactor. The oxygen carrier is typically granulated and sintered powder mixture of inert part and active part. The active part suffers the repeated oxidation/reduction cycle while the inert part is stable. The oxygen carrier suffers slight thermal cycling but mechanical agitation and chemical cycling is the most damaging mechanisms. In this test facility oxygen carrier's response to chemical cycling is tested.

Tampere, January 2015

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15.1.2015

1 Introduction

Small scale fluidized bed tests facility was planned and constructed. Chemical looping combustion CLC normally consist of two fluidized bed reactors where oxidation of the oxygen carrier powder occurs in the other and reduction of it by the fuel in the other reactor. The oxygen carrier is typically granulated and sintered powder mixture of inert part and active part. The active part suffers the repeated oxidation/reduction cycle while the inert part is stable. The oxygen carrier suffers slight thermal cycling but mechanical agitation and chemical cycling is the most damaging mechanisms. The oxygen carrier granules are sintered for mechanical stability but with maximum reactivity in mind. This is a task of opposite aims, optimized sintering temperatures are needed. The composition and structure of the oxygen carrier also affects both the reactivity and mechanical agitation resistance.

The apparatus constructed was planned to test oxygen carrier`s chemical cycling resistance i.e. oxidation/reduction resistance by artificial aging. Aim of the test procedure was to test the chemical cycling resistance separately with minimum mechanical agitation. The test facility contains fluidized bed in order to oxidize/reduce the while sample volume thoroughly but without heavy agitation. Indirect testing regime of the oxygen carrier was planned to test the effect of chemical cycling. Planned tests are: particle size distribution measurement, flowability measurements, X-ray diffract, crushing strength measurements, morphology studies by SEM and thermal analysis (DTA, TG). In such way the effect of chemical cycling is revealed. Second addition to the test facility would be separate fluidization resistance tests (Powder Technology, Volume 256, April 2014, Pages 75–86, Measuring attrition resistance of oxygen carrier particles for chemical looping combustion with a customized jet cup, Magnus Rydén, Patrick Moldenhauer, Simon Lindqvist, Tobias Mattisson, Anders Lyngfelt).

1.1 Test bed design

The test fluidization bed was designed to contain small amount of oxygen carrier granules but sufficiently to perform the tests listed previously. The sample amount was kept small in order to keep process gas usage low, the bigger the sample amount the bigger the fluidization tube should be. Figure 1 presents the calculated minimum gas flow rates for different size oxygen carrier as function of temperature, calculation were done for 28 mm fluidization tube. Gas volume increases as function of temperature which eases the fluidization. Temperature range planner for the tests is 700-950ºC. Figure 2 presents schematic picture of the fluidized bed construction. Heat resistant stainless steel was used for fluidization tube, sintered filters and gas inlet and outlet tubes. Function of the sintered filters was to distribute the fluidization gas homogeneously and to keep all of the oxygen carrier material in the tube. Installation of the filters is presented in the figure 3.

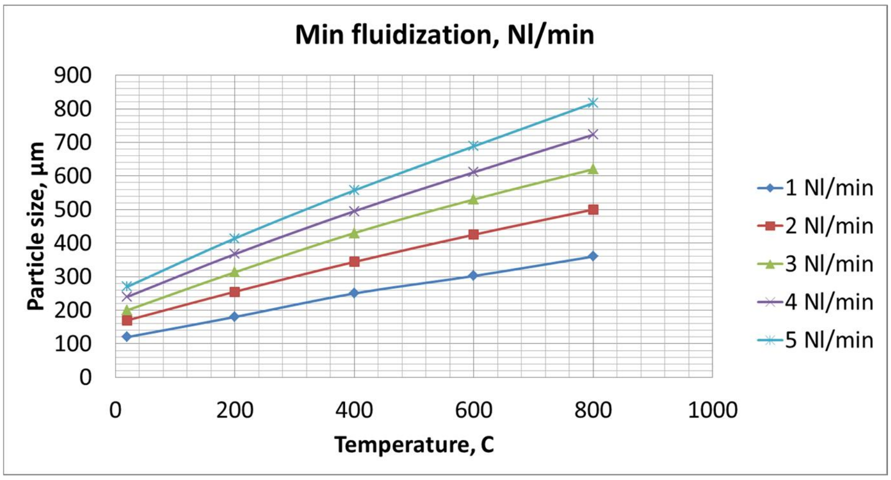


Figure 1. Minimum fluidization particle sizes for different gas speeds as function of temperature.

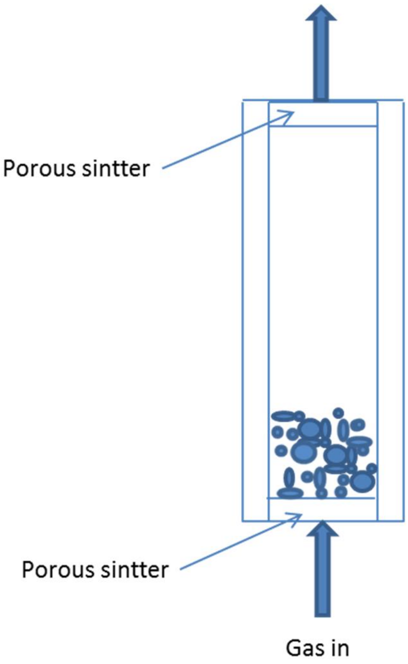


Figure 2. Schematics of the fluidized bed construction. Tube inner diameter is 28 mm. Blue dots represent the fluidized oxygen carrier under test.

Both ends of the fluidization tube are sealed with a Termiculite^{TM} class ceramic seal. The construction is build using bolts made of heat resistant stainless steel. Tests apparatus is installed to large chamber furnace. Coil of gas inlet tube ensures the heating of the gas prior to entering the fluidization tube (figure 4.).

Figures 4-9 present the configuration of the tests apparatus. The fluidization tube is mounted to vertical position by adjustable pedestal in order to ensure even fluidization. Process gas feeding and evacuation is in a such way that leakages in possible mall function will be save. Premixed bottled gas is used as fuel (CH_4+CO_2), nitrogen as rinsing gas and filtered pressurized air as oxidizing gas.

Figure 3. The sintered filters installed to fluidization tube cover and to fluidization tube bottom.

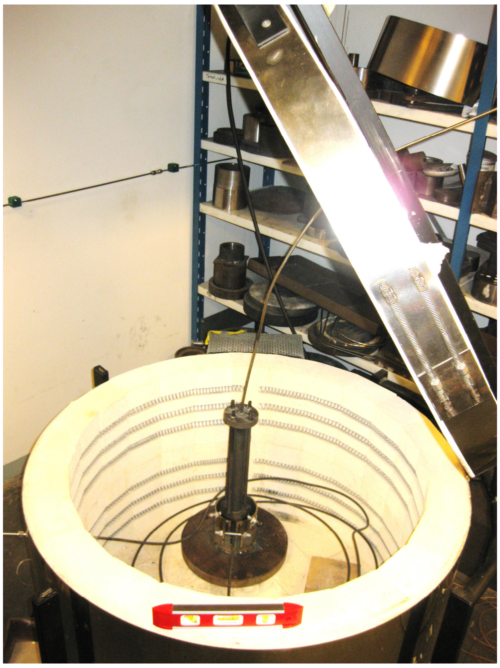


Figure 4. Tests apparatus installed to large furnace. Coil of gas inlet tube ensures the heating of the gas prior to entering the fluidization tube.

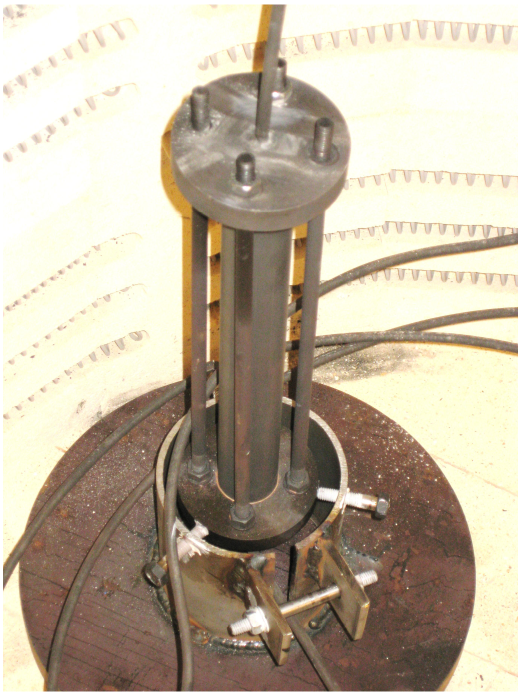


Figure 5. Fluidization tube installation inside the furnace. The tube is installed in a way that it can be adjusted to upright position to ensure homogeneous fluidization.

Figure 6. The furnace in operating position. Gas outlet comes out from an opening of the furnace lid. Ventilation hood connected to powered roof ventilation ensures gas evacuation in the case of mall function. Methane alarm system will be installed before oxygen carrier testing work.

Figure 7. Gas manifold with back pressure regulated flow meters. The automated valve system is regulated by automatized valves, control in lower picture.

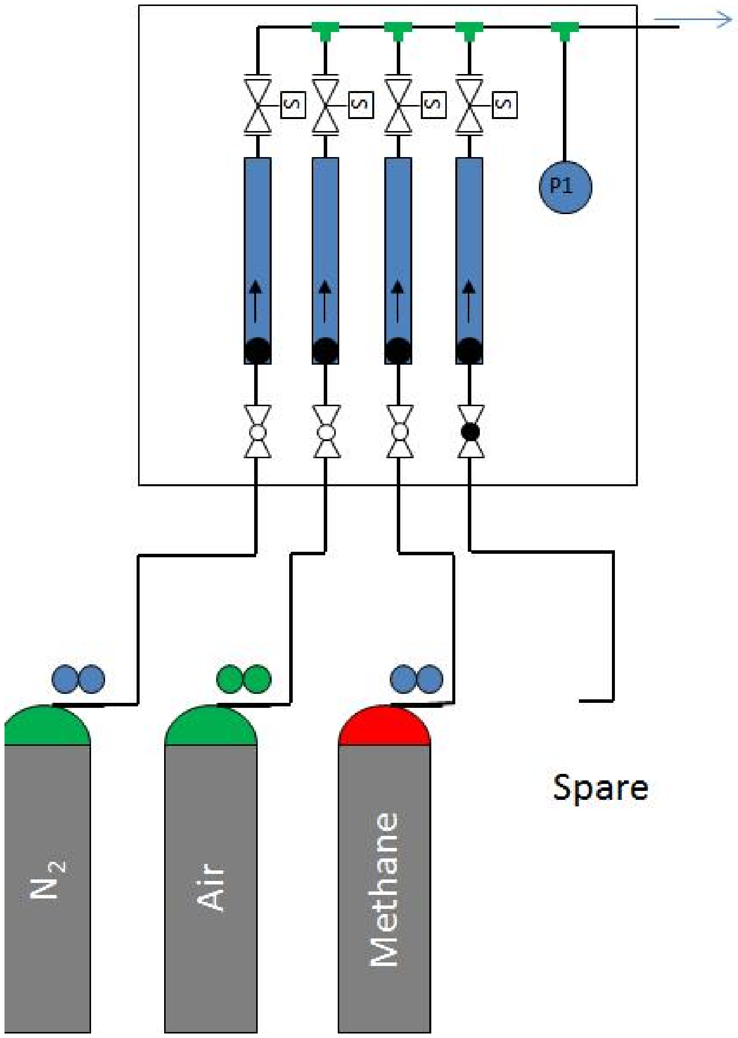


Figure 8. Schematics of the automated gas manipulation system.

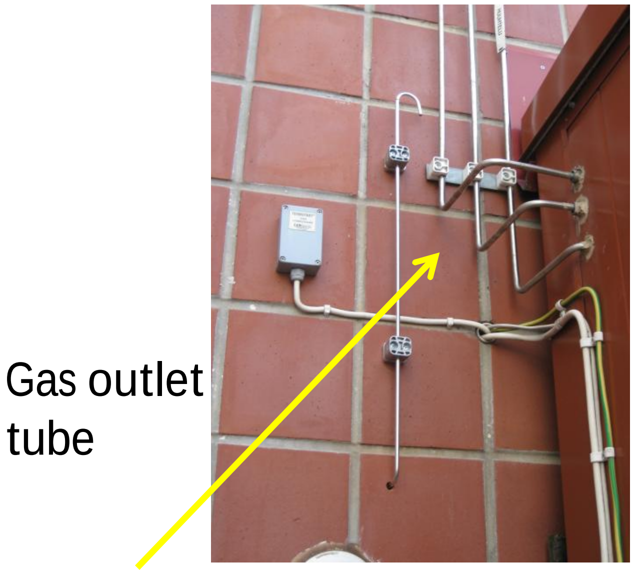


Figure 9. Gas outlet tube connected outside to avoid burning gas collection inside the laboratory.

2 Testing

The whole system was found out to be gas tight using Termiculite^{TM} glass ceramic gasket. Each test cycle must be started with leakage test as burning gases are involved. The fluidization of oxygen carrier granules was tested and nice bubbling fluidization was observed. The vertical position is essential to ensure homogeneous bubbling and subsequent fast and homogenous oxidation/reduction cycle. Oxidation rate and reduction rate studies will start as soon as possible.

2.1 Inspection and proofing

The gas feeding valve, rotameter and tubing system was constructed by accredited company. The gas tightness and methane gas alarm system was tested and reported. Inspection report is attached.

2.2 Tests plan

The tested oxygen carrier`s properties will be tested prior and after thermal cycling. Basic tests include particle size distribution, x-ray diffraction (phase structure), crushing strength by die pressing test, flowability measurement by hall flow method, packing density and morphology by SEM.

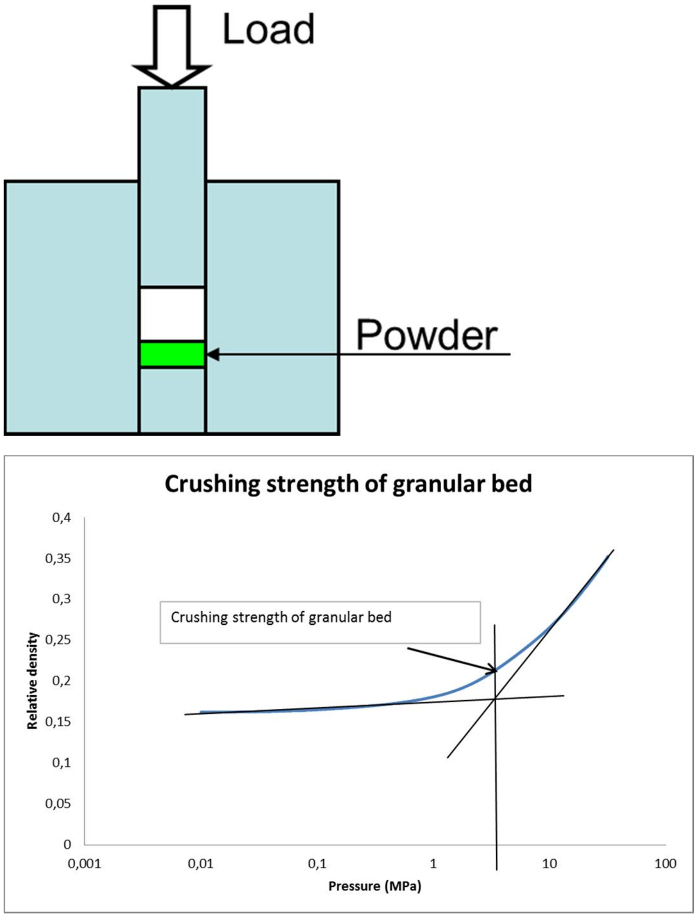


Figure 10. Principle of the die pressing test and a graphic example of the mechanical strength results

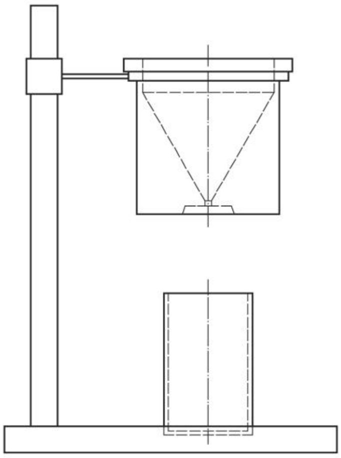
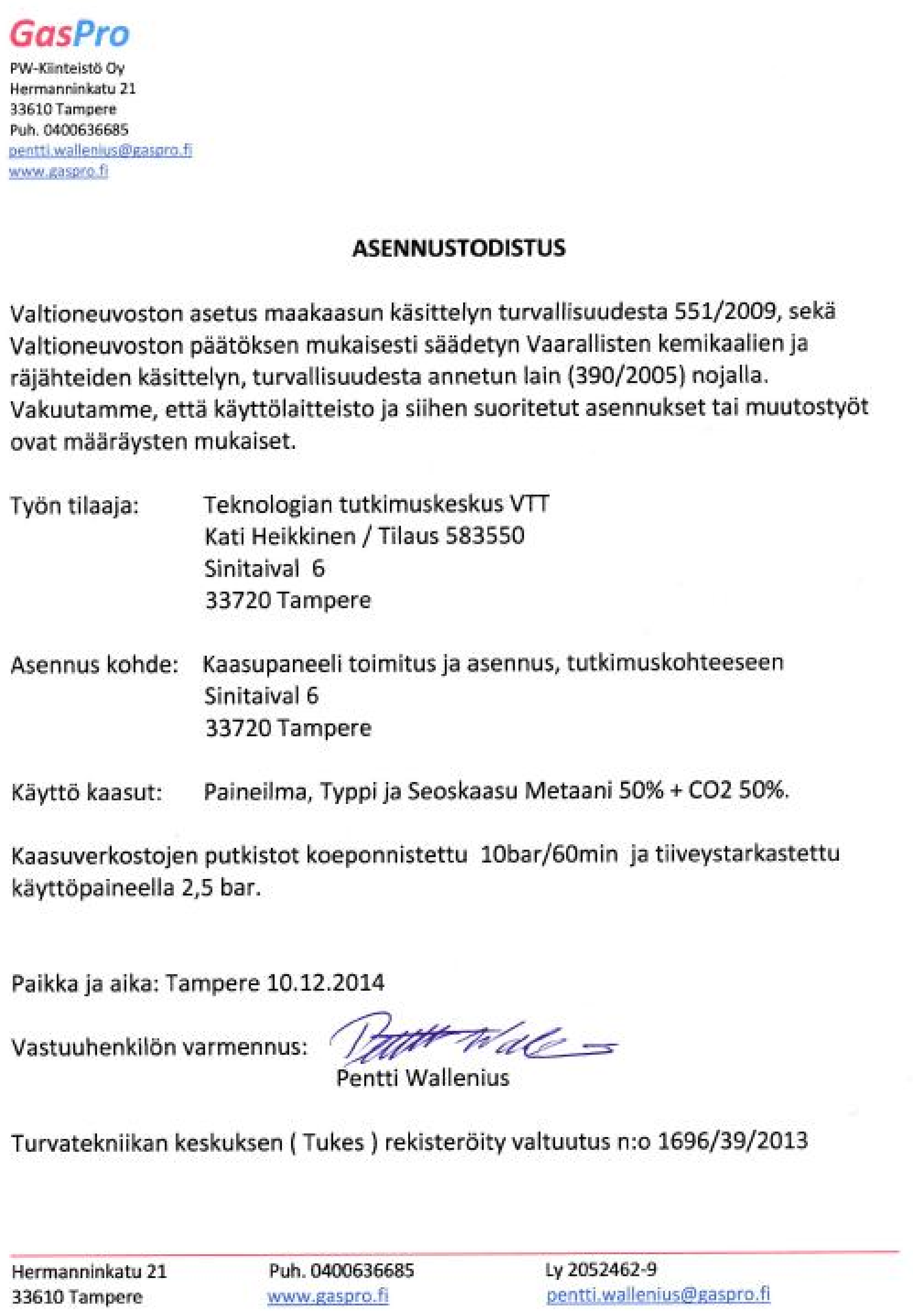


Figure 11. Hall flow method funnel and 25 ml vessel. Feed rate of powder and apparent packing density can be measured.

In chemical cycling tests cyclic oxidation and reduction can be conducted. Reactivity of the oxygen carriers may change due to repeated oxidation reduction reactions. Different thermal analysis DTA and thermo gravimetry TG can be used to evaluate oxygen carrying capacity, reacted volume fraction of the oxygen carrier and the rate of the reactions. As the number of the loops is quite large is difficult to perform such tests in DTA/TG due to long test times. With the test facility constructed, chemical cycling and thermal analysis can be done separately. DTA/TG can be measured before and after the cycling test giving the change as a result. Simultaneously the other tests can be done also as the sample volume is considerably larger than in DTA/TG.

Attachment 1.



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ASENNUSTODISTUS

Valtioneuvoston asetus maakaasun käsittelyn turvallisuudesta 551/2009, sekä Valtioneuvoston päätöksen mukaisesti säädetyn Vaarallisten kemikaalien ja räjähteiden käsittelyn, turvallisuudesta annetun lain (390/2005) nojalla. Vakuutamme, että käyttölaitteisto ja siihen suoritetut asennukset tai muutostyöt ovat määräysten mukaiset.

Työn tilaaja: Teknologian tutkimuskeskus VTT
Kati Heikkinen / Tilaus 583550
Sinitaival 6
33720 Tampere

Asennus kohde: Kaasupaneeli toimitus ja asennus, tutkimuskohteeseen
Sinitaival 6
33720 Tampere

Käyttö kaasut: Paineilma, Typpi ja Seoskaasu Metaani 50% + CO2 50%.

Kaasuverkostojen putkistot koeponnistettu 10bar/60min ja tiiveystarkastettu käyttöpaineella 2,5 bar.

Paikka ja aika: Tampere 10.12.2014

Vastuuhenkilön varmennus:
Pentti Wallenius

Turvatekniikan keskuksen (Tukes) rekisteröity valtuutus n:o 1696/39/2013

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