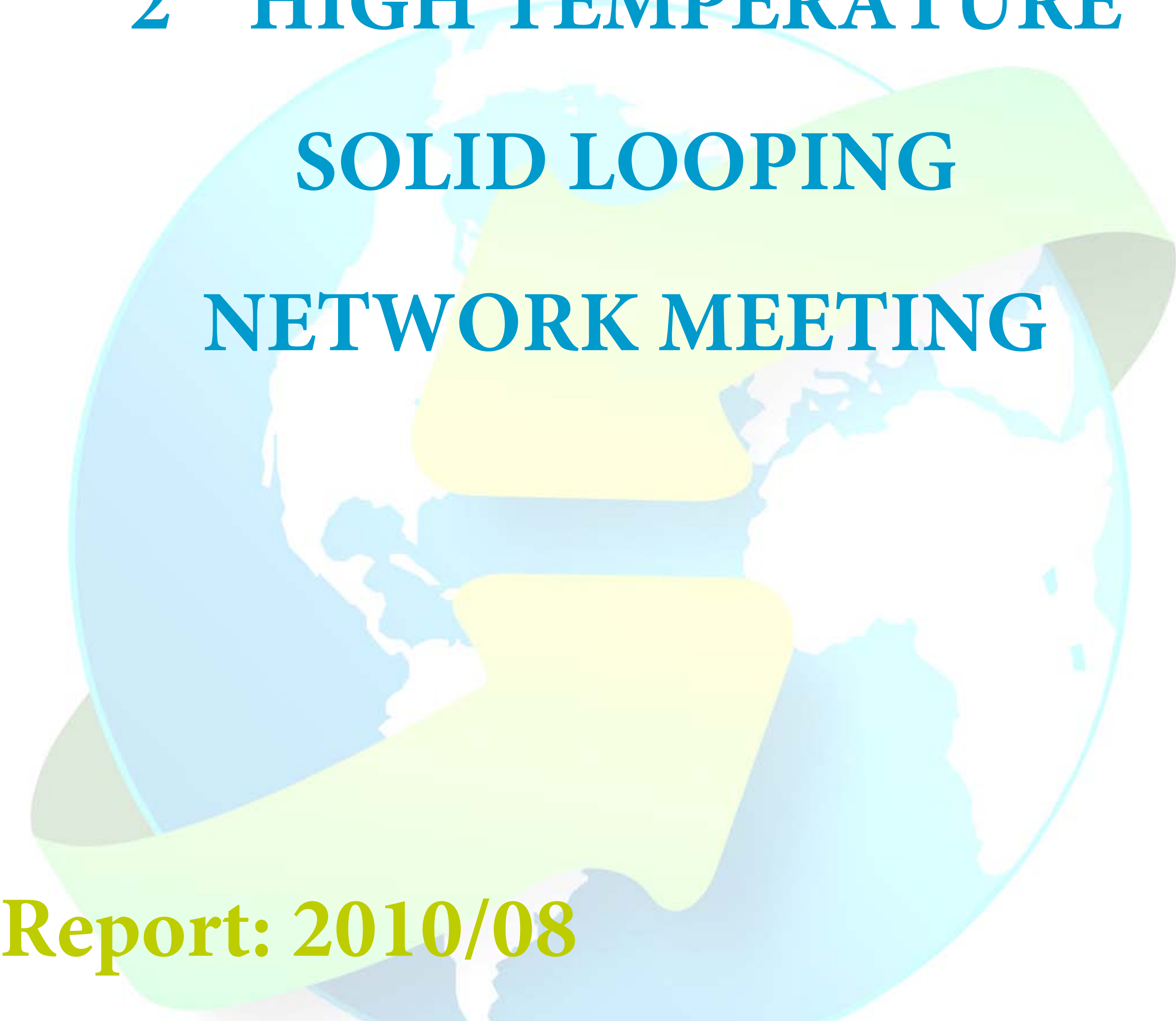
2nd HIGH TEMPERATURE SOLID LOOPING NETWORK MEETING



Report: 2010/08

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DISCLAIMER AND ACKNOWLEDGEMENTS

IEAGHG supports and operates a number of international research networks. This report presents the results of a workshop held by one of these international research networks. The report was prepared by IEAGHG as a record of the events of that workshop.

The 2nd international research network on the high temperature solid looping network was organised by IEAGHG in co-operation with ECN. The organisers acknowledge the financial support provided by TNO, Array Industries and Agentschap for this meeting and the hospitality provided by the hosts at Petten, the Netherlands.

A steering committee has been formed to guide the direction of this network. The steering committee members for this network are:

Paul Cobden, ECN Petten, The Netherlands
Tobias Proell, Technical University of Vienna
Paul Fennell, Imperial College, London
Douglas Harrison, Louisiana State University
Ben Anthony, CANMET
Carlos Abanades, INCAR-CSIS
Mike Haines, IEAGHG

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2nd High Temperature Solid Looping network meeting
ECN Petten,
The Netherlands,
15th – 17th September 2010

Executive Summary

The second meeting on the high temperature solid looping network was held at the Netherlands energy research centre at Petten from 15-17th September 2010. Approximately 70 delegates attended the meeting of which about 25% were representing industry. 24 papers were presented and discussed during the event and in addition 7 posters were also on display. ECN is actively engaged in research in this area and delegates were able to see first hand the large pilot plant constructed under the EU framework 6 and CCP 2 CACHET project to demonstrate an integrated sorbent enhanced reforming and water gas shift process using multiple fixed beds. Also included in the site tour were ECN's fluid bed biomass gasification pilot plants, the latest of which is a large 0.8 MW unit large enough to operate without external heat jacketing to compensate for heat losses.

Slow and steady progress has been made with the looping processes over the last year with improvements in the understanding of attrition and activity loss as well as incremental improvements in performance. All eyes are on larger scale demonstration plants under construction in Spain and Germany both slated to be started up and delivering performance information before next years network meeting.

The slides of the presentations and posters can be viewed in the members section of the network website along with a brief written summary of each. Highlights of the progress and learning are noted in brief below.

Contents

Summary of Presentations

Session 1: Hydration

1.1 Ruud van der Brink ECN

ECN is developing a sorbent enhanced water gas shift (SEWGS) process. This can be considered as a Hot PSA process. Process studies show it is a good route for reducing parasitic power losses during CO_2 capture. On the practical side ECN are running experiments in a side stream of the Buggenum IGCC plant. Work on NGCC is supported by the EU and the CCP2 consortium under the CACHET project. An experimental set up of multiple fixed bed reactors is used. The first step in the cycle is gas reforming and simultaneous shift and adsorption of CO_2 carried out in a bed filled with a potassium promoted hydrotalcite. This material has been found to have sufficient activity as a reforming catalyst so no additional catalyst is required. The bed is subsequently rinsed with steam and depressured to release the CO_2. By having several pressure equalisation steps losses are reduced, product purity of both the hydrogen and CO_2 streams is improved and rinse steam requirements are minimised.

1.2 Robert Symonds CANMET

CANMET has been exploring the effect of steam on calcination and sulphation of limestones. Addition of steam increases the rate and extent of reaction. There appears to be an optimum of about 10% steam beyond which there is little further improvement. Carbonation is enhanced by steam up to temperatures of about 600C beyond which $Ca(OH)_2$ is not stable. Similar effects were found for sulphation of limestone for example increasing utilisation from 50-60% without steam to over 95% when stem is added. The effects are limited to the later diffusion rate controlled period of reaction. Steam also restores limestone utilisation for carbonation where the limestone has been partially sulphated probably by helping to overcome the effects of the $CaSO_4$ blocking layer which forms. The effects are confined to the 450-650 C temperature range and are observed in a number of different limestones. However in one case, a high sodium content limestone, the effects were absent.

1.3 Vlatko Materic Industrial research Ltd NZ

Hydration based reactivation of limestones has been applied every few cycles in the carbonation/calcination process. Applying hydration in this way restores capacity to the limestone but also has the effect of weakening the particles. An attrition tester is used to measure the mechanical performance under CFB conditions. A small fluidised bed is fitted with a centrally placed vertical riser tube above which is placed a metal target plate. Particles passing up the riser impact on the plate. The sample placed in the bed is circulated after which the extent of attrition is measured by particle size analysis. IRC claims to have refined the hydration process so that much fewer fines and far greater activity restoration is achieved.

Session 2: Sorbent Enhanced Reforming (SER)

2.1 Angelika Lemonidou, Universty of Thessaloniki

Described experiments to combine CO_2 sorption and reforming for production of hydrogen in one bed. The sorption material chosen was a CaO/CaAluminate mix with an *%/15 ration proving to give the best performance. This material was tested for about 13 cycles over 60 hours with absorption at 650 regeneration at 850C with only small loss in activity. Composite materials were then prepared by addition of Ni to provide reforming activity. This was done by mixing an aqueous solution of nickel and aluminium nitrates with a suspension of calcium oxide followed by drying calcination at 500C, water addition and further calcination at 900C. The best material was found to be one containing about 16% Nickel. In tests this was found to have about 30% CO_2 absorption capacity and reasonable methane conversion capability. This was however only about 80% and dropped off sharply when nickel content was less than or greater than this amount. Reforming activity is however gradually lost as the number of cycles increases and investigations revealed that this is mainly due to sintering but formation of a thin NiO layer may also be contributing.

2.2 Richard Blom, SINTEF

Combined methane reforming and CO_2 absorption were tested in a circulating fluid bed reactor. The endothermic reforming coupled with exothermic water gas shift and carbonation reactions are almost energy neutral. Initial experiments were conducted in a fixed bed reactor system. CO_2 and steam/CO_2 mixtures with various catalyst/sorbent combinations were conducted. Three catalyst additions consisting of 2%Ni/MgAl(O), 2%Ni/$NiAl_2O_4$ and 2%Ni/Ni/Al_2O_4 with 0.5%Rhenium were used. Residence time was about 5min and a steam carbon ratio was 4 and all experiments were at atmospheric pressure. Initial conversion of methane was quite low (15-25%) and for non-promoted catalysts dropped close to zero after 2 cycles.

Further experiments were conducted in a small circulating CFB system with a reforming reactor running at 570C and a regeneration reactor at 880C. Quite high yields of hydrogen were obtained and capture efficiency of CO_2 generated was a maximum of about 70% in these experiments. Run time constraints did not allow steady sate operation to be reached and further experiments are needed. Noble metal promotion of the nickel catalysts seems to be essential.

2.3 Nikos Giannakeas, Leeds University

Described experiments on the chemical loop reforming of waste tyre pyrolysis oil (WTPO) using a mixture of catalysts and sorbant. Experiments so far have been conducted in the presence of around 80% nitrogen diluent. The cycle is in three steps. Starting with a reduced catalyst steam and WTPO are reformed at 750 C to produce hydrogen. The sorbent becomes loaded with CO_2. The nickel in the catalyst is then oxidized with air but without raising the temperature enough to release CO_2. Finally the

WTPO feed is resumed and this reduces the nickel as well as heating up the sorbent to release the captured CO_2. Reforming starts once about 90% of the nickel is reduced and hydrogen in then produced in this part of the cycle. So far the experiments have shown that the nickel can be successfully regenerated but both catalyst and sorbent capacity degraded steadily over the 6 cycles of the experiments. So far experiments have been conducted diluted with nitrogen and have aimed at production of purer hydrogen and not capture of CO_2.

Session 3: Alternatives

3.1 Erin Kimball, TNO

TNO have tested CLC of syngas in 10w, 100w and now 1Kw fixed bed test rigs. Results are encouraging showing much sharper breakthroughs with larger reactors. Some information towards scaling rules for design of large scale applications has been generated. Iron and copper oxide carriers have been tested. The use of fixed beds would make it easier to operate at pressure and have different pressures for oxidation and reduction.

3.2 Rowena Ball, CALIX/ Australian National University, Australia

Described the mathematics of coupling simultaneous endothermic and exothermic reactions. Where rates and heats of reaction are in balance an extremely thermally stable configuration results. Heat from the exothermic reaction has to transfer through the reactor walls to the endothermic reaction. The concept is easily scalable. Also presented were results for the analysis of the coupling of natural gas or syngas reforming to hydrogen in the presence of a calcium oxide sorbent coupled with calcium carbonate decomposition. Theoretically the efficiency of power generation using this concept was calculated to be only marginally less than NGCC/IGCC processes without capture. A consortium has been formed to further develop this “ENDEX flexifuel carbon capture process”

Session 4: Sorbents

4.1 Roger Molinder, Leeds University

Work has been carried out to better understand the behaviour of sorbants. X-ray scans of partially hydrated CaO powders reacting with CO_2 gas mixtures were performed. This allowed the structure of the powder to be followed as the reaction progressed. This showed that $Ca(OH)_2$ starts to disappear from 200-400C but does not finally go until 600C. They also showed that $Ca(OH)_2$ has a much higher surface area than CaO but that CaO derived from $Ca(OH)_2$ also has a very high surface area.

4.2 Alexandra Charitos, IFK University of Stuttgart

Recent results from operation of a 10kw double fluidized bed reactor capturing CO_2 using two different Swabian limestones were presented. Experiments used flue gas containing 11-14% CO_2 and calcium/CO_2 ratios varying from 3 to 23 were tested.

Experiments used both bubbling and circulating fluidized beds (BFB and CFB). Carbonation was at 630-700C and regeneration using 21-40% oxygen at 850-900C. Solid gas contacting in a BFB is much less than in a CFB and for carbonation a space time value 5 times higher was needed. Sorbant decay was measured and plotted on the basis of capacity v cumulative CO_2/CaO ratio which is the equivalent of number of cycles of a batch experiment. Several sizes of lime (0.1, 0.3, 0.6 mm) were used, precalcined for an hour. Initial conversion was only 12-18% reducing over time to below 10%. Key design parameters were identified. For the regenerator the ratio % carbonation/ residence time needs to be >2.5 hr^{-1} to achieve >90% conversion. For the carbonator a CFB space time of 0.01hr is needed to reach >90% of equilibrium conversion) but for a BFB 0.05hr is needed.

4.3 Bo Feng, University of Queensland

Work continues on development of materials for calcium looping gasification of coal and natural gas. A highly effective sorbent has been developed as reported last year and is a mixture of 100nm CaO particles and 20nm inert particles. The optimum ratio is 82 % CaO which produces a material stable over many cycles which retains a capacity of 40-60 % over many cycles because the nano-particles cannot sinter together. So far this material has been produced by wet mixing and a key task is to produce larger granules of about 30 microns with the same nanostructure for commercial use. Two classes of commercially producible particles are under development; naked particles for use with natural gas and coated particles for use with coal. One of three potential production methods has been tried so far without success. Two other methods will be tried in the coming year.

4.4 Roberta Pacciani, Air Products-MATGAS

Described research into use of lithium orthosilicates as CO_2 absorbants in preference to calcium oxide. Li4SiO4 reacts with CO_2 at temperatures above 300C absorbing about 25% weight with a reaction energy of 70kj/mol to form $Li_2SiO_3 + Li_2CO_3$. The reaction is enhanced by the presence of water. Tests were performed in the temperature range 450 – 700C and pressures of up to 25bar. The effects of SO_2 in concentrations from 20ppm - 0.95%vol were also measured. Tests were performed using a magnetic balance holding a wire with a hook holding the sample. Maximum absorption was found at 550C reducing to very low values by 700C. Uptake was fast, typically about 10 minutes. The material is very stable when cycled at 550C and 600C. Exposure to SO_2 reduced activity progressively. With 0.95% SO_2 activity is lost in 10 cycles whereas at lower concentrations there is steady but nevertheless inexorable reduction. In conclusion the material is a potential sorbent but susceptible to permanent deactivation by SO_2.

4.5 Li Zhenshan, Tsinghua University

Experiments on the rate of carbonation and sulphation of Kelly limestone were conducted. Theseshow the classical relation ship between temperature and extent of reaction which has been measured by other researchers. However it was found that stepwise increases in the temperature and exposure to steam both increase the reaction rate and degree of conversion to extents greater than would be predicted. Atomic field microscopy revealed an explanation for this phenomenon. In the case of sulphation, MgSO4 deposits formed as many tiny islands covering the surface. When the temperature was increased the islands coalesced to become larger and expose more reactive surface. This results in an increase in the rate but also an increase in the amount of material available for reaction. Application of steam was found to have a similar effect on the carbonation reaction.

4.6 Nicholas Florin, Imperial College London

Described results from work on 3 approaches to improvement of sorbent performance. Doping with Magnesium Chloride was shown to have variable effects depending on the type of limestone used. Good results were obtained with Havelock limestone where doping with 0.05M $MgCl_2$ was found to have the optimal effect approximately doubling conversions and improving reaction rate. However higher levels of dopant 0.1M and 0.5M lead to reduced conversion. Mechanical stability as measured by mass loss due to fines formation was improved. However results with other limestones showed different results so that this technique would have to be tailored to the type of limestone. Further research is aimed at understanding the mechanisms through which the dopants work.
A second approach investigated was hydration. Previous work used hydration with humid environment at room temperature but despite a small increase in activity resulted in increased mass loss due to attrition. Experiments in which hydration was done with steam at 200C and 400 C were performed. There was a positive effect at 200C but not at 400C. Mass loss was reduced but was still high especially after the subsequent calcination.

A third approach is to prepare a synthetic sorbent. Material derived from precipitated CaCO3 has a very high activity but is not suitable for use in a fluid bed reactor unless pelletised or combined with a binder. Experiments with pure precipitated material and finely mixed $CaCO_3/Al_2O_3$ derived by mixed precipitation were performed. The optimum performance was from material with 15% binder which showed triple the conversion and about 2.5 times capacity of Havelock limestone. However mechanical stability was poor but could be improved somewhat by a thermal pre-treatment.

Session 5: Chemical Looping Combustion

5.1 Carlos Abanades, INCAR, Spain

Presented the concept of a process for producing hydrogen from natural gas using cyclic operation of a fixed bed reactor containing CaO absorbent and CuO oxidant. The cycle is in three steps and starts with step A in which the bed contains CaO and reduced Cu. Natural gas and steam is fed to the bed which is at about 550C and 20bar and is reformed. The temperature rises to 700C across the reaction front and CO_2 is absorbed by the CaO and this causes the shift reaction to occur simultaneously so that the main products are hydrogen and steam. In step B air is fed at high pressure to the reactor and the Cu is oxidized raising the temperature across the reaction front to 850 C. Nitrogen is recirculated to control oxygen concentration and hence the outlet temperature. These conditions are chosen so that the CaCO3 formed in the first step does not decompose significantly. The hot gas from step B can be expanded in a gas turbine. In the final step C fuel and steam is fed to the reactor which is now run at low pressure, atmospheric or lower. The temperature rises to around 850C due to oxidation of the fuel by the CuO. Calculations show that the process is thermally balanced with a Cu/Ca molar ratio of about 3.1. Initial simulations indicate that the capture efficiency could be 84% and that the thermal efficiency of conversion of the fuel to hydrogen is 58%. All of the remaining energy from the fuel is available at high temperature and thus capable of raising steam at state of the art supercritical conditions for power generation. A patent EP093821692 has been applied for. Funding has been obtained to conduct preliminary lab experiments.

5.2 Tobias Proell, University of Vienna

Presented considerations for scaling up CLC to burn gas and coal. In gas fed systems good methane conversion up to 99% have been obtained in an atmospheric double circulating fluid bed system using NiO based oxygen carrier. Up to 96% capture of CO_2 has been achieved. However the technology has to compete with gas turbine combined cycle which is inherently very efficient. It would be necessary both to scale up and go to pressurized operation to complete.

With respect to coal there are three key steps 1) oxidize volatiles, 2) burn the char, 3) remove ash. Two key problems exist in a circulating fluid bed system. Firstly volatiles in the coal form bubbles of gas in the bed and may rise out of the bed without enough contact with the solids. Secondly because the beds are well mixed unreacted char is carried from the oxidation reactor to the air reactor. A modified reactor configuration is under development to address both these issues. The fuel reactor contains diameter restrictions at intervals, three restriction rings have been used in cold flow model tests. This creates zones of high and low density solids and creates some plug flow characteristics. Now volatiles have to pass through low velocity high solids density zones greatly increasing solid gas contact. As the reactor is less well mixed because of the zoning loss of unreacted char to the air reactor should be reduced. Further investigation with cold flow models are ongoing and future plans would be to build a 150-200Kw system primarily for gasification experiments but with the possibility to test CLC as well.

5.3 Mehrdokht Nikoo, Alberta Innovates Technology Futures

Described specific needs for steam assisted gravity drainage (SAGD) of heavy bitumen from reservoirs. This requires from 2.5 to as much as 5 bbl/steam/bbl bitumen costing upwards of 1.06 Mcf. of Natural Gas per bbl. bitumen. A technique to raise this steam with carbon dioxide capture is needed with an estimated future thermal capacity of up to 12GW. CLC is preferred because despite a higher capital cost the operating cost is only a third of that of the alternatives post or oxy combustion. It also promises a higher efficiency, Post/Oxy/CLC - 69/76/84 respectively. A field demonstration at the 1-10MW capacity is planned in the next 2-3 years.

5.4 A Bischi, NTNU

Presented the BIGCLC project. Work on development of an oxygen carrier based on Manganese ore $MnO\text{-}Mn_3O_4$ is ongoing. Reversible capacity up to 4.9% at 1000C has been measured (7% theoretical) but volume change of 15% causes stresses which embrittle the material. Addition of calcium has been investigated resulting in slightly reduced cyclic capacity of 4.9% but improvements in mechanical strength and reactivity. Addition of Titanium is currently under investigation. A cold flow model has been used to investigate the performance of the dual circulating bed system. This incorporates a divided loop seal for solid flowing from the fuel reactor, part of which are returned to a point partway up the reactor. Staged air injection into the air reactor has also been evaluated. The effects of lateral air injection and bubble cap type injection nozzles have also been tested. The results from the cold flow model testing and materials screening will be incorporated into the design of a 150KW demonstration plant.

Session 6: Large Scale Demonstrations

6.1 Borja Arias, INCAR

Presented the EU framework 7 CaOling project. This is a 1.7MW capture plant using calcium oxide looping technology to be installed in a slip-steam of flue gas from Hunosa's 50MW CFB coal power plant at La Perida in Northern Spain. Main partners are Hunosa, Endesa, CSIC and Foster Wheeler with additional support from Imperial College London, Lappeenranta University of Technology Finland , IFK Stuttgart and CANMET (Canada). The La Pereda double circulating fluid beds will have gas velocities of 3-5m/s similar to that in CFB's and recirculation flows will be controlled in the loop seals. Reactors will be equipped with removable cooling bayonet tubes to allow heat extraction to be varied. The coal fired calciner can be fed either air or oxygen. The unit should become operational around mid 2011. Thereafter conceptual design for a larger 20-30MW unit will be prepared. This is the next step envisaged in the development path and could be implemented as soon as 2014. Following successful operation at that capacity sufficient data should be available to design and construct a full scale commercial capture plant.

6.2 Matteo Romano, Politecnico di Milano

Outlined simulation work done on combining coal power plant with CaO looping CO_2 capture and cement plant. Spent CaO from the coal plant is directed into the kiln of the cement plant reducing overall fuel consumption and CO_2 emissions. The limiting factor is likely to be the content of components from the ash in the spent CaO. In the example calculated Al_2O_3 would be the limiting component but even so around 50% of the clinker production could come form the power plant and fuel consumption of the cement plant would be reduced to 65%.

6.3 Shiying Lin, Japan Coal Energy Centre

Presented recent results from the HyPr-RING project (Hydrogen Production by Reaction Integrated Novel Gasification). Their process runs with a coal gasifier operating at 30bar and a regenerator operating at 1 bar. Solids are fed and removed using lock hoppers. Calcining with steam recycle was found to be better than using a CO_2 recycle. Lower temperatures are need and the activity of the material is higher due to the effects of hydration. Hydration of the lime is also occurring in the gasification section which operates at 650C. Observation of the particles reveals that their bulk shape does not change with time but many cracks in the surface of particles are evident. In this process the sorbent retains its activity over many cycles but the strength of the sorbent declines steadily – from 28 to 15 kg/cm^2 over 20 cycles. Hydration is thus proving to be highly effective in retaining activity. Measurements show that nearly all the ash present in the coal feed can be recovered in concentrated form from the cyclone and filter downstream of the calciner. Most of the sulphur present in the fuel also ends up in this material. Particle size analysis on samples from various points in the process suggests that the sorbent is not significantly reduced in size in the gasifier and undergoes only minor attrition in the calciner. The material collected by the calciner cyclone and filter is much finer.

6.4 Bernt Epple, University of Darmstadt

Outlined the potential efficiency advantages of both CLC and carbonate looping namely that CLC could achieve as low as 4% points lass and Carbonate looping 6% with much of this (around 3%) being for CO_2 compression. Unit costs for capture are estimated as low as €10/ton using CLC and €20/ton for carbonate looping. A feasibility study for full scale 1GW plant shows that 2 calciners of 38 m^2 cross-section and one carbonator of 132 m^2 would be required.

A 1MW test unit is currently in the hot commissioning phase and should become operational shortly. The vessels are a carbonator or air reactor 10m high 600mm dia. And a calciner or fuel reactor 10.5 m high and 400mm dia. Operation is possible in either CLC or carbonate looping mode. The facility includes a water cooled 1MW combustor for biomass or coal. This is 7m in height with and internal diameter of 750mm. The project, short name “ÉCLAIR” is EU funded for the period 2008-2012. Results of experimental operation will be available soon and the intention is to report these in the forthcoming 2nd international conference on chemical looping to be held at TU Darmstadt in Sep/Oct 2012.

6.5 Kumar Patchigolla, University of Cranfield

Reported on experiments in which calcination and carbonation of CaO was carried out with varying amounts of steam at a total pressure of a 15 bar. Steam was found to have a number of beneficial effects. It improved degree of carbonation in the first cycle and also the degree of calcination, though this latter effect may simply be due to the reduction in CO_2 partial pressure. The degree of reaction was measured for multiple cycles and showed a steady decrease with number of cycles. Observations seem to indicate that presence of steam results in larger pores. There is also evidence of transient formation of $Ca(OH)_2$ from XRD measurements.

6.6 Craig Hawthorne, IFK Stuttgart.

Described the design and commissioning of a 200kw pilot plant for calcium oxide looping. The unit comprises 10m high carbonator and regenerator designed for gas velocities of 4-7m/s. The CaO/CO_2 ratio can be varied between 4 and 14 and the looping ratio is controlled by cone valves in the loop seals. These have already been tested by circulation at ambient temperature which shows that the ratio can be varied reliably from 3-15 by changing the valve openings from 10% -45%. The reactors have been hot commission to cure the refractory. The plant is extensively instrumented and start up to establish baseline sorbent performance is planned in last quarter of 2010.

Session 7: Posters

7 posters were on display during the meeting. 3 covered sorbent. ECN showed results of investigations into the structural changes which occur to K promoted hydrotalcites during cyclic SEWGS. In a second poster the effects of magnesium on sorbent performance were shown indicating that loading with a suitable amount can improve performance. ZSW at Baden Wurttenburg, the centre for solar energy and hydrogen research showed the capabilities they have developed for testing limestone sorbent material properties including mechanical strength tests, ash/sorbent melting behaviour and reactivity.

The University of Thessaloniki showed results of sorbent enhanced reforming using:

$$CaO\text{-}Ca_{12}Al_{14}O_{33} .$$

This was found to have good activity over 60 hours and 13cycles with capacity dropping from 55% to 49%. CSIC presented results of work on reactivation of limestone sorbants using hydration with steam. Main conclusions were that the best point to reactivate is after calcination mainly because exposure temperatures above 750C tend to reverse the effect. For a given quantity of reactivation steam it is better to use a high concentration in a small slip stream than to try to re-activate a larger stream.

IFK and EnWB Kraftwerke AG showed details of the 200kw dual bed facility also covered by a full presentation. Cranfield University showed details of a proposed 25kw cold model (1/3 scale) of their hot capture rig for production of hydrogen using sorbent enhanced reforming. The work is undertaken as part of the Joint UK-China Hydrogen Production Network

Session 8: Wrap up Session

It was agreed that the next meeting would be hosted by technical university of Vienna and would be held in the week end August/beginning September. For 2012 the offer last year from China would be followed up. If this does not materialize offers from other no-European destinations will be considered. It was generally agreed that the format with 2 full days of meeting should be continued.