Membrane-Assisted Chemical Looping Reforming reactor for combined ultra-pure H_2 production and CO_2 capture

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Highlights

- Combination of two technologies in a new reactor that is here fully developed
- Ultra-pure H_2 (>99.99%) is recovered using Pd-based membranes
- Environmentally friendly concept with cost of H_2 similar to conventional technology
- Efficient heat integration that allows 91% CO_2 capture

1. Introduction

Globally, more than 95% of the hydrogen is worldwide produced from fossil fuels [1], with the steam methane reforming of methane (SMR) as the most used technology. Around 90% of this hydrogen is used for ammonia production (50%), methanol and refining industries. Giving a closer look to the ammonia industry, about 80% is used for fertilizers, which points out the strong effect of cost of H_2 production on basic products in the agriculture sector. Besides, the SMR technology is a highly energy-intensive process, thus also responsible for a large contribution to the anthropogenic emissions to the atmosphere. With the aim of reducing the CO_2 emissions associated with hydrogen production, a new reactor concept that combines advantages of two different technologies, namely membrane reactors for H_2 production and Chemical Looping for CO_2 capture, has been proposed and demonstrated at lab scale [2]. This reactor concept is named Membrane-Assisted Chemical Looping Reforming (MA-CLR), and has been demonstrated using natural gas as fuel source, though it could also be powered by renewable fuel sources like biogas (thus even leading to negative carbon emissions). Chemical Looping is a technology where a solid (metal) oxide circulates and transports heat and oxygen between two reactors operated at different atmospheres, oxidizing (air reactor) and reducing (fuel reactor), and avoids at any point in the process the mixing of fuel and air, which facilitates CO_2 sequestration. In the fuel reactor the endothermic fuel conversion occurs, and by integrating H_2 selective membranes, the hydrogen produced can be recovered as pure product. This hydrogen recovery allows achieving high yields at lower temperatures by shifting the equilibrium reactions towards the products. In this work the main results of the experimental demonstration of the new reactor concept will be presented and discussed together with a detailed techno-economic evaluation of the process.

2. The reactor concept

The entire reaction system consists of two fluidized bed reactors connected by a riser column and gas sealing units. The air reactor is 15 cm in height and 5 cm in diameter, and is in its turn connected to the riser column of 2 m in height in order to drive the solids towards the fuel reactor, which is 20 and 6.5 cm in height and diameter, respectively. Inside this unit, a commercial Ni-based catalysts supplied by Johnson Matthey is used as heat and oxygen carrier and as catalyst for the reforming reaction occurring in the fuel reactor. Immersed in the fuel reactor, three metallic supported Pd-based membranes prepared at Tecnalia (Spain) have been used for the selective H_2 separation. These membranes have been previously tested for several hundred hours to verify their stability in time. Experiments have been carried out at different operating conditions, viz. temperature, inlet composition and inlet gas flow rates, and the results have been compared with an in-house developed phenomenological model. Furthermore, a techno-economic assessment of this technology has been performed using Aspen Plus, and the results have been compared for the same plant size as the conventional SMR technology with and without integration of CO_2 capture.

3. Results and discussion

The influence of the different operating conditions has been assessed through a detailed experimental study. Some of these results are presented in Figure 1, where the influence of the temperature and the amount of oxygen fed in the air reactor is evaluated in terms of fuel conversion. The use of membranes largely improves the performance of the reactor, and H_2 recoveries above 30% have been measured. This recovery could be increased by working at higher pressures and with more membranes. In the same research line, a detailed techno-economic assessment has been carried out by investigating the cost of H_2 production of the reactor concept projected at industrial scale and comparing the results with conventional SMR technology [3]. The results show that the overall efficiency of the process would be 82%, which is very similar to what is achieved with state-of-the-art conventional technology without CO_2 capture (81%) and much higher than the conventional technology when integrating CO_2 capture (67%). When referring to costs, H_2 could be produced in the MA-CLR at a cost of 0.213 €/Nm^3, almost identical as produced nowadays (0.216 €/Nm^3) with the advantage that 91% of the carbon emissions would be avoided. If CO_2 is to be captured in the conventional SMR, then the cost of H_2 production would increase to 0.282 €/Nm^3, indicating the potential of the here presented and demonstrated MA-CLR.

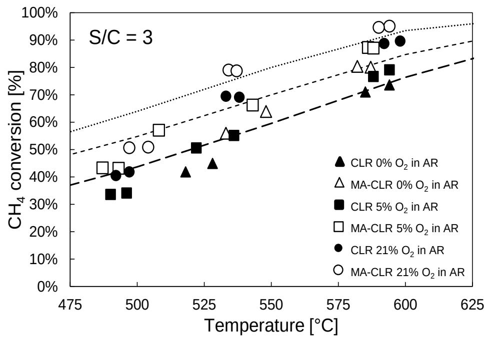

Figure 1. Fuel conversion as function of different operating conditions for the unit operated with (MA-CLR) and without (CLR) membranes

4. Conclusions

In this work a new reactor concept has been proposed and successfully demonstrated at lab scale. This technology has the potential to produce ultra-pure H_2 at operating conditions far from conventional reformer units, with the addition that it achieves high carbon capture rates. Despite the fact that the technology is still in a first stage of development, these promising results, also supported by the results from a techno-economic analysis advise further research in order to bring the technology to the market. The recommended guidelines for further development involve the improvement of existing membranes in order to allow operation at slightly higher temperatures, and more fundamental understanding and engineering development of pressurized circulating beds.

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Keywords

H2 production; Membrane Reactors; CO2 capture; Chemical Looping.

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Dr. in Chemical Engineering

My profile

- Chemical Engineer with experience in R&D on the field of novel processes for H_2 production and their subsequent scale up.
- Multi-task capabilities and always willing to participate as team-worker.
- Very good communication skills, with many presentations in international events as well as teaching/supervision related activities in academia.
- Used to write and handle reports in time.
- More than 18 scientific publications and more than 25 conference contributions.

Office and computing

- Microsoft Office: Advanced level
- Aspen Hysys and Aspen Plus: Advanced level
- Matlab: User level

Languages

Language	Speaking	Reading	Writing
Spanish		Mother tongue	
English	C1	C1	C1
Dutch	B1	B1	B1

Personal grants and awards

- **2017:** PhD with the distinction 'cum laude' at Eindhoven University of Technology. More information can be found here.
- **2017:** Best poster presentation at International Conference on Catalytic Membrane Reactors (ICCMR13) in Houston, USA.
- **2015:** Best oral presentation at International Conference on Catalytic Membrane Reactors (ICCMR12) in Szczecin, Poland.
- **2013:** Personal competitive grant for four years contract at University of Zaragoza (ES) as doctoral candidate as published the 8th February 2013 in the Official Circular in Aragon (BOA).
- **2012:** Second best average grade in Chemical Engineering at University of Zaragoza (ES).

Work experience (more details in the appendix)

- **January 2017-Up to date. Researcher at Eindhoven University of Technology (NL).**
  Participation in the following projects: 1) "MEMERE: Methane activation via integrated membrane reactors" EU Horizon 2020 call SPIRE-05-2015; 2) "PROMECA: Process intensification via through the development of innovative membranes and catalysts" EU Horizon 2020
  Main activities: Techno-economic assessment of novel processes for ethylene and hydrogen production and comparison to benchmark technologies in Aspen Plus.
- **January 2013-January 2017. Researcher at Eindhoven University of Technology (NL) with a doctoral fellowship.**
  Project associated: "ClingO2: Chemical looping reforming for pure hydrogen production with integrated CO2 capture" Project (VIDI 12365)
  Main activities: Development of a new membrane reactor from fundamental research to lab-scale demonstration. In this period I have done activities related to process optimization, techno-economic analysis, development of experimental techniques and experimental operation among others. I have also been involved in process design, P&ID of the novel concept and in preparation of documentation and reports.
- **July 2011-December 2012. Research assistant at University of Zaragoza (ES)**
  Projects participation: (1) "New process to obtain aromatics employing methane from natural gas" and (2) "Process integration by reactor development for glycerol reforming and selective oxidation reactors" Project (CTQ2010-15568).
  Main activities: In this period I have done activities related to catalyst preparation and characterization, process optimization and experimental evaluation at lab scale of a reactor concept patented at the University of Zaragoza.

Teaching activities at Eindhoven University

- **Process Design course (Bachelor).** Lecturer and responsible of tutorials
- **Advance Separation Technology (Master).** Responsible of tutorials
- **Separation Technology (Bachelor).** Responsible of tutorials

Academic background

- **2013-2017: PhD in Chemical Engineering at Eindhoven University of Technology (NL)**

- **2011-2012: Post-Master in Introduction to Research in Chemical Engineering and Environmental Technologies at University of Zaragoza (ES)**
  Average mark: 8.86/10 and Post-Master Thesis qualified with Cum Laude (9.7).

- **2009-2011: Master on Chemical Engineering at University of Zaragoza (ES)**
  Average mark: 7.97/10 and Master Thesis qualified with Cum Laude (9.7).
  Award: 2nd best average mark in Chemical Engineering promotion 2011.

- **2005-2009: Bachelor on Industrial Technical Engineering; specialized in Industrial Chemistry at University of Zaragoza (ES)**
  Average mark: 7.06/10 and Bachelor Thesis qualified with Excellent (9.0).

References

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Scientific publications

Journal articles

1. V. Spallina, I. Campos, **J.A. Medrano**, H. Reza, M. van Sint Annaland, F. Gallucci. "Techno-economic assessment of different routes for olefins production through the oxidative coupling of methane (OCM): Advances in benchmark technologies", submitted to Energy Conversion and Management.
2. **J.A. Medrano**, I. Potdar, J. Meléndez, V. Spallina, D.A. Pacheco-Tanaka, M. van Sint Annaland, F. Gallucci. "The Membrane Assisted Chemical Looping Reforming concept for efficient H2 production with inherent CO_2 capture: experimental demonstration and model validation", submitted to Applied Energy.
3. B.S. Patil, F.J.J. Peters, **J.A. Medrano**, F. Gallucci, W. Wang, A. Bogaerts, Q. Wang, G. van Rooij, J. Lang, V. Hessel. "Fertilizing with the wind: Plasma Assisted Nitric Oxide Production from Air in a Renewable Energy Driven Pulse Powered Gliding Arc Reactor for a Containerized Fertilizer Plant", submitted to Energy and Environmental Science.
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7. **J.A. Medrano**, F. Gallucci, F. Boccia, N. Alfano, M. van Sint Annaland. "Determination of the bubble-to-emulsion phase mass transfer coefficient in gas-solid fluidized beds using a non-invasive infra-red technique". Chemical Engineering Journal 325 (2017), 404-414.
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1. F. Gallucci, E. Fernandez, **J.A. Medrano**, D.A. Pacheco Tanaka, M. van Sint Annaland. "Pd-based membranes for high temperature applications: current status", Austin Chemical Engineering 3(1) (2016), 1025.

Book Chapter

1. F. Gallucci, A. Arratibel, **J.A. Medrano**, E. Fernandez, M. van Sint Annaland, D.A. Pacheco-Tanaka. "Hydrogen production with membrane systems". In Hydrogen Generation Technologies, Willey, USA (2017).

2. **J.A. Medrano**, V. Spallina, M. van Sint Annaland, F. Gallucci. “Chemical looping for hydrogen production and purification” In Membrane Engineering for the Treatment of Gases, 2nd Edition, Royal Society of Chemistry, Cambridge, UK (2017)

Other research-related activities

Conferences (* contributions presented by myself)

1. **J.A. Medrano***, M.A. Llosa, M. van Sint Annaland, F. Gallucci, D.A. Pacheco-Tanaka. “Biogas upgrading with carbon membranes: experimental demonstration and benefits evaluation”, in 13th International Conference on Catalysis in Membrane Reactors (ICCMR13), 10-13 July 2017, Houston, USA. Poster presentation. ***Selected as best poster presentation***
2. S. Wassie, **J.A. Medrano***, M. van Sint Annaland, F. Gallucci. “Hydrogen production with integrated CO_2 capture in a novel membrane assisted chemical switching reforming (CSR) reactor”, in 13th International Conference on Catalysis in Membrane Reactors (ICCMR13), 10-13 July 2017, Houston, USA. Oral presentation.
3. **J.A. Medrano***, I. Potdar, J. Meléndez, V. Spallina, M. van Sint Annaland, D.A. Pacheco Tanaka, F. Gallucci. “*Experimental demonstration of a 2 kW Membrane-Assisted Chemical Looping Reforming system for hydrogen production with CO_2 capture”*, in 13th International Conference on Catalysis in Membrane Reactors (ICCMR13), 10-13 July 2017, Houston, USA. Oral presentation.
4. V. Spallina, **J.A. Medrano**, M. van Sint Annaland, H. Godini, F. Gallucci, *“Techno-economic assessment for C_2H_4 production using OCM integrated with membrane and membrane reactor”*, in 13th International Conference on Catalysis in Membrane Reactors (ICCMR13), 10-13 July 2017, Houston, USA. Oral presentation
5. **J.A. Medrano***, V. Spallina, M. Van Sint Annaland, F. Gallucci. *“Chemical looping membrane reformer concept for H2 production and CO2 capture”*, in Process Intensification Network-Netherlands (PIN-NL) spring session, 13th April 2017, Enschede, The Netherlands. Oral presentation. ***Invited speaker***
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9. **J.A. Medrano***, M. Nordio, F. Gallucci, M. van Sint Annaland. “*Measurement of solids circulation rates with optical techniques in circulating beds and comparison to pressure drop methods”*, in Fluidization XV, 22-27 May 2016, Montebello, Quebec, Canada. Oral presentation.
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11. E. Fernandez, A. Helmi, **J.A. Medrano**, K. Coenen, A. Arratibel, J. Melendez, V. Spallina, J.L. Viviente, J. Zuñiga, M. van Sint Annaland, D.A. Pacheco Tanaka, F. Gallucci. “*Palladium based membranes and membrane reactors for hydrogen production and purification*”, in World Hydrogen Energy Conference 2016, 13-16 June 2016, Zaragoza, Spain. Oral presentation.
12. E. Fernandez, A. Helmi, **J.A. Medrano**, A. Arratibel, J. Melendez, J.L. Viviente, J. Zuñiga, M. van Sint Annaland, D.A. Pacheco Tanaka, F. Gallucci, *“Palladium based membrane reactors for hydrogen*

production", in the 12th International Conference on Membrane Science and Technology (MST2015) 1-3 November 2015, Tehran, Iran. Oral presentation

13. V. Spallina, A. Battistella, **J.A. Medrano**, I. Campos, F. Gallucci, M. van Sint Annaland. "*Design and Modelling of novel membrane-assisted reactor concepts for pure H_2 production with near zero CO_2 emissions*", in 6th World Hydrogen Technologies Convention (WHTC), 11-14 October 2015, Sydney, Australia. Oral presentation.
14. R. Voncken, **J.A. Medrano**, I. Roghair, F. Gallucci, M. van Sint Annaland, *"Hydrodynamics and mass transfer phenomena in fluidized bed membrane reactors"*, in 12th International Conference on Catalysis in Membrane Reactors (ICCMR12), 22-25 June 2015, Szczecin, Poland. Oral presentation.
15. **J.A. Medrano***, N. de Nooijer, A. Helmi, F. Gallucci, M. van Sint Annaland. *"Development of a novel experimental technique for the study of mass transfer in fluidized bed membrane reactors"*, in 12th International Conference on Catalysis in Membrane Reactors (ICCMR12), 22-25 June 2015, Szczecin, Poland. Oral presentation. ***Selected as best oral presentation***
16. A. Helmi, **J.A. Medrano**, M. van Meurs, T. Rijmakers, F. Gallucci, M. van Sint Annaland. *"On the concentration polarization in packed bed and fluidized bed membrane reactors"*, in 12th International Conference on Catalysis in Membrane Reactors (ICCMR12), 22-25 June 2015, Szczecin, Poland. Oral presentation.
17. E. Fernandez, **J.A. Medrano**, J. Melendez, J.L. Viviente, D.A. Pacheco Tanaka, M. van Sint Annaland, F. Gallucci. *"Metallic Supported palladium alloy membranes for high temperature (fluidized bed) applications"*, in 12th International Conference on Catalysis in Membrane Reactors (ICCMR12), 22-25 June 2015, Szczecin, Poland. Oral presentation.
18. **J.A. Medrano***, R.J.W. Voncken, I. Roghair, F. Gallucci, M. van Sint Annaland. *"Effect of gas extraction through membranes on the hydrodynamics in fluidized bed membrane reactors"*, in J.M.Burgersdag 2015, 15 January 2015, Delft, the Netherlands. Oral presentation.
19. R.J.W. Voncken, **J.A. Medrano**, I. Roghair, M. van Sint Annaland, F. Gallucci. *"Two fluid modelling of fluidized bed membrane reactors"*, in J.M.Burgersdag 2015, 15 January 2015, Delft, the Netherlands. Poster presentation.
20. **J.A. Medrano***, V. Spallina, M. van Sint Annaland, F. Gallucci, "*The Membrane Assisted Chemical Looping Reforming concept as efficient reactor for H2 production and CO2 capture: a comparison to benchmark technology"*, in NPS14 conference, 3-5 November 2014, Utrecht, The Netherlands. Oral presentation.
21. **J.A. Medrano***, R.J.W. Voncken, I. Roghair, M. van Sint Annaland, F. Gallucci. *"Experimental and numerical study on gas pockets surrounding horizontally immersed membranes in fluidized beds"*, in XXI International conference on Chemical Reactors CHEMREACTOR-21, 22-25 September 2014, Delft, the Netherlands. Oral presentation.
22. **J.A. Medrano***, P. Hamers, M. Ortiz, A. Ramirez, M. van Sint Annaland, G. Williams, F. Gallucci. *"NiO/CaAl2O4 as active oxygen carrier for low temperature chemical looping applications"*, 3rd International Conference on Chemical Looping, 9-11 September 2014, Goteborg, Sweden. Oral Presentation.
23. **J.A. Medrano***, V. Spallina, M. Van Sint Annaland, F. Gallucci. *"Chemical looping membrane reformer concept for H2 production and CO2 capture"*, in 22nd Process Intensification Network (PIN) Meeting, 21 May 2014, Newcastle, UK. Oral presentation.
24. **J.A. Medrano***, V. Spallina, M. van Sint Annaland and F. Gallucci, *"The membrane-assisted chemical looping reforming concept (MA-CLR) for efficient hydrogen production with integrated CO2 capture"*, in 11th International Conference on Catalysis in Membrane Reactors (ICCMR11), 7-11 July 2013, Porto, Portugal. Poster presentation.
25. **J.A. Medrano***, I. Julian, J. Herguido, M. Menendez. "Dual process intensification: membrane reactor coupled to a two-zone fluidized bed reactor (TZFBR) for the catalytic propane dehydrogenation", in 11th

International Conference on Catalysis in Membrane Reactors (ICCMR11), 7-11 July 2013, Porto, Portugal. Oral presentation.

26. **J.A. Medrano***, V. Spallina, M. van Sint Annaland, F. Gallucci. *"A novel reactor concept for hydrogen production with CO2 capture: Membrane assisted chemical looping reforming"*, in Fluidization XIV, 26-31 May 2013, Noordwijkerhout, The Netherlands. Oral presentation.
27. **J.A. Medrano***, I. Julian, J. Herguido, M. Menendez. "Dual process intensification: membrane reactor coupled to a two-zone fluidized bed reactor (TZFBR) for the catalytic propane dehydrogenation", in 9th European Congress of Chemical Engineering (ECCE2013), 21-25 April 2013, The Hague, The Netherlands. Poster presentation.
28. J. Lasobras, **J.A. Medrano**, J. Soler,J. Herguido, M. Menéndez, A. Jimenez, M. da Silva, M.J. Franco, I. Barrio, J. Lázaro. "Mo/H-ZSM-5/bentonite as a catalyst for methane aromatization: optimization of the preparation method", in 11th European Congress on Catalysis (EuropaCat 2013), 1-6 September 2013, Lyon, France. Oral presentation.
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33. **J.A. Medrano**, I. Julián, F.R. Garcia-Garcia, K. Li, J. Herguido, M. Menéndez. "Two Zone fluidized bed reactor with hollow fiber palladium membrane for Catalytic Propane Dehydrogenation", in I International Congress of Chemical Engineering (ANQUE), 24-27 June 2012, Seville, Spain. Oral presentation.
34. **J.A. Medrano***, I. Julián, F.R. Garcia-Garcia, K. Li, J. Herguido, M. Menéndez. "Reactor de lecho fluidizado de dos zonas con membrana permeable al hidrogeno para deshidrogenación catalítica de propano", in I Jornadas de Jóvenes Investigadores I3A, 26-27 April 2012, Zaragoza, Spain. Oral presentation.
35. **J.A. Medrano**, I. Julián, F.R. Garcia-Garcia, K. Li, J. Herguido, M. Menéndez. "Two Zone fluidized bed reactor with hollow fiber palladium membrane for Catalytic Propane Dehydrogenation", in International Workshop on Inorganic Membrane Technology-Advanced Production & Design, 27-28 March 2012, Montpellier, France. Poster presentation.

Courses

1. Course on Process Economics and Cost Engineering, National Research School in Process Technology OSPT, 8-11 November 2016, Eindhoven University of Technology, Eindhoven, The Netherlands.
2. Course on Advanced Thermodynamics, National Research School in Process Technology OSPT, 29 February-1 March 2016, University of Delft, Delft, The Netherlands.
3. Course on Particle Image Velocimetry, 10-14 March 2014, German Aerospace Center (DLR), Gottingen, Germany.
4. Course on Computational Fluid-dynamics, National Research School in Process Technology OSPT, 18-22 November 2013, Eindhoven University of Technology, Eindhoven, The Netherlands.

5. Course on Numerical Methods for Chemical Engineers, National Research School in Process Technology OSPT, 29 February-1 March, Eindhoven University of Technology, Eindhoven, The Netherlands.
6. Course on Scientific integrity, 2013, Eindhoven University of Technology, Eindhoven, The Netherlands.
7. Course on Supervising Master students, 2013, Eindhoven University of Technology, Eindhoven, The Netherlands.

Workshops

1. "Third European workshop on membrane reactors: Membrane Reactors for Process Intensification – MR4PI2017, 9-10 March 2017, Verona, Italy
2. "International SAOT Workshop on Optical Metrology: in situ optical diagnostics for process technology", 23-24 March 2015, Erlangen, Germany.
3. "Scale up of Pd Membrane Technology: from fundamental Understanding to Pilot Demonstration", 20-21 November 2014, Petten (ECN), The Netherlands.

Supervision of students

Name	Type of project	Affiliation	Dates
Chris Ki	Master thesis	TU/e (The Netherlands)	09/17 – 05/18
Javier Noriega	Master thesis	TU/e (The Netherlands)	09/17 – 05/18
Lukas Burg	Research project	Bremen University (Germany)	09/17 – 02/18
Philipp Kenkel	Research project	Bremen University (Germany)	09/17 – 02/18
Timo Hemmers	Research project	Bremen University (Germany)	09/17 – 02/18
Serena Fausto	Research project	University of Salerno (Italy)	03/17 – 08/17
Valentina Cecheto	Master thesis	Politecnico di Milano (Italy)	02/17 – 11/17
Federico Basile	Master thesis	University of Calabria (Italy)	02/17 – 07/17
Cristina Herrero	Master thesis	Universidad de Zaragoza (Spain)	09/16 – 02/16
Ishan Potdar	Master thesis	TU/e (The Netherlands)	08/16 – 06/17
Willy Genuard	Research project	Clermont-Ferrand (France)	06/16 – 09/16
Nadia Alfano	Master thesis	University of Salerno (Italy)	04/16 – 09/16
Mustafa Tasdemir	Master thesis	TU/e (The Netherlands)	09/15 – 06/15
Francesca Boccia	Master thesis	University of Salerno (Italy)	09/15 – 04/16
Maria Nordio	Master thesis	Politecnico di Milano (Italy)	08/15 – 03/16
Pauline Gadert	Research project	Clermont-Ferrand (France)	06/15 – 09/15
Alban Gerbault	Research project	Clermont-Ferrand (France)	06/15 – 09/15
Niek de Nooijer	Master thesis	TU/e (The Netherlands)	12/14 – 9/15
Madeline Foussier	Research project	Clermont-Ferrand (France)	06/14 – 09/14
Marina Pilz	Bachelor thesis	TU/e (The Netherlands)	02/14 – 06/14
Jasper van Kampen	Bachelor thesis	TU/e (The Netherlands)	12/13 – 04/14
Steven Rademakers	Master thesis	TU/e (The Netherlands)	09/13 – 06/14
Tim Wilke	Master thesis	TU/e (The Netherlands)	09/13 – 05/14
Arthur Boedec	Research project	National Polytechnic Institute (FR)	06/13 – 09/13
Leo Pasquier	Research project	National Polytechnic Institute (FR)	06/13 – 09/13
Aditi Potdar	Master thesis	TU/e (The Netherlands)	05/13 – 03/14