Integrate for a more efficient future

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Integrate for a more efficient future

Prof. dr. Eng. Fausto Gallucci
February 22, 2019

Inaugural Lecture

Integrate for a more efficient future

TU/e
EINDHOVEN UNIVERSITY OF TECHNOLOGY

Department of Chemical Engineering and Chemistry
Introduction

Mr. Rector Magnificus, ladies and gentlemen, family and friends,

Let me start from the very beginning. As a young boy, living in a village of 3000 people, my dream was to travel (through the universe) and discover new planets and new things. Although I was not able to fulfill the first part of the dream (yet), the discovery of new things almost every day is surely what makes me extremely happy with my work.

During my life, I have met several people who have helped me to realize that so much is unknown about the planet on which we live, enough to fulfill the desire for discovery of millions of researchers.

During my studies, I was fascinated by research on membranes, from the description of permeation in the Transport Phenomena course to the application of membranes for water desalination. I was also fascinated by reaction engineering, although the research in this field was not extremely exciting in the department in which I was studying. I was thus very happy when, while asking for an MSc assignment, I was offered the chance to work on membrane reactors for a reaction that I was told could be interesting in the future. Since then, I have always had some sort of ongoing activity related to membrane reactors.

The research in my group is on integrated reactors, including plasma reactors, with the aim of creating more efficient processes. However, in this inaugural lecture I will start by introducing you to my original and main field of research, membranes and membrane reactors, and I hope that at the end of the lecture you are all convinced of how cool it is to integrate unit operations in a single, smaller reactor. I would also like to share my opinion on education at the university level and on the way the research is carried out.
Why efficiency?

There is large consensus that there is a correlation between the high concentration of greenhouse gases in the atmosphere (CO₂ in particular) and the observed climate change [1]. With the increase in the CO₂ concentration in the atmosphere higher than 400 ppm (see Figure 1), it is increasingly evident that a reduction of greenhouse gas emissions is required to decrease the human impact on the observed climate change. I think there are only three possible solutions to the problem: 1) reduce the number of people; 2) reduce energy use (through the use of renewables and improved efficiency) and 3) capture the CO₂ (at the production point but also in the atmosphere). Obviously, the first one is not a practical solution but will be the only consequence if we continue business-as-usual.

I hope most of you agree that a graph like this indicates the effect of human activities and, I’m sad to say, of human technology on the CO₂ concentration in the atmosphere.

But where is this CO₂ coming from?
In its fifth assessment (see figure), the Intergovernmental Panel on Climate Change reported on 2010’s biggest emitters [3]. 25% of the total emissions were due to production of electricity and heat. This is generally achieved through the burning of coal, natural gas and oil for electricity and heat, which is the largest single source of global greenhouse gas emissions. We should consider that these emissions are, in reality, secondary emissions from the other sectors. Improving the efficiency of the conversion and adding carbon capture technologies would decrease these emissions dramatically. 24% of the total was due to Agriculture, Forestry, and Other Land Use. Greenhouse gas emissions from this sector come mostly from agriculture and deforestation. Improving the efficiency of fertilizer production would decrease these emissions as well.
21% of the total was due to industrial processes. This includes the conversion of fossil energy to chemicals. Improving any conversion process would obviously decrease these emissions. Better still would be to improve efficiency while integrating CO₂ capture technologies and to use alternative feedstocks to fossil. 14% of the total was due to transportation. These emissions are due to the fossil fuels used in engines. These are probably the most difficult emissions as they are too distributed and capturing the CO₂ is not feasible. Thus, we should improve the efficiency of the engines and also produce fuels not based on fossil resources.
6% was due to emissions coming from local heat generation and energy conversion in buildings. These can be reduced if efficient combined heat and power generation systems are used.
10% was associated with other processes. It is interesting to know that at present 2% of these emissions are due to the internet and ICT (a figure which could...
increase to 20% by 2025). So, each time we send and read an email, watch a video or go on Facebook, we are contributing to an increase in these emissions.

These emissions and their increases are all associated with our lifestyles. I used the tool available at http://www.footprintcalculator.org to evaluate what the total consumption would be if all people behaved like I do.

The results were shocking for me. If all people lived like I do, we would annually consume more than 7 times the resources available on Earth. If we look at the results in more detail, most of this is caused by mobility, food (especially packaging) and services. These can all be decreased, as discussed above.

Inorganic Membranes and Membrane Reactors

In 2016, Sholl and Lively reported in Nature that there are 7 industrial separations that are very energy-consuming and can be greatly improved by using membranes instead of conventional processes (see Figure 4) [6]. Their final conclusion was “…current training of chemical engineers and chemists in separations often places heavy emphasis on distillation. Exposure to other operations – such as adsorption, crystallization and membranes – is crucial to develop a work force that is able to implement the full spectrum of separations technologies that the future will require.”

Figure 3. Graphical representation of how I am contributing to the destruction of our world (and why I should do something to remedy this)[5].

Figure 4. US Energy Consumption [6].
But what is a membrane? A membrane is a permeable phase acting as a selective barrier. The transport processes through this barrier depend on a driving force, which can be a gradient of pressure, concentration, pH, etc. Figure 5 shows a typical inorganic membrane (a thin Pd-Ag dense foil), permeable to hydrogen and driven by a difference in hydrogen partial pressure.

As the driving force is generally pressure or concentration, the separation is always less energy intensive than other types that require a phase change or large temperature swing.

Different kinds of membrane do exist, and the first general classification is based on membrane material; we distinguish between polymeric and inorganic membranes. Both have advantages and drawbacks. My research focuses on inorganic membranes. However, TU/e covers a large part of the total research on membranes, as my colleague Prof. Nijmeijer focuses on polymeric membranes.

A membrane can have different functions in a process, as shown in Figure 6. The best-known is membrane separation, in which one component of a mixture is selectively separated by permeation through the membrane. Here, one can also see the difference between a membrane and a filter. Membranes can indeed also separate molecules which are very similar to each other in the same phase - think about the separation of oxygen from nitrogen through a high-temperature ceramic membrane. Membranes can be used as contactors between two phases (gas/liquid), especially because membranes can have contact areas of up to 10,000 m²/m³, compared to 100-500 m²/m³ for well-packed columns. Membranes are used as immobilization media, for instance for enzyme immobilization. And, finally, they can be used for controlled release, such as for drug release or for feeding a reactant to a reactor.

I like inorganic membranes because my research focus is on the integration of reaction and separation, and most of the reactions of industrial interest are carried out at temperatures at which other types of membrane would not survive. Although everything is relative, the typical reactions I study are carried out at high temperatures (up to 800-1000 °C).

All the membrane functions described above can be integrated with reactions in multifunctional systems called membrane reactors. Thus, as a definition, a membrane reactor is a reactor system in which reaction and separation (through a membrane) occur simultaneously.

The idea behind integrating the two functions is that the typical thermodynamic equilibrium limitations of the conventional reactors can be circumvented, so smaller systems are required alongside lower energy requirements. Typically, membrane reactors are extremely helpful when thermodynamic limitations affect either the conversion or the yield of conventional systems (see Figure 7).
Examples of membrane reactors (capturing CO$_2$, converting CO$_2$)

THE CASE OF HYDROGEN PRODUCTION

Hydrogen is a very important chemical for industry. Despite being the most abundant gas in the universe, it does not exist (for long) as a gas on Earth as it easily escapes the atmosphere. This means that hydrogen needs to be produced, and it is generally produced through fossil fuel conversion. The importance of hydrogen is even increasing. It is considered a very good energy carrier [7] as its conversion is very efficient and does not produce CO$_2$ (at the point of use). For example, hydrogen can be used as a storage medium for peak electricity production; in automotive applications, combining electromotors with hydrogen-powered fuel cells also shows an overall efficiency (40-55%) that is significantly higher than internal combustion engines (13-30%).

Pt-based catalysts are commonly used in Polymer Electrolyte Membrane (PEM) Fuel Cells, with the performance of the catalyst decreasing dramatically if carbon monoxide (a main product of most conversion technologies) or hydrogen sulfide are present. For this reason, interest in the production of ultra-pure hydrogen has strongly increased in recent years. It has been demonstrated that by using pure hydrogen produced by membrane reactors in co-generation units, the total efficiency will be increased by roughly 10% [8][9].

Traditionally, hydrogen is produced via steam reforming (SR) of hydrocarbons such as methane, naphtha oil or methanol/ethanol. But on an industrial scale, most of the hydrogen (more than 80%) is currently produced by SR of natural gas, carried out in large multi-tubular fixed-bed reactors. In small-scale applications, two other main alternatives are generally considered along with SR: partial oxidation reactions, with a significantly lower efficiency than SR, and auto-thermal reforming, in which partial oxidation (exothermic reaction) and SR (endothermic reaction) are carried out in the same reactor.

The main drawbacks of conventional SR, partial oxidation and auto-thermal conventional reactors are that all these reactions are equilibrium-limited and
(even in the case of complete fuel conversion) produce a hydrogen-rich gas mixture containing carbon oxides and other by-products. Consequently, in order to produce pure hydrogen, these chemical processes are carried out in a number of reaction units (typically high-temperature reformers, high and low temperature shift reactors) followed by separation units (mostly for pressure swing adsorption). The large number of different process steps decreases the system efficiency and makes scaling down uneconomical. A typical reaction process scheme is shown in Figure 8.

My group and I have been working on hydrogen for a very long time (my PhD work was on pure hydrogen production in membrane reactors). In Eindhoven, with thanks to NWO for the VIDI support, I was able to develop a completely new reactor that combines Chemical Looping (see figure) and membrane reactors with the potential for merging direct CO₂ capture and (ultra) pure H₂ production in an efficient way. I named this Assisted Chemical Looping Reforming (MA-CLR).

The idea behind Chemical Looping is to divide the conversion of the fuel into separated oxidation and reduction stages by means of solid material transferring oxygen. If the reduction and oxidation stages are carried out in different locations, this would be called a metal fuel system (as my colleagues in the Department of Mechanical Engineering are investigating).

In this technology, the separation of oxygen from air is accomplished by fixing the oxygen to a metal (oxide), as schematically presented in Figure 9. This (often supported) metal/metal oxide particle is referred to as an oxygen carrier. While in the so-called air reactor, the oxygen carrier is oxidized (regenerated) and it releases the oxygen needed in the fuel reactor, which works on reducing atmospheres in the presence of the fuel.

The combination of Chemical Looping and membrane reactors has the potential to merge direct CO₂ capture and (ultra) pure H₂ production in an efficient way. A typical reaction process scheme is shown in Figure 8.

Using this process, high hydrogen yields are achieved, but costly high-temperature heat exchangers and complex energy integration among different process units are required to obtain the hydrogen at the desired high level of purity.

Among the different technologies related to the production, separation and purification of H₂, membrane technologies seem to be the most promising, and membrane separation is nowadays increasingly considered as a good candidate to replace conventional systems. The specific thermodynamic constraints that limit traditional reactors can be circumvented by using membrane reactors.

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proper heat balance. On the other hand, when the oxygen carrier is reduced in the membrane reactor, it behaves as a catalyst for fuel reforming. A representation of the MA-CLR concept is shown in Figure 10. In this concept, both catalytic and gas-solid reactions are occurring simultaneously.

The research carried out in Eindhoven has allowed me (with a strong contribution from Dr. Medrano and Dr. Spallina) to develop the technology from a sketch on the VIDI proposal through all the stages, from TRL1 to TRL4-5 (prototype at a lab scale [10][11]), using European terminology. Figure 11 shows some pictures of the setup constructed and tested at TU/e.

When integrating Pd-membranes and Chemical Looping in a single reactor, we are able to produce ultra-pure hydrogen at a lower temperature in an autothermal fashion with integrated capture of CO₂. One would be very happy with these results, but this is very dangerous. It is clear that every time we develop a new reactor, it is often more interesting, higher performing and in some cases ‘nicer’ than a conventional one. But this reactor will only be used if it can be proven that it brings benefits in terms of efficiency and ultimately costs. The main challenge is that a new reactor generally also means that the plant around it has changed, and the only way to calculate the cost of the product is to perform a detailed process design.

We also performed the complete process design of the new concept, using Aspen as the software. A scheme of the plant developed for the new reactor is shown in Figure 12.

To compare the results, one should also carry out simulations of the benchmark technology using the same cost bases. The results of this are shown in the table below. They show that we can be more efficient (and cost-effective) than the conventional process allows, while also capturing (and thus decreasing) 91% of CO₂ emissions. We are now in the process of further scaling-up the system.
Coming back to my membrane reactors for CO$_2$ hydrogenation, methanol synthesis is indeed an equilibrium system that is best represented by the following reactions:

\[
\begin{align*}
\text{CO} + 2 \text{H}_2 &\rightleftharpoons \text{CH}_3\text{OH} \quad \Delta H_{298}^\circ = -90.7 \text{ kJ/mol} \quad (1) \\
\text{CO}_2 + \text{H}_2 &\rightleftharpoons \text{CO} + \text{H}_2\text{O} \quad \Delta H_{298}^\circ = 41.19 \text{ kJ/mol} \quad (2) \\
\text{CO}_2 + 3 \text{H}_2 &\rightleftharpoons \text{CH}_3\text{OH} + \text{H}_2\text{O} \quad \Delta H_{298}^\circ = -49.51 \text{ kJ/mol} \quad (3)
\end{align*}
\]

CO$_2$ is always present, either in the syngas or produced in situ. The third reaction is also very interesting as it is the basis for CO$_2$ reuse into chemical building blocks.

To circumvent the thermodynamic limitation that reduces the yield of conventional systems, two possible routes can be taken: i) smart recycling of the unconverted synthesis; ii) in situ removal of the products of the reaction.

In situ product removal can be achieved by selective adsorption of one or both liquid products. While removing only the methanol would simplify the final product purification, the removal of water is very important as well. By removing water, one can indeed achieve higher methanol production (as Le Chatelier’s effect on the equilibrium reactions is higher) and at the same time can prevent unwanted methanol reforming reactions and deactivation of the catalyst (as water accelerates the crystallization of Cu and ZnO contained in a Cu/ZnO-based catalyst) [12].

My approach was to use zeolite membranes to separate methanol and water through capillary condensation in the well-defined pores of the zeolites. Generally, zeolite membranes are supported on ceramic porous supports. The function of the supports is to mechanically sustain the thin zeolite layer. It is mesoporous or, more often, microporous, so that no external mass transfer resistances are induced by the support itself (see Figure 13).

Methanol production in zeolite membrane reactors has also been studied from an experimental point of view, as reported in one of my first papers [14].
change phase from vapor to gas when exceeding 238 °C. Above 238 °C, there is a weaker increase in CO₂ conversion versus temperature. In fact, at 255-263 °C, both membrane reactors and conventional reactors show similar CO₂ conversion.

The research was very successful (I achieved my MSc degree, a couple of papers and a patent, at least) but the main drawback was related to the difficulty in reproducibly preparing zeolite membranes without inter-crystal defects.

What we are doing at the moment and what we are planning for the future is the application of different kinds of membranes. One type is carbon-based membranes. These are produced by depositing a polymer (or polymer precursor) on a support, followed by the carbonization of the polymer at a high temperature. By controlling the temperature of carbonization and the atmosphere during carbonization, we are able to prepare membranes with different pore sizes and different functional groups in the pores. This will allow us to produce membranes to be used in CO₂ hydrogenation reactions. This will (also) be done in an EU project in which Eindhoven is very well represented, with 2 PhD students and 2 groups involved and a spin-off (Dens) as a partner. A typical example of a carbon membrane, developed in collaboration with TECNALIA, is shown in the following picture.

Another line of research I will follow in the future, already started in the InSciTe Horizontal project, is the use of liquid-supported membranes prepared with new solvents (deep eutectic solvents - DES) in porous membranes (see Figure 16). This would also open opportunities for collaboration within TU/e.
Scaling-up: a way to valorize while generating more research questions

I will be personally satisfied as a researcher if the reactors and systems I am developing end up being used in industry. This will not happen if we do not go through the process of scaling-up.

While it is very exciting to carry out research at a very low TRL level, it is also true that the proof that a technology works or doesn’t is achieved when we move a bit further away from a ‘clean’ lab environment.

In many projects, we have industrial partners that take our technology and try to scale-up. Collaborating with these companies and even testing lab technologies on a somehow larger scale solves technical issues that are otherwise fatal for the technology.

However, technical issues and serendipity are also initiators of new ideas. A typical example is the use of palladium membrane reactors. For a long time, we were able to produce up to 1 m$^3$/h of H$_2$ with a high purity in our lab and the membranes could withstand continuous operation for more than 1000 hr; however, when given to companies they were either ruined or the reactors did not reach this purity and production rate. To solve this problem, we considered new kinds of membrane that are still thin (low amount of palladium) but resistant to ‘technicians and professors’.

We came back to square 1 and developed (after 2 years of research) a new kind of membrane that is ‘protected’ and thus much more feasible for industrial applications. The membrane in Figure 17 has been named double skin membrane and has even been patented. The interesting thing is that while the highest selectivity achieved with previous membranes was 200,000, the new membrane allows up to 3,000,000, and even allows us to repair and upgrade old membranes.

The development of such a system would require i) generating fundamental understanding of DES formation by applying detailed DFT and thermodynamic modelling, for which help from my colleague Tuinier would be fundamental; ii) gaining knowledge of the liquid-supported membrane preparations to be carried out with my colleague Nijmeijer; iii) studying in great detail the prevailing phenomena in gas/vapor separation through novel membranes and being able to model this separation; iv) developing detailed modelling tools for the design and optimization of membrane reactors based on the new membranes and validating these models through dedicated experimental investigation and v) creating a proof-of-principle of the novel membrane reactor concept for methanol production from CO$_2$ and H$_2$.

The following picture shows the approach proposed in this research line:

Figure 16. Schematic representation of the project. From DES formation and membrane production to CO$_2$ hydrogenation in a novel membrane reactor.
Education

One of my preferred mottos in research is “if it is not published, it is not done”, especially because until then we cannot cite our state-of-the-art results. In the same way, I like to say “if it is not taught, it is lost”. Indeed, the nicer part of our job is transferring knowledge to students. I see this as the easiest way to disseminate our knowledge to industries. The students, PhD students and PDEng students that we educate in our university are the future leaders of the (chemical) industry in Europe.

It is also a fact that an excellent university produces so much knowledge that it cannot be contained in a single standard MSc program, so it becomes difficult (if not impossible) to efficiently transfer all knowledge to our students.

I like Strategy 2030 a lot as there is flexibility in the study program. However, there should be a strong backbone (minimum fundamental knowledge) connected to a high flexibility component.

If I take Chemical Engineering as an example, I cannot imagine a chemical engineer who has not had Transport Phenomena and Reaction Engineering as part of their education. I would then allow the students to attach different subjects with different weights (in terms of ECTS) to these two ‘backbone subjects’ until he/she has reached the sufficient ‘knowledge-ECTS’ required for the degree. All the components of the tree should also be given to people who want to enrich their knowledge and just want to have a certificate in a certain area. A representation is shown in figure 18.

In an ideal case, we should also be able to make use of digital examinations in such a way that students can take exams as many times as they want in order to improve their knowledge. At the end of the day, we should be happy as a university if students have achieved the knowledge required to start working as engineers, and not simply if they have passed an exam.

We should also be able to move past the standard 0-10 grading used until now. As Singapore’s Education Minister said, “learning is not a competition” [17].
Research at university

A few years ago, my parents and I decided to go for a week or so to Rome for a short holiday. It was my first time there. One day, the weather was not very good, few people were around in the streets and we were walking to visit a monument. We decided to go through the smaller streets and, while walking, we could hear the sound of water flowing. At a certain point, we entered a very small square and I saw where the sound was coming from: Fontana di Trevi. It was the most beautiful feeling I have ever had in front of a monument. Every time I go to Rome (too often lately), I try to go there and recall the feeling I had.

In October, we organized a workshop in Rome related to an EU project, and one of the social activities was a guided walk to Fontana di Trevi. The guide asked if we knew who designed the monument. Most of us immediately answered Bernini, the
closing words

first, i would like to thank the executive board of eindhoven university of technology and the board of the department of chemical engineering and chemistry for their trust and support during this appointment. and i want to thank emiel hensen, dean of my department, for the great help in my promotion, the continuous support and the pleasant working relationship.

during this time, i have met several people who have influenced my career and to whom i would like to say thanks.

i was introduced to the world of membranes by prof. enrico drioli, to whom i am very grateful. he was also my chemistry teacher, who started the first lecture of the course with atoms, molecules and orbitals and, after 25 minutes, was discussing membrane desalination plants in japan. he also asked me to be a tutor and then teacher of the chemistry course, which has helped me quite a lot in my career.

during my time at the institute on membrane technology, i was working with dr. angelo basile and i learned a lot from him. this opened many doors in my later career.

i am in the netherlands because of a colleague and friend of mine, dr. giampaolo manzolini (politecnico di milano). he called me and told me i absolutely had to apply to go to ecn (in petten). i did, and somehow my cv reached twente, where i soon started as a postdoc.

in twente, i was working in the group of prof. hans kuipers, and in particular with prof. martin van sint annaland. after 1 year as a postdoc, halfway through my contract, i received the position of tt assistant professor, and we then all moved to eindhoven together. i have learned a lot from both of you.

while working on my network, i met very important people for my career. above all, a group of people that became not only close partners but also close friends. i would like to thank dr. jose luis viviente sole. i have learned a lot from you about the management of projects, consortia, meetings etc. i hope to continue to learn for many years to come. and thank you for supporting my jokes.
Dr. Alfredo Pacheco Tanaka, I was reading your papers while in Italy and it was a privilege for me when we started working together. I have discovered some of your 1001 scientific skills and I hope to discover the rest. It is a pleasure to talk with you, whether it is about a new membrane or about when we decided to produce catalysts from frozen potato.

Jon Zuniga, I should write a book on you. In short, thanks for all the work you have done. If I achieved so much, it is also because of you. I do not know how many 1000s of mails, messages and calls we have had in the past years. It was a pleasure to see your membrane group grow and become one of the biggest and more active groups in Europe. And thanks for letting me feel like a part of Tecnalia. Anyway, my offer for you to become my PhD student is always valid.

Part of my lecture was about research and big names. Well, I would not be a professor without the excellent work of all the students, PhD students and postdocs that have worked or are working with me. Every one of you has contributed to my career. It was a pleasure to be your mentor and, most importantly, your colleague during your stay at Eindhoven. And I want to thank Joris Garenfeld, too many times I told you I just need “a kind of tube” and finally it was a fully automatic setup.

A successful career starts in the childhood, un grosso grazie ai miei genitori Anna e Guido che mi hanno sempre supportato sin dai primi anni di studio. My father literally travelled the 7 seas (several times in nearly 40 years) to make our lifestyle and education possible.

Ai miei Suoceri, Mela e Toto’, che mi hanno sempre aiutato, supportato e sopportato da quando ci siamo conosciuti. Ci rendete sempre la vita facile, pur essendo lontani.

A Simona, hai cambiato casa (molte volte) e lavoro, ti sei reinventata sempre per segurmi e far si che potessi seguire il mio sogno. Non sarei qui senza di te.

A Edward e Carmen, la cosa piu’ meravigliosa che ho. Quelli che pagano di piu’ per i miei molti viaggi e lunghe giornate ma che riempiono il cuore con sorrisi e amore. Il futuro e’ nei vostri occhi, ed e’ bellissimo.

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Curriculum Vitae

Prof. Dr. Eng. Fausto Gallucci was appointed full professor of Inorganic Membranes and Membrane Reactors at the Department of Chemical Engineering and Chemistry at Eindhoven University of Technology (TU/e) on February 1, 2018.

Fausto Gallucci studied Chemical Engineering at the University of Calabria (UNICAL, Arcavacata di Rende, Italy), where he obtained his MSc (2001) and PhD (2006) degrees. He performed his PhD research on hydrogen production from methanol in membrane reactors. In 2007, after having held a position as a postdoctoral researcher at the Research Institute on Membrane Technology (in Calabria, Italy), Gallucci moved to the research group Fundamentals of Chemical Reaction Engineering at the University of Twente (Enschede, The Netherlands). In 2009, he was appointed Assistant Professor (tenure track) at the same university. The following year, Gallucci moved to the Chemical Process Intensification laboratory at Eindhoven University of Technology (TU/e, The Netherlands), where he was appointed Associate Professor in 2015, leading experimental work on Multiphase Reactors. In 2018, he was appointed full Professor at the chair Inorganic Membranes and Membrane Reactors.

He has contributed to over 190 peer-reviewed scientific publications and 6 patents and patent applications. He obtained several research grants, including an NWO-VIDI, an NWO-Demonstrator, an NWO-Perspectief and 17 European projects (2 of which are starting in 2019), all of them on integrated reactors.
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