

VOL. 52, 2016



DOI: 10.3303/CET1652090

Guest Editors: Petar Sabev Varbanov, Peng-Yen Liew, Jun-Yow Yong, Jiří Jaromír Klemeš, Hon Loong Lam Copyright © 2016, AIDIC Servizi S.r.I., ISBN 978-88-95608-42-6; ISSN 2283-9216

Novel Process for Hydrogen Production Through the Sorption Enhanced Reforming of Methane Combined with Chemical Looping Combustion

Jose R. Fernández*, J. Carlos Abanades

Spanish Research Council, INCAR-CSIC, C/ Francisco Pintado Fe, 26, 33011, Oviedo, Spain jramon@incar.csic.es

A novel process scheme is analysed for the production of hydrogen from methane, while obtaining a separate stream of CO₂, which is suitable for a subsequent reuse and/or for its permanent geological storage. This system comprises three interconnected fluidized-bed reactors operating at atmospheric pressure. H₂ is produced in a sorption enhanced reforming (SER) stage, where the presence of a CaO-based sorbent allows the CO₂ to be removed as it is formed. The main novelty of the process concerns the regeneration of the CaCO₃, required to carry out a multicycle operation. A redox chemical loop supplies the heat necessary for the calcination. The CaCO₃ formed in the SER is put in contact with a very high-temperature solids stream, which is mainly composed of Fe₂O₃ resulting from the exothermic oxidation of Fe₃O₄ with air in a separate reactor (at 950 °C - 1,200 °C). The calciner also acts as a fuel reactor, where Fe₂O₃ is reduced again to Fe₃O₄ by feeding a fuel gas, which is converted to CO₂ and steam. A preliminary thermodynamic assessment of the proposed system has been carried out reaching H₂ equivalent efficiencies of up to 79 %, which are around 9 % points above the efficiency of a reference H₂ production plant based on conventional steam reforming including CO₂ capture with MDEA. Carbon capture efficiencies of around 98 % can be obtained, which are significantly higher than those obtained in the reference plant that uses MDEA absorption (around 85%). These results confirm the potential of this novel system for a future development as hydrogen production process with CO₂ capture.

1. Introduction

Hydrogen has long been an important raw material for the manufacture of commodity chemicals such as ammonia and methanol. Increased quantities of H₂ are now being used in petroleum refining, metallurgical processes and also in recent applications as fuel cells and combustion engines, which could result in large H₂ demands in a medium-to-long term (IEA, 2006). There is also global concern about reducing anthropogenic CO₂ emissions to the atmosphere to mitigate global warming. Among the different options presented, carbon capture and storage arises as a robust and viable solution to drastically reduce global CO2 emissions (IPCC, 2014). Steam Methane Reforming (SMR) is the most widely used technology to produce H₂ at commercial scale. SMR is typically carried out in Fired Tubular Reformers (FTR) at high temperatures and high pressures to overcome the thermodynamic constraints of reforming reaction and thus reach high H₂ yield. Moreover, SMR requires a complex arrangement composed of high number of stages with different catalysts and consumes large amount of energy due to the high endothermicity of the reforming reaction (Li et al., 2016). In the Sorption Enhanced Reforming (SER), the use of a CaO-based material combined with the reforming catalyst allows the CO₂ to be removed as soon as it is formed from the gas phase, generating CaCO₃. The equilibrium is then shifted to H_2 formation, with the result that almost complete conversion of the fuel gas to H_2 is achieved in one single stage under moderate conditions of pressure and temperature. CaO-based sorbents can be obtained from a wide range of abundant, natural and inexpensive sources, such as limestone, dolomite or calcium hydroxide and their presence in the reformer minimizes coke formation and diminishes the steam requirements of the process (Harrison, 2008). The CO₂-sorbent needs to be continuously regenerated to carry

out a multicycle operation. The calcination of CaCO₃ is a highly endothermic reaction that demands temperatures around 900 °C in pure CO₂ and at atmospheric pressure. The main challenge to develop the SER technology at a large scale lies in finding a process scheme that allows the energy required for the calcination to be supplied with minimum energy penalties. In this work, a Fe2O3/Fe3O4 chemical loop is proposed to supply the heat for the calcination on the basis on the scheme described by Fernández and Abanades (2016), which was envisaged for the calcination of CaCO₃ in cement plants. The Chemical Looping Combustion (CLC) principle consists in the transfer of oxygen from air to the fuel using a solid oxygen carrier (typically a metal oxide), avoiding any direct contact between the fuel and air. In a first stage (fuel reactor), the fuel is oxidized to CO₂ and steam by a metal oxide that is reduced. In a second stage (air reactor), the reduced solid is oxidized with air and the material is regenerated to start a new cycle. The net chemical reaction over the two stages is the same to direct combustion of the fuel with air. Therefore, it is possible to carry out a combustion process with inherent CO₂ separation with minimum energy penalties (Adanez et al., 2012). Iron-based oxygen carriers are especially suitable for this application because they are cheap materials and present good stability under repeated oxidation/reduction cycles at high temperatures. This work proposes a new process for H₂ production with CO₂ capture using a CaO-based sorbent, where the calcination of CaCO₃ is carried out by means of indirect heating using iron-based carriers that come at very high temperature from the air reactor of a CLC system. The main operating conditions and design parameters are discussed using the available knowledge in the literature on the key reactions involved.

2. Process description

The H₂ production process proposed in this work (see Figure 1) comprises three main reaction stages, which are carried out in a compact arrangement of interconnected fluidized-bed reactors operating at atmospheric pressure. In the first stage, a low pressure H₂-rich gas (stream 5 in Figure 5) is produced through the sorption enhanced reforming (SER) of CH4 (stream 2) in the presence of a Ni-based reforming catalyst combined with CaO, that acts as CO₂ sorbent. This stage is expected to be fast and efficient in a circulating fluidized bed (Reformer Carbonator) at a temperature around 650 °C, as it is the case in CFB carbonator reactors in calcium looping post-combustion systems (Arias et al., 2013). Under these conditions, both reforming and carbonation reactions are highly favoured, so that almost complete conversion of CH4 to H2 and a drastic CO2 removal from the gas phase can be achieved (Harrison, 2008). The product gas will contain around 90 - 95 % vol. of H₂ (depending on the steam-to-carbon ratio in the feed) and less than 1 % vol. of CO and CO₂. After cooling and heat recovery, gas from the reformer is compressed to be purified in a pressure swing adsorption (PSA) unit, where high pressure pure H₂ (stream 6) is obtained. After the separation from the product gas in a downstream cyclone, the carbonated particles formed in the SER (stream 7) come into a second fluidized bed (Fuel Reactor Calciner) to carry out the regeneration of the CO₂ sorbent. The heat required for the calcination of the CaCO₃ is supplied by a very high-temperature stream of iron-based oxygen carrier (stream 17), which is mainly composed of Fe₂O₃ resulting from the exothermic oxidation of Fe₃O₄ with air in a separate reactor (Air Reactor) at temperatures higher than 950 °C. During the calcination stage, Fe₂O₃ is reduced again to Fe₃O₄ by feeding a fuel gas, which is converted to CO2 and steam. The use of the low pressure PSA off-gas (stream 10), which still contains large fractions of H₂, CO and CH₄ (Table 1), as reducing agent significantly reduces the need to feed additional CH4 to the Fuel Reactor Calciner, which leads to a higher overall efficiency. The typical temperatures required for the rapid calcination of CaCO₃ (860 °C - 910 °C) are favourable the reduction of Fe₂O₃ to Fe₃O₄ to be effective for a wide range of fuels (Yan et al. 2015). The product gas (stream 11) will be mainly composed of CO₂ and steam, but low amounts of unburnt compounds resulting from the incomplete oxidation of the fuel gases can be also present. In this case, an additional oxygen polishing step to complete the combustion of the gas is required in order to obtain a final concentrated stream of CO₂ at the exit of the compression and purification unit (CPU), which is suitable for reuse or purification, compression and storage. In order to reduce the energy demand of the calcination stage, a preheating system composed of at least two cyclones in series is proposed to increase the temperature of the carbonate particles (Romano et al., 2011) before entering the fuel reactor calciner. The solids enter at the top side and while they are passing through the cyclones are heated by a gas stream at a higher temperature that flows in countercurrent. The hightemperature gas (stream 18) generated in the Air Reactor can be used in a first heating stage and the CO2rich gas obtained in the fuel reactor (stream 11) can be used in the last cyclone to heat the solids up to temperatures around 750 °C avoiding any previous carbonate decomposition (Fernandez and Abanades, 2016). From the reduction/calcination stage, a mixed stream of solids mainly containing Fe₃O₄ and CaO is obtained. The use of a downstream segregator will highly improve the performance of the system, since large amounts of iron oxide would act as thermal ballast in the SER stage, while the oxidizing atmosphere and the high temperatures achieved in the Air Reactor would negatively affect the reactivity of the reforming catalyst and the CaO (Gonzalez et al., 2008), respectively, after multiple cycles.



Figure 1: Scheme of the proposed H₂ production process combining SER and Chemical Looping Combustion

The segregator needs to be operated under bubbling-bed regime in order to facilitate the segregation of the Fe-solids from the calcium-based and reforming catalyst before they enter the subsequent reaction stages of the process. The driving force for the separation of the solids will be the ratio of particle densities and of particle sizes between the solids (Naimer at al., 1982). This is a similar operation to that envisaged for carbon strippers under testing in other CLC systems with solid fuels to separate unconverted char from the oxygen carrier (Adanez et al., 2012). Under suitable fluidization conditions, very high separation efficiencies can be achieved (up to 95 %, comparable to those obtained in carbon strippers of CLC), since the CaO and reforming catalyst particles (porous solids with apparent density of around 1,800 kg/m³) will tend to float, while the denser Fe-based particles (with apparent density of around 4,000 kg/m³) will sink in the reactor. The solid stream rich in CaO and reforming catalyst (stream 14) is directed to the Reformer Carbonator for a subsequent SER stage, while the solid stream rich in iron oxide (stream 15) is fed into the air reactor, which operates as a circulating fluidized-bed (CFB), being oxidized with air (stream 16). The oxidation is carried out with a little excess of oxygen to ensure the complete oxidation of the Fe-based particles. The maximum temperature achieved in the Air Reactor will depend on the active content of the Fe-based solids, which will affect the heat transport capacity form the Air Reactor to the Fuel reactor Calciner. The O2-depleted air released at very high temperature (stream 18) is a high quality source of thermal power that can be used for preheating requirements and/or also for power generation in a steam cycle. Meanwhile, the oxidized solids (stream 17) will be fed into the Fuel Reactor Calciner to complete the cyclic operation.

3. Results and discussion

Mass and energy balances involved in every stage of the proposed process were solved including a complete thermal integration between the solids and gas streams included in Figure 1. Adiabatic conditions in all the units were assumed taking the thermodynamic properties of the substances involved in the process from Barin (1989). Commercial Fired Tubular Reformer (FTR) plant produces around 30,000 Nm³/h of H₂ (about 90 MW) at 29 bar, which is a common size for H₂ plants installed in refineries. A reference case assuming this production of H₂ in the SER stage of the proposed process is analyzed to theoretically demonstrate its viability. Reference H₂ production plant with CO₂ capture performed by means of methyldiethanolamine (MDEA) chemical absorption is considered in this analysis for purposes of comparison. A detailed description including a comprehensive thermodynamic assessment of both reference H₂ plants with and without CO₂ capture can be found elsewhere (Martínez et al., 2014). Different performance indexes have been defined to analyze global operation of the proposed process and compare it with those of the reference plants with and without CO₂ capture. Hydrogen yield is defined as moles of H₂ produced per mole of CH₄ fed into the SER stage. Overall H₂ yield is referred to the total moles of CH₄ introduced into the process. Hydrogen production efficiency (η_{H2}) is the ratio between the energy of the H₂ output and the energy of the CH₄ fuelled into the plant (both based on LHV). To consider the contribution of the electricity and heat flows exchanged with the exterior, an equivalent H₂ production efficiency ($\eta_{eq,H2}$) has been defined (Martínez et al., 2014). The CO₂ capture efficiency is evaluated using the carbon capture ratio (CCR) calculated as the ratio between the mass flow rate of CO₂ arising from the CO₂ compression and purification unit (CPU) and the mass flow rate of CO₂ associated to the total CH₄ fed into the plant.

Fable 1: Temperature	, flow rate and co	omposition of the	main gas stre	eams of the propo	sed process in Figure	1
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Streams	1	2	3	4	5	6	10	11	12	13	16	18	19
T, ⁰C	25	350	350	25	650	35	35	870	742	30	25	975	711
Flow,mol/s	131	119	296	13	528	372	73	217	217	126	364	293	293
Composition, vol.%													
CH ₄	100	100	-	100	2.3	-	16.3	1.8	1.8	-	-	-	-
H ₂ O	-	-	100	-	15.8	-	-	39.4	39.4	-	-	-	-
H ₂	-	-	-	-	78.3	100	56.7	-	-	-	-	-	-
CO	-	-	-	-	2.6	-	18.4	-	-	-	-	-	-
CO ₂	-	-	-	-	1.2	-	8.7	59.4	59.4	97.0	-	-	-
O ₂	-	-	-	-	-	-	-	-	-	2.0	21.0	1.8	1.8
N ₂	-	-	-	-	-	-	-	-	-	1.0	79.0	98.2	98.2

In this study, the Ca-based material is assumed to contain 20 wt.% of active CaO and the Fe-based solid consists of 30 wt.% of Fe₂O₃ over alumina, which are conservative values compared to the usual active contents of these materials in CLC systems (Ge et al., 2016) and calcium looping applications (Martínez et al., 2014). A typical Ni-based reforming catalyst over alumina with an active phase of 10 wt.% is adopted. A catalyst/sorbent ratio present in the Reformer Carbonator of about 0.3 will ensure the operation close to SER equilibrium (Martínez et al., 2014). PSA is assumed to separate 90 % of inlet H₂ as high pressure and pure gas (Martínez et al., 2014). Due to the large difference in solids densities, very high separation efficiency (about 95% in lighter solids and 100 % in denser solids) can be expected in the segregator (Fernández and Abanades, 2016). As a result, the solids stream coming to SER stage from the segregator (stream 14) will not contain Fe-based particles, while the solids stream guided to the Air Reactor (stream 15) will only contain less than 2 wt.% of CaO and reforming catalyst. Table 1 indicates the temperature, flow rate and composition of the main gas streams obtained by solving the mass and heat balances at each stage of the proposed process. As can be seen in Figure 2a, the SER operation must be carried out at temperatures no higher than 650 °C to ensure a product gas with a very high content of H₂ (higher than 90 vol.% on a dry basis) and negligible amounts of CO_2 . High steam-to-carbon molar ratios (S/C) promote the conversion of CH_4 to H_2 at the expense of higher steam requirements (and therefore higher energy demand).



Figure 2: a) Effect of steam-to-carbon molar ratio (S/C) and temperature on sorption enhanced reforming (SER) equilibrium at atmospheric pressure. b) Effect of the S/C molar ratio (using PSA) on the H₂ yield

Performing the SER stage at 650 °C with a S/C molar ratio of 2.5 (119 mol/s of inlet CH₄ in the reference case), a flow gas of 528 mol/s is produced in the Reformer Carbonator containing around 93 vol.% of H₂, on a dry basis (see Table 1). A flow of solids of 31.6 kg/s (stream 14) comes from the segregator at 870 °C. Under these conditions, both inlet CH₄ and steam need to be preheated up to 350 °C to carry out the SER stage al 650 °C. The product gas is cooled to 35 °C and subsequently compressed up to 30 bar to be purified in a PSA unit. A flow of 372 mol/s of pure H₂ at 29 bar (stream 6) arises from the PSA, while 73 mol/s of an off-gas (stream 10) is obtained at near atmospheric pressure. The sensible heat of the O₂-depleted air leaving the air reactor allows the mixture of carbonated particles and reforming catalyst coming from the SER stage (35.5 kg/s, stream 7) to be heated up to 711 °C in a first preheating stage. Subsequently, the sensible heat of the CO₂-rich gas obtained in the Fuel Reactor (containing 59.4 vol.% CO₂) is sufficiently high to heat stream 8 up to 742 °C while avoiding any carbonate decomposition (PCO_{2eq,720°C}=0.08). A minimum temperature of 870 °C (PCO_{2eq,870°C}=0.65) in the Fuel Reactor is assumed in order to ensure the complete calcination of the calcium carbonate fed into this reaction stage (i. e., 6 °C above the temperature corresponding to the equilibrium for

the CO₂ partial pressure of 0.59 bar present at the fuel reactor outlet in the reference case). In this case, the heat flow required to carry out the operation is provided by a stream of 229 kg/s (mainly containing Fe₂O₃based particles) arriving from the Air Reactor at 975 °C. As can be seen in Figure 2b, lower S/C molar ratios reduce CH₄ conversion to H₂ and therefore lower H₂ yields in the SER reactor are achieved. However, the reducing capacity of the PSA off-gas increases and a lower flow of additional CH₄ is needed for the reduction of the Fe-based particles, improving the overall H₂ yield of the process. Moreover, the use of the PSA off-gas as reducing agent highly improves the CO₂ capture efficiency, since the carbon associated to the CH₄ fed into the SER stage is finally emitted as CO2 easily purified that leaves the Fuel Reactor. For the reference case, a CCR of about 74 % would be achieved in case the PSA off-gas was not fuelled in the Fuel Reactor. Using this gas in the reduction/calcination stage the CCR increases up to 98 %. An adequate trade-off has to be found for the S/C molar ratio in SER stage in order to minimize the flow of additional CH4 fuelled in the Fuel Reactor while avoiding excessive costs associated to PSA operation. A more detailed heat integration of the process accompanied by an analysis of costs would be required, but this exercise is out of the scope of this work. At 870 °C, the complete reduction of Fe₂O₃ to Fe₃O₄ is feasible. Moreover, H₂ and CO fed into the Fuel Reactor will be totally oxidized to CO₂ and steam, while CH₄ can achieve a very high conversion of around 80 % (Adanez et al., 2012). As explained above, a solids flow rich in Fe₃O₄ (227 kg/s for the reference case, stream 15) is obtained in the segregator and subsequently directed to the Air Reactor. An excess of 7 vol.% of O_2 is assumed for the oxidation stage to ensure the complete conversion of the Fe-particles to Fe₂O₃. Under these conditions, a flow of 364 mol/s of air needs to be supplied (stream 16), obtaining a flue gas of 293 mol/s containing 1.8 vol.% of O₂ (stream 18). High proportions of inert in the iron-based particles will lead to lower oxidation temperatures in the Air Reactor, since inert phase will act as thermal ballast. However, there will be an increase in the heat transport capacity from the Air Reactor to the Fuel Reactor, at the expense of a higher circulation of solids between the reactors to fulfill the mass balance of the system. Consequently, the energy demand for the CaCO₃ calcination will be reduced, improving the energy efficiency of the process (see Figure 3). For the reference case (i. e., 30 wt.% of Fe₂O₃ in the iron-based particles), a maximum temperature of 975 °C is achieved. A solid/gas ratio of around 20 is needed in the Air Reactor in these conditions, which is within the usual operational range of large-scale circulating fluidized beds reported in the literature (Reh, 1995).



Figure 3: Effect of the active content of the iron-based material on the energy efficiency of the process

Table 2 summarises plant performance indexes obtained for the reference plants with and without CO₂ capture (Martínez et al., 2014), together with those obtained for the proposed H₂ production process combining SER with CLC. The FTR plant without CO₂ capture operates with relatively low S/C molar ratios because the resulting off-gas from the PSA is burned in the FTR furnace to reduce the need of additional CH4 as fuel. In the reference FTR plant with CO₂ capture, the S/C molar ratio must be significantly higher to promote the conversion of CH₄ to H₂ in the reformer and subsequently reach a high CO₂ capture efficiency in the MDEA chemical absorption unit. In both plants, there is excess heat from the flue gases, which is recovered producing electricity through a steam turbine. For the commercial FTR plant, a neq.H2 of around 79 % is achieved, while the value for a FRT plant with MDEA absorption is significantly lower (around 70 %) due to the energy penalty associated to the CO₂ capture system. In the proposed SER process combined with CLC, there is no excess heat available from the flue gases once steam and preheating gas requirements are fulfilled. However, a neq,H2 of 78.8 % (comparable to that obtained in a plant without CO2 capture) can be achieved while avoiding more than 98 % of carbon emissions to atmosphere, mainly due to the better H₂ efficiency of the SER process and the very low energy penalties associated to the chemical looping system arranged for the regeneration of the CO₂ sorbent. A more detailed integration of the process will be carried out in future works including a sensitivity analysis of the key design variables in order to determine the conditions that lead to optimal performance in terms of energy consumption and CO₂ capture efficiency.

	Ref. FTR plant*	FTR plant with CO2 capture*	SER+CLC process
S/C molar ratio	2.7	4.0	2.5
Thermal input, MW	121.9	130.8	-
Steam turbine output, MW	3.3	3.8	-
Air Separation Unit consumption, MW	-	-	0.1
CO ₂ compression, MW	-	2.2	2.2
H ₂ compression, MW	-	-	5.7
Other plant auxiliaries, MW	1.0	1.4	0.4
Heat output, MW	8.6	4.1	-
H ₂ yield in Reformer	3.1	3.6	3.5
Overall H ₂ yield	2.5	2.3	2.8
H ₂ production efficiency,%	73.9	68.8	85.2
Equivalent H ₂ production efficiency,%	79.0	70.0	78.8
Carbon capture ratio, %	-	84.9	98.7

Table 2: Performance parameters associated to reference H_2 production plants with and without CO₂ capture and the proposed SER process combined with chemical looping combustion (after Martínez et al., 2014)

4. Conclusions

A detailed process design of a novel H₂ production process which combines SER and CLC has been described assuming a set of reasonable operating conditions. The presence of a CO₂-sorbent during CH₄ reforming enhances H₂ production and simplifies its purification. A redox chemical loop for the calcination of the CO₂-sorbent allows a separate stream of CO₂ to be obtained with minimum energy penalty. H₂ equivalent efficiencies up to 78.8 % (close to that obtained in commercial FRT plants) can be achieved with carbon capture ratios of around 98 %. These results demonstrate the potential of this process as a H₂ production technology with CO₂ capture compared to conventional processes that uses commercial capture technologies.

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