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Initial testing of an inside-out type palladium membrane reactor for recovery of hydrogen from hydrocarbons or water

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ABSTRACT

The ITER Tokamak Exhaust Processing (TEP) system relies on palladium membrane reactors (PMRs) for tritium recovery. The PMR consists of a palladium/silver membrane permeator filled with a catalyst, that can be used to recover hydrogen species (most importantly tritium) from methane and water impurities present in fusion reactor exhaust emissions. Two superimposed phenomena simultaneously occur inside the PMR: a chemical reaction onto the catalyst liberating hydrogen contained in molecules and permeation of this molecular hydrogen through the membrane. Applying a vacuum on the permeate side allows recovery of pure hydrogen from the molecules (including tritium). The inside-out configuration of the PMR improves vacuum conditions at the permeate side, thus enhancing decontamination factor and tritium recovery performances.

This paper presents the design, construction and initial commissioning of a newly erected test rig at UKAEA to perform experiments with protium on an inside-out PMR prototype in support of the ITER TEP. The efficiency of the PMR is assessed by both measuring flowrate through the permeate gas stream and analysing the composition of the retentate gas stream using gas chromatography. Permeation rate is assessed for protium for flowrates between 10 and 100 ml/min. The methane/water ratio, crucial for methane steam reforming tests, is assessed to optimise decontamination of hydrogen and minimise coking of the PMR. The hydrogen recovery fraction and decontamination factor are assessed for methane and water from flow rates of 10 to 60 ml/min.

1. Introduction

A process to recover tritium from hydrocarbons, water, and other tritiated fusion exhaust is required for the sustainable fuelling of a fusion reactor. The ITER fuel cycle deals with, amongst other crucial functions, the exhaust gases through the Tokamak Exhaust Process (TEP) system [1]. A palladium membrane reactor (PMR) recovers tritium from fusion exhaust [2] and has been highlighted as a critical component of the ITER TEP system for dealing with air-like and water-like hydrogen processing [3]. Testing has been completed for outside-in type PMRs [4,5] designed for high throughput-low hydrogen recovery, however little research has been conducted on inside-out type PMRs designed for low throughput-high hydrogen recovery. An outside-in type PMR has a catalyst housed in a vessel, with permeation into an isolated palladium/silver (Pd/Ag) tube; an inside-out type PMR has a catalyst housed in a Pd/Ag tube, with permeation into an isolated vessel.

The PMR is a combined permeator and catalytic reactor used to promote reactions from hydrocarbon and water to molecular hydrogen. These reactions include: the water gas shift reaction (WGS),

$$Q_2O + CO \rightarrow Q_2 + CO_2 \quad (1)$$

And methane steam reforming (MSR),

$$CQ_4 + Q_2O \rightarrow 3Q_2 + CO \quad (2)$$

These reactions occur simultaneously, complementing the process to form (where successful) the combined reaction,

$$CQ_4 + 2Q_2O \rightarrow 4Q_2 + CO_2 \quad (3)$$

where Q represents the hydrogen isotopes Protium (H), Deuterium (D), and Tritium (T).

Constant removal of hydrogen through the Pd/Ag membrane overcomes thermodynamic equilibrium limitations and improves yield. Two product streams leave the PMR: a permeate stream consisting of

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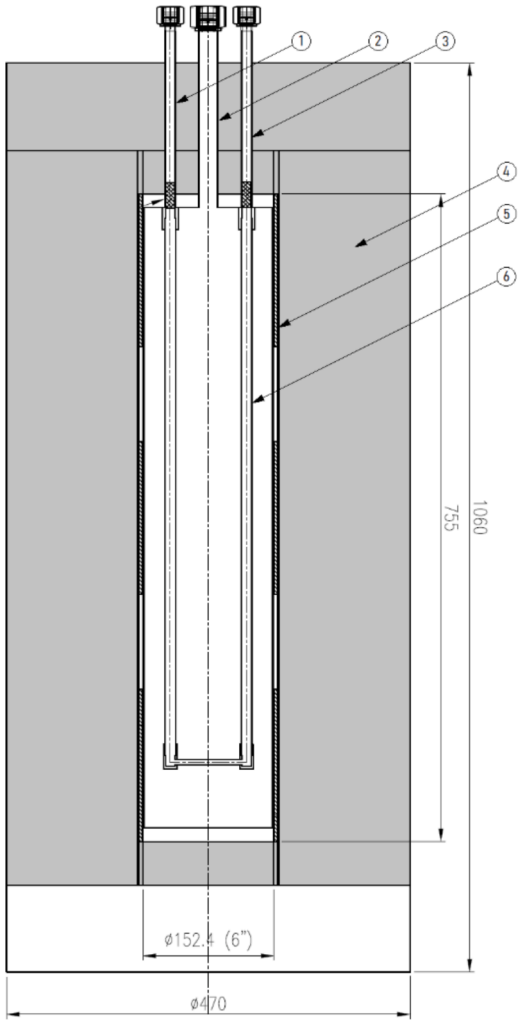

Fig. 1. Palladium membrane reactor design. (1) 12.7 mm OD Pd/Ag tube inlet, (2) 25.4 mm OD permeate outlet, (3) 12.7 mm OD Pd/Ag tube outlet, (4) 150 mm insulation, (5) 152.4 mm ID, 755 mm vacuum vessel, (6) 12.7 mm ID Pd/Ag tube.

ultrapure hydrogen; and a retentate stream consisting CO, CO_2, unreacted feed gases, and any un-permeated hydrogen. This experiment is conducted cold (non-tritiated), using protium only.

2. Experimental setup

2.1. PMR

The PMR, designed and supplied by GE Healthcare, featured a 152.4 mm ID (Inside Diameter) by 755 mm stainless steel (316 L) vacuum vessel (Fig. 1, label (5)) with a single outlet of 25.4 mm OD (Outside Diameter) (2) through which a vacuum could be drawn. The Pd/Ag membrane (75%Pd/25%Ag) (6) has an OD of 12.7 mm, and the tube has an inlet through (1) and outlet through (3), with a thickness of 0.345 mm. The inside of the Pd/Ag tube (6) was packed with catalyst (0.5% Pt on Alumina in 3.175 mm cylinder form). A total weight of 180.26 g of catalyst was added.

Three eltherm® ELK-MI/AY-T 14.5 m heating elements provide 2.28 kW heating each for a total maximum of 6.84 kW heating input, operated using on-off cycling to achieve a desired, steady rate of heating power. Six thermowells are spread across three heights, one set of three for measuring temperature, the other for overtemperature safety. To maintain the required process temperatures, the PMR is insulated with 150 mm of formed MICROTHERM® insulation resulting in a surface temperature below 40 °C when the PMR vessel temperature is at its maximum experimental temperature (550 °C, 823 K).

2.2. Inlet system

Five high purity gas feeds supplied simulated fusion exhaust to the PMR system, individually controlled by Brooks GF040 mass flow controllers (MFCs) calibrated from 0 to 100 ml/min (converted from mass to volumetric flow rate equivalent at 293.15 K and 101,325 Pa and applicable throughout), with stated ±1% accuracy of the full scale. The gases included Methane (99.995%), Hydrogen (99.9995%), Carbon Monoxide (99.97%), 10% Ammonia in Argon (99.9995%), and Argon (99.9995%). Water was introduced in liquid form through a Bronkhorst L01V12 liquid mass flow controller and vaporised along 1.5 m of heat traced ¼" stainless steel 316 L tubing with the temperature stabilised at 393 K to ensure no liquid water was fed to the PMR, shown to the left of the PMR in Fig. 2. ¼" stainless steel 316 L tubing was utilised for the inlet system.

2.3. Retentate system

To ensure water was removed from the PMR, heat tracing extended from the retentate outlet of the PMR to a water trap, where it could be removed (if present) before potentially damaging the Gas Chromatograph (GC) (shown in the top right of Fig. 2). Vaisala HMP7 humidity sensors are positioned before and after the water trap, first to indicate post-PMR water content (i.e. unreacted water) and second to ensure removal of water to levels that will not damage the GC and not lead to build up of liquid water in pipes or the vacuum pump (when evacuating the system). A Brooks GF040 MFC was positioned on the retentate line to indicate retentate flow rate. ¼" stainless steel 316 L tubing was utilised for the retentate system.

2.4. Permeate system

A vacuum was drawn on the permeate (shell) side of the PMR via an

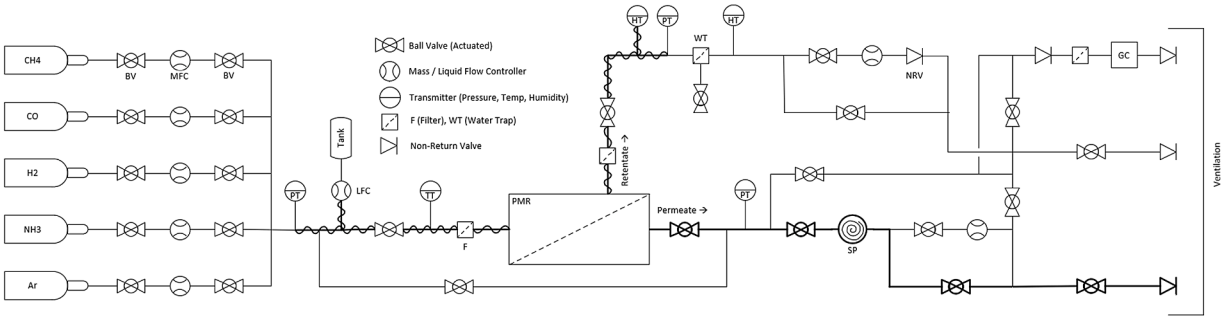

Fig. 2. Simplified process & instrumentation diagram (P&ID) of the PMR experimental setup (SP: scroll pump, GC: gas chromatograph).

Edwards nXDS10i scroll pump, with 25.4 mm stainless steel 316 L tubing to promote conductance. A Brooks GF040 MFC was positioned on the permeate line to indicate permeate flow rate. The permeate MFC showed a base flow rate prior to the introduction of system gases of 0.56 ± 0.05 ml/min averaged over 30 min which was then deducted from the recorded permeate flow rate.

2.5. Gas analysis

Analysis of the retentate gas by GC is the highest accuracy measurement of PMR efficacy available on this experiment (detection limits below 0.05% stream composition for Agilent 490 Micro GC, available for multiple gas species in a single sample, while mass flow controllers typically do not differentiate gas species and (for Brooks GF040) have a stated standard deviation of 0.2%). High ratios of carbon dioxide indicate successful conversion of methane and water through the water gas shift reaction and methane steam reforming. Presence of carbon monoxide indicates a partial reaction, potentially due to a high flow rate, or due to lower temperatures promoting the water gas shift but not methane steam reforming. Presence of hydrogen indicates failure to permeate, either due to high flow rate or coking of the membrane (but not the catalyst). Presence of methane indicates reaction failure, or potentially membrane coking leading to re-combination of carbon and hydrogen after the initial molecular decomposition. As the GC does not detect (and is de-sensitised by) water, high methane presence also infers water in the retentate stream; any methane should react with water, therefore its presence implies water also did not react.

Although isolated from each other during normal operation, the permeate and retentate streams could be routed to the GC for gas analysis. The entire retentate flow stream was routed through the GC filter/bypass which allowed the GC to sample a highly representative retentate stream at regular intervals. An adjustable non-return valve was set to approximately 125 kPa and placed after the GC to increase the retentate and inlet pressure to process relevant levels and provide pressure for the GC inlet. The GC's baseline column pressure was set to 200 kPa and the carrier gas utilised was Argon (99.9995%) to better identify hydrogen.

The GC was fitted with two columns: a Molsieve 5A column designed to separate and detect hydrogen, carbon monoxide, methane; and a PoraPlot U column designed to separate and detect hydro- and halocarbons, and carbon dioxide. For ammonia detection, one column can be replaced with a CP-Volamine column used for separating amines. Calibration of the GC was conducted using Agilent Universal Calibration Gas (5184-351) as well as interval concentrations of pure gases (Hydrogen, Methane, and Carbon Monoxide).

3. Methods

3.1. Pd/Ag membrane preparation

As received, the membrane may not be permeable to hydrogen even at elevated temperatures. Activation and conditioning of the membrane are steps to achieve optimal performance of the PMR unit [5]. Conditioning of the membrane is achieved in two steps: 1) activation of the membrane using air; and 2) conditioning of the membrane using hydrogen. Previous studies have shown that exposure to air (50% air / 50% argon in one study [4] and 10% air mixture in another [5]) increases permeation rate. Further, it both reduces the probability of a decrease in permeation over time and the rate of decrease. Hydrogen conditioning also aids permeation during methane and other process gas feed experiments [4].

3.2. Permeation of the membrane

Quantification of the ability for hydrogen to permeate the Pd/Ag membrane is done by feeding pure hydrogen to the PMR in dead-end mode and measuring the permeate flow rate. The permeation coefficient, the measure of membrane permeability, is calculated using the Eq. (4) with the variables described in Table 1:

Table 1
Permeation coefficient variable definitions.

	Variable	Value	Unit
$P(H_2)$	Permeation flow rate (H_2)	measured	$mol \cdot s^{-1}$
A	Permeation surface area	0.0586	m^2
t	Membrane thickness	0.000345	m
p_f	Pressure (H_2 feed)	measured	Pa
p_p	Pressure (H_2 permeate)	measured	Pa
Π	Permeability	calculated	$mol \cdot m^{-1} \cdot s^{-1} \cdot Pa^{-0.5}$

Table 2
HRF and DF variable definitions.

	Variable	Value	Unit
HRF	Hydrogen Recovery Fraction	calculated	$mol \cdot s^{-1}$
F_{H2O}	Feed flow rate of water	measured	$ml \cdot min^{-1}$
F_{CH4}	Feed flow rate of methane	measured	$ml \cdot min^{-1}$
$F_{ret}(x_{H2})$	Retentate flow rate of hydrogen	measured	$ml \cdot min^{-1}$
$F_{ret}(x_{CH4})$	Retentate flow rate of methane	measured	$ml \cdot min^{-1}$
$F_{ret}(x_{H2O})$	Retentate flow rate of water	measured	$ml \cdot min^{-1}$

$$\boldsymbol{P}(\boldsymbol{H}_2) = \boldsymbol{\Pi} \cdot \frac{\boldsymbol{A}}{\boldsymbol{t}} \cdot \left(\boldsymbol{p_f}^{0.5} - \boldsymbol{p_p}^{0.5} \right) \quad (4)$$

The theoretical permeability has been calculated for H_2 as Π_T = 3.85E-8 exp(−5730/RT) = 1.38E-8 $mol \cdot m^{-1} \cdot s^{-1} \cdot Pa^{-0.5}$ (at 673 K), where R is the gas constant (8.31 $J \cdot K^{-1} \cdot mol^{-1}$) and T is the measured temperature of the reactor (K).

Permeation measurement was conducted by evacuating the system to a pressure less than 1 Pa with the PMR temperature stabilised at 673 K. The retentate and permeate were closed off so any gas introduced through the feed would reside within the pipework. The permeation coefficient was calculated at each collected data point (one second intervals). Permeation measurement was conducted pre- and post-activation of the Pd/Ag membrane.

3.3. Activation of the membrane

The system was evacuated to a pressure less than 5 Pa from the system exhaust non-return valves to the Argon inlet MFC and the temperature of the PMR stabilised at 673 K. Air was introduced through the Argon inlet MFC until the pressure in the Pd/Ag tube reached approximately 10 kPa, whereupon the air inlet was stopped and the pressure held for 30 min. The system was evacuated of gas afterwards. This process is also used for de-coking, which (regardless of requirement) is completed regularly, with permeation measured before and after activation / de-coking.

3.4. Conditioning of the membrane

The system was evacuated to a pressure less than 5 Pa from the system exhaust non-return valves to the Hydrogen inlet MFC and the temperature of the PMR stabilised at 683 K. Hydrogen was introduced through the hydrogen inlet MFC until the pressure in the Pd/Ag tube reached approximately 10 kPa, whereupon the hydrogen inlet was stopped and the pressure held for 45 min. The system was evacuated of gas afterwards.

3.5. Hydrogen recovery fraction and decontamination factor

The hydrogen recovery fraction (HRF), a measure of the relative total of hydrogen recovered from the feed, can be calculated based on feed and retentate measurements using Eq. (5) during WGS experiments and

Eq. (6) during MSR experiments with the variables described in Table 2:

$$HRF_{WGS} = 100\left(1 - \frac{F_{ret}(x_{H2} + 2x_{CH4} + x_{H2O})}{F_{H2O}}\right) \quad (5)$$

$$HRF_{MSR} = 100\left(1 - \frac{F_{ret}(x_{H2} + 2x_{CH4} + x_{H2O})}{2F_{CH4} + F_{H2O}}\right) \quad (6)$$

The accuracy of the GC far surpasses that of the permeate MFC, therefore Eqs. (5) and (6) were used to determine the hydrogen recovery fraction throughout this experiment. As water is removed before entering the GC due to damage possibilities, the retentate flow rate of water, $F_{ret}(x_{H2O})$, was calculated from the total flow, and corroborated with humidity data and the presence of methane and carbon monoxide in the retentate flow.

The decontamination factor (DF) is a ratio of the total hydrogen in the feed against the total hydrogen in the retentate. It is calculated for Water Gas Shift experiments using Eq. (7) and Methane Steam Reforming experiments using Eq. (8) [4].

$$DF_{WGS} = \frac{F_{H2O}}{F_{ret}(x_{H2} + 2x_{CH4} + x_{H2O})} \quad (7)$$

$$DF_{MSR} = \frac{2F_{CH4} + F_{H2O}}{F_{ret}(x_{H2} + 2x_{CH4} + x_{H2O})} \quad (8)$$

3.6. System readiness for process gases

Firstly, the system is initialised into a state ready for the introduction of reactive gases. If in a cold state, the system is evacuated to a pressure less than 5 Pa and the temperature of the PMR raised at a rate of 5 K per minute to protect against thermally induced mechanical stresses to between 673 K and 823 K. Between experiments, the PMR feed and retentate is purged with Argon for 30 min to remove residual gases, including hydrogen, while the permeate is exhausted, to protect against palladium expansion with temperature drop [6] in case of power loss. The PMR is kept at a maximum of 773 K during hot-shutdown events and a pressure of approximately 100 kPa, with the feed and retentate isolated to prevent the catalyst from drying out. The maximum operating temperature is 823 K, above this temperature there is a risk of damaging the PMR. The temperature of the feed and retentate heat tracing is raised to above 393 K to ensure no liquid water is fed to the PMR, which can cause corrosion or damage via a pressure increase onset by rapid water vaporisation. Before process gas introduction, the system control software is initiated, logging pressures, temperatures, valve positions, actual MFC & LFC flow rates, pump demand, and humidity at one second intervals. The retentate and permeate streams are separated, with the retentate stream routed to the GC and then exhausted, and the permeate stream routed through the permeate MFC to the exhaust ventilation. Argon (99.9995%) is introduced at 100 ml/min. Once the system equilibrates, the GC can be initiated, with samples taken in 4-minute intervals (to balance residence time for identification of carbon dioxide). Water is introduced before hydrocarbons or carbon monoxide to reduce the chance of coking.

3.7. Carrier gas effects

To aid in the speed of achieving steady state, a carrier gas can be used to increase the flow rate of the experimental gases. The effect of the carrier gas is assessed by varying the flowrate of the carrier gas without altering the experimental gas flow.

3.8. WGS and MSR operation

The PMR temperature is stabilised between 673 K and 823 K for WGS reactions, and 723 and 823 K for MSR reactions. Below these temperatures, reaction may not occur. Water is then introduced up to 2 g/h (41.5 ml/min steam flow rate). Shortly after water introduction and stabilisation (between 10 and 30 s), carbon monoxide is introduced for WGS experiments at a ratio of 1.09:1 ($CO:H_2O$), or methane is introduced for MSR experiments at a ratio of 1:1.84 ($CH_4:H_2O$). The WGS ratio promotes complete reaction of water and thus hydrogen recovery, essential in tritium operation. Although the range of ratios in MSR is considered broad (good results obtained in previous study consider 1.75–1.84 [5]), 1:1.84 for MSR balances the risk of coking with a lower ratio to detrimental hydrogen recovery fractions with a higher ratio. The argon feed is reduced to compliment the process gases and can be eliminated.

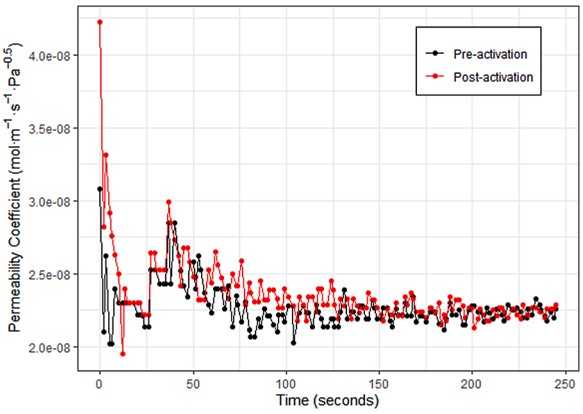

Fig. 3. Permeation coefficient calculated from feed and permeate pressures (Eq. (4)) against time for pre-and post-activated membrane.

The length of an experimental run is a minimum of 2 h, with consideration given to extending this whether equilibrium is attained. Selected runs are tested for an extended period of between 10 and 12 h with a further few tested for longer.

4. Results and discussion

Characterisation and testing of the experimental setup and process strategy included assessing the membrane permeation efficiency and performing initial experimental runs at standard length (approximately 2 h, or more than seven times process gas replacement).

4.1. Permeation of the membrane

Characterisation of the Pd/Ag membrane permeation rate is presented in Fig. 3 with respect to introduction of hydrogen with time and the response in permeability coefficient.

Before activation, the permeation coefficient was $2.22 \times 10^{-8} \pm 5.2 \times 10^{-10}$ $mol \bullet m^{-1} \bullet s^{-1} \bullet Pa^{-0.5}$, while after activation the permeation coefficient was $2.26 \times 10^{-8} \pm 6.1 \times 10^{-10}$ $mol \bullet m^{-1} \bullet s^{-1} \bullet Pa^{-0.5}$. Both measurements were higher than the theoretical permeation coefficient value, stabilizing at 160.4 ± 0.04%, representing ideal permeation conditions. This PMR has been used previously and had been activated and conditioned in the previous months; the lack of any tangible difference pre- and post-activation may show there had been no degradation or coking of the membrane in that time, and its comparison with theoretical values shows it is in good working order. Helium leak testing was conducted to prove membrane integrity.

4.2. Initial tests

4.2.1. Carbon monoxide experiment

Initial standard experimental runs were conducted with carbon monoxide and water to test PMR response to WGS reactions, a component of the fully reacted methane experiments. With the PMR stabilised at 723 ± 0.2 K, carbon monoxide feed at 10.43 ± 0.01 ml/min, water

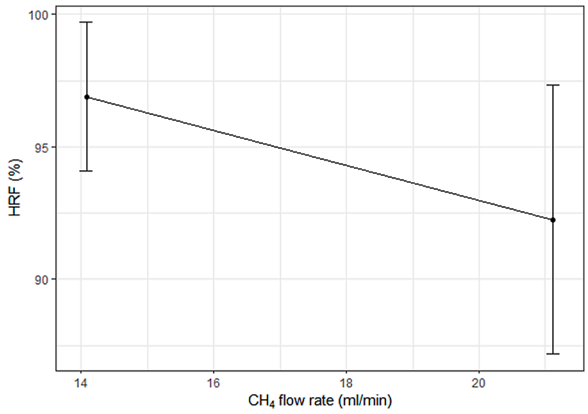

Fig. 4. GC data showing hydrogen recovery fraction (HRF) for varying methane inlet flow rates.

Table 3
Feed parameters for initial CH_4/H_2O experiments.

CH_4 ml/min	H_2O ml/min	Argon ml/min	Inlet Pressure kPa	PMR temp (av.) K
14.1 ± 0.01	25.9 ± 0.06	59.9 ± 0.74	135 ± 2	822 ± 8.60
21.1 ± 0.01	38.9 ± 0.06	39.9 ± 0.01	128 ± 2	830 ± 10.1

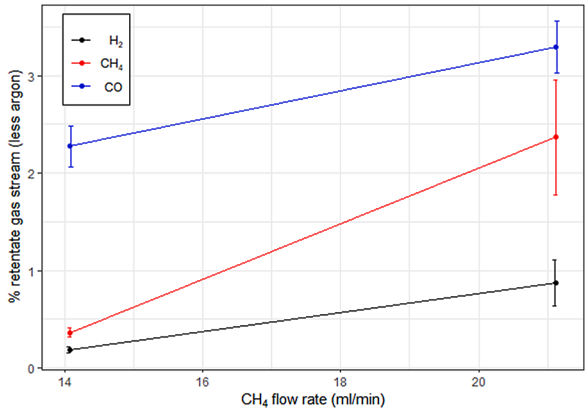

Fig. 5. GC data showing percentage of gases other than CO_2 in the retentate gas stream (less Argon, meaning 100% equals all gases minus argon) for varying methane inlet flow rates.

feed at 9.57 ± 0.01 ml/min (0.46 g/h), and argon at 80.19 ± 0.01 ml/min, HRF and DF were measured at 99.38 ± 0.07% and 164.0 ± 16.4 respectively over a 2-hour period.

4.2.2. Methane experiment

Initial standard experimental runs were conducted for varying flow rates of methane and water to assess the efficacy of the experimental setup.

Data in Fig. 4 is averaged, once steady state is achieved, over 2–3 h continuous operation. Standard deviation varied from ± 1.25% for 14.1 ml/min CH_4 feed to ± 1.30% for 21.1 ml/min CH_4 feed. Inlet parameters were as per Table 3 (shown as returned values).

The CH_4:H_2O ratio was maintained at 1.84 ± 0.005. Permeate pressure rose quickly above the maximum range of the high accuracy permeate pressure sensor (max. range 133.3 Pa). The permeate pressure on the low accuracy-wide range pressure sensor averaged 515 ± 147 Pa and 779 ± 91 Pa, however with the proximity to the bottom of the sensor range these often show higher (300 to 400 Pa) than dedicated low pressure (less than 200 Pa full range) sensors. The decontamination factor spiked early in the 14 ml/min CH_4 experiment, rising above 2000 for a short time before settling at 22.1 ± 8.1 after 1 hour of operation, comparable with the second experiment, 21 ml/min CH_4, with a decontamination factor of 19.3 ± 11.2. The hydrogen recovery fraction was 96.9 ± 2.8% and 92.2 ± 5.1% in the 14 and 21 ml/min CH_4 experiments respectively; arguably equal considering uncertainty. As a high hydrogen recovery fraction indicates, the ratio of CO_2 to all other process gases in the retentate stream – another indicator of successful reaction – equalled 97.2 ± 3.2%.

The relatively large increase in CO in Fig. 5 compared to H_2 and CH_4 indicates partial completion of the reaction. Increase in membrane saturation may cause H_2 to remain present for longer and recombine with O_2.

5. Conclusions

Results achieved were comparable with previous experiments [4,5] and corroborated internally with secondary instrumentation (i.e. GC results matched permeate MFC flow rates). System data-capture facilitates in-depth analysis of relevant hydrogen recovery information, including pressure, temperature, humidity, and flow rate at a minimum capture rate of one datapoint per instrument per second. The GC adequately analyses retentate gas composition.

Initial water gas shift and methane steam reforming results indicate the equipment setup meets experimental aims. Further data capture during the full experimental campaign will highlight hydrogen recovery and decontamination changes of hydrocarbons, water, and ammonia with respect to temperature, carrier gas flow rate, and process gas feed flow rate.

Data availability

Datasets related to this article are available upon request.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgement

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