Experimental study of oxygen transport membranes for oxy-fuel combustion reactors

Ryan FALKENSTEIN-SMITH* and Jeongmin AHN*
*Syracuse University
255 Link Hall Syracuse, NY 13244, USA
E-mail: rlfalken@syr.edu

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Abstract

There is a growing pressure on industry to reduce carbon dioxide emissions from combustion processes while meeting the growing energy demand, resulting in an increase in the development of carbon capture technology. Current practices available, such as chemical looping combustion (CLC) or cryogenic air separation units, separate oxygen from the atmosphere and feed it into combustor to eliminate any nitrogen in the reactor and produce a rich CO$_2$ exhaust that can be captured and contained for future use. However, by implementing these technologies there is a significant energy penalty. One potential alternative is an oxygen transport membrane reactor that has the potential to provide a large amount of high purity oxygen at minimal energy costs. This work investigates the performance of perovskite-type La$_{0.6}$Sr$_{0.4}$Co$_{0.2}$Fe$_{0.8}$O$_{3-δ}$ (LSCF) and SrSc$_{0.1}$Co$_{0.9}$O$_{3-δ}$ (SSC) membrane reactors for the combustion of methane in various configurations. The ceramic membranes exploited here are oxygen semi-permeable, dense ceramic membranes with mixed oxygen ionic and electronic conductivity at high temperatures. Planar and tubular reactors were fabricated and studied with a methane fuel. The oxygen permeability feasibility of the membrane reactors were studied and confirmed. The CO$_2$ selectivity at various test conditions were also reported with the maximum selectivity achieved 87.0% selectivity.

Key words: Oxy-fuel combustion, Carbon capture, Oxygen transport membranes

1. Introduction

Natural gas containing primarily methane is one of the most important energy resources in the world, as its consumption is expected to grow by 50% over the next 20 years (Cavenati et al., 2004). However, over the past two decades there has been a growing resistance to the usage of fossil fuels, since it is estimated that 30 Gt of carbon dioxide is produced each year from combustion processes (Canadell et al., 2007). Already, there has been extensive studies demonstrating the damaging impact that CO$_2$ and other greenhouse gases, which stem from the energy production systems, have on the environment and ultimately human wellbeing. This creates one of the biggest challenges for industry, which is forced to find a way to maintain current and future energy demand while significantly reducing harmful combustion emissions (Herzog et al., 2000). One solution that has been gaining popularity is the idea of integrating carbon capture and sequestration (CCS) technologies into power facilities.

CCS technology consist of several different forms that have shown to capture and sequester carbon at a minimal energy penalty, but one form that has shown more appeal is oxy-fuel combustion due to its ability to be retrofitted into most existing plant designs (Zheng, 2011). Oxy-fuel combustion consists of delivering pure oxygen into a furnace, where it can react with fuel, excluding the presence of nitrogen, and produce higher flame temperatures at a lower fuel consumption rate (Borgert and Rubin, 2013). More importantly, when designed correctly, oxy-fuel combustion has the potential to produce a high concentration of CO$_2$ flue gas which can be captured and sequestered for future use (Borgert and Rubin, 2013). Although the idea is novel, one of the most challenging aspect of oxy-fuel combustion is developing an air separation unit (ASU) that can deliver a high purity of oxygen, without sacrificing a significant amount of power.
Recent research has shifted focus towards chemical looping combustion (CLC), a process that uses a dual fluidized bed system to reduce a metal oxide in a fuel reactor, then re-oxidize said metal in an air reactor, thus avoiding any contact between the fuel and the air (Lyngfelt et al., 2001). Despite its novelty, this process is difficult to repeatedly ensure the same amount of energy transfer to and from both reactors and narrowly limits the selection of available metal oxides.

Alternatively, there has been a growing interest in oxygen transport membranes (OTM), which have the potential to revolutionize the process of delivering a high purity of oxygen to a combustion reactor. Specifically, OTMs are oxygen semi-permeable, dense ceramic membranes based on the composite oxides with mixed ionic and electronic conductivity at high temperatures (Andersen et al., 1994; Bouwmeester et al., 1992; Bouwmeester et al., 1994). When under high temperature conditions (>700°C), OTMs have the capability of creating a chemical potential gradient that filters oxygen from atmosphere and introduces it into a fuel rich environment. This occurs because of the perovskite lattice structure (ABO₃), which facilitates the formation of oxygen vacancies allowing oxygen to move through the membrane via defect reactions and be charge compensated from electron transfer via electron-holes in the reverse direction. After the high purity oxygen is separated from the atmosphere and more importantly nitrogen, it can react with the fuel to produce a higher temperature combustion reaction with a rich CO₂ flue gas which can be capture, stored, and potentially used for other purposes (Ishida et al., 1987).

![Diagram of oxygen transport membranes using methane sweeping gas](image)

**Fig. 1** Concept of oxygen transport membranes using methane sweeping gas where oxygen ions transport through the membrane using oxygen vacancy sites (V_O⁻) in the cubic structure. Upon reaching the sweeping side (fuel rich side), oxygen deionizes and reacts with fuel. Meanwhile electron transfers back through the membrane via electrons holes (h) to further reduce oxygen on the permeating side.

As the demand for ASUs become more pressing, more research has been focused on studying dense perovskite structure’s oxygen permeation performance under high temperatures, but little experimental work has been done that deals with investigating OTM performance for the purpose of oxy-fuel combustion (Ahmed et al., 2014; Mansour et al., 2013; Nemittallah et al., 2013). Consequently, this work focuses on the influence that OTM reactor design has on its performance. Under modified boundary conditions, such as reactor temperature and fuel flow, the OTM reactor’s oxygen permeability and CO₂ selectivity can be studied and provide better insight into OTM oxy-fuel combustion performance.

2. Experimental Design

2.1 Material Selection

In order to properly identify OTM performance under high temperature conditions, it was essential to identify materials that would exhibit strong oxygen permeation performance. Since its initial results showed a high oxygen
2.2 Membrane Reactor for Methane Combustion

Tubular and planar reactors were selected as reactor configurations to further understand OTM geometry relations to oxygen permeation performance. Planar reactors were fabricated using a dry pressing technique which pressed selected powders into disk membranes with diameters of 1.5 cm. Due to its ability to densify at lower temperatures, SSC was sintered at 1100°C for 5 hours, while LSCF was sintered at 1250°C for 5 hours. After sintering, planar membranes shrunk to a diameter of 1.2~1.3 cm and a thickness of 0.04 cm. In order to prevent any leakage, the dense disk membranes were then sealed, using a high temperature adhesive, to a quartz tube with an inner diameter of 0.91 cm. In doing so, edges of the planar membrane and a small portion of the surface was covered. In addition, to ensure a uniform surface area on both sides of the planar membrane, the membrane was left with an exposed area of 0.65 cm² on the feed side in compliance with the exposed surface area on the permeation side.

![Planar reactor experimental setup using methane or helium as a sweeping gas.](image)

Figure 2 provides a detailed example of the planar reactor’s experimental setup. After being sealed to the quartz tube and placed in the center of a vacuum split furnace, a smaller inner quartz tube, with an inner diameter of 0.4 cm, would be able to feed methane, acting as a sweeping gas, beneath one side of the reactor while the other side was exposed to ambient air. The methane flow rate, controlled by a digital mass controller, would then create a chemical potential gradient and allow for oxygen to permeate through the reactor to the sweeping gas side. The high purity of oxygen would then react with methane and the exhaust would be fed into a gas chromatography (GC) system.
In both the planar and tubular reactor setups, temperature was monitored using a K-type thermocouple placed at the center of each reactor and each of the incoming flow rates were measured at STP. After the exhaust was fed into the GC, the results showed the composition only consisted of unburned methane, carbon dioxide, carbon monoxide, ethylene, and ethane. Each composition mole fraction was measured by the GC and used to calculate the oxygen permeation flux based on the corresponding stoichiometric ratios of consumed oxygen. The calculation for this is seen in Eq. (1):

\[
J_{O_2} = \frac{F_{\text{out}}(X_{O_2} + 2X_{CO_2} + 1.5X_{CO} + X_{C_2H_4} + 0.5X_{C_2H_6})}{A_{\text{membrane}}}
\]

where \( F_{\text{out}} \) is the flow rate out in mL.min\(^{-1} \), \( X \) is the mole fraction composition provided by the GC, \( A_{\text{membrane}} \) is the effective surface area in cm\(^2 \), and \( J_{O_2} \) is the oxygen permeation in mL.min\(^{-1}.cm\(^{-2}. It should also be noted that the coefficients in front of the provided mole fractions correspond to the specified species balance with oxygen under methane combustion. Furthermore, the effective surface area for the planar reactor can be calculated based on the exposed area to the surrounding environment, which was stated before to be 0.65 cm\(^2 \). In consistent with previous research, the tubular effective surface area (\( A_{\text{Tubular Membrane}} \)) for oxygen permeation was calculated based on Eq. (2):

\[
A_{\text{Tubular Membrane}} = \frac{\pi(D_o - D_i)L}{\ln\frac{D_o}{D_i}}
\]

Here, the \( D_o \) and \( D_i \) are the outer and the inner diameters of the tubular reactor (cm), and \( L \) is the effective length (cm). Using this equation, which is consistently used in previous tubular oxygen permeation studies, the calculated tubular effective area is 2.89 cm\(^2 \) (Tan et al., 2005a).

\( CO_2 \) selectivity was also calculated based on the composition in the exhaust stream. It was calculated by dividing...
the amount of CO$_2$ in the exhaust flow rate by the total amount of converted methane in the exhaust stream. The total amount of converted methane was calculated by subtracting the incoming flow rate reactor by the exhaust flow rate multiplied by the methane mole fraction. This calculation for this is seen in Eq. (3):

$$S_{CO_2} = \frac{F_{out}X_{CO_2}}{F_{in}-F_{out}X_{CH_4}}$$

(3)

where $F_{out}$ is the flow rate out in mL.min$^{-1}$, $F_{in}$ is the flow rate out in mL.min$^{-1}$, and $X$ is the mole fraction composition provided by the GC. Through establishing trends of the oxygen permeation and CO$_2$ selectivity performance under various reactor configurations, key parameters could be identified between OTM reactors and oxy-fuel combustion.

3. Results and Discussion

3.1 LSCF Planar Reactor

Oxygen permeation and CO$_2$ selectivity performance was investigated through modifying a sweeping gas flow rate from 5 to 20 mL.min$^{-1}$ and modifying temperature from 800°C to 950°C. It should be noted that 950°C was the maximum temperature to maintain the integrity of the reactor’s sealant, in which any higher temperature would severely degrade the seal and jeopardize the interior reactor division from the surrounding atmosphere. The reactor exhaust composition was measured by the GC to provide each component’s mole fraction. The exhaust gas was comprised of unburned methane, carbon dioxide, carbon monoxide, and ethylene. More importantly the GC detected no amount of nitrogen in the exhaust gas signifying an ideal hermetic seal for oxygen permeation.

![Fig. 4 Oxygen permeation flux for LSCF planar reactors at varying temperatures and methane flow rates.](image)

Figure 4 depicts the relationship that varying methane flow and increased reactor temperature have on the performance of a LSCF planar reactor. A maximum value of 1.32 mL.min$^{-1}$.cm$^{-2}$ at a temperature of 950°C and a flow rate of 20 mL.min$^{-1}$ while a minimum value was achieved at 0.33 mL.min$^{-1}$.cm$^{-2}$ at a temperature of 800°C and 5 mL.min$^{-1}$. It is interesting to note that Fig. 4 shows that as the temperature of reactor increases, the amount of oxygen permeating through the material increases as well. It can also be seen that as the flow rate of methane introduced into the sweeping side of the reactor is increased, there is also a higher amount of oxygen permeating through the reactor. Since there is more oxygen permeating through the reactor with an increase in fuel flow rate, it can be assumed that the partial pressure of oxygen on the sweeping side is maintained at relatively low value (~0). This is further seen by the absence of oxygen in the analyzed GC composition.
In order to further understand this phenomenon even more, it is crucial to examine the steady state oxygen permeation flux, derived by Xu et al (1999).

\[ J_{O_2} = \frac{D_v k_r (P'_{O_2}^{0.5} - P''_{O_2}^{0.5})}{2L k_f (P'_{O_2} P''_{O_2})^{0.5} + D_v (P'_{O_2}^{0.5} - P''_{O_2}^{0.5})} \]  

(4)

Equation 4 shows the theoretical calculation of the oxygen permeation flux correlating the partial pressure across the reactor where \( D_v \) is the effective diffusivity of oxygen vacancy (cm\(^2\)/s), \( k_r \) is the reverse surface exchange reaction rate constant (mol.cm\(^{-2}\).s\(^{-1}\)), \( k_f \) is the forward surface exchange reaction rate constant (cm.Pa\(^{-0.5}\).s\(^{-1}\)), \( P'_{O_2} \) is the partial pressure of oxygen on the feed side of the reactor (Pa), \( P''_{O_2} \) is the partial pressure of oxygen on the sweeping side of the reactor (Pa), and \( L \) is the membrane thickness (cm). Equation 4 also demonstrates that oxygen permeation is driven by bulk diffusion and the surface reaction rate. Typically, bulk diffusion is driven by partial pressure gradient across the membrane, meaning that as more methane sweeping gas is introduced into the system, the partial pressure gradient will significantly increase, thus depicting the trend in Fig. 4. Surface reaction kinetics are severely driven by temperature, meaning that as reactor temperature increases, the faster reaction rate of oxygen being deliver to the system can happen, which is confirmed in Fig. 4 as well. A similar trend can be also found when examining CO\(_2\) selectivity.

Fig. 5 CO\(_2\) selectivity of LSCF planar reactor at varying methane flow rates and temperatures.

Figure 5 shows the CO\(_2\) selectivity as a function of temperature and flow rate and its improvement as both factors increase. For example, when the flow rate is set at 20 mL.min\(^{-1}\) and the temperatures at 800°C the CO\(_2\) selectivity achieved is small, achieving a value of only 35.2 %, but as the reactor temperature is increased to 950°C and the flow remains constant, the CO\(_2\) selectivity increases to 46.5 % of the exhaust stream. The same growth can be seen when increasing the methane sweeping gas, when at a constant temperature of 950°C, achieves a CO\(_2\) selectivity of 39.0 % at a low flow of 5 mL.min\(^{-1}\), but able to achieve a higher selectivity value of 46.5 % at 20 mL.min\(^{-1}\). It could then be summarized that the introduction of a higher amount of oxygen content allows the reactor to operate at leaner combustion conditions, which is supported by the increase in CO\(_2\) selectivity. The maximum CO\(_2\) selectivity achieved was 46.5 %, meaning that approximately half of the converted flue stream consisted of CO\(_2\). Looking back at Eq. (4), there is no modification that can be made to the reactor composition and configuration in its current setup that will further enhance its performance. However, through modifying the configuration of the reactor to allow more surface interaction with both fuel and the surrounding atmosphere, further enhancement of CO\(_2\) selectivity at the given conditions could be achieved.
3.2 LSCF Tubular Reactor

Through modifying an OTM configuration to a tubular reactor setup, more surface area can be exposed to the surrounding atmosphere and inlet fuel, further improving the oxygen permeation and CO\textsubscript{2} selectivity. Paralleling the planar reactor setup, methane flow rate and temperature were modified to investigate oxygen permeation and CO\textsubscript{2} selectivity trends. The temperatures varied from 850°C to 950°C and flow rate ranged from 7 to 10 ml/min. An inner LSCF catalyst layer was also selected and applied to further enhance the oxygen permeation performance and CO\textsubscript{2} selectivity. The oxygen permeation and CO\textsubscript{2} selectivity were also calculated using Eq. (1) and Eq. (3) as previously used for the planar reactor, with the addition of Eq. (2) to compensate for the change in effective surface area.

![Oxygen permeation flux of LSCF tubular membrane with impregnated catalyst at varying temperatures and flow rates](image)

Figure 6 shows the oxygen permeation performance of the LSCF tubular reactor. Consistent with the trends found in the planar data, the oxygen permeation increases with temperature and methane flow rate achieving a maximum value of 0.171 mL.min\textsuperscript{-1}.cm\textsuperscript{-2} at 950°C and 10 mL.min\textsuperscript{-1} methane sweeping flow rate. This raises some concern as to why the planar reactor, when held at the same condition was able to achieve a higher value (0.662 mL.min\textsuperscript{-1}.cm\textsuperscript{-2}), despite the tubular reactor having a significant amount more of effective surface area compared to the planar reactor. An explanation can be found when examining the steady state oxygen permeation flux through a tubular reactor (Liu et al., 2006; Tan et al., 2002; Tan et al., 2005b).

\[
J_{O_2} = \frac{k_f (R_0^{0.5} - R_\text{in}^{0.5})}{(R_m/R_0)(P_{O_2}^{0.5} - P_{O_2}''^{0.5}) + (2k_f(R_0 - R_\text{in})/D_V)(P_{O_2}''^{0.5} + (R_m/R_0)(P_{O_2}^{0.5}))} \tag{5}
\]

Similar to Eq. (4), the same nomenclature is represented here with the addition of inner radius $R_\text{in}$ (cm) and outer radius $R_0$ (cm). $R_m$ is defined as the logarithmic radius and is calculated using the equation below:

\[
R_m = \frac{(R_0 - R_\text{in})}{\ln \left( \frac{R_0}{R_\text{in}} \right)} \tag{6}
\]

When compared to Eq. (4), the calculation for oxygen permeation for tubular reactors is comparable, being driven
by diffusivity and the surface reaction kinetics, but also takes into account the modified geometry. If it were to be assumed that the partial pressure on the sweeping side of the reactor was maintained at approximately zero, then Eq. (5) would reduce by $Rm/Rin$ or in our case a third, based on the size of the tubular reactor (O.D./I.D.: 0.215 cm/0.105 cm). Still, the results in Fig. 6 show a reduction in oxygen permeation compared to planar reactor data. It can then be suggested that the additional loss in performance can be contributed from the bulk diffusion of oxygen through the membrane. The planar reactor exhibited a uniform thickness of 0.04 cm while the tubular reactor had a thickness of 0.11 cm. The greater thickness of the tubular reactors was a direct consequence of the reactor’s fabrication process, which limited the minimum thickness of the extruded tube in order to maintain mechanical integrity, thus creating a trade off in the process.

Although the tubular reactor showed a lower oxygen permeation flux compared to the planar reactor, the investigation into the CO$_2$ selectivity provided an additional understanding as to how OTM technology operates as an oxy-fuel combustion system. The GC analysis of the tubular exhaust showed that the composition found in the exhaust stream for the planar reactor were comparable, consisting of unburned CH$_4$, CO$_2$, CO, C$_2$H$_4$, and C$_2$H$_6$. CO$_2$ selectivity was calculated using Eq. (3).

As seen in Fig. 7, an increase in temperature directly increases the CO$_2$ selectivity, as found in Fig. 5. However, the relationship between methane flow rate and CO$_2$ selectivity differs from what was previously found; as the fuel flow rate increases from 7 ml/min to 10 ml/min, the CO$_2$ selectivity decreases. It has already been established that as more fuel is introduced into the reactor, the chemical potential gradient increases, causing more oxygen to permeate through the reactor and form a combustion reaction with the fuel. As more oxygen is introduced into the system, the rich fuel environment becomes leaner, approaching stoichiometric conditions where a complete combustion reaction can occur. When that is the case, there is a higher amount of CO$_2$ content in the exhaust stream, thus improving CO$_2$ selectivity. In contrast, by increasing the fuel content, a richer combustion reaction occurs in which the total amount of oxygen provided, which is also limited by the material composition, structure, and set boundary conditions, either remains the same or diminishes, consequently reducing CO$_2$ selectivity.

Furthermore, Fig. 7 also demonstrates one of the major achievements that tubular reactors have over planar reactors. The tubular reactor was able to achieve a maximum CO$_2$ selectivity of 87.15 % at a flow rate of 7 ml.min$^{-1}$ while the planar reactor only achieved a high value of 46.5 % at 20 ml.min$^{-1}$. This indicates that despite its limitations set by fabrication techniques, tubular reactors have a better potential of achieving stoichiometric combustions that ideally would be put in practice for industrial power plants.
3.3 Planar Reactor Composition Investigation

After completing an extensive investigation concerning LSCF reactors in different configurations, recently developed SSC was selected as a candidate for a preliminary comparison to the oxygen permeation performance of LSCF planar reactors. SSC powder was dry pressed and sintered to produce a dense disk membrane that matched the thickness and effective surface area of the previously formed LSCF planar reactor. The conditions of the experimental setup and measurement were identical to the previously performed investigation, with a slight modification to the reactor temperature and flow rate. The reactor temperature was selected at a high operating temperature of 900°C, which would serve as a baseline temperature for an initial material comparison study. Second, in consistent with other oxygen permeation studies, helium was selected as an inert sweeping gas in order to solely characterize the oxygen permeation performance of the newly developed material and alternatively to using methane as a reactive sweeping gas. In order to compensate for the diminished chemical potential gradient, helium was fed at much higher flow rates ranging from 5 mL.min⁻¹ to 100 ml/min. The results were as followed:

![Oxygen permeation flux vs. Helium flow rate](image_url)

Fig. 8 Planar reactor composition comparison between LSCF and SSC at varying helium flow rates and 900°C.

Figure 8 shows the comparison between SSC and LSCF planar reactors with identical thicknesses and a constant reactor temperature of 900°C. As previously confirmed, both plots show that through increasing the amount of sweeping gas introduced into the system, a higher oxygen permeation flux can be achieved. Fig. 8 also shows the SSC has a significant better performance, achieving a value of 3.7 mL.min⁻¹cm⁻² at 100 mL.min⁻¹, while LSCF only achieves a value of 0.35 mL.min⁻¹cm⁻² also at 100 mL.min⁻¹. The remarkable improvement that SSC has over LSCF, provides some interesting insight regarding the future of OTM development, specifically that there are higher performing compositions that make commonly available and frequently used materials irrelevant. This figure also suggests further study of the performance of SSC reactors at a wider range of temperatures, similarly to the reported LSCF. After underlining the key parameters that make up the foundation of OTM reactor design for oxy-fuel combustion purposes, more work should begin to shift focus on better performing materials and their integration in oxy-fuel combustion systems as OTM reactors.

4. Conclusion

The work presented studies an OTM reactor for carbon capture purposes using an oxy-fuel combustion process. Key parameters concerning OTM reactor designs were established through examining the oxygen permeation flux and CO₂ selectivity performance. These parameters included reactor composition and temperature, incoming sweeping gas...
flow rate, and reactor geometry. It was concluded that both an increase in temperature and methane flow rate, directly improved oxygen permeation flux and CO₂ selectivity for the planar reactor, achieving a maximum value of 1.32 mL.min⁻¹.cm⁻² and 46.5 % at 20 mL.min⁻¹ and 950°C. The tubular reactor, despite showing similar trends in terms of oxygen permeation, showed an indirect relationship between methane flow rate and CO₂ selectivity. This was due to the limitation set by the reactor which provided a fixed amount of oxygen under set conditions, ultimately signifying that an increase in methane flow rate would cause a richer combustion reaction and reduce the amount of CO₂ selectivity in the exhaust stream. However, the tubular membrane was able to achieve a much higher percentage of CO₂ selectivity (87.15 %) at a flow rate of 7 mL.min⁻¹ and 950°C compared to the planar reactor, signifying that tubular reactors are ideal candidates for producing more efficient combustion reactions with rich CO₂ exhaust stream that can be contained for further use.

Additionally, reactor composition using the commonly available LSCF material and the newly developed SSC material, was compared using an inert sweeping gas at a fixed temperature. The results showed that although LSCF is known for its strong performance, SSC showed a much higher oxygen permeation flux, achieving a maximum value of 3.7 mL.min⁻¹.cm⁻² at 100 mL.min⁻¹. Future work will focus on further investigating SSC material used for OTM reactor composition. In doing so, it could be possible to implement cost saving oxy-fuel combustion technologies and offset harmful greenhouse gas emissions for a cleaner tomorrow.

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