Sr$_{2}$Fe$_{1+x}$Mo$_{1-x}$O$_{6-d}$ as anode material of cathode–supported solid oxide fuel cells

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Abstract

In this study, perovskite-type oxides of Sr$_{2}$Fe$_{1+x}$Mo$_{1-x}$O$_{6-d}$ (x = 0, 0.2, 0.35, 0.5) were synthesized and the phase stabilities both in oxidizing and reducing atmosphere were investigated. When calcined in the air atmosphere, pure perovskite phase can be obtained for the composition of Sr$_{2}$Fe$_{1.3}$Mo$_{0.7}$O$_{6-δ}$ while a large portion of SrMoO$_4$ second phase was detected in other compositions. Further reducing the above powders showed a pure phase for Sr$_{2}$Fe$_{1.1}$Mo$_{0.9}$O$_{6-δ}$ (x = 0, 0.2, 0.35) while minor amount of Fe was precipitated from the Sr$_{2}$Fe$_{1.5}$Mo$_{0.5}$O$_{6-δ}$. Due to the highest electrical conductivity and lowest polarization resistance of Sr$_{2}$FeMoO$_{6-δ}$, such composition has been applied as the cell anode for a cathode supported solid oxide fuel cell (CS-SOFC). Maximum power densities of 463 and 331 mW cm$^{-2}$ have obtained for the fuel cell when measured at 800 °C using hydrogen and propane as fuel, respectively. The durability test in propane indicated that Sr$_{2}$FeMoO$_{6-δ}$ could be a potential anode material with good tolerance ability toward coking formation.

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Introduction

Solid Oxide Fuel Cells (SOFCs) can directly convert the chemical energy of fuels like hydrogen, natural gas and propane into electricity in a highly efficient and environmental friendly way [1]. The high operation temperature of SOFC facilitates the utilization of low cost hydrocarbon fuels [2–6]. Nickel/yttria-stabilized zirconia (Ni/YSZ) cermet has been widely used as an excellent anode material due to its high catalytic activity toward hydrogen oxidation [7]. However, when hydrocarbon fuels are directly fed into Ni/YSZ anode, the nickel particles tend to catalyze the carbon deposition, resulting in quick performance degradation of the cells [8]. In order to solve this issue, many perovskite materials such as La$_x$Sr$_{1-x}$TiO$_3$(LST) [9], La$_{0.75}$Sr$_{0.25}$Cr$_{0.5}$Mn$_{0.5}$O$_{3-δ}$ (LSCM) [10], La$_{0.75}$Sr$_{0.25}$Cr$_{0.5}$Fe$_{0.5}$O$_{3-δ}$ [11] and Sr$_x$Mn$_{1-x}$O$_{3-δ}$ (M = Mg, Co, Ni) [12–14] have been investigated as potential anode materials.

In recent years, Sr$_{2}$Fe$_{1.5}$Mo$_{0.5}$O$_{6-δ}$ (SFMO) has gradually become the hotspot in the research of both cathode [15–17] and anode materials [18–20]. SFMO has a perovskite structure and could be stable in a wide temperature range in both reducing and oxidizing atmosphere [21]. This material shows the characteristic of mixed ionic and electronic conductivity due to the hybridization of Fe$^{2+}$/Mo$^{6+}$ and Fe$^{3+}$/Mo$^{5+}$ [22]. When measured in oxidizing atmosphere, the ionic conductivity of SFMO is higher than that of the traditional La$_0.8$Sr$_{0.2}$MnO$_3$ and is even comparable to that of the...
L_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3} [23]. Meanwhile, SFMO exhibits comparable electronic conductivity to the conventional nickel-cermet anode when measured in reducing atmosphere [22]. Furthermore, SFMO has also shown good anti-carbon deposition effects. A direct methanol SOFC all perovskite showed a power density of 391 mW cm \(^{-2}\) at 800 °C and no carbon formation was found in the cell's anode after operating in methanol [24]. Wang et al. also reported a SOFC with Sr\(_2\)FeMoO\(_6\) anode which exhibited a power density of 604.8 mW cm \(^{-2}\) in CH\(_3\)OH at 850 °C [25]. The durability test demonstrated that the stability of the cells with Sr\(_2\)FeMoO\(_6\) anode was much better than with Ni based anodes when operated in dry CH\(_3\)OH. In addition, Sr\(_2\)Fe\(_{1.33}\)Mo\(_{0.67}\)O\(_6\) have also been studied as the anodes for SOFCs with anti-carbon deposition ability [26]. Although all these reports indicated that Sr\(_2\)Fe\(_{1+\delta}\)Mo\(_{1-\delta}\)O\(_6\) with different composition have good carbon resistance and excellent catalytic activity for the oxidation of the hydrocarbon fuels, the influence of the ratio of Fe:Mo on the anode performance has not been comparably well studied.

In this paper, the phase stability of Sr\(_2\)Fe\(_{1+\delta}\)Mo\(_{1-\delta}\)O\(_6\) (\(\delta = 0, 0.2, 0.35, 0.5\)) series materials in both oxidizing and reducing atmosphere is studied. With the main interest in anode, the conductivity in reducing atmosphere and electrochemical performances of SOFCs with the impregnated Sr\(_2\)Fe\(_{1+\delta}\)Mo\(_{1-\delta}\)O\(_6\) anodes are also investigated.

**Experimental**

**Powder preparation**

Sr\(_2\)Fe\(_{1+\delta}\)Mo\(_{1-\delta}\)O\(_6\) (\(\delta = 0, 0.2, 0.35, 0.5\)) powders were synthesized by a combined EDTA and citric acid complexing method with Sr(NO\(_3\))\(_2\), Fe(NO\(_3\))\(_2\), H\(_2\)O, (NH\(_4\))\(_2\)MoO\(_4\) and H\(_2\)O as the raw materials [25]. All the starting chemicals were of analytical grade and purchased from Sinopharm Chemical Reagent Co., Ltd. EDTA was added into the aqueous ammonia to form a homogenous solution, followed by dissolving a certain amount of (NH\(_4\))\(_2\)MoO\(_4\) 4H\(_2\)O while heating and stirring. The stoichiometric amounts of Sr(NO\(_3\))\(_2\), Fe(NO\(_3\))\(_2\), H\(_2\)O were first dissolved into the deionized water and then added into the EDTA-NH\(_4\)H\(_2\)O solution. After stirring, proper amount of citric acid was introduced. The mole ratio of EDTA: citrate: total metal ions was controlled to be 2:2:1. The pH value of the solution was adjusted to about 8 via the addition of NH\(_3\)H\(_2\)O. The solution was then dried and calcined at 850–1100 °C in air for 2–5 h to form the desired Sr\(_2\)Fe\(_{1+\delta}\)Mo\(_{1-\delta}\)O\(_6\) phase. The obtained Sr\(_2\)Fe\(_{1+\delta}\)Mo\(_{1-\delta}\)O\(_6\) powders were further reduced in 97% H\(_2\)-3% H\(_2\)O at 800 °C for 2–20 h.

**Single cell fabrication**

The scaffold of cathode-supported SOFC ((La\(_{0.9}, Sr_{0.1})_{0.95}MnO_3 \text{(LSM)} \) support/porous 8 mol% Y\(_2\)O\(_3\)-stabilized ZrO\(_2\) (YSZ)/YSZ/porous YSZ) was fabricated by laminating four tape-casted green tapes under a pressure of 3000 psi at 75 °C for 10 min [27]. The laminated green tapes were then co-sintered at 1250 °C for 4 h in air [28]. Wet chemical impregnation was introduced to build the active catalysts for both the cathode and the anode. The cathode catalyst La\(_{0.6}Sr_{0.4}FeO_3\) was introduced into the porous YSZ backbone (close to LSM support layer) by impregnating nitrate solution containing La(NO\(_3\))\(_2\), 6H\(_2\)O, Sr(NO\(_3\))\(_2\), Fe(NO\(_3\))\(_3\), 9H\(_2\)O, Sc(NO\(_3\))\(_3\), 9H\(_2\)O and citric acid with the mole ratio of citric acid to metal ions being 1:1. After drying, anode catalyst of Sr\(_2\)FeMoO\(_6\) was introduced into the porous YSZ backbone (opposite to the LSM support layer) by impregnating the precursor solution as shown in section 2.1. Both the cathode and anode catalysts were calcined at 850 °C for 2 h in air. The impregnation/heat treatments cycle was repeated to achieve the desired loadings of 30 wt. % both for cathode and anode. For the symmetrical anode cell, the tri-layer electrolyte supported scaffold (YSZ/YSZ/YSZ) was also manufactured by laminating three tape-casted green tapes and co-sintering at 1400 °C for 4 h. Sr\(_2\)Fe\(_{1+\delta}\)Mo\(_{1-\delta}\)O\(_6\) catalysts were introduced into the porous YSZ layer in the same way as the single cell. The effective area of both the single and symmetrical cells were 0.35 cm\(^2\).

**Characterization**

Phase structure of Sr\(_2\)Fe\(_{1+\delta}\)Mo\(_{1-\delta}\)O\(_6\) powders were identified by the X-ray diffraction technique at room temperature using a Rigaku XRD diffractometer with monochromatic CuK\(_α\) radiation. For electrical conductivity measurement, Sr\(_2\)Fe\(_{1+\delta}\)Mo\(_{1-\delta}\)O\(_6\) powders with different stoichiometric compositions were pressed into rectangular bars and sintered at 1350 °C in air for 5 h, respectively. The relative density of the bars was 0.932, 0.958, 0.963, 0.982 for Sr\(_2\)Fe\(_{1+\delta}\)Mo\(_{1-\delta}\)O\(_6\) (\(\delta = 0, 0.2, 0.35, 0.5\)), respectively. The electrical conductivity were measured by the DC four probe method in a temperature range from 550 to 800 °C in 97% H\(_2\)-3% H\(_2\)O atmosphere. For electrochemical characterization of the SOFC, silver grids were coated onto the electrodes as the current collector with silver wires attached as the lead of current and voltage. The fuel cells were tested over the temperature range of 700–800 °C with the flow rate of the fuel (H\(_2\) or CH\(_3\)OH) being 30 scm while ambient air was used as the oxidant. Both the current–voltage curves and electrochemical impedance spectra were obtained using an IM6 Electrochemical Workstation (ZAHNER, Germany). The electrochemical impedance spectra were collected at open circuit over the frequency range from 0.01 Hz to 100 kHz with a 20 mV perturbation. Impedance measurements were also carried out on the symmetrical anode cells. The microstructure of the cells were examined by scanning electron microscopy (SEM) using a Hitachi S-4800-Il microscope.

**Results and discussion**

Fig. 1 shows the XRD patterns of the synthesized Sr\(_2\)Fe\(_{1+\delta}\)Mo\(_{1-\delta}\)O\(_6\) (\(\delta = 0, 0.2, 0.35, 0.5\)) powders calcined at 1100 °C for 5 h in air. As shown, pure phase Sr\(_2\)Fe\(_{1+\delta}\)Mo\(_{1-\delta}\)O\(_6\) was obtained while a larger portion of SrMoO\(_4\) second phase was detected in Sr\(_2\)Fe\(_{1+\delta}\)Mo\(_{1-\delta}\)O\(_6\) (\(\delta = 0, 0.2, 0.35\)). The diffraction peaks of SrMoO\(_4\) became more obvious when the content of Mo increased. This indicates that the ratio of Mo had a significant influence on the stability of the...
Sr$_2$Fe$_{1+\delta}$Mo$_{1-\delta}$O$_6$ powders calcined in air. The experimental results are consistent with the previous study which showed that Sr$_2$Fe$_{1+\delta}$Mo$_{1-\delta}$O$_6$ could be synthesized in the oxidizing environment while Sr$_2$FeMoO$_6$ could only be synthesized in a reducing environment [22].

Fig. 1 – XRD patterns of the Sr$_2$Fe$_{1+\delta}$Mo$_{1-\delta}$O$_6$ powders calcined at 1100 °C for 5 h in air: (a) $x = 0.5$, (b) $x = 0.35$, (c) $x = 0.2$, (d) $x = 0$.

Powders obtained in air were then reduced in 97% H$_2$–3% H$_2$O at 800 °C for 10 h and the XRD patterns are shown in Fig. 2. A pure perovskite phase of Sr$_2$Fe$_{1+\delta}$Mo$_{1-\delta}$O$_6$ ($x = 0, 0.2, 0.35$) was formed after reduction. Meanwhile, minor amount of Fe

Fig. 2 – XRD patterns of Sr$_2$Fe$_{1+\delta}$Mo$_{1-\delta}$O$_6$ powders calcined at 1100 °C for 5 h in air followed by reducing at 800 °C for 10 h: (a) $x = 0.5$, (b) $x = 0.35$, (c) $x = 0.2$, (d) $x = 0$. 

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precipitated from Sr$_2$Fe$_{1.5}$Mo$_{0.5}$O$_6$–0.3 was found. Wang et al. pointed out that irradiation-induced reducing atmospheres could cause the Sr$_2$Fe$_{1.5}$Mo$_{0.5}$O$_6$ to decompose to Fe and Sr$_2$FeMoO$_3$–0.3 [29]. However, more researches reported that Sr$_2$Fe$_{1.5}$Mo$_{0.5}$O$_6$ could be stable in reducing atmosphere without the precipitation of Fe [22,25,30]. Reasons of the conflicting conclusions are not known. To further study the influence of calcining duration on the precipitation of Fe, powder obtained above was reduced in 97% H$_2$–3% H$_2$O at 800 °C for another 10 h. However, Sr$_2$Fe$_{1.5}$Mo$_{0.5}$O$_6$–0.3 (x = 0, 0.2, 0.35) oxides were still stable in reducing atmosphere. The diffraction peak of precipitated Fe did not show obvious change and the reduced Sr$_2$Fe$_{1.5}$Mo$_{0.5}$O$_6$–0.3 oxide could retain the perovskite structure. It indicates that the amount of the metallic Fe is not increased and the reduced Sr$_2$Fe$_{1.5}$Mo$_{0.5}$O$_6$–0.3 oxide is stable after the extended exposure in reducing atmosphere.

The Sr$_2$Fe$_{1.5}$Mo$_{1-x}$O$_6$–0.3 anodes were prepared by impregnation in this work. Due to its reactivity and poor chemical capability with YSZ at high temperature [22,30], the calcining temperature of the impregnated anode was limited to only 850 °C, because a higher calcining temperature would result in the growth of impregnated particles and cause the performance degradation of cells’ electrode [31,32]. To confirm the phase structure of the impregnated Sr$_2$Fe$_{1.5}$Mo$_{1-x}$O$_6$–0.3 anode applied in this study, XRD patterns of Sr$_2$Fe$_{1.5}$Mo$_{1-x}$O$_6$–0.3 (x = 0, 0.2, 0.35, 0.5) powders calcined at 850 °C for 2 h in air was measured. As shown in Fig. 3(a), all components showed the major impurity phases of SrMoO$_4$ and SrFeO$_3$–0.3 and no perovskite phase was detected. To simulate the cell operation environment, powders calcined at 850 °C in air were further reduced in 97% H$_2$–3% H$_2$O at 800 °C for 2 h. After the reducing process, pure perovskite phase was obtained for the components with Fe: Mo ratio being 1.2:0.8 and 1:1, while minor impurity of metallic Fe was detected for the components with Fe: Mo ratio being 1.35:0.65 and 1.5:0.5 (Fig. 2(b)).

Fig. 4(a) shows the temperature dependence of total electrical conductivities of Sr$_2$Fe$_{1.5}$Mo$_{1-x}$O$_6$–0.3 (x = 0, 0.2, 0.35, 0.5) in 97% H$_2$–3% H$_2$O. It can be seen that the electrical conductivity of Sr$_2$Fe$_{1.5}$Mo$_{1-x}$O$_6$–0.3 (x = 0, 0.2, 0.35, 0.5) is increased with the increasing content of Mo over the whole temperature range. Sr$_2$FeMoO$_6$–0.3 showed the highest electrical conductivity, e.g., 186.9 S cm$^{-1}$ at 800 °C. Note that in the temperature range of 550–800 °C, Sr$_2$FeMoO$_6$–0.3 exhibited a metallic behavior while other compositions showed a semiconducting behavior [33], which means different stoichiometric Sr$_2$Fe$_{1.5}$Mo$_{1-x}$O$_6$–0.3 would follow different conduction mechanisms under different oxygen partial pressures. Fig. 4(b) shows the Arrhenius curves of the electrical conductivities and linear relationships can be observed for different samples.

![Fig. 3](image3.png)  
**Fig. 3** – XRD patterns of Sr$_2$Fe$_{1.5}$Mo$_{1-x}$O$_6$–0.3 (x = 0.5, 0.35, 0.2, 0) powders: (a) Calcined at 850 °C for 2 h in air, (b) Calcined at 850 °C for 2 h in air followed by reducing in 97% H$_2$–3% H$_2$O at 800 °C for 2 h.

![Fig. 4](image4.png)  
**Fig. 4** – (a) Electrical conductivities and (b) Corresponding Arrhenius plots of the Sr$_2$Fe$_{1.5}$Mo$_{1-x}$O$_6$–0.3 (x = 0.5, 0.35, 0.2, 0) measured in 97% H$_2$–3% H$_2$O at 800 °C.
This means that the electrical conductivity behavior may be primarily determined by the small polaron conduction mechanism.

Fig. 5 shows the Nyquist plots of the impedance spectra data for symmetrical anode cells impregnated with different stoichiometric of Sr$_{2}$Fe$_{1+x}$Mo$_{1-x}$O$_{6-d}$ ($x = 0, 0.2, 0.35, 0.5$) measured in 97% H$_2$–3% H$_2$O at 800 °C. The ohmic response of the electrolyte and electrodes were removed while the resistances were divided by 2 to account for the contributions of two symmetrical electrodes. As shown, the polarization result was 0.235, 0.193, 0.176 and 0.152 Ω cm$^2$ for the component with x = 0.5, 0.35, 0.2 and 0, respectively. The polarization resistance of cells' anode decreased with the increasing amount of Mo, which means that the content of Mo has a serious effect on the performance of the cell. All the spectra consist of two

<table>
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<td>0.054</td>
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<tr>
<td>0.2</td>
<td>0.11</td>
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<td>0.35</td>
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<td>0.5</td>
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Fig. 6 — Ohmic and polarization resistances of the impregnated Sr$_{2}$Fe$_{1+x}$Mo$_{1-x}$O$_{6-d}$ ($x = 0, 0.2, 0.35, 0.5$) symmetrical anode cells measured at 800 °C.

Fig. 7 — Cross-section SEM images of: (a) The single cell, (b) The LSFSc impregnated YSZ cathode, (c) The Sr$_{2}$FeMoO$_{6-d}$ impregnated YSZ anode.
sемисфер, которые можно отделить согласно частоте. Экспериментальные данные были получены с использованием эквивалентной схемы, состоящей из \( R_{ohm} \) (РФ) и \( R_{CPE} \) (РФ) [34]. \( R_{ohm} \) является общим омическим сопротивлением, включающим электролитическое сопротивление и омическое сопротивление катода. \( R_{HF} \) и \( R_{LF} \) являются постоянными факторами, соответственно, и \( R_{HF} \) и \( R_{CPE} \) относятся к различным химическим реакциям, соответствующим различным частотам, соответственно. Результаты анализа представлены в Таблице 1, которая иллюстрирует влияние содержания Mo на высокие и низкие значения частот омического сопротивления в МОФ, соответственно. Изменение частоты между различными слоями приводит к созданию дENSE электролитного слоя в катодно-поддерживаемой МОФ. В данной работе использовался катод поддерживаемый, который состоит из плотного электролитного слоя, располагаемого между двумя пористыми электродами YSZ. Важным свойством является то, что с увеличением содержания Mo, как увеличивается электрическая проводимость, так и уменьшается омическое и поляризационное сопротивления. Подобное поведение может быть обусловлено различными химическими реакциями, соответствующими различным частотам.

Электрохимические характеристики МОФ с использованием \( \mathrm{Sr}_2\mathrm{Fe}_{1-x}\mathrm{Mo}_{1+x}\mathrm{O}_{6.5} \) как анодного материала катодно-поддерживаемой МОФ с водородом при 750 и 800 °C показаны на рис. 8(a). Максимальная плотность энергии (МПД) была 324 и 462 мВт см\(^{-2}\) при 750 и 800 °C соответственно. Это значение является сравнительным с результатом, полученным в работе [24]. Различные структуры МОФ могут быть ассоциированы с переменным содержанием Mo в электролитическом слое, что влияет на работу катода. Использование \( \mathrm{Sr}_2\mathrm{Fe}_{1-x}\mathrm{Mo}_{1+x}\mathrm{O}_{6.5} \) как анодного материала приведет к уменьшению поляризационного сопротивления в сравнении с \( \mathrm{Sr}_2\mathrm{Fe}_{1.8}\mathrm{Mo}_{0.2}\mathrm{O}_{6.5} \), что может быть обусловлено различными химическими реакциями, соответствующими различным частотам.

Рис. 8 — Электрохимические характеристики МОФ с использованием \( \mathrm{H}_2 \) и \( \mathrm{C}_2\mathrm{H}_6 \) при 750 и 800 °C соответственно. (c) электрохимическая характеристика МОФ с использованием \( \mathrm{C}_2\mathrm{H}_6 \) как топливо, при 0.6 V и 750 °C.

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331 mW cm$^{-2}$ was obtained at 750 and 800 °C, respectively. Electrochemical impedance spectra (EIS) of the single cells measured at open circuit and 750 °C using wet hydrogen and propane as the fuel are shown in Fig. 8(c). The ohmic resistance corresponds to the high-frequency real-axis intercept and the electrode polarization resistance is taken by subtracting the ohmic resistance (high-frequency intercept at real-axis) from the total resistance (low-frequency intercept at real-axis). The ohmic resistance was 0.31 and 0.25 Ω cm$^2$ for cell feeding with hydrogen and propane, respectively. In contrast, the polarization resistance was 0.52 and 2.11 Ω cm$^2$, respectively. Obviously, the impedance spectra difference of the cell using H$_2$ and C$_3$H$_8$ as fuel is mainly from the polarization loss, especially at the low-frequency. This difference may be caused by the variation of the activation and diffusion processes in the anode.

Fig. 8(d) shows the stability of the single cell operated in propane at 750 °C and 0.6 V. No obvious degradation was found during the 530 min test. This indicates that Sr$_3$FeMoO$_6$-a impregnated porous YSZ anode showed good resistance against the carbon deposition. An extended measurement is needed to further study the cell stability in propane.

**Conclusions**

Phase stability of the Sr$_3$Fe$_{1.8}$Mo$_{1.2}$O$_{6.8}$-a (x = 0, 0.2, 0.35, 0.5) powder was investigated in this study. After calcining at 1100 °C in air, pure Sr$_3$Fe$_{1.8}$Mo$_{0.5}$O$_{6.8}$-a phase could be obtained while a large portion of SrMoO$_4$ second phase was detected in Sr$_3$Fe$_{1.8}$Mo$_{0.5}$O$_{6.8}$-a (x = 0, 0.2, 0.35). Pure phase for Sr$_3$Fe$_{1.8}$Mo$_{1.2}$O$_{6.8}$-a (x = 0, 0.2, 0.35) were obtained after reducing the above powders in 97% H$_2$-3% H$_2$O at 800 °C for 10 h, while minor amount of Fe was precipitated from the Sr$_3$Fe$_{1.8}$Mo$_{0.5}$O$_{6.8}$-a. The extending reducing time made no obvious influence on the phase structure. Compared to other components, Sr$_3$FeMoO$_6$-a impregnated YSZ anode showed the lowest polarization resistance, e.g. 0.152 Ω cm$^2$ at 800 °C. Cathode-supported SOFC employing the Sr$_3$FeMoO$_6$-a impregnated YSZ anode reached the maximum power density of 462 and 331 mW cm$^{-2}$ at 800 °C, using H$_2$ and C$_3$H$_8$ as the fuel, respectively. Durability test of the fuel cell in C$_3$H$_8$ showed a relatively stable performance.

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