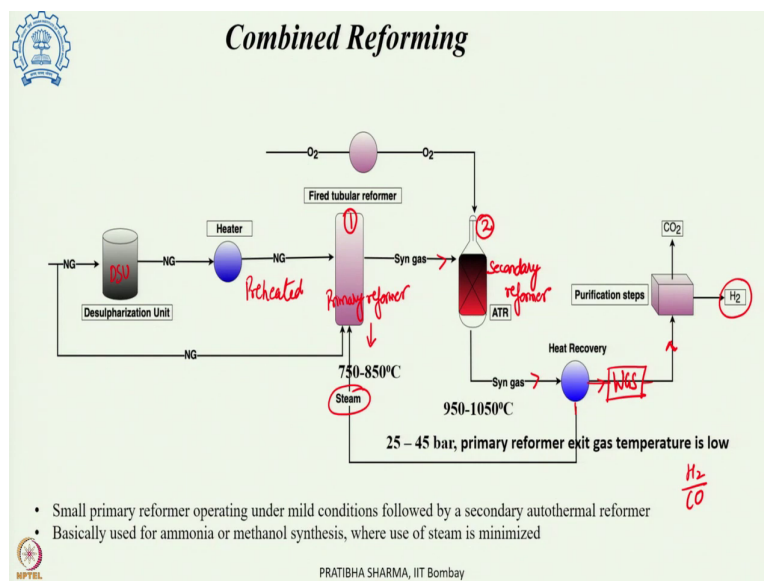


Hydrogen Energy: Production, Storage, Transportation and Safety
Prof. Pratibha Sharma
Department of Energy Science and Engineering
Indian Institute of Technology, Bombay

Lecture - 11
Combined, Dry, Bi and Tri Reforming

So, in the earlier class we have seen the 3 processes, steam methane reforming, partial oxidation, auto thermal reforming and their comparison. Now, any of these processes can be combined and then we can have a combined reforming as well, bi reforming, tri reforming based on what are the oxidants, how these are combined, all those we will see in today's class. In combined reforming, we have a primary reformer which is a smaller fire tubular reactor and then we have a secondary reformer.

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So, the partially reformed gas from the primary reformer goes to the secondary reformer and we can get the desired gas composition. Now, let us look at the process. Natural gas is pretreated. So, in the desulfurization unit after removal of the sulfur containing contents it is preheated and along with the steam it is passed into the primary reformer. So, this is the primary reformer wherein it undergoes the reforming reaction.


So, the syn gas which is partially being reformed is passed through the auto thermal reformer. The secondary reformer undergoes various processes that we have already learned now. The

outlet gas from the secondary reformer is to be cooled. So, excess of heat which is there in the syn gas can be utilized for steam generation and the remaining.

So, the syn gas thereafter can undergo the various gas cleaning steps, water gas shift reaction so as to finally get the desired product. Now, after the water gas shift, it will undergo the purification steps to separate the various other constituents, other than hydrogen. There is reformer, primary reformer then there is a secondary reformer.

Now, this primary reformer is a steam methane reformer, which operates under mild set of conditions than partially reformed gas goes to the secondary reformer, which is an auto thermal reformer. Usually, the method of combined reforming wherein two different processes are combined together so as to get the desired H₂ by CO ratio and that could be used for various applications, like whether it is ammonia synthesis, whether it is methanol synthesis and with this the use of steam could be reduced.

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Dry Reforming (DR)

$$\checkmark \quad \checkmark \quad \text{Syngas} \\ \underline{CH_4} + \underline{CO_2} \rightarrow \underline{2CO} + \underline{2H_2} \quad \frac{H_2}{CO} = 1 \quad \Delta H^0 = +247 \text{ kJ/mol}$$

- CO₂ reforming or stoichiometric reforming
- Both greenhouse gases used
- If natural gas have both in high concentration then DR
- One is highly reduced form and one is highly oxidised form of C
- Can be used for processes where CO rich gas required, H₂/CO ratio = 1
- For production of acetic acid, synthesis of dimethyl ether, direct conversion of syngas into olefins through the Fischer Tropsch process to olefins reaction (FTTO).
- Highly endothermic process

$\frac{H_2}{CO} = 1$

$\left. \begin{matrix} CH_4 \\ CO_2 \end{matrix} \right\} \text{inert}$



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Another method of reforming is dry reforming, this is also known as carbon dioxide reforming or stoichiometric reforming. The difference between the earlier processes and dry reforming is, here carbon dioxide is used as the oxidant. So, feedstock that is methane is reacting with the oxidant which is carbon dioxide to produce syn gas.

The important thing here is that both the feedstock and oxidant are greenhouse gases. So, if we are using them then we are in fact reducing the carbon footprint or if the natural gas which

is used for the dry reforming it has a higher CO₂ content. So, the natural gas if it is already having a higher CO₂ content rather than going for gas cleaning, that can directly be used for dry reforming process.

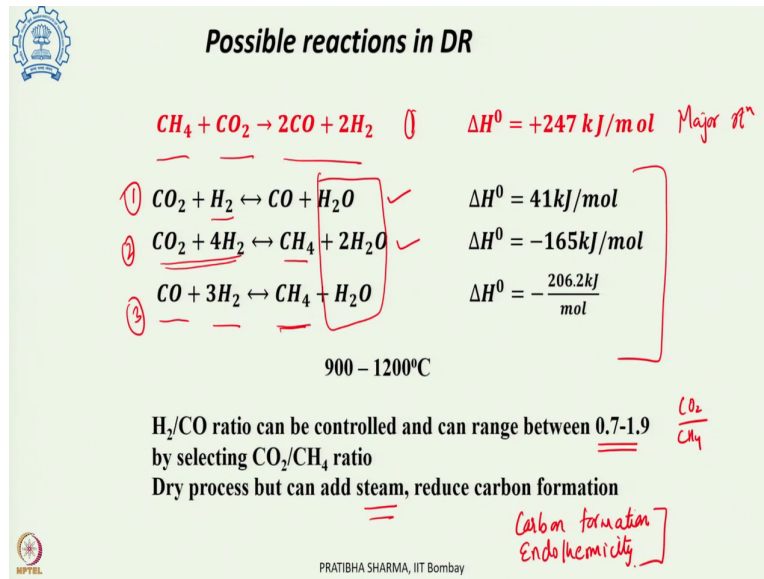
Now, one of the thing that needs to be seen here is methane which is a highly reduced form of carbon, carbon dioxide which is highly oxidized form of carbon are reacting together so as to give the syn gas. The hydrogen to CO ratio as can be seen is 1 or it can be less than 1.

In ideal case it is 1, but it could be less than 1 also, we will see under what conditions. The reaction delta H value suggests that it is endothermic and positive sign shows it is endothermic. Value suggest that it is even more endothermic than the steam methane reforming reaction.

The reason for high endothermicity is both the feedstock and the oxidant. They are relatively inert. So, this is the stabilized form, if they have to react, then an activation energy is required to make them to react. So, that is why high energy is required and it is in a highly endothermic process. With this H₂ by CO ratio of 1, it may not be a desired method for hydrogen production.

However, this is the method of hydrogen production wherein this syn gas which is being produced can directly be used for various processes, various oxo processes like production of acetic acid, synthesis of dimethyl ether, syn gas can be converted into olefins using Fischer trope process to olefin reactions. These are the applications wherein the syn gas which is obtained after dry reforming can be used for.

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If you see the possible reactions that occur in dry reforming of methane, so, methane reacting with carbon dioxide producing syn gas, that is the primary reaction that is the major reaction that occurs. Other than that the various other reactions that can occur include the reaction wherein certain amount of hydrogen is being consumed in the process. And the carbon dioxide which is oxidant also consumes the hydrogen which is obtained to produce CO and H₂O. The other reaction consuming more hydrogen. Now, this reduces the hydrogen in the product obtained. CO formed can also consume hydrogen to produce methane. So, this is the methanation reaction, the second and third are methanation reaction.

So, dry reforming of methane occurs in the temperature range of 900 to 1200 degree centigrade and the hydrogen to CO ratio if we look at the reaction it is ideally is 1, but this can be somewhere lying between 0.7 to 1.9. And this can be selected based on what is the oxidant to feedstock ratio, based on what is the CO₂ to CH₄ ratio, this can range somewhere between 0.7 to 1.9.

So, although it is a dry process the name itself suggesting dry reforming of methane, it is a dry process, but we can see that certain amount of water is obtained on the product side. At the same time we can also add some amount of steam to the process and that has an advantage, that addition of steam will reduce the carbon formation.

Since carbon dioxide is used as an oxidant so the carbon formation is the major bottleneck. So, both endothermicity of the process and carbon formation, these are the two major bottlenecks towards the industrial use of the process.

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Catalyst and support

Non-noble catalysts - Ni or Ni based catalyst, Ni/Al₂O₃, NiMgAl or NiCe have shown good methane conversion, Ni with K promoter shows increased life
 Co, Cu and Fe shown activity but they are preferably employed in bimetallic catalyst
 Ni and Co with noble metals on the Al₂O₃ support

Noble Metals - The order of reactivity : Rh > Ru > Ir > Ni ✓ *higher activity, better conversion, stability, low cost*
 Pt, Pd > Co > Fe, Cu *Rh/Al₂O₃ lower coke deposition prone to coking and sintering*

by adding small amounts of noble metals like Rh, Ru, Ir, Pt, Pd, Au to non-noble catalysts Ni, Co, Cu, Fe results in good stability, catalytic activity, higher conversions and reduced cost
 Ni-Fe, Ni-Cu good stability Ni-Co higher conversion, Ni-Pt higher activity and reduced coking
Ni/Al₂O₃ - good conversion
Ni/ZrO₂ - good oxygen mobility

Perovskites with noble metal doping

Support materials are Al₂O₃, TiO₂, ZrO₂, Nb₂O₅, CeO₂, Ta₂O₅, La₂O₃, ZrO₂-La₂O₃, MgO, Al₂O₃-MgO, SiO₂, Y₂O₃, Zeolites, SBA-15.
Ni/MgO-Al₂O₃ better stability & activity

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This carbon formation can be reduced with use of appropriate catalyst, support and promoter. It can also be reduced by including steam. It can be reduced by having proper dispersion of the catalyst onto the support, by using promoters. Now, the various catalysts which are used in the dry reforming process are, they can be either non-noble metal catalyst, like nickel or other nickel based catalyst which can be on supports like Al₂O₃, this is the most widely used catalyst and support combination.

It can be nickel, magnesium, aluminium or nickel cerium. All these nickel based catalyst have shown to have a very good methane conversion. The major problem in the nickel based catalyst is they have a very good initial methane conversion, they have good activity but the coke formation is higher in nickel based catalyst.

And we can add up certain promoters like potassium to increase its life and stability. There are other non-noble metal catalyst like copper, cobalt, iron they also show good activity and they can be used preferably in a bimetallic catalyst, like they can be used in together with a noble metal.

Like for example, nickel and cobalt they are used along with noble metals as a bimetallic catalyst on an alumina support. Compared to non-noble metals, noble metals have a higher activity. They provide better conversion and they have good resistance towards coking and sintering. So, they have a good thermal stability and mechanical stability, they are less prone to coking and sintering.

If we see among the various noble metals the order of their reactivity could be rhodium higher than ruthenium higher than iridium and it is more than that of the even the nickel, which is used widely as a catalyst. Platinum, palladium they have higher activity compared to the cobalt, iron and copper. So, noble metals definitely have better activity, they have stability, they provide good conversion and selectivity, but the cost is the major problem here.

To address these issues like to get all these simultaneously and cost effectively as well, a small amount of noble metal like rhodium, ruthenium, iridium, platinum, palladium or gold can be added to the non-noble metal catalysts like nickel, cobalt, copper, iron and that can provide together a stability, catalytic activity, higher conversion at a reduced cost.


Now, there are several catalyst combinations bimetallic catalysts like nickel iron, nickel cobalt, they show very good stability. Nickel cobalt they show higher conversion, nickel platinum they show higher activity and reduced coking. And other than these noble and non-noble metal catalysts, transition metal based catalyst, perovskites are also used as catalyst with noble metal doping. We know that other than catalyst, support is also very important, because it serves several purposes.

The various supports which have been used for dry reforming of methane includes alumina Al_2O_3 , TiO_2 , ZrO_2 , Nb_2O_5 , CeO_2 , Ta_2O_5 , La_2O_3 , ZrO_2 a combination of the supports ZrO_2 , La_2O_3 , magnesium oxide, alumina along with magnesium oxide, silica, yttrium Y_2O_3 , zeolites and SBA-15.

Now, all these have a separate characteristic. For example, if we see nickel on alumina, then these have good methane conversion. Nickel can be on a zirconia support. So, it has good oxygen mobility. Nickel when it is with magnesium oxide and alumina, they have better stability as well as activity. And similarly, the others noble metal based catalyst like rhodium on alumina they show lower coke deposition.

So, there are various combination of catalyst and support, which could provide good performance in terms of activity, selectivity, conversion, reduced coking and sintering.

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Catalyst deactivation

Boudouard reaction

$$2CO \leftrightarrow CO_2 + C \quad \Delta H^0 = -172 \text{ kJ/mol}$$

Exothermic reaction so favourable at lower temperatures

2. Reduction of CO

$$CO + H_2 \leftrightarrow H_2O + C \quad \Delta H^0 = -131 \text{ kJ/mol}$$

Although occurs at low temperature but with higher Gibbs energy not referred route except cases when there is high partial pressure of H_2

3. Methane Decomposition

$$CH_4 \leftrightarrow C + 2H_2 \quad \Delta H^0 = +74.6 \text{ kJ/mol}$$

Favoured at higher temperatures

4. Reduction of CO_2


$$CO_2 + 2H_2 \leftrightarrow C + 2H_2O \quad \Delta H = -90 \text{ kJ/mol}$$

$\frac{CO_2}{CH_4} = 1 \quad T > 1100^\circ C$

$\frac{CO_2}{CH_4} = 2 \quad T > 730^\circ C$

$\frac{CO_2}{CH_4} = 3 \quad T > 700^\circ C$

Reverse of any of these can result in removal of coke formed, gasification of coke, the nature of support desirable is the basicity of the support, lower at higher T and higher CO_2/CH_4 ratio



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Now, we are using carbon dioxide as oxidant here. So, the problems associated with carbon deposition are more severe in case of dry reforming of methane. There are various reactions that can result into catalyst deactivation by means of carbon deposition, like the Boudouard reaction, wherein there is CO is disproportionation can give CO_2 and carbon deposition. Now, we can see that this is a reaction wherein it is favourable at lower temperature.

Reduction of CO can result into carbon formation, this again occurs at a lower temperature, but the Gibbs free energy of this reaction is not favourable. So, it is not a preferred route for carbon formation, there can be methane decomposition favoured at a higher temperature. Even reduction of carbon dioxide can occur to produce carbon.

So, there are several ways in which carbon can formed, but this carbon which is formed can also be removed. Any of the reverse reactions out of these can result into removal of the carbon formation. There could be gasification of coke which is formed, which can reduce the carbon formation.

The use of appropriate support, promoter and catalyst can reduce the carbon formation. The support can provide desired basicity which instead of supporting carbon formation like decomposition of the various species, can support carbon gasification. So, the appropriate

support catalyst and promoter use can reduce the carbon formation. At the same time it is observed that it depends upon what is the temperature and carbon dioxide to methane ratio.

It has been reported carbon dioxide to methane ratio if it is stoichiometric, say it is 1, 1 is to 1, then the carbon formation is lower for temperatures higher than 1100 degree centigrade. If this ratio is 2, then the temperatures above 750 degree centigrade for reduced coke formation.

If it is 3, then temperatures above 700 degree centigrade results into lower coke formation. So, both the temperature as well as the CO₂ to methane ratio are important in how much amount of carbon is being formed with the various reactions taking place in the process. The carbon which is formed can be of different type.

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Catalyst deactivation

- Use of CO₂ so possibility of C formation more → *aged catalyst*
- Three types of Carbon formed – (1) Whisker carbon (2) Pyrolytic carbon (3) Gum *C₂ ~~carbon~~ support*
- Whisker and coke (or pyrolytic carbon in case of higher hydrocarbons) at higher temperatures, while gum at lower temperatures, gum covers the catalyst by layers of graphene type carbon
- Most problematic is whiskers formed at higher T, low S/C ratio and presence of aromatics
- Which species depends on thermodynamics and properties of catalyst
- Small size of catalyst and high dispersion desirable, appropriate operating conditions

MPTEL PRATIBHA SHARMA, IIT Bombay

It could be either whisker form of carbon being formed and this whisker type of carbon is formed usually in the catalyst which are aged which has undergone several cycles. So, after catalyst aging, this whisker type of carbon becomes more prominent. And this carbon which is formed diffuses. The problem with the whisker carbon is that as it is formed on to the catalyst surface it diffuses inside the catalyst. It reaches the catalyst and support interface.

So, as it is formed on the surface, it diffuses inside the catalyst, it reaches the interface between the catalyst and support and with time this lifts the catalyst. So, catalyst is lifted from the support. So, there is a breakage of catalyst that will happen and that will also block the catalytic bed as the catalyst will be removed from the support.

And this is the most problematic form of carbon which is formed in the dry reforming of methane, the other form of carbon is pyrolytic carbon and this is usually formed in case of higher hydrocarbons. And the third one is the gum which is formed. So, this is in fact, a several layers of carbon is being formed CHX material, these are layers of graphene type of carbon which is formed on the catalyst.

Now, out of these three forms of carbon the gum carbon is formed at a lower temperature and the whiskers and pyrolytic carbon or the coke, these are formed at a higher temperature. But the most problematic out of the three carbon formed is the whisker type of carbon. And this is promoted at a higher temperature, lower steam to carbon ratio.

So, if we use higher steam to carbon ratio the coking tendency will reduce and in the presence of aromatics. Now, which of these species out of these whisker carbon or coke or gum will be formed that all depends upon the thermodynamics of the process. It also depends upon the catalyst, catalyst substrate interaction all these are going to decide the species which are formed in the process.

At the same time, we know that what is the shape of the catalyst, what is the size of the catalyst, how good is the dispersion, how good is the support catalyst interaction, what promoters we have used, all those also determine the catalyst deactivation or coking tendency. Like if small size of catalyst is being used, high surface area is being used we know that if the dispersion is higher, then the coke formation will be lower.

So, small size of catalyst and high dispersion is desirable to reduce the coke formation and we require appropriate operating conditions, temperatures and pressures so that the catalyst deactivation should be lower. But then there are challenges that we have seen in detail in the steam methane reforming, that if we use very small size of catalyst, then the pressure drop will be there.

And so, there should be an optimum between the size of the catalyst and like it should not be very high and it should not be very low and that is determined by the heat transfer as well as the pressure drop inside the bed. There are two processes of dry reforming, which have reached to the commercialization state.

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DR Processes

Two processes based on dry reforming have reached to the commercialization stage - CALCOR and SPARG.

① CALCOR process - designed for achieving higher CO content (H₂/CO ratio of 0.43) using Ni based catalysts and under CO₂ partial pressures

② SPARG process - coke deposition is prevented with sulphur poisoning resulting in wide range of possible H₂/CO ratio. $\frac{H_2}{CO}$


NPTEL PRATIBHA SHARMA, IIT Bombay

These are CALCOR and SPARG process. Now, in the first process which is CALCOR process, it is designed to achieve a particular H₂ by CO ratio which could be used for various downstream applications. So, the H₂ by CO ratio which is preferred in the process is 0.43 and the nickel catalyst is used under the carbon dioxide partial pressures. The second process which is SPARG process, this is the process wherein wide range of H₂ by CO ratios are achieved.

And this is achieved in a way wherein sulphur poisoning is done intentionally to reduce the ensemble size. To get a wider range of H₂ by CO ratio in this process, the activity is altered of the catalyst, wherein intentionally sulphur poisoning is done by means of H₂S so that the ensemble size becomes smaller and the possible H₂ by CO ratio, that can be achieved could be varied in that process.

But then such processes have issues like, if later catalyst and all have a tendency towards sulphur poisoning, then that can lead to sulphur poisoning of the downstream process catalyst. At the same time it will reduce also the catalyst activity.

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Bireforming

Oxidant - H₂O and CO₂

$3CH_4 + CO_2 + 2H_2O \leftrightarrow 4CO + 8H_2 \quad \Delta H^0 = +712 \text{ kJ/mol} \quad (\text{BRM})$ H₂/CO = 2

Other reactions:

$CO + H_2O \rightarrow CO_2 + H_2 \quad \Delta H^0 = -41 \text{ kJ/mol} \quad (\text{WGS})$

$CH_4 + CO_2 \rightarrow 2CO + 2H_2 \quad \Delta H^0 = +247 \text{ kJ/mol} \quad (\text{DRM})$

$CO_2 + H_2 \rightarrow CO + H_2O \quad \Delta H^0 = +41 \text{ kJ/mol} \quad (\text{RWGS})$

$CH_4 + \frac{1}{2}O_2 \rightarrow CO + 2H_2 \quad \Delta H^0 = -38 \text{ kJ/mol} \quad (\text{POX})$

$2CO \leftrightarrow C + CO_2 \quad \Delta H^0 = -172 \text{ kJ/mol} \quad (\text{Boudouard reaction})$


$CH_4 \leftrightarrow C + 2H_2 \quad \Delta H^0 = +74.6 \text{ kJ/mol} \quad (\text{Methane Decomposition})$

H₂/CO ratio = 2, can be adjusted changing the H₂O/(H₂O+CO₂) ratio, T and P

Operating T = 700-950°C and P = 10-30 bar.

Advantage -

- use of greenhouse gases to produce hydrogen like DRM
- ratio of H₂/CO appropriate for various downstream production processes
- less of carbon formation and reduced catalyst deactivation than DRM



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So, that was about the dry reforming of methane. Now, we can have several other combinations of oxidant being used to perform various reforming for certain selective applications, like bi reforming. In bi reforming oxidant which is used is steam and carbon dioxide. So, if we see the bi reforming for feedstock which is methane, it reacts with carbon dioxide and steam to produce syn gas.

And the syn gas which is produced is in the H₂ by CO ratio of 2 here. Now, this is the primary reaction which can occur in bireforming of methane. Other than that, there are several other reactions that can also occur. The reaction wherein carbon monoxide reacts with steam to produce carbon dioxide and hydrogen. This is the well known process which we have seen earlier also several times water gas shift reaction.

It can undergo dry reforming in the presence of carbon dioxide which is an oxidant being present. So, methane can react with carbon dioxide producing syn gas again, dry reforming of methane. So, the carbon dioxide which is oxidant can react with the hydrogen product to produce carbon monoxide and steam. So, this is the reverse of water gas shift reaction that can also occur. Methane can react with sub stoichiometric amount of oxygen to undergo partial oxidation, producing syn gas.

Carbon monoxide disproportionation can occur, that can lead to carbon formation. Methane can decompose to give carbon and hydrogen. So, other than the primary reaction that we have seen, there can be several other possible reactions that can occur in during the bi reforming of


methane. The H₂ by CO ratio is 2 and this can be further adjusted by selecting the steam and steam to carbon dioxide ratio.

Or we can say the steam and methane or carbon dioxide and methane ratio can be selected such that we can get the required H₂ by CO ratio. The operating temperature in the process is between 700 to 950 degree centigrade and pressure between 10 to 30 bar.

Now, the advantage of bireforming is that here again we are using the greenhouse gases to produce hydrogen or H₂ syn gas in the desired ratio of hydrogen to carbon monoxide. And this hydrogen to carbon monoxide is such that the required application can be used directly for the various downstream production processes.

Compared to the dry reforming of methane, here the carbon formation is comparatively less and thus the reduced catalyst deactivation will be obtained, because we are using steam in the process as an oxidant as well and that reduces the coke formation in the entire process. So, herein we have used two oxidants in the bireforming, there are two processes they are occurring simultaneously. So, it is the reforming with steam reforming as well as dry reforming.

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Tri-reforming

Oxidant - O₂, H₂O, CO₂


$CH_4 + H_2O \rightarrow CO + 3H_2$	$\Delta H^0 = +206\text{kJ/mol}$ SMR
$CH_4 + CO_2 \rightarrow 2CO + 2H_2$	$\Delta H^0 = +247\text{kJ/mol}$ DRM
$CH_4 + 2O_2 \rightarrow CO_2 + 2H_2O$	$\Delta H^0 = -891\text{kJ/mol}$ oxidation

Other reactions that occur in TRM are as follows:

$CO + H_2O \rightarrow CO_2 + H_2$	$\Delta H^0 = -41\text{kJ/mol}$
$CH_4 + \frac{1}{2}O_2 \rightarrow CO + 2H_2$	$\Delta H^0 = -38\text{kJ/mol}$ (Pox)
$2CO \leftrightarrow C + CO_2$	$\Delta H^0 = -172\text{kJ/mol}$
$CH_4 \leftrightarrow C + 2H_2$	$\Delta H^0 = +74.6\text{kJ/mol}$

Operating temperature of 700-1000°C and pressure of around 20 bar, H₂/CO ratio achieved is 1.2-1.5

Catalysts - Ni, Pd, Ir, Pt, Rh, support - Al₂O₃, MgO, Al₂O₃ etc.



PRATIBHA SHARMA, IIT Bombay

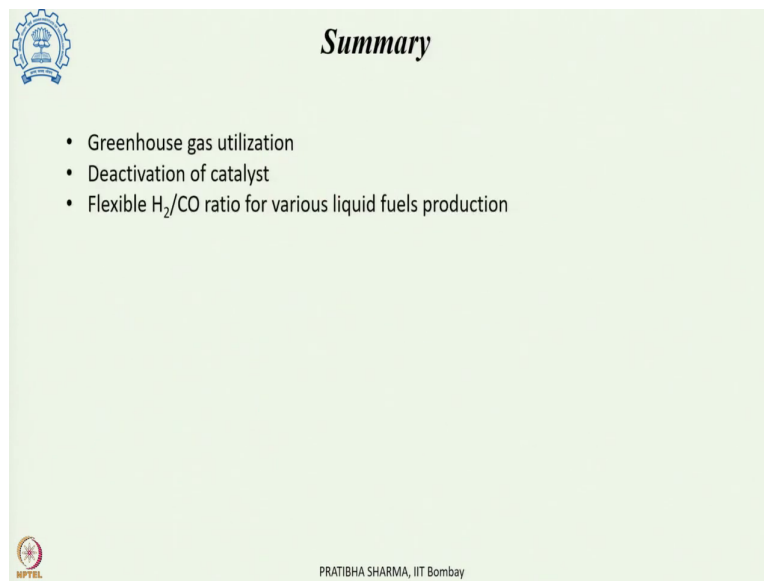
Now, if oxygen, steam and carbon dioxide all three are used together, so, it can undergo either partial oxidation, it can undergo combustion, it can undergo steam reforming, it can undergo dry reforming. So, methane if it is the feedstock, it can undergo steam methane reforming,

producing syn gas, can undergo dry reforming reacting with carbon dioxide to produce syn gas or it can undergo oxidation reaction to produce carbon dioxide and steam.

So, these are the major reactions occurring in the tri-reforming other than these three reactions mentioned there can be several other possible reactions that can occur. CO reacting with steam to produce CO_2 , methane undergoing partial oxidation to produce syn gas, carbon monoxide disproportionation reaction, methane can undergo decomposition. So, other than the primary reactions these are the other possible reactions which can occur.

Now, the operating temperature for tri-reforming is somewhere 700 to 1000 degree centigrade in between and the pressure of operation around 20 bar. The H_2 to CO that can be achieved can be within 1.2 to 1.5 and there are various catalysts that can be used for the tri-reforming process, like nickel, palladium, iridium, platinum, rhodium and supports like aluminium oxide, magnesium oxide can be used for the process.

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Summary

- Greenhouse gas utilization
- Deactivation of catalyst
- Flexible H_2/CO ratio for various liquid fuels production

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To summarize what we have seen today we have seen that various greenhouse gases can be utilized for reforming process, either to produce hydrogen if the H_2 to CO ratio is not appropriate, then that can be used for various other production various other chemicals or various other Fischer-Tropsch liquid fuels production processes. But when we are using carbon dioxide as an oxidant, we have seen the major problem is related to the deactivation of catalyst.

However, that can be reduced by appropriate selection of catalyst, promoter and support. We have seen that the steam to carbon ratio can be adjusted. We can see that the operating temperatures also defines how much will be the carbon formation or coke deposition. And we can have in through these processes either combined refining or dry reforming or bi and tri reforming, various H₂ to CO ratio processes that can be used for various downstream processes.

Thank you.