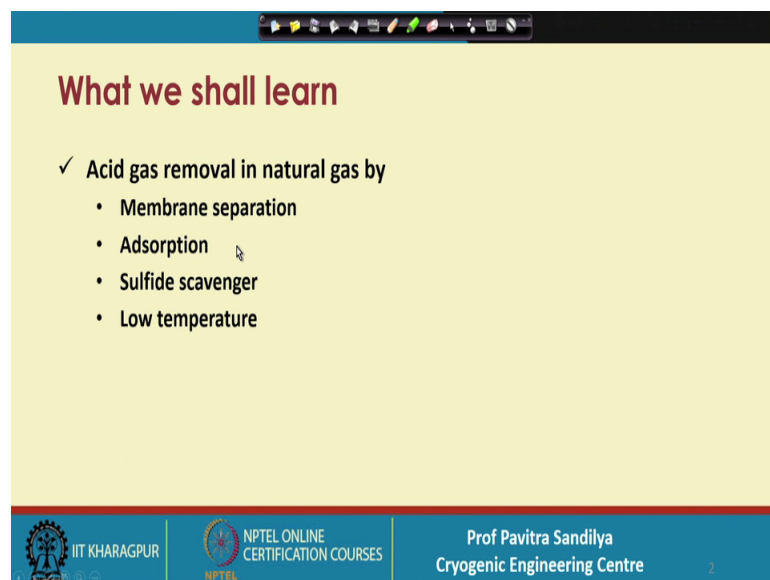


Upstream LNG Technology
Prof. Pavitra Sandilya
Department of Cryogenic Engineering Centre
Indian Institute of Technology, Kharagpur

Lecture – 44
Acid gas removal in natural gas system- II

Welcome, now we shall be learning some more about the acid gas removal something we learnt earlier also. So, in this case we are these part 2 on the Acid gas removal from natural gas.

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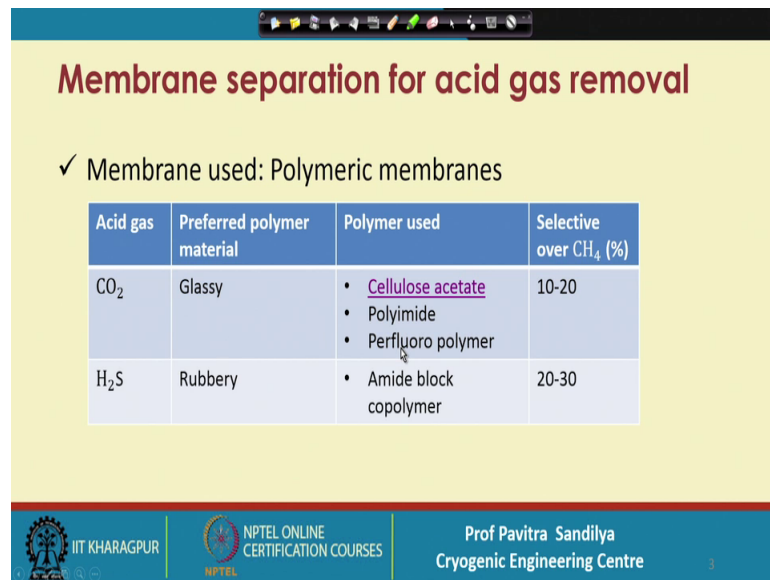
What we shall learn

- ✓ Acid gas removal in natural gas by
 - Membrane separation
 - Adsorption
 - Sulfide scavenger
 - Low temperature

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In this we shall be learning about the membrane separation, adsorption separation, use of sulfide scavenger and the low temperature for the removal of the acid gases.

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Membrane separation for acid gas removal

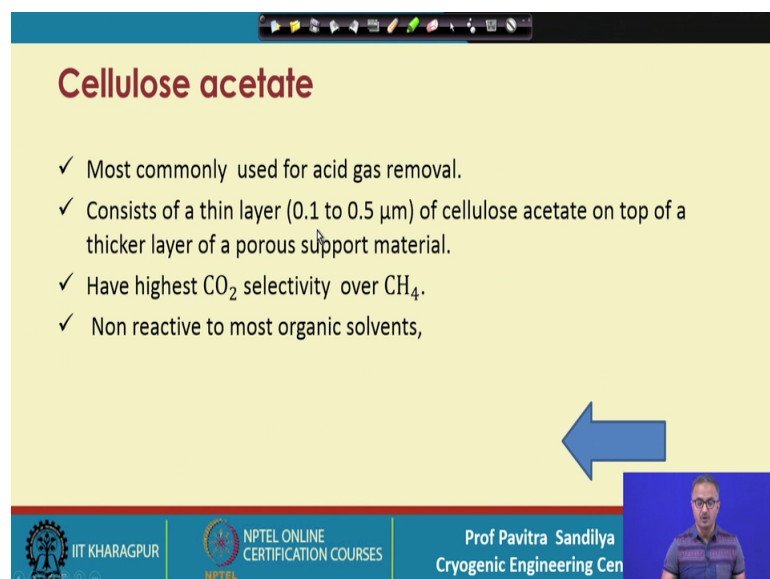
✓ Membrane used: Polymeric membranes

Acid gas	Preferred polymer material	Polymer used	Selective over CH ₄ (%)
CO ₂	Glassy	<ul style="list-style-type: none">Cellulose acetatePolyimidePerfluoro polymer	10-20
H ₂ S	Rubbery	<ul style="list-style-type: none">Amide block copolymer	20-30

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So, first we come to the membrane separation of the acid gas. Now, here we are using the membrane which is organic membrane some polymeric membrane is there. And the acid gases are CO₂ and H₂S and we have several types of membrane which are broadly classified based on their structures as glassy and rubbery. And then we have the one is the polymer used for carbon dioxide removal is the cellulose acetate polyimide and for perfluoro polymer.

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Cellulose acetate

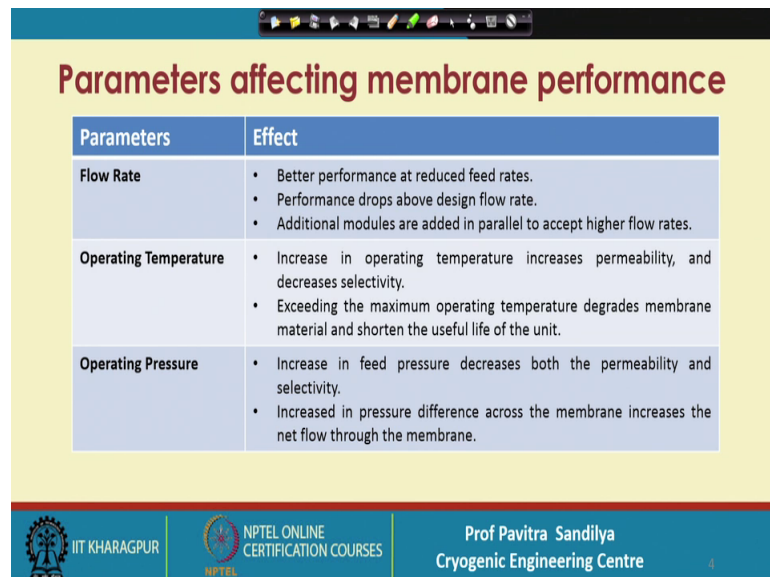
- ✓ Most commonly used for acid gas removal.
- ✓ Consists of a thin layer (0.1 to 0.5 μm) of cellulose acetate on top of a thicker layer of a porous support material.
- ✓ Have highest CO₂ selectivity over CH₄.
- ✓ Non reactive to most organic solvents,

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And let us look at those acetate which is very commonly used for acid gas removal. And it is the asymmetric membrane we learnt earlier that is the skin is about 0.5 to 0.1 to 0.5 micron of cellulose acetate which is kept on a thick layer of porous material. And which has highest selectivity of carbon dioxide over methane and it is non reactive to most organic solvents.

And then the selectivity over methane goes from 10 to 20 whereas, for the H₂S this rubbery kind of polymeric materials are there for the membrane. We are using amide block copolymer and their selectivity is 20 to 30 percent over methane.

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Parameters	Effect
Flow Rate	<ul style="list-style-type: none"> Better performance at reduced feed rates. Performance drops above design flow rate. Additional modules are added in parallel to accept higher flow rates.
Operating Temperature	<ul style="list-style-type: none"> Increase in operating temperature increases permeability, and decreases selectivity. Exceeding the maximum operating temperature degrades membrane material and shorten the useful life of the unit.
Operating Pressure	<ul style="list-style-type: none"> Increase in feed pressure decreases both the permeability and selectivity. Increase in pressure difference across the membrane increases the net flow through the membrane.

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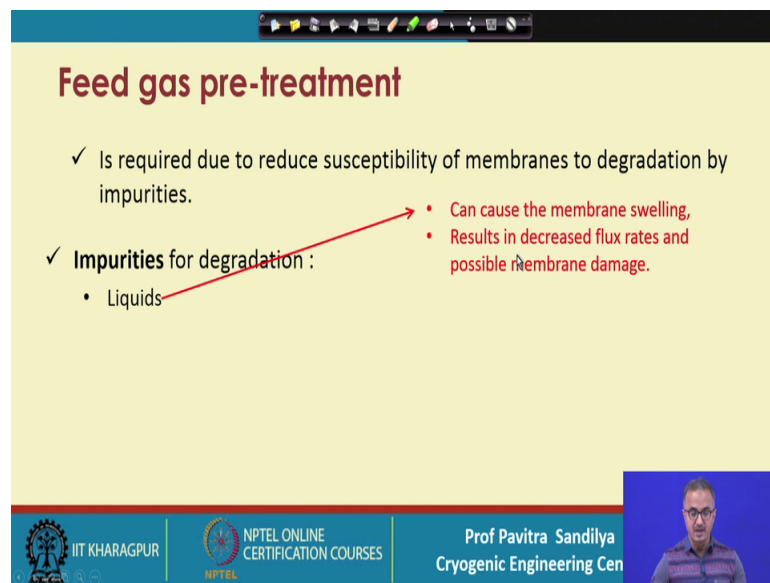
Now, here we are listing the basic parameters operative parameters which affect the performance of the membrane; here we have the flow rate operating temperature and operating pressure. So, we find that the flow rate a dictates that if we have higher flow rate then we find that the membrane performance degrades.

So, for better membrane performance we need to operate at lower flow rate; then performance is also seen to drop when the we operate above the design flow rate. And we need additional modules of the membrane; if we have to handle larger flow rates. Then the operating temperature has this kind of effects that if a increase the operating temperature; it increases permeability, but it decreases the selectivity.

So, permeability means we shall be getting higher production, but then we will not be able to separate out the various components which is dictated by the selectivity. And if we exceed the operating temperature maximum operating temperature, we find it may degrade the membrane material. And there by it can shorten the useful life of the membrane unit. In case of operating pressure if the operating pressure is high then both the permeability and the selectivity increases.

And if the difference between the pressures on the permeate side and the retentate side is very high, then we find that the net flow through the membrane will increase, but at the same time we have to keep in mind that the membrane can have enough; then should have enough mechanical stability to withstand that kind of high pressure difference across it.

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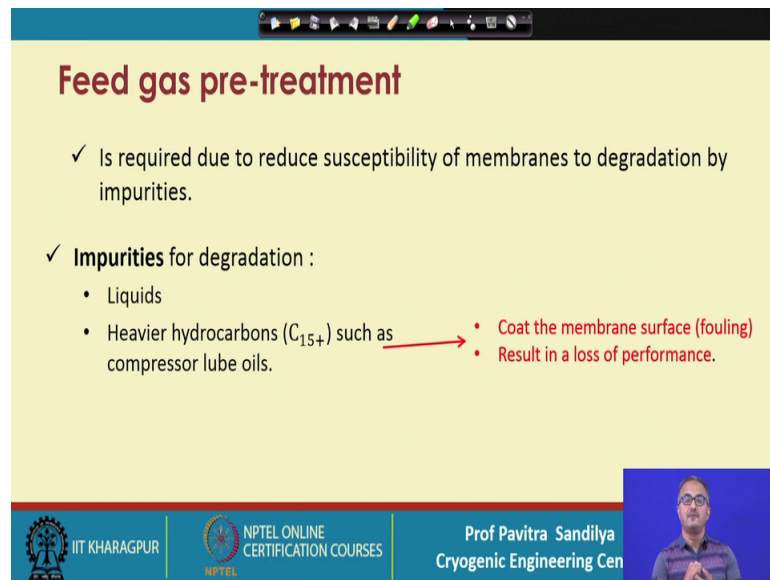
Feed gas pre-treatment

- ✓ Is required due to reduce susceptibility of membranes to degradation by impurities.
- ✓ **Impurities for degradation :**
 - Liquids
- Can cause the membrane swelling,
- Results in decreased flux rates and possible membrane damage.

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In case of membrane, we need to pre treat the gas because the membrane force may get chocked by some particulate matters. So, it is require to pre treat the membrane to reduce the susceptibility of the membranes by for by degradation in the presence of some impurities. And there can be several types of impurities; suppose here the some liquid impurity and this liquid impurity may cause the membrane swelling, membrane can get swelled and then it may results in the decreased flux rate and the possible membrane damage.

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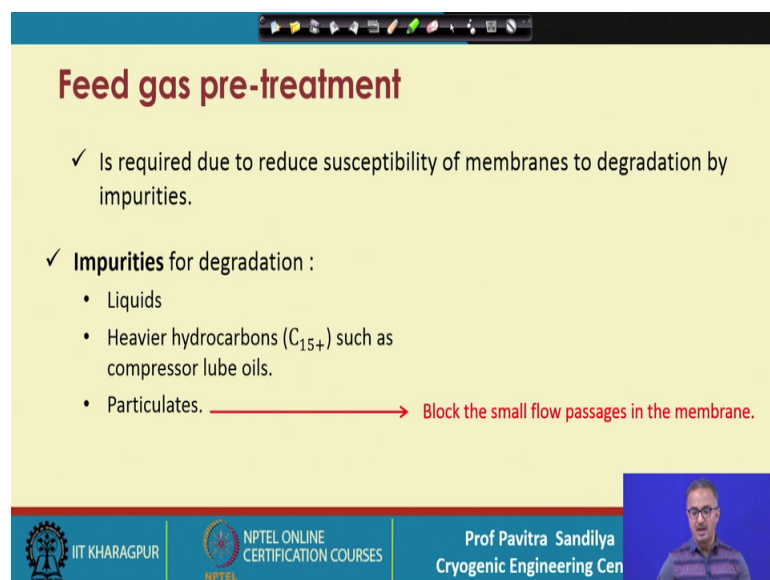
Feed gas pre-treatment

- ✓ Is required due to reduce susceptibility of membranes to degradation by impurities.
- ✓ **Impurities** for degradation :
 - Liquids
 - Heavier hydrocarbons (C_{15+}) such as compressor lube oils. →
 - Coat the membrane surface (fouling)
 - Result in a loss of performance.

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Then we have the presence of some heavier hydrocarbons and some especially lube oils; what will they do? They will coat; that means, they will cause some kind of fouling over the membrane surface. And if there is fouling or this coating by this heavier hydrocarbons; then the flux will decrease. So, the flux decreases means the membrane performance will come down.

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Feed gas pre-treatment

- ✓ Is required due to reduce susceptibility of membranes to degradation by impurities.
- ✓ **Impurities** for degradation :
 - Liquids
 - Heavier hydrocarbons (C_{15+}) such as compressor lube oils.
 - Particulates. → Block the small flow passages in the membrane.

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Then if we have some particulates what is happen? They will block the pores through which the solutes will pass through and thereby they will degrade the performance of the membrane.

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Feed gas pre-treatment

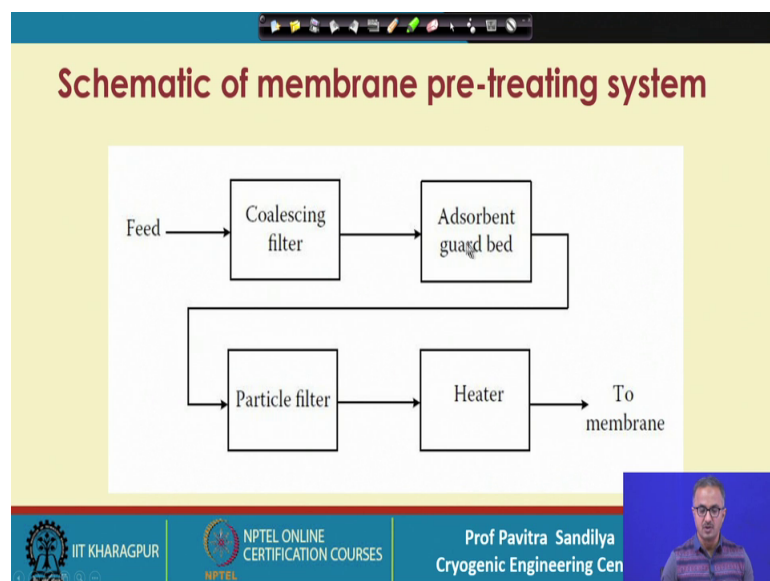
- ✓ Is required due to reduce susceptibility of membranes to degradation by impurities.
- ✓ **Impurities** for degradation :
 - Liquids
 - Heavier hydrocarbons (C_{15+}) such as compressor lube oils.
 - Particulates.
 - Corrosion inhibitors and well additives.

→ Destructive to membrane material

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And if there are any kind of corrosion inhibitors and well additives then they will they made destruct the membrane materials. So, that is while to remove all this things we need to pre treat the feed before it is allowed to go to the membrane module.

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Now, here we have a typical membrane system configuration that the feed comes here; then we have all these filters are used for. So, this mechanical filters and adsorbent filters they are used for pre treating the things and then heat it is heated and then it is goes to the actual membrane module. So, these are the pre treating system.

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Feed gas pre-treatment

- ✓ Is required due to reduce susceptibility of membranes to degradation by impurities.
- ✓ Impurities for degradation :
 - Liquids
 - Heavier hydrocarbons (C₁₅₊) such as compressor lube oils.
 - Particulates.
 - Corrosion inhibitors and well additives.

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Performance of some membranes for CO₂ removal

Membrane	Permeation experiment	% CO ₂	Permeability (Barrer)		Selectivity CO ₂ /CH ₄	Reference
			CO ₂	CH ₄		
PPM-γ-CD-425	Mixed gas	50	3976	174	22.85	Askari et al. (2012a,b)
PPM-β-CD-425	Mixed gas	50	2911	133	21.85	Askari et al. (2012a,b)
PPM-α-CD-425	Mixed gas	50	2392	117	20.44	Askari et al. (2012a,b)
Dense film TR-PBO	Single gas	—	3575	80.8	44	Kim et al. (2012)
TR-PBO Hollow fiber ^a	Single gas	—	1938	137.1	14	Kim et al. (2012)
TR-PBI	—	—	1624	35	46.1	Han et al. (2010)
Addition-type poly(trimethylsilylnorbornene (PTMSN) with Si(CH ₃) ₂ side groups	—	—	4480	990	4.5	Yampolskii et al. (2010)
Spiroisindane polymers	Single	—	3496	360	9.7	Staiger et al. (2008)
Poly(dimethyl siloxane) (PDMS)	Pure gas	—	2850	823	3.5	Nakagawa et al. (2002)
Silicone (polydimethyl siloxane, or PDMS)	—	—	3250	950	3.4	MedArray, Inc.

^a Permeance is in GPU.

1 GPU (Gas Permeation Unit) = 1 x 10⁻⁶ cm³(STP)/cm² s cmHg

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And here comes the performance of some of the membranes for the carbon dioxide removal. So, these are some commercial membranes.

So, we find that these difference these membranes we have and they have been done with different mixed gas and single gas all this things. That means, this is mixture of carbon dioxide with other gases and this is one carbon dioxide and these are the percent of the carbon dioxide in this mixed gas. And we find that these are the permeabilities given in barrier for carbon dioxide and methane; that means, these membranes are more selective towards carbon dioxide. So, that is why we find the this selectivity is a 22 to about 4 percent it differs. So, we can get the this kind of membranes can be used to separate carbon dioxide from the methane.

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Advantages of membrane separation

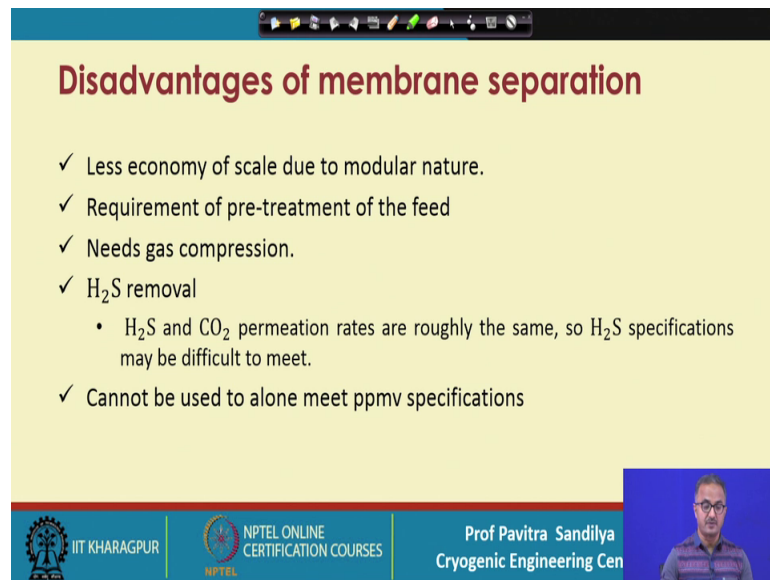
- ✓ Low capital investment when compared with solvent systems.
- ✓ Ease of operation.
- ✓ Ease of installation (Units are normally skid mounted)
- ✓ Simplicity (No moving parts for single-stage units)
- ✓ High turndown ratio.
- ✓ High reliability and on-stream time.
- ✓ No chemicals needed.
- ✓ Good weight and space efficiency.

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The advantages of the membrane separation are that they have low capital investment when we compare with the solvent system for the acid gas removal. Then they have easy to operate; their installations are easy because they come in small small modules. And they have simplicity of the construction and especially because they do not involve any kind of moving part ah.

And then they have high turndown ratio that is the ratio between the highest to the lowest feed rate; they can handle. Then high reliability and on stream time and then we do not need any kind of chemicals. And they have good weight and space efficiency that is they do not occupy much of space and they are not very heavy.

(Refer Slide Time: 07:43)



Disadvantages of membrane separation

- ✓ Less economy of scale due to modular nature.
- ✓ Requirement of pre-treatment of the feed
- ✓ Needs gas compression.
- ✓ H₂S removal
 - H₂S and CO₂ permeation rates are roughly the same, so H₂S specifications may be difficult to meet.
- ✓ Cannot be used to alone meet ppmv specifications

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On the other hand, this membrane systems has several disadvantages also. In this case we find that because we need several modules and these modules have to be collected externally by some pipelines. So, over all we find that by using the moderate structure the capital cost increases and along with that the operating cost or maintenance cost also increase.



Then we need feed pre treatment before the mixture is same to the membrane module. Then the gas compression is needed because the membrane can work well when we have high pressure difference between the permeate side and the retentate side. And then we have the, we need to remove the H₂S removal that in the H₂S and carbon dioxide permeation rates are roughly the same.

So, H₂S specifications may be difficult to meet; that means, it is difficult to separate out the carbon dioxide and the H₂S separately. And cannot be used to can alone to meet the ppmv; that means, if we a very very small amount required in the of stream, then only membrane cannot be used it can be added with the other acid removal process.


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Adsorptive acid removal

- ✓ Less popular than absorption.
- ✓ At ambient temperatures substantial quantities of both gases are adsorbed even at low partial pressures.
- ✓ Adsorbent used
 - Molecular sieves
 - Zeolites
 - Activated carbon
 - Metal oxide
 - Metal organic frameworks (MOF)

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Now, next we come to the adsorptive acid removal; now in this case this is less popular than absorption and is getting developed also. And at ambient temperatures substantial amounts of both the gases and are adsorbed even at low partial pressure that is both H₂S and CO₂ can get adsorbed at low partial pressure low concentrations.

And these are the type of adsorbents used molecular sieves which may be zeolitic or may be activated carbon and may be have the metal oxide what we call metal organic framework MOF.


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Performance of some adsorbents

Adsorbent	Adsorbate	T (°C)	P (mmHg)	Loading (mol/kg)	Regeneration Method
Activated carbon	CO ₂	25-300	500	1.5-2.0@25°C 0.1-0.2@300°C	PSA
5A zeolite	CO ₂	25-250	500	~3.0@25°C 0.2@250°C	PSA
Titanosilicates	CO ₂	25-200	760-6x10 ⁶	-	PSA
HTiC (K-promoted)	CO ₂	300-400	200-700	0.4-0.7	PSA
Solid amine (supported PEI)	CO ₂	75	760	1.5 - 3.0	PSA
Double-layer hydroxides	CO ₂	375	230	1.5	PSA
Alumina (un-doped)	CO ₂	400	500	0.06	PSA
Alumina (doped w/ Li ₂ O)	CO ₂	400	500	0.52	PSA
alumina(basic)	CO ₂	300	500	0.3	PSA
Li zirconate	CO ₂	500	760	3.4-4.5	TSA
CaO	CO ₂	500	150	4-8@500°C 7@500°C	TSA

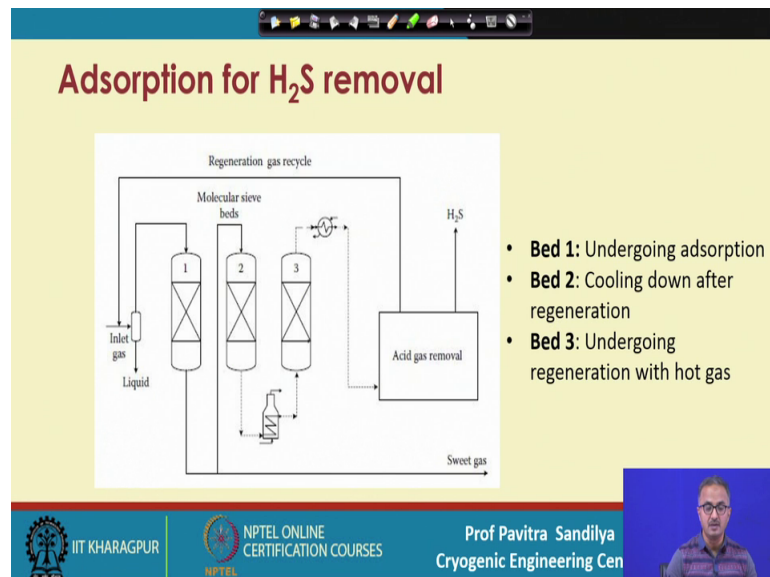
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And here we find the performance of various adsorbents. So, these are the kind of adsorbents which are used activated carbon zeolite, titanosilicate etcetera alumina etcetera. And here we have the carbon dioxide and we find that these are the operating temperatures for all these kinds of adsorbents.

And these are the pressures of the carbon dioxide and these are loading and these are the regeneration method; that means, and most of the cases we find the regeneration is done by the pressures in adsorption and in some cases we have these temperatures in adsorption.

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And here we have a typical membrane separation system for H₂S removal what we find? Here we have the feed gas which passes through various beds, but mind it that all the beds are not performing adsorption at the same time here we have that the say typical; suppose bed 1 is undergoing adsorption.

But then bed 3 is undergoing regeneration whereas, bed 2 is being cooled and we getting prepared for adsorption. So, here we are finding these 3 beds it may be 2 bed minimum is 2 bed, but it may be 3 or 4 depending on the different times for regeneration for cooling down. So, in this typically we find we are using 3 adsorption beds to carry out the H₂S removal.


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Adsorption of acid gases by molecular sieve

- ✓ Can reduce H₂S levels to the 6 mg/Sm³ specification.
- ✓ Reduction requires regeneration of the bed at 600°F(315°C) for extended time with the potential for COS formation if 4A* is used.




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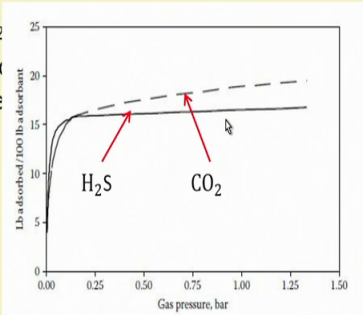


Now, when we talk of molecular sieve for removal of the acid gases; this molecular sieve can reduce it to very small amount about 6 milligram per standard cubic meter. And they need regeneration of the bed at a quite high temperature for extended time with the potential of COS formation carbonyl sulfide; if this kind of zeolite is use 4 a star.


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Adsorption of acid gases by molecular sieve


- ✓ Can reduce H₂S
- ✓ Reduction requires regeneration of the bed at 600°F(315°C) for extended time with the potential for COS formation if 4A* is used.



Adsorption of H₂S and CO₂ on type 5A molecular sieve at 25°C




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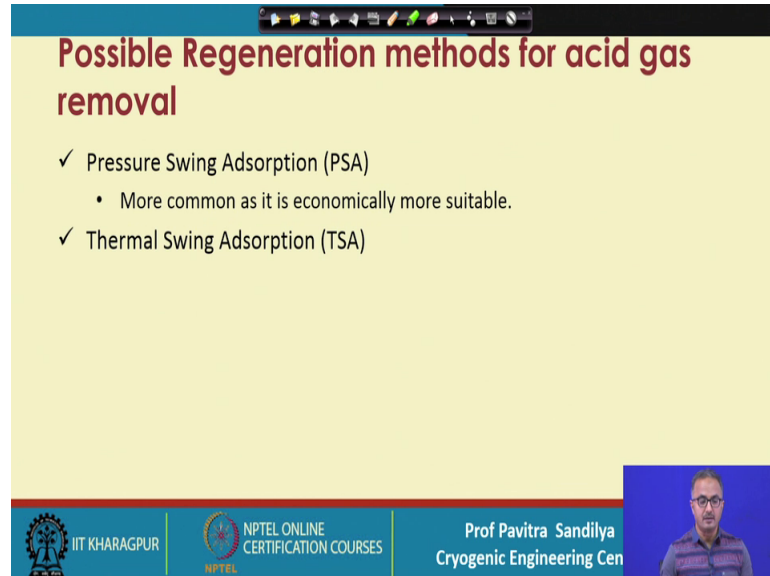
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And here we find how H₂S and CO₂ get adsorbed on this molecular sieve in here we find that this kind of molecular sieve is having more affinity towards carbon dioxide.

And rest for H₂S though the difference is very not very much and it increases at higher gas pressure.

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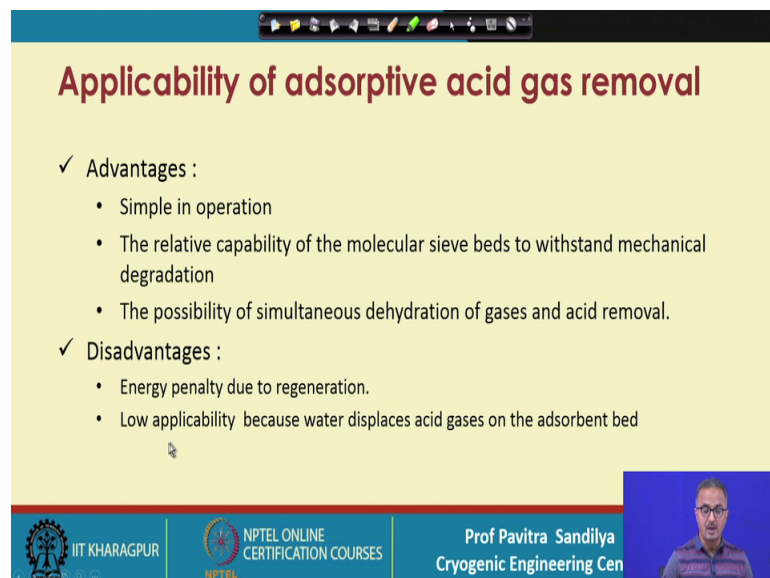
Possible Regeneration methods for acid gas removal

- ✓ Pressure Swing Adsorption (PSA)
 - More common as it is economically more suitable.
- ✓ Thermal Swing Adsorption (TSA)

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And now we know that there are several methods for regeneration; one is a pressure swing adsorption another is a thermal swing. In this case pressure swing is more common because we are talking of handling high amount of the gases.

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Applicability of adsorptive acid gas removal

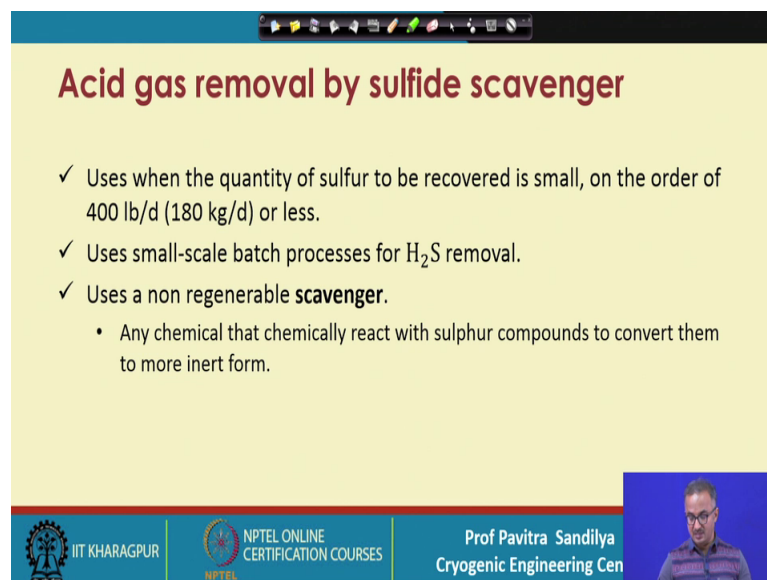
- ✓ Advantages :
 - Simple in operation
 - The relative capability of the molecular sieve beds to withstand mechanical degradation
 - The possibility of simultaneous dehydration of gases and acid removal.
- ✓ Disadvantages :
 - Energy penalty due to regeneration.
 - Low applicability because water displaces acid gases on the adsorbent bed

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And these are the advantages of the membrane separation that it there simple and their relative capability of the molecular sieve beds can withstand the mechanical degradation; that means, they may not be getting attrition.

So; that means, they will not from powder and they can with stand high pressure and the possibility of simultaneous dehydration of gases and the acid removal; that means, we can remove water and carbon dioxide and H₂ S simultaneously. This advantages are that these a energy penalty for the regeneration; that means, we need energy for the regeneration of this catalyst and sorry adsorbent and the low applicability because water displaces the acid gases on the adsorbent bed because the water will get accumulated in the bed.

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Acid gas removal by sulfide scavenger

- ✓ Uses when the quantity of sulfur to be recovered is small, on the order of 400 lb/d (180 kg/d) or less.
- ✓ Uses small-scale batch processes for H₂S removal.
- ✓ Uses a non regenerable **scavenger**.
 - Any chemical that chemically react with sulphur compounds to convert them to more inert form.

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Now, acid gas is sulfide scavenger when then this scavengers are used when the quantity of sulfur to be recovered is small of the order of say 100 kg per day or less. So, it uses small scale batch processes for H₂ S removal and the scavengers are non regenerable; that means, only once they can be used.

Because they react chemically and this scavengers are the chemical that chemically react with the sulphur compounds to convert them to more inert form. Because they are getting chemically reacted; so, it is not possible to regenerate the scavengers.

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Some desired features of a scavenger

- ✓ Should react completely and rapidly with the sulphides.
- ✓ Ability to react in a wide range of chemical and physical environments like pH, temperature, pressure etc.
- ✓ Non-corrosive.
- ✓ Easy and economical availability.
- ✓ Safe and no risk to health of personnel or pollute the environment.
- ✓ Environmentally friendly.

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So, these are the desirable properties on the scavenger that they should react completely and rapidly with the sulphides. They should be able to react in a wide range of chemical and physical environment like pH, temperature, pressure etcetera. They should be non corrosive and they should be economically and easily available. And there should not be any kind of risk to health, to safety and it should be environmental friendly.

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Sulfide scavengers in natural gas system

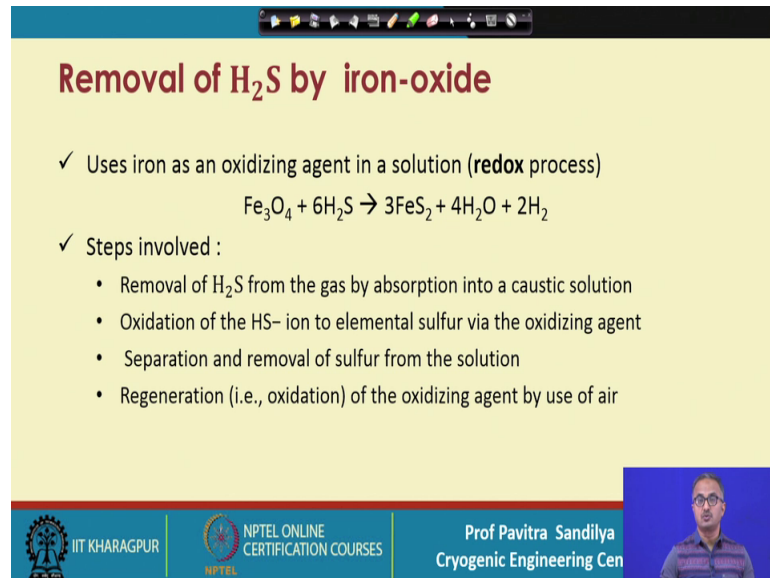
Type of scavengers processes	Material used
Solid based	<ul style="list-style-type: none">• Iron oxide• Zinc oxide
Liquid based	<ul style="list-style-type: none">• Nitrite• Amine aldehyde condensate• Caustic scrubbing• Aldehyde• Oxidizer• Metal oxide slurry

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Now, here we have a few scavengers and the some are solid based, some are liquid based. So, solid based we have iron oxide zinc oxide; liquid we have all these

scavengers. So, among all these scavengers the iron oxide is the most common and we have this.

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Removal of H₂S by iron-oxide

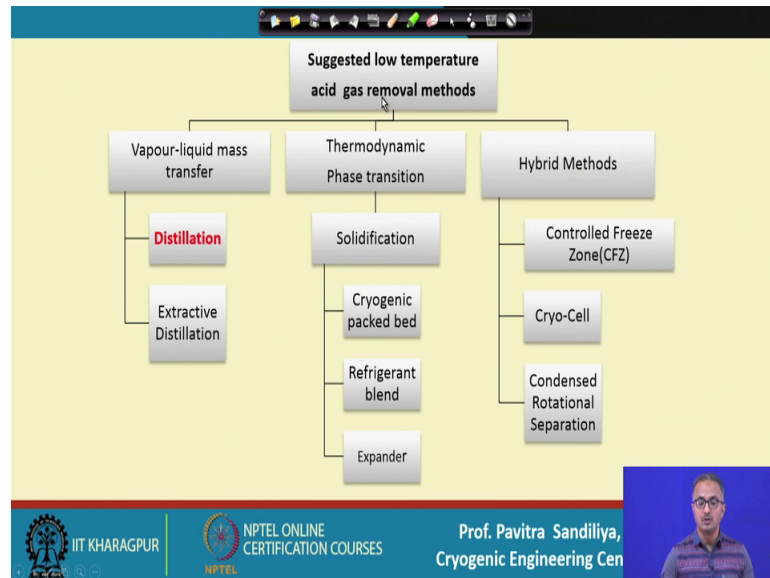
- ✓ Uses iron as an oxidizing agent in a solution (**redox** process)
$$\text{Fe}_3\text{O}_4 + 6\text{H}_2\text{S} \rightarrow 3\text{FeS}_2 + 4\text{H}_2\text{O} + 2\text{H}_2$$
- ✓ Steps involved :
 - Removal of H₂S from the gas by absorption into a caustic solution
 - Oxidation of the HS⁻ ion to elemental sulfur via the oxidizing agent
 - Separation and removal of sulfur from the solution
 - Regeneration (i.e., oxidation) of the oxidizing agent by use of air

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For the iron this iron oxide we have the redox reaction takes place and this is the reaction with the iron oxide. And we find that which reacts with the H₂S to give this FeS₂ this is precipitates and we have water and hydrogen.

And the steps involved the, that removal of H₂S from the gas by absorption into a caustic solution. Then oxidation of the HS ion to elemental sulfur via some oxidizing agent and separation and removal of the sulfur from the solution and we regeneration that is oxidation of the oxidizing agent by use of air. So, these are the several steps which are involved in the scavenging of the H₂S by iron oxide.

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And lastly we come to some of the low temperature method; the low temperature methods are getting developed. And the several broad categories of this some are the vapor liquid mass transfer is involved; some are just some thermodynamic phase transition and some of them are hybrid methods.

So, all this methods are also getting developed and some of them have been proposed. And like we have distillation, extractive distillation, then their solidification, cryogenic packed bed, refrigerant blend, expander. Then on the hybrid we have controlled freeze zone cryo cell and condensed rotational separation.


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Distillation

- ✓ Good for removing both CO_2 and H_2S from natural gas,
 - Because the vapor pressures of the principal components are considerably different.
- ✓ Faces operating problems during CO_2 separation from different natural gas components




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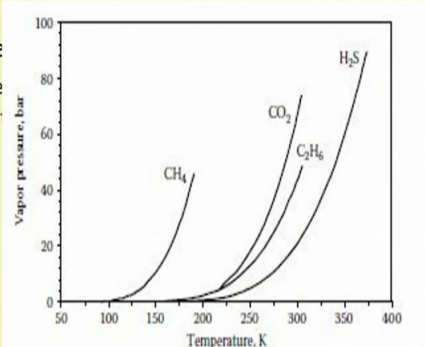


So among them we shall be talking about distillation which is most common among the load temperature removal of these gases. So, they are good remove both carbon dioxide and H_2S from natural gas because the vapor pressures are considerably different from the other natural gas components.

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Distillation

- ✓ Good for re
 - Because differen



Vapor pressure, bar

Temperature, K

CH₄


CO₂

C₂H₆


H₂S

considerably

Vapor pressure of light natural gas components




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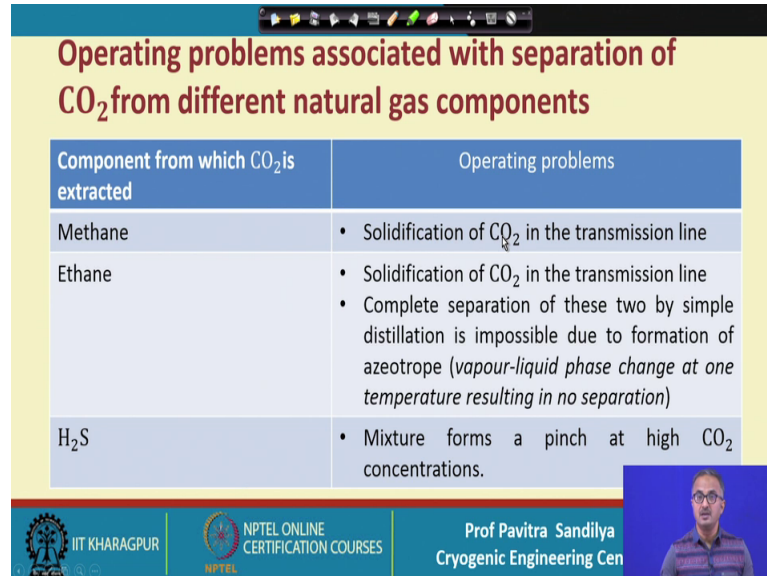
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And here we have the vapor pressure chart for these gases that we find that these carbon dioxide H_2S ; they are their vapor pressures are quite different from that of the methane.

And they the face the, these distillation faces problem operating problem during carbon dioxide separation from different natural gas components.

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Component from which CO ₂ is extracted	Operating problems
Methane	<ul style="list-style-type: none">• Solidification of CO₂ in the transmission line
Ethane	<ul style="list-style-type: none">• Solidification of CO₂ in the transmission line• Complete separation of these two by simple distillation is impossible due to formation of azeotrope (<i>vapour-liquid phase change at one temperature resulting in no separation</i>)
H ₂ S	<ul style="list-style-type: none">• Mixture forms a pinch at high CO₂ concentrations.

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And here we have the operating problem is methane that methane may gets solidified means solidification the carbon dioxide in the transmission line. So; that means, if the carbon dioxide solidified solidifies then it will chock the flow then for ethane also same thing happens carbon dioxide can gets solidified.

And there could be complete separation of these 2 by simple distillation may not be there because it azeotrope. Azeotrope means we have some vapor liquid phase change at single temperature. Vapor liquid phase change we have learned earlier that when we have a mixture of components; then generally this vapor liquid phase change that is either evaporation or the condensation takes place over a range of temperature.

That is the dew point temperature and the bubble point temperature and within these temperatures we find that we get some separation. But if these components from some azeotrope then the this phase change would occur only at a single temperature and then we will not be able to get any kind of separation. So, that is why we find that they from azeotrope and we cannot get any separation and in case of H₂S mixture forms a pinch at high carbon dioxide concentration.

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Commercially used methods for acid gas removal

- ✓ [Ryan/Holmes method](#)
- ✓ CFZ

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Then we have these commercially used methods; like Ryan Holmes and the controlled freezing zone process Ryan zone Holmes is quite popular.

(Refer Slide Time: 18:42)

Ryan/Holmes method

- ✓ Is an extractive distillation process that uses hydrocarbons to significantly alter the behaviour of the system .
 - The hydrocarbons are normally mixtures of propane and heavier hydrocarbons obtained from the feed mixture.
- ✓ Effectively eliminate the distillation problems.
- ✓ No additional separations are required.

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And this is a typical method that it has an extractive distillation process that uses hydrocarbons to significantly alter the behavior of the system.

The hydrocarbons are normally mixtures of propane and heavier hydrocarbons; they are obtained from the feed mixtures and they can effectively eliminate the distillation problems and no additional separations are required.

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Ryan

Four Column Ryan Holmes

✓ Is a
alter
•
✓ Effe
✓ No

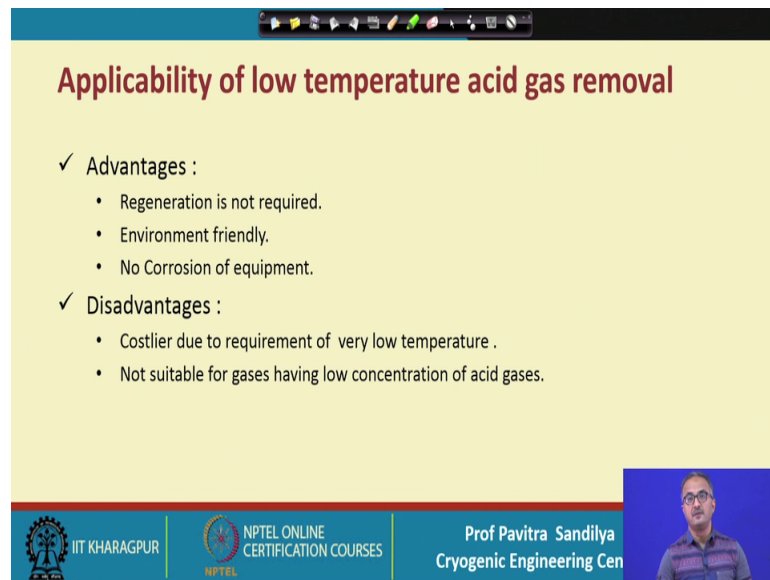
Significantly hydrocarbons

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And this is a typical Ryan Holmes process in which we find the natural gas is coming after compression, dehydration; it is passing through several columns and in each of the columns we are able to recover the various gases. And ultimately we are able to produce the natural gas and here we find that we are getting the NGL that is the natural gas liquid. And the sulfur gas is obtained here and the other gases are been recovered from this. And the ultimately at the last stage we are able to get this C 4 plus compounds here.

So, this is a typical 4 columns Ryan Holmes process to take out the H 2 S and carbon dioxide from the natural gas.

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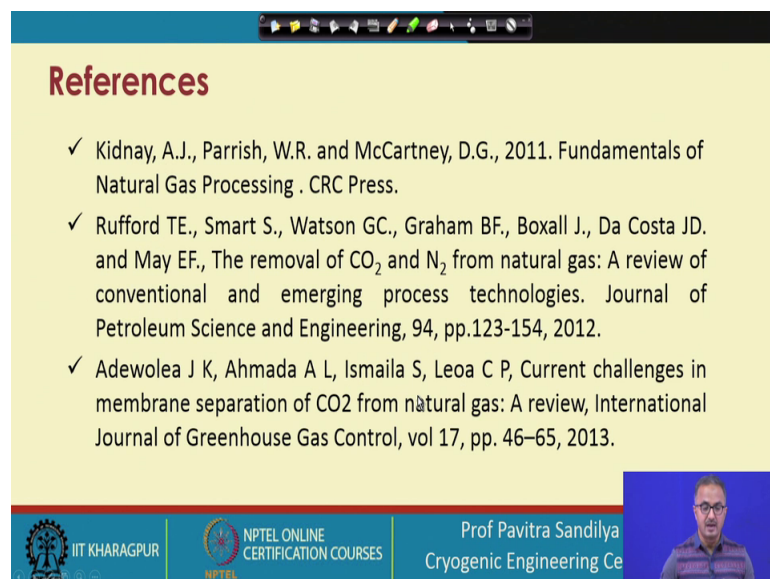
Applicability of low temperature acid gas removal

- ✓ Advantages :
 - Regeneration is not required.
 - Environment friendly.
 - No Corrosion of equipment.
- ✓ Disadvantages :
 - Costlier due to requirement of very low temperature .
 - Not suitable for gases having low concentration of acid gases.

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So, this low temperature acid gas removal have some advantages like we do not need any kind of regeneration; they are environmental friendly and there is no corrosion of the equipment. Whereas, disadvantage are there costlier due to requirement of low temperature and these low temperature need different types of refrigeration systems. And especially this we need to have some kind of cryogenic systems for this which we are not talking in this particular lecture and they are not suitable for gases have been low concentration of acid gas.

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And here we have the references which will talk in more detail about these various processes. We have learned about the acid gas removal that is the removal of the carbon dioxide and H₂S.

Thank you.