

Inorganic Chemical Technology
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Lecture - 08
Industrial Gases

Welcome to the MOOCS course Inorganic Chemical Technology the title of today's lecture is Industrial Gases. There are a few gases which are very essential for many of the industrial purposes such gases are known as a industrial gases and these gases are also produced or manufactured industrially, right.

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Industrial Gases

- Industrial gases are manufactured for the use in industries
- They include oxygen, nitrogen, hydrogen, carbon dioxide and noble gases
- Oxygen, nitrogen and hydrogen are often used as raw materials for production of other chemicals
- Packaged foods flavour (by reducing chemical action leading to rancidity) preserved by N_2
- Oxygen and helium are essential medicaments
- Many of these gases, their liquids and solids (for example: liquid CO_2 and dry ice) have a common application in creating cold*
- By absorbing heat upon evaporation; by performing work; or by melting
- New division of cryogenics engineering has aroused with modern expansion of industry

Handwritten notes in red: "later temperature" circled next to the "creating cold" bullet point.

What are these gases these are oxygen, nitrogen, hydrogen, carbon dioxide and noble gases. All of them are having one or other kind of a applications and in some of these things are already known to us. Let us say carbon dioxide in compressed form it is used as a kind of fire extinguisher in solid form like dry ice it is used as dry ice whereas, the hydrogen is often used for many synthesis purposes.

Nitrogen is used for the production of ammonia synthesis gas, ammonia urea and other kind of a nitrogen fertilizers it is sometimes also used as a kind of a inert atmosphere for conducting a few reactions where inert atmosphere is essential. Oxygen is often we know it is used for oxidation partial oxidation or combustion this kind of reaction it is often used it is also used as a kind of medical requirement as well many times ok.

So, as we understand that oxygen nitrogen and hydrogen are often used as raw materials for production of other chemicals. Let us say N₂ is also used for preserving the flavour of packed material packed food material especially. How does it prevents the flavour of the packaged food by reducing the chemical action occurring on them over the time of storage. So, that may lead if there is a chemical action then that may lead to rancidity which is not good from the food point of view or packaged food point of view.

So, it is also used for the preservation of food items. Oxygen and helium are essential medicaments. Many of these gases their liquids and solids for example, liquid CO₂ like used for fire extinguisher and dry ice often used for many purposes have a common application in creating cold, right. So, they are used for creating cold how they do creation of cold for a required applications they do by absorbing the heat upon evaporation or by performing work or by melting right.

So, since we understand this creating of cold; that means, there is something which appear or happen at very low temperatures. So, something is happening. So, that whatever the things are happening like you know both production and utilization of these gases that now we can understand that they are happening at low temperatures.

So, because of such applications at lower temperatures associated with these gases there is a special or a new division called cryogenic engineering right. So, what is this cryogenic, that is what we are going to see.

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Cryogenics

- It deals with production and utilization of low temperature in the region from absolute zero to 123 K; and "cryogenics" term is extensively used
- Use of liquid hydrogen, oxygen and fluorine in missiles for military and space applications display the importance of this technology (x)
- Advanced cryogenic techniques are also used for storage, handling and shipment of very cold liquids and gases
- Cryogenic liquids are stored and shipped in tanks built in multiple-walled vacuum bottle (O₂)
- An example of economic advantage of using cryogenic liquids: (4)
 - A cylinder weighing 113 kg contains 169 kg of (liquid) O₂ (equivalent to 126 m³ of gaseous O₂ at NTP)
 - Eighteen small cylinders of gaseous O₂ contain a total of 166 kg of O₂ at 15MPa but they weigh 1090 kg
- Cryogenic (super-cold) temperatures cause fundamental changes in properties of materials

Cryogenics as the name indicate it deals with the production and utilization both not only production, but also utilization also takes place at low temperatures, what is the range of low temperature it is in general absolute 0 to 123 kelvin. So, this production and utilization occurring at such low temperatures are often associated with this cryogenics and then cryogenics term is extensively used.

So, what is so, great about cryogenics how do we understand, how much it is important to study as a separate division? So, some of the applications we see now we know that use of liquid hydrogen oxygen and fluorine are used in missiles for military purpose and then space application purpose also they are used something like rocket propulsion etcetera. So, these are the two important applications if you see from these applications point of view we can understand that how much it is important.

Because the missiles for military or you know any component associated with the space technology are very expensive and they need to be given at most care for each and every element. So, for those industries if these are going to be useful so, then we must study in detail about this cryogenics. Some advanced application techniques are also developed in the cryogenics engineering division and then these techniques are often used for storage handling and shipment of very cold liquids and gases.

So, they are not only just applications kind of thing, but for the storage also they are very important handling and then shipment of very cold liquids and gases also they advanced cryogenic techniques are developed. Cryogenic liquids are often stored and shipped in tanks built in multiple-walled vacuum bottle.

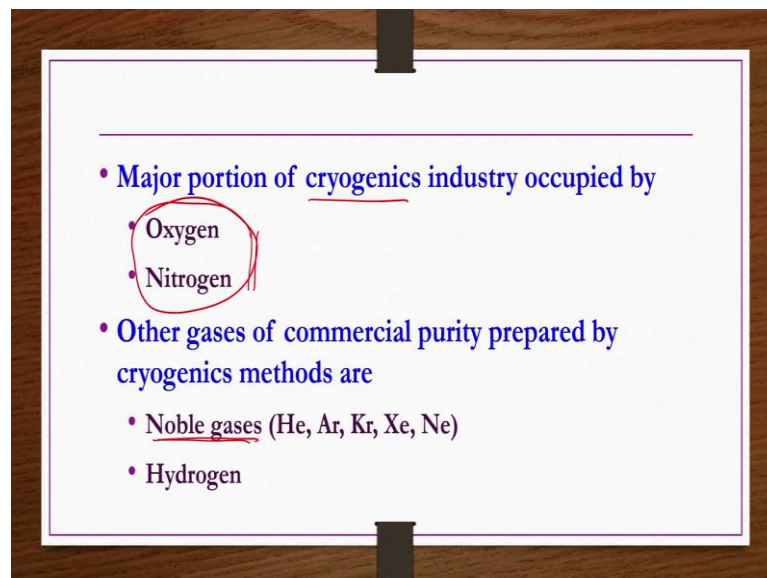
So, what is so important? Because now; obviously, whatever the equipment or storage vessels that are available for storing at the atmospheric conditions cannot be used the same thing for the for storing the cryogenic material right. So, then we need to have a separate you know equipment design.

So, that to you know one can easily store and transport this cryogenic materials ok. So, we see one example how much it is important to have a knowledge of cryogenic engineering for example, we take a oxygen right, we take oxygen right. If you wanted to store 166 kgs of gaseous oxygen at 15 mega pascals you need to have 18 small cylinders which may be weighing approximately 1090 kgs.

But the approximately same weight or slightly higher than the 166 that is 169 kgs of liquid O₂ if you wanted to store then you need a cylinder which would be weighing only 113 kgs; that means, if you are the same amount of same weight of oxygen if you are storing in gases from normal conditions then the storing vessels may be weighing almost 10 times higher compared to the same if you are doing at the cryogenic or liquid oxygen conditions ok.

So, this is one example to indicate how much it is essential from the economics point of view also not only from the application point of view. Cryogenic or super cold temperatures cause fundamental changes in properties of materials because of that one only such kind of a changes or advantages we may have.

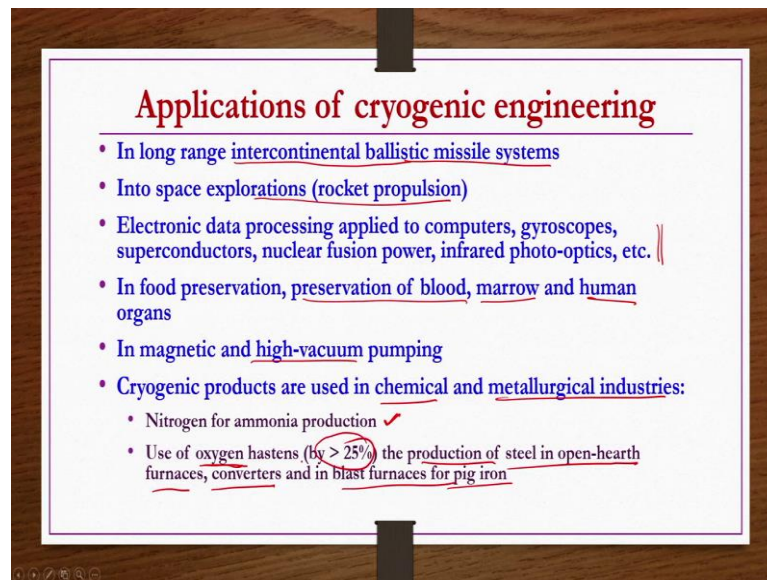
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Now, we see major portion of cryogenics industries are found to be occupied by oxygen and nitrogen. Though there are also like hydrogen and then carbon dioxide in addition to that some noble gases are also there these are also produced commercially right using cryogenic methods.

So, what are we going to see in this lecture and then coming lecture we are going to study the production or purification of air to get oxygen and nitrogen separately ok. And then we will be discussing the production of carbon dioxide and hydrogen in the next class.

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So, before going to the production of this cryogenic oxygen and nitrogen what we do we see a few applications of cryogenic engineering as I already mentioned that in long range intercontinental ballistic missile system you need them right. So, in space explorations also something like rocket propulsion also you need them there are also other applications like electronic data processing applied to computers, gyroscopes, superconductors, nuclear fusion power, infrared photo-optics etcetera.

In all these applications also you need a knowledge of cryogenic engineering or you know this cryogenic material. In food preservation also preservation of blood, marrow and then human organs also it is used. Also used in magnetic and high vacuum pumping the performance of some materials required you know you need to maintain a low temperature and under such low temperatures if you are pumping the material maybe become waxy kind of let us say wax if you wanted to transport.

If you transport at low temperature that may become much more solidified and then it become very difficult to transport. So, then you can do you know you can apply this magnetic and high vacuum pumping system to transport such kind of material. Cryogenic products are used in chemical and metallurgical industries as well for example, nitrogen for ammonia production as explained.

And then use of oxygen low purity oxygen itself if you use then the production of steel in open hearth furnaces converters and in blast furnaces for pig iron increases almost by

more than 25 percent ok. Now, let us say how do you do this one if you wanted to convert a gas into the liquid form what are the things are you supposed to know.

So, one important thing that you need to understand is liquefaction of gases let us say vapours of substances which are liquids at atmospheric conditions if you want to condense them you can condense them by cooling right that we all know.

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Liquefaction of gases

- Vapours of substances, which are liquids at atmospheric conditions, are condensed by cooling ✓
- Liquefaction of substances, which are liquid at lower temperatures, is possible by compression or a combination of compression and cooling
- Pressure required for liquefaction can be reduced by cooling
- Examples of such gases:
 - Ammonia, sulfur dioxide, methyl chloride, fluorocarbons, chlorine and hydrogen sulphide, etc.
- For liquefaction of permanent gases (O₂, N₂, H₂ and He), in addition to compression and cooling, expansion may also be required ||

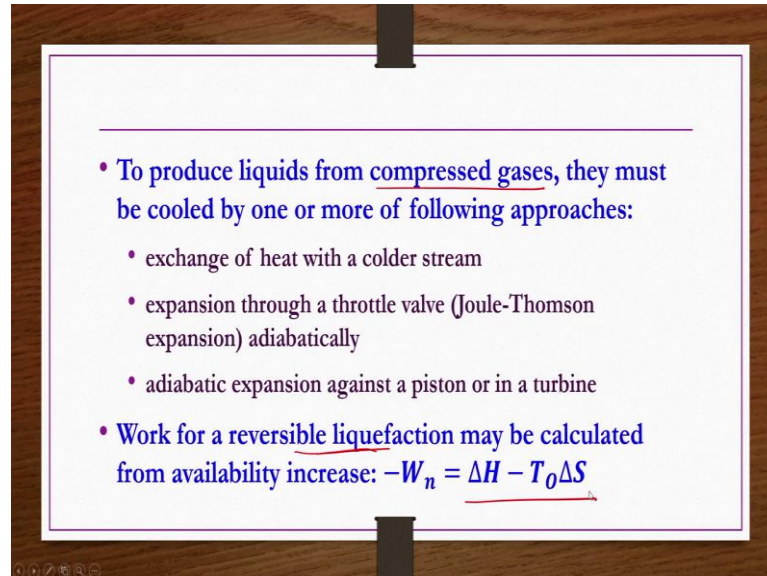
Let us say; however, liquefaction of substances if you wanted to those substances are liquid at lower temperature then it is possible to further do the liquefaction of such substances by compression or by a combination of compression and cooling ok. So, pressure required for liquefaction can be reduced by cooling as well.

Actually liquefaction occurs by increasing the pressure right let us say if you take a gas and then if you wanted to liquefy what you have to do? You have to give the pressure high pressure you have to apply to that gas so, that gas can be liquefied. And then that requirement of high pressure may be reduced if the process is accompanied by cooling as well ok. Some examples of such gases are ammonia, sulphur dioxide, methyl chloride, fluorocarbons, chlorine, hydrogen sulphide, etcetera.

For liquefaction of permanent gases like O₂ N₂ H₂ and helium, in addition to compression and cooling, it may also be required to have expansion in the process. So,

these are actually some cycles are there in cycles these are these kind of compression expansion cooling etcetera occurring so, accordingly we have to design a system.

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To produce liquids from compressed gases, they must be cooled by one or more of the following approaches: before starting compressing them. So, what are they exchange of heat with a colder stream otherwise expansion through a throttle valve adiabatically, which is also known as Joule Thomson expansion. Also adiabatic expansion against a piston or in a turbine is also another approach in order to get the liquids from the compressed gases ok.

Work for reversible liquefaction if at all is required may be calculated from the availability increase that is what is the value of delta H minus T delta S. Now, what we do we are going to discuss manufacturing of oxygen and nitrogen. So, as any chemical manufacturing that we have been doing whenever we are discuss start discussing production of any chemical what are we doing we starting with the properties of the material and then raw materials and then quantitative requirements flow sheet etcetera, that is how we are progressing. So, here also we are going to do the same thing.

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Pertinent properties of oxygen and nitrogen

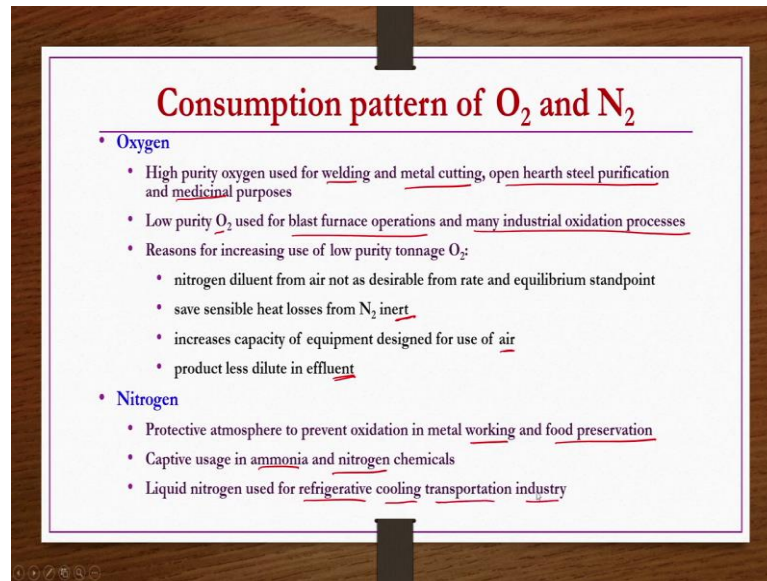
- **Oxygen**
 - Mol. Wt. 32.00
 - Melting pt. -218.8°C
 - Boiling pt. -183°C
 - Grades: High purity 99.5% O_2 and 0.5% Ar
Low purity 90 – 95% O_2 , 4 – 5% Ar & balance $\text{N}_2, \text{CO}_2, \text{H}_2\text{O}$
- **Nitrogen**
 - Mol. Wt. 28.02
 - Melting pt. -210°C
 - Boiling pt. -195.8°C
 - Grades: Technical 99% N_2 balance Ar and O_2

So, let us see properties of oxygen and nitrogen - oxygen molecular weight is 32, melting point is minus 218.8 degree centigrades, boiling point is minus 183 degree centigrade, grades high purity oxygen if you want then that should be 99.5 percent or more pure and then remaining is balanced by the argon, low purity oxygen if you want then it should purity should be between 90 to 95 percent and then 4 to 5 percent would be argon and then imbalances that is because of balances due to the N_2 , CO_2 and H_2O ok.

Coming to the nitrogen molecular weight is 28.02, melting point is minus 210 degree centigrade, boiling point is minus 195.8 degree centigrade, grades technically 99 percent pure N_2 is available in general balances ArO_2 or both of them may be present in remaining 1 percent.

So, now we see consumption pattern of O_2 and N_2 right. So, if not sufficient consumption is there. So, then why do we need to you know produce all of them. So, what we have to do we have to understand the consumption pattern a little bit if not all applications or usage of these two.

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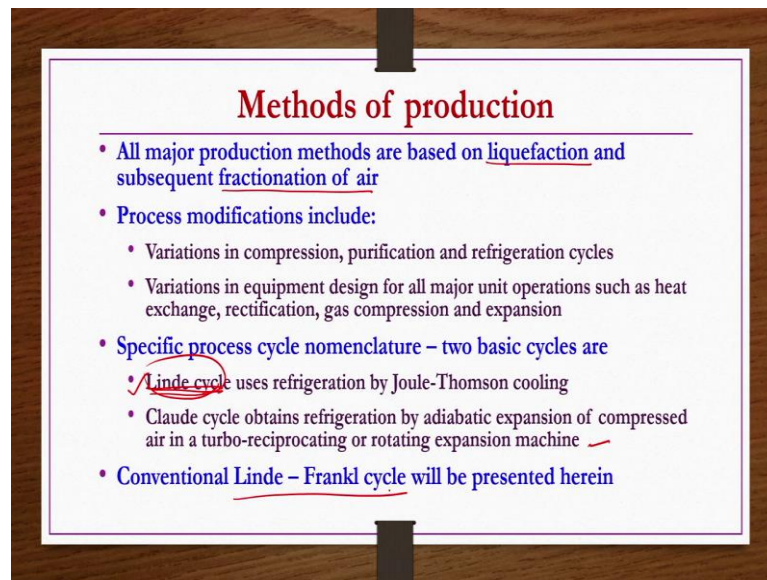
Consumption pattern of O₂ and N₂

- **Oxygen**
 - High purity oxygen used for welding and metal cutting, open hearth steel purification and medicinal purposes
 - Low purity O₂ used for blast furnace operations and many industrial oxidation processes
 - Reasons for increasing use of low purity tonnage O₂:
 - nitrogen diluent from air not as desirable from rate and equilibrium standpoint
 - save sensible heat losses from N₂ inert
 - increases capacity of equipment designed for use of air
 - product less dilute in effluent
- **Nitrogen**
 - Protective atmosphere to prevent oxidation in metal working and food preservation
 - Captive usage in ammonia and nitrogen chemicals
 - Liquid nitrogen used for refrigerative cooling transportation industry

Let us start with oxygen high purity oxygen used for welding, metal cutting, open hearth steel purification and as well medicinal purposes. And then low purity oxygen used for blast furnace operations and many industrial oxidation processes use often low purity oxygen. Reasons for increasing use of low purity tonnage oxygen compared to the high purity oxygen or because the nitrogen diluent from air not as desirable from rate and the equilibrium standpoint and then it saves sensible heat losses from N₂ inert.

Also increases capacity of equipment designed for use of air then product less dilute in effluent. Coming to the nitrogen it is used as a protective atmosphere to prevent oxidation in metal working and food preservation, captive usage is in ammonia and then nitrogen chemicals production liquid nitrogen used for refrigerative cooling transportation industry as well ok. Now we will see methods of production of O₂ and N₂.

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All major production methods are based on liquefaction followed by subsequent fractionation of air. So, if all the methods are based on the same principles. So, then what are the difference or you know why do we need to study all different methods. So, there may be some variations small small variations from one plant to the other plant.

And most importantly these O₂, N₂ are often produced on site especially from industrial applications point of view this O₂ and N₂ are produced on site especially for industrial applications view point ok. So, from one industry to the other industry situations may be different or from one application to the other application may be very different.

So, depending on these kind of variations there may be slight variations in production methods from one industry or production of this O₂ and N₂ from one onsite production to the other onsite productions. So, what are these possible variations? Variations in compression purification and refrigeration cycles then variations in equipment design for all major unit operations such as heat exchange, rectification gas, compression and expansion.

So, specific process cycle nomenclatures are there which are very well established we see couple of them - one is the, Linde cycle that uses refrigeration by Joule-Thomson cooling, another one is the Claude cycle obtains refrigeration by adiabatic expansion of compressed air in a turbo-reciprocating or rotating expansion machine right. So, but;

however, this is very conventional one and then we are going to see this Linde-Frankl cycle for the production of low purity tonnage oxygen.

So, Linde-Frankl cycle for low purity oxygen that process we are going to see now, ok.

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Linde - Frankl cycle for low purity O₂

- **Raw materials**
 - Air by usual composition by volume: N₂ (78.03%), O₂ (20.97%), Ar (0.94%), H₂ (10⁻⁴%), CO₂ (0.02 - 0.07) and H₂O (0.01 - 0.03%)
 - Minor quantities of NaOH, NH₃ and silica gel
- **Quantitative requirements:**
 - (a) **Basis: 1 ton of 95% oxygen in 300 tons/day plant**
 - Air 3600 Nm³
 - Steam 1.75 tons
 - Cooling water 5 tons
 - Electricity 450 - 480 kWh
 - (b) **Plant capacities: 50 - 500 tons/day**
 - **Chemical reaction for CO₂ scrubbing: 2NaOH + CO₂ → Na₂CO₃ + H₂O**

So; obviously, raw materials is the starting point what is the raw material for production of oxygen and nitrogen any plant you take the naturally available air is a raw material ok. So, the air that is having composition usually N₂ - 78.03 percent, O₂ - 20.97 percent right remaining are argon, hydrogen CO₂ and then H₂O etcetera would be there right.

So, the natural air is taken as raw material minor quantities of sodium hydroxide, ammonia and silica gel are also used. In general in application why they are used for purification or for removing the CO₂, H₂O purpose these are used these chemicals are required in minor quantities only. Quantitative requirements point of view if you see if you wanted to produce one 1 of 95 percent pure oxygen in a 300 tons per day plant. So, then how much air is required 3600 normal cubic meters of air is required.

Steam 1.75 tons required cooling water 5 tons required electricity 450 to 480 kilowatt hours required, plant capacity is usually between 50 to 500 tons per day ok. So, for removing of CO₂ some chemical reactions may also take place with NaOH for example, then 2NaOH plus CO₂ giving rise to sodium carbonate along with the water this is the

chemical reaction often involved that is also if there is a CO₂ and then if you wanted to remove it by scrubbing with sodium hydroxide solution ok.

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Process description

- Feed air is compressed to 4-5 atm., then cooled with water prior to passing it into regenerative exchangers
- These exchangers, operating in pairs, are cylindrical pressure vessels packed with aluminium spirals
- One exchanger is used for air cooling while cold product gas (containing N₂ and O₂) is removing sensible heat from the packing in the other exchanger
- Cycle is reversed by automatic valves after 2 to 4 minutes for effective use of exchangers
- Water and carbon dioxide must be removed from air before sending it to fractionation section
- After removing these impurities, air enters fractionation section operates at -183 to -195°C to prevent plugging
 - These impurities are removed on cold packing throughout the exchanger
 - Then re-evaporated by product gas which starts with zero concentration of H₂O and CO₂
 - Thus provides an equilibrium driving force for the vaporization process

Now, we see process description then we are going to see the same thing in flow sheet as well ok. The feed air is compressed to 4 to 5 cool after compressing it is cooled with water prior to passing it into regenerative exchangers. These regenerative exchangers are very important aspects of the process because in other process or probably up to this exchangers step whatever is there; that means, to only different from one process to the other process.

After crossing this exchangers step whatever the remaining fractionation of air etcetera are there they are same. Let us say now we are discussing low purity oxygen production right using this Linde-Frankl cycle right if you wanted to produce high purity oxygen using the Kellogg cycle then what we will be having only this regenerative exchangers would be replaced by recuperative exchangers and then after that whatever the fractionation of air etcetera are there that is same here and then other process that is Kellogg process as well.

So, what are these regenerative exchangers? They are actually operating in pairs these are from the equipment or from the unit operations point of view these are physical pressure vessels packed with aluminium spirals. So, they are the cylindrical in shape and

then inside this pressure vessels there is a aluminium spirals packing as well. So, both of them are having some purpose ok.

Now, one exchanger is used for air cooling ok because I said you know they are operating in pairs. So, 2 are there ok one is used for air cooling while the cold product gas which is containing N₂ and O₂ is removing sensible heat from the packing in the other exchanger what is the packing aluminium spirals. So, from them sensible heat is being removed you know by the product gases in the other exchanger because there are 2 exchangers they are operating in pairs, ok.

And then these cycles are reversed by automatic walls after 2 to 4 minutes depending on the plant to plant for effective use of exchangers that is first you know let us say exchanger one is used for air cooling, exchanger two is used for a removing the sensible heat from the packing material when we do the reversing of the cycle the exchanger one would be used for a removing the sensible heat from the packaging that is present in the first exchanger.

Whereas the second exchanger would be used for air cooling because both of them are having aluminium spirals packing and then design such a way ok. And then water and carbon dioxide should not be there in the air they must be removed before sending it to the fractionation section. Basically crudely in a Layman language if you wanted to understand this process what is this process of production of O₂ and N₂ you have two steps one is the exchanger steps and then another one is the fractionation step.

Before the exchanger step there is a compressor is used for the drying of the air right after drying the air is cooled and then sent to the exchangers, in the exchangers 2 actions are taking place air is being cooled in one exchanger and another exchanger sensible heat is being removed right.

So, then cycles interchange after every 2 to 4 minutes. So, after that whatever the product gases N₂, O₂ are there. So, they should be sent to the fractionation section they should be sent to fractionation section, but what we have to make sure that before sending to the fractionation section water and carbon dioxide should be removed.

Not only water and carbon dioxide if at all any hydrocarbons also present then they should also be removed from what is the source of this hydrocarbons you may be

thinking, but as I mentioned most of these plants are installed on site wherever let us say you have ammonia production plant.

There you need pure nitrogen hydrogen maybe you getting by different methods of the synthesis gas production that we have seen in the previous section nitrogen you have to get by this method or the other method that we are going to study after this method. So, there if since the onset it is there or it is required for a partial oxidation oxygen pure low purity oxygen is required for the partial oxidation or combustion of some of the components in the fuel industry.

So, then; obviously, the surrounding atmosphere may be contaminated with the hydrocarbons as well. So, then there is a possibility of hydrocarbons present as well in the air. So, they should also be removed ok. So, how do we do that part of the removal of this can also be done in the exchangers how that we see, after removing these impurities air enters the fractionation section operates at minus 183 to minus 195 degree centigrades to prevent plugging.

So, how this removal takes place these impurities are removed on cold packing throughout the exchanger whatever the exchanger that we have then in cold packing these impurities are removed and then re evaporated by product gas whatever N₂, O₂ product gases are there which starts with zero concentration of H₂O and then CO₂ then thus this will provide an equilibrium driving force for the vaporization process as well.

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- In this type of cycle, often impurities cannot be completely removed and thus only low purity product gas (O₂ and N₂) can produced
- Air leaving the regenerative HEs is cooled to -170°C and fed to reboiler section of the double column where further cooling takes place
 - Double column (or two distillation columns) with a low pressure (1.4 atm.) column standing on top of high pressure (5.7 atm.) column
 - Reboiler of upper column working as condenser of vapours from lower column
 - In high pressure section, more volatile N₂ reaches to top of the column and is condensed inside the tubes of reboiler of low pressure column by liquid O₂ surrounding the tubes
 - This is accomplished because the temperature of liquid oxygen at 1.4 atm. is lower than condensation temperature of saturated nitrogen vapor at 5.7 atm.
 - This condensed nitrogen is then sprayed into the top of low pressure column for reflux
 - Less volatile oxygen still containing 50% N₂ is pumped to middle of low pressure column where final rectification takes place *

So, often in this type of cycle impurities cannot be removed completely and then; obviously, you can produce low purity product gas product may be O₂ or N₂ or both ok. So, air leaving the regenerative heat exchangers is cooled to minus 170 degree centigrade and fed to reboiler section of the double column where further cooling takes place what is this double column right that we are going to see now, ok.

So, double column or 2 distillation columns that is 2 distillation column are there one is mounted on to the other ok. So, one is standing on to the other with low pressure column which is operating at 1.4 atmospheric conditions pressure conditions that is standing on a top of high pressure column that is operating at 5.7 atmosphere column.

Re-boiler of upper column working as condenser of vapours from the lower column ok in high pressure section more volatile N₂ out of this O₂ and N₂, N₂ is more volatile and then it reaches the top of the column easily and is condensed inside the tubes of reboiler of low pressure column by liquid O₂ surrounding the tubes. Let us say simply we are going to see the flow sheet also.

So, there is one column another column is there like this. So, in between there is a heat exchanger these columns are provided with a some kind of trace number of trace or stages required for distillation that one has to calculate all those things from the mass transfer principle that is different thing.

Now, this is operating at 5.7 atmosphere this is operating at 1.4 atmosphere. So, whatever the O₂, N₂ is there. So, that is entering from the reboiler section of the lower column and then since N₂ is the low volatile thing. So, that moves up like this right. So, this more volatile N₂ reaches the top of the column and is condensed inside the reboiler of low pressure column here it actually here itself there is a reboiler should be there.

So, that will be you know in that it is getting condensed right how it is getting condensed because liquid O₂ is surrounding the tubes right. So, this is accomplished because the temperature of liquid oxygen at 1.4 atmosphere is lower than the condensation temperature of saturated nitrogen vapour at 5.7 atmosphere.

So, because of this region you need to have a condensation temperature of standard nitrogen is required condensation of standard nitrogen is required. So, that you can get the saturated nitrogen vapour and then that is possible at 5.7 atmosphere that is the

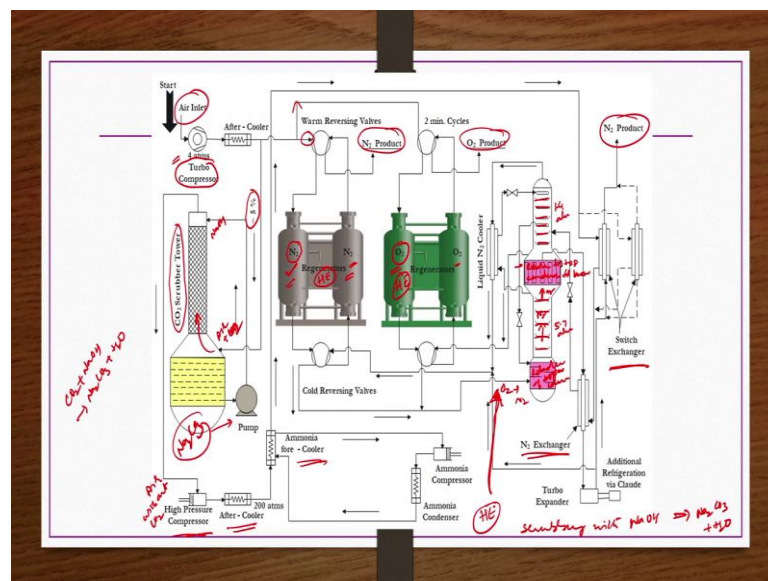
reason lower column is operating at the 5.7 atmosphere and then nitrogen as it moves up and then reaches the top of the lower column it get condensed in the reboiler of the top column ok.

So, the condensers are provided at the top usually reboilers are provided at the bottom end usually at the bottom ok. So, for both of the columns there would be a condenser there would be a reboiler. So, now, here it is a reboiler is there. So, whatever the condenser is there or whatever the reboiler of the top column is there they are same they are doing the combined operation for them for one column it is operating as a kind of reboiler, other one it is for the top one it is operating as a reboiler for the bottom one it is operating as a condenser right.

So, because of that this pressure difference you know the nitrogen gets saturated you get the saturated nitrogen and then that is being condensed in the reboiler of the top column that is top column is nothing but the low pressure column and then it is get condensed how it is get condensed because the surrounding tubes are occupied by the liquid O₂.

This condensed nitrogen is then spread into the top of low pressure column for reflux as well less volatile oxygen still containing 50 percent N₂ is pumped to the middle of low pressure column that is top column where final rectification takes place, right.

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So, next slide is the flow sheet is there. So, here we can clearly see ok. So, let us say we have a air that has to be compressed through turbo compressor at 4 atmosphere. So, that to dry it after drying when you do the drying water vapours kind of things would be removed by this compression process.

And then that after removing the water vapours etcetera that will be passed through a cooler and then after cooling you know you have a two options one option is that the air is going to end to regenerator this is nothing but whatever the exchangers regenerative exchangers that we have discussed in the text previous slides these are nothing but this one.

They work in the pairs. So, for one N₂ one pair for O₂ another pair so, whatever the air comes here. So, that will be you know regenerated and then here in this case in this first let us say first this is the first column here cooling is taking place in the second column what will happen it will be removing the sensible heat from the aluminium spirals that are present. These aluminium spirals are present in both the columns here oxygen also the same thing.

So, that after removing the sensible heat from the second column and two product is collected if it is of sufficient purity, otherwise it is sent back to the regenerative heat exchanger again and the cycle continues. Similarly whatever the air is there cooled air after removing the water vapours etcetera that will also go to oxygen regenerative exchangers right.

So, these exchangers now here again same process takes place here cooling of air takes place and then here the removing of sensible heat from this spirals takes place and this cycle continues and from this one if the outlet is having sufficient purity oxygen you can take collect it as a product otherwise you can send back, right. So, one cycle continues like this for 2 to 4 minutes after that they interchange their duties.

Now, the first one would be used for a sensible heat removal second one would be used for a cooling the air same is here also this cycle continues right. So, now, once you get the sufficiently dry air without H₂O etcetera then this product O₂, N₂ would be taken to the fractionation column right, where are we feeding it we are feeding it in the this is nothing but a reboiler of bottom column.

Fractionation column we understand it is to double column or two distillation columns lower one is operating at 5.7 atmosphere the top one is operating at 1.4 atmosphere right. So, when this O₂ and N₂ are the products they are they actually coming here. So, when it is coming N₂ is more volatile.

So, it passes through right and then here when it pass through this reboiler of the top column right now this section is reboiler for top column and then condenser for lower column. So, since it is a condenser for lower column. So, then what happens. So, whatever the pure nitrogen while it is passing through it will be condensed because of the surrounding liquid oxygen.

Liquid oxygen is surrounding the tubes. So, when the nitrogen passes through the tubes that will be condensed right and then that you can take it as a product or you can take liquid N₂ cooler and then send to the cold reversing walls and then warm reversing walls and this cycle continues like this right. Or you can take them to the N₂ exchange and then further passing through the switch exchanger you can get the N₂ product.

So, more purified N₂ you get it here and then liquid O₂ you will be getting in the reboiler of the or towards the reboiler of the lower column you will be getting the pure O₂ ok this cycle continues like this. Sometimes what happens you know on the air that we are taking that may also have CO₂ also.

So, that has to be removed. Let us say CO₂ if you wanted to remove there are one option is that after this process after this heat exchangers or regenerative exchangers after this step you can have this scrubbing with NaOH before feeding it to the fractionation column before feeding it to the fractionation column you can have this section here and then remove CO₂ in the form of Na₂CO₃ and then plus H₂O ok one other option is that if the CO₂ is very less.

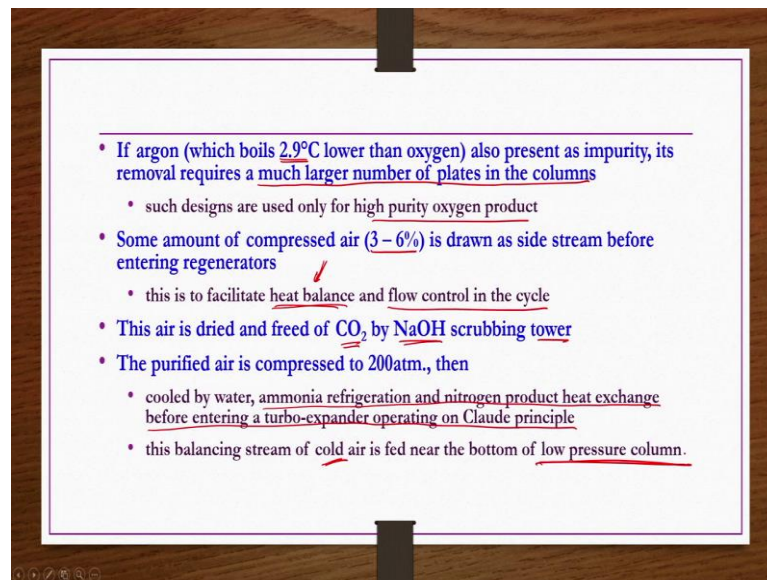
So, then what you can do you can bypass some amount 5 percent of air after cooling from the inlet you know you are compressing and then cooling it. After this cooling whatever the air is there 5 percent you can take and then it pass it through CO₂ scrubbing tower right. Here from the top you give NaOH solutions by pump and then from the bottom you give the air which is containing CO₂ also.

So, that CO₂ would be get absorbed and then as a solution you get Na₂CO₃. So, from the top you get air without CO₂ and then you pass through this air high pressure compressor for the removing any water etcetera that is present. Because when this reaction is taking place to remove CO₂ by NaOH we have seen the reaction not only Na₂CO₃ is forming, but also H₂O forming.

When it is forming? So, then it is obviously, it is possible that air may be wet. So, that has to be dried how you do in drying of the air by passing through high pressure compressor. So, when you do this drying so; obviously, air would become hot. So, then that has to be cooled and then it has to be passed through the circle again or you can take it to the ammonia core cooler and then ammonia condenser etcetera as per the applications.

Whereas, the sodium carbonate solution whatever is there that is pumped through and then recycle after regeneration etcetera that continues it ok. So, this is the process to get low purity tonnage oxygen production in addition to nitrogen production as well.

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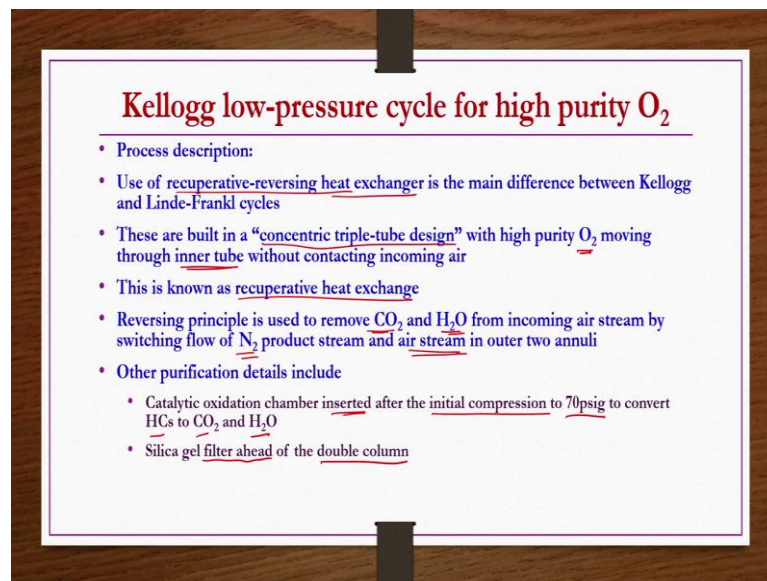


So, if argon which boils at 2.9 degree centigrade lower than oxygen is also present as impurity its removal requires much larger number of plates in the columns. In the fractionation column these are nothing but the these indicating the plates large number of plates may be required if you wanted to go for the you know high purity any of the chemicals that you wanted to purify by distillation.

If the required purity is very high so, then distillation column height would be high very large value and then number of plates would also be large. Such designs are used only for high purity oxygen product that we are going to see next slide. Some amount of compressed air is drawn as side stream before entering the regenerators this is to facilitate heat balance not only for removing CO₂ as we have shown in the previous slide, but also it is suitable or you know very useful for heat balance heat balance has to be maintained in the plant ok.

And flow control in the cycle as well the air is dried and freed of CO₂ by NaOH scrubbing tower as explained previous slide. The purified air is compressed to 200 atmosphere then cooled by water and then ammonia refrigeration and nitrogen product heat exchange before entering a turbo expander operating on Claude principle. This balancing stream of cold air is fed near the bottom of lower pressure column that is at the middle between the 2 columns.

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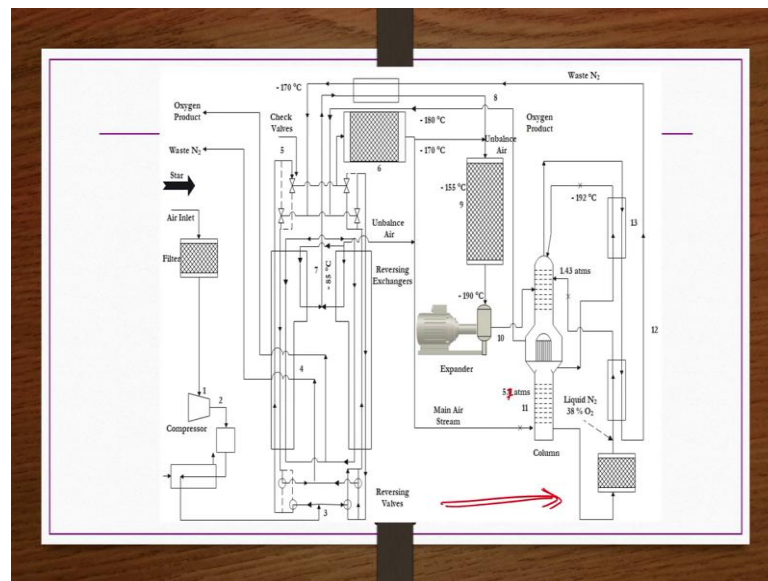
Kellogg low-pressure cycle for high purity O₂

- Process description:
- Use of recuperative-reversing heat exchanger is the main difference between Kellogg and Linde-Frankl cycles
- These are built in a "concentric triple-tube design" with high purity O₂ moving through inner tube without contacting incoming air
- This is known as recuperative heat exchange
- Reversing principle is used to remove CO₂ and H₂O from incoming air stream by switching flow of N₂ product stream and air stream in outer two annuli
- Other purification details include
 - Catalytic oxidation chamber inserted after the initial compression to 70psig to convert HCs to CO₂ and H₂O
 - Silica gel filter ahead of the double column

Now, what we do we see Kellogg low pressure cycle for high purity oxygen. So, process description as I mentioned use of recuperative reversing heat exchanger is the only or main difference between Kellogg cycle that we are going to see and then Linde-Frankl cycle that we have already seen in the previous slide. These are built in a concentric triple tube design with high purity O₂ moving through inner tube without contacting incoming air this is known as recuperative heat exchanger.

Reversing principle is used to remove CO₂ and then H₂O from the incoming air stream by switching flow of N₂ product stream and air stream in outer two annuli other purification details include catalytic oxidation chamber inserted after the initial compression to 70 psig to convert hydrocarbons to CO₂ and H₂O if at all hydrocarbons are present in the air. Silica gel filter ahead of the double column is used to remove if any water or CO₂s are present.

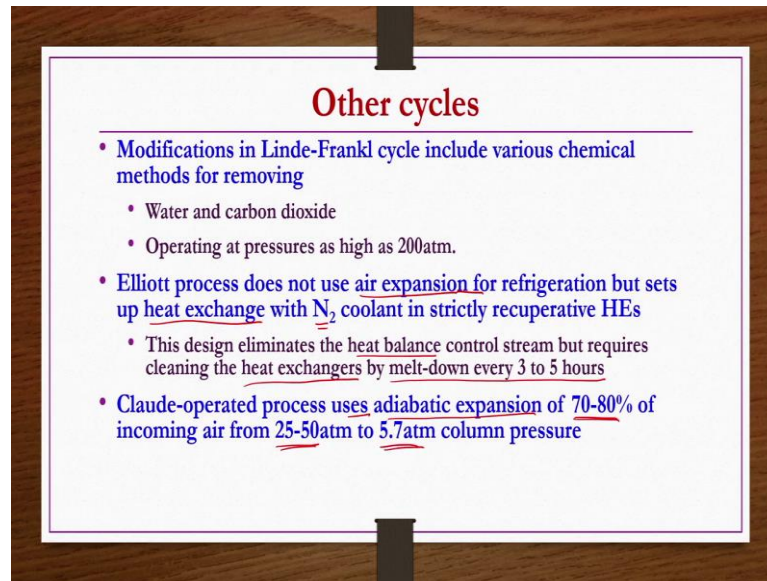
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Let us say the same thing is schematically shown here see after this recuperative heat exchanger unit. So, whatever this section is there that is same that is same as the previous one that is double column distillation or 2 distillation columns one on to the other all those things are same again here also ok.

So, this should be 5.7 atmosphere right. So, in addition to that one what we have see these are the concentric triple tube design recuperative heat exchangers through the center one purified O₂ is passing through and then the outermost one and air is flowing through the outermost one without interacting with the purified oxygen. So, that is the advantage of this one after this this process is again the same as what we have seen in the previous slide.

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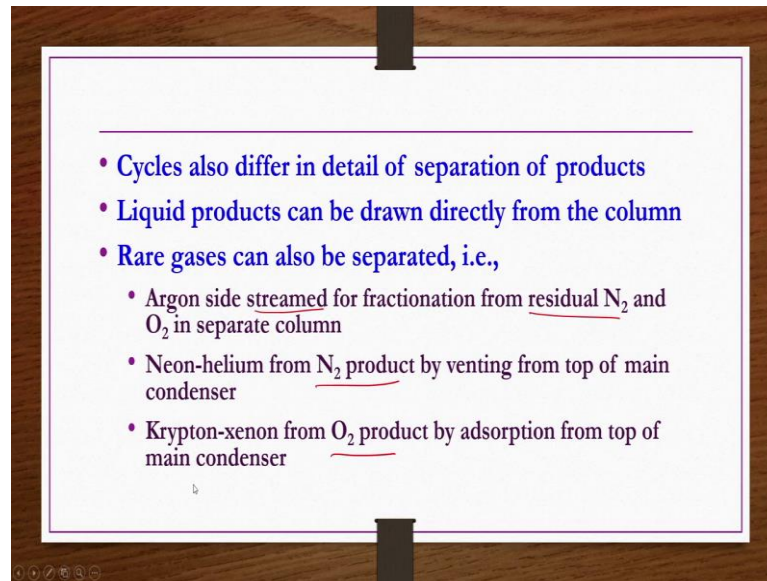
Other cycles

- **Modifications in Linde-Frankl cycle include various chemical methods for removing**
 - Water and carbon dioxide
 - Operating at pressures as high as 200atm.
- **Elliott process does not use air expansion for refrigeration but sets up heat exchange with N₂ coolant in strictly recuperative HEs**
 - This design eliminates the heat balance control stream but requires cleaning the heat exchangers by melt-down every 3 to 5 hours
- **Claude-operated process uses adiabatic expansion of 70-80% of incoming air from 25-50atm to 5.7atm column pressure**

Other cycles are also there some modification something like that modification in Linde Frankl cycle include various chemical methods for removing water and carbon dioxide and then operating at pressures as high as 200 atmospheres; however, another process that is Elliott process does not use air expansion for refrigeration, but sets up heat exchange with O₂ coolant in strictly recuperative heat exchangers.

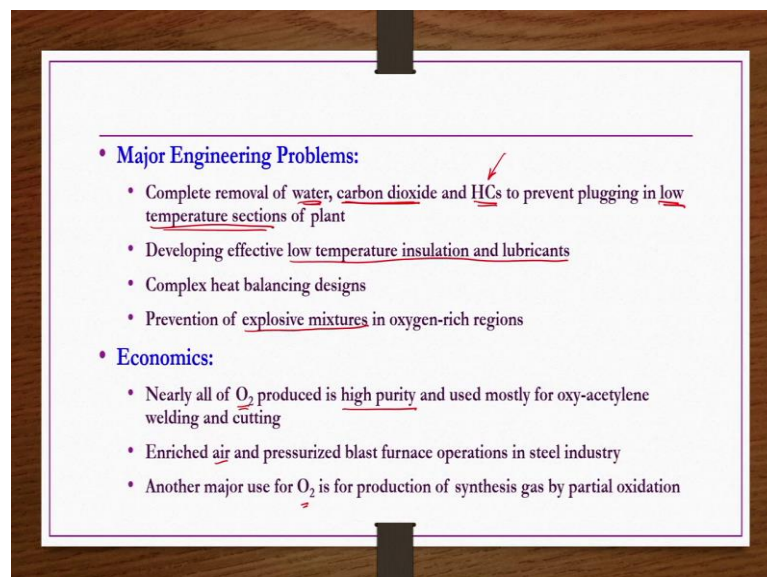
This design eliminates the heat balance control stream, but requires cleaning the heat exchangers by melt-down every 3 to 5 hours. Claude operated processes use adiabatic expansion of 70 to 80 percent of incoming air from 25 to 50 atmosphere to 5 to 7 atmosphere column pressure.

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Cycles also differ in detail of separation of products how the separation is being done. So, liquid products can be drawn directly from the column rare gases can also be separated that is argon side streamed for a fractionation from residual N₂ and then O₂ in separate column or neon helium from N₂ product by venting from top of main condenser or krypton-xenon from O₂ product by adsorption from top of main condenser, ok.

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So, whatever the cycles that we have seen for the production of low purity oxygen or high purity oxygen now, they seems to be simple and then having only a compression

and expansion kind of units, but however, there are certain kind of engineering problems associated with these cycles as well and then one must be careful about them what are the complete removal of water is one problem.

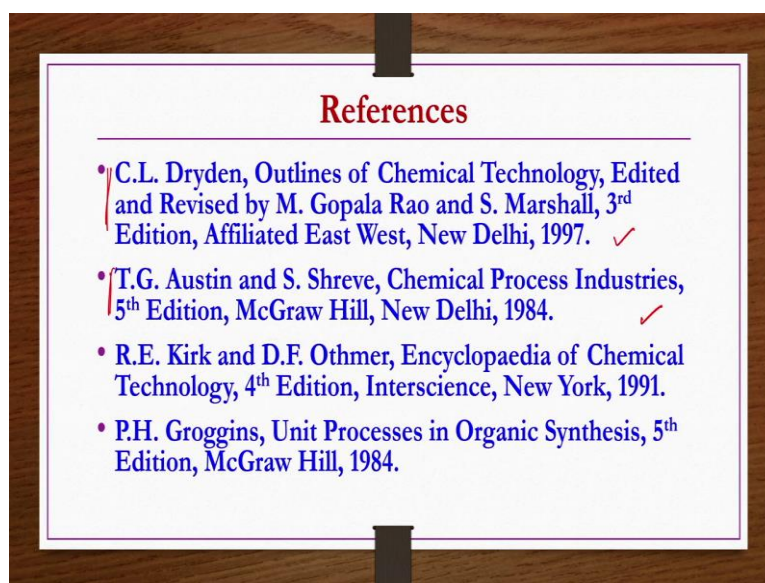
Similarly, complete removal of carbon dioxide is also another problem and then hydrocarbons to prevent plugging in low temperature sections of plant is also in a problem had it not been a low temperature process then removal of these things might not be a difficult at all right.

Since the process are low temperature cryogenic processes the removal of these impurities become very difficult and especially these impurities like hydrocarbons etcetera are dangerous if they are present in oxygen use that is going to be used for medical requirements.

Developing effective low temperature insulation and lubricants also sometimes you need lubrication, design from the connections etcetera, but the temperature if very low temperature then these lubricants may become solidified. So, that is one problem complex heat balancing designs is also another problem and then prevents enough explosive mixtures in oxygen rich regions is another very big danger.

From the economics point of view nearly all of O₂ produced is high purity and used mostly for oxy-acetylene welding and cutting processes. Enriched air and pressurized blast furnace operations in steel industry also require this oxygens. Another major use of O₂ is for production of synthesis gas by partial oxidation and then nitrogen is used for the production of ammonia and then several types of nitrogen fertilizers etcetera, ok

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The references for this lecture are provided here, but most of the notes is prepared from these two reference books.

Thank you.