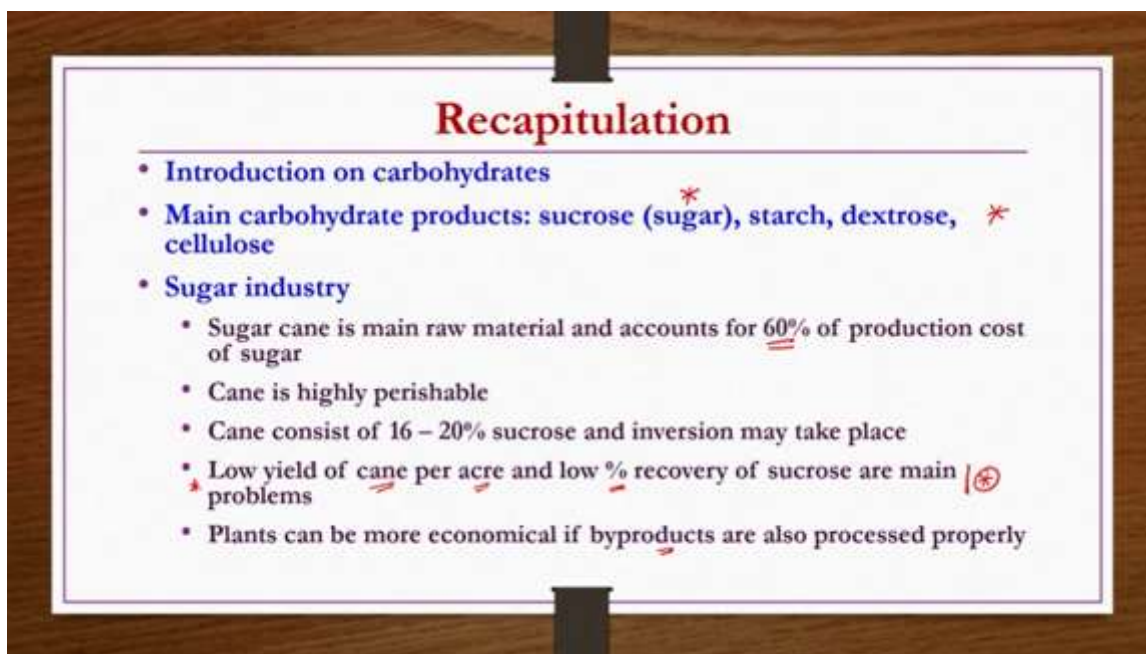


Lec 11: Carbohydrates Industry – Beet Sugar and Starch.

Welcome to the MOOCs course organic chemical technology. The title of today's lecture is carbohydrates industry, beet sugar and starch. In this particular lecture, we are going to discuss about the manufacturing of the beet sugar, corn sweeteners and then starch and its derivatives etc.

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Recapitulation

- Introduction on carbohydrates
- Main carbohydrate products: sucrose (sugar), starch, dextrose, cellulose
- Sugar industry
 - Sugar cane is main raw material and accounts for 60% of production cost of sugar
 - Cane is highly perishable
 - Cane consist of 16 – 20% sucrose and inversion may take place
 - Low yield of cane per acre and low % recovery of sucrose are main problems
 - Plants can be more economical if byproducts are also processed properly

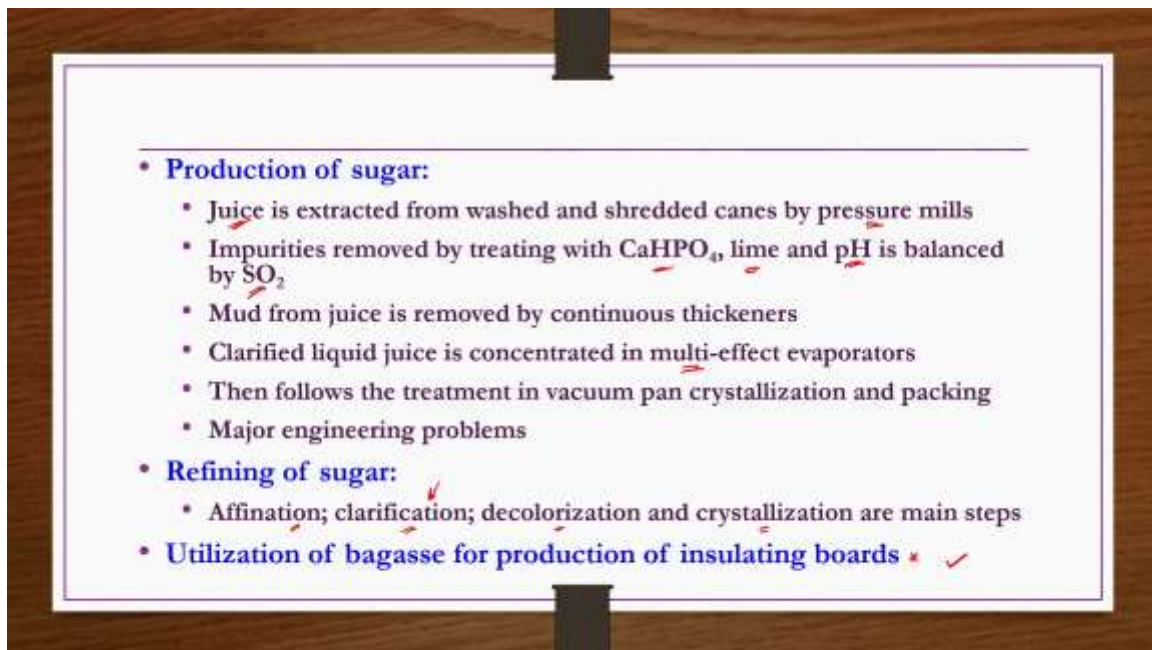
But however, before going into the details of today's lecture, we will have a recapitulation of what we have discussed in carbohydrates industries in last couple of lectures, okay? We started introduction on carbohydrates. Carbohydrates or hydrates of carbons are nothing but the natural products obtained by the plants by photosynthesis reaction where the plants utilizes the carbon dioxide and then moisture available in the environment and then energy from the sun in order to do the required photosynthesis reaction and then products coming out of such photosynthesis reactions are nothing but the carbohydrates.

These carbohydrates primarily consist of C, H and O atoms only that also hydrogen atoms should be twice the number of oxygen atoms and then we also studied that the carbohydrates are the main footsteps not only for the human beings but also for the animals, right? So, these carbohydrates also serves as a main raw material, one of the main source of raw material for the production of different types of chemicals, okay? Then we listed out different types of carbohydrate products out of which important are sucrose, starch, dextrose and cellulose. There may be n number of carbohydrates are available, we cannot cover all of them. So, then we listed some of them and then we started discussing their production process. So, first we started with the sugar or sucrose.

Sugar industry, for the sugar industry sugarcane is a primary raw material and then it accounts almost up to 60% of the production cost of the sugar that much important it is actually from the sugarcane you extract the juice and then remove the impurities and do the concentrating of the syrup followed by the crystallization that is what crudely sugar production process is. So, mostly most of the sucrose or sugar whatever is there that is coming out from the sugarcane juice and then sugarcane juice is having roughly 16 to 20% of sucrose only. So, then yield of juice or sucrose from the sugarcane is also plays an essential role in the success of the sugar industry. Further cane is highly perishable. If it is not utilized within 2 days after cutting from the cane cultivating field, it will undergo some kind of inversion reaction and then sugar may be inverted into the D glucose and D fructose as we have seen.

So that is the reason it is necessary for the sugar industry to be closer to the cane fields or cane field should be developed near to the sugar industries. They should be within economic distance of each other. Then other problems of sugar industries are low yield of cane per acre of the land as well as the low percentage recovery of sucrose from the sugarcane. These are the 2 important factors if you are improving or able to improve these factors then sugar industry is going to be very profitable. Plants can be more economical if byproducts are also processed properly something like bagasse you can use to make the insulation boards etc. as we discussed in the previous lecture or the molasses you can use in fermentation industries for the production of ethanol etc.

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The slide is a presentation slide with a white background and a purple border. It contains two main sections: 'Production of sugar' and 'Refining of sugar'. The 'Production of sugar' section lists six steps: juice extraction from washed and shredded canes by pressure mills; impurities removal by treating with CaHPO_4 , lime, and pH balanced by SO_2 ; mud removal by continuous thickeners; concentration of clarified liquid juice in multi-effect evaporators; treatment in vacuum pan for crystallization and packing; and major engineering problems. The 'Refining of sugar' section lists one step: affination, clarification, decolorization, and crystallization as main steps. A final bullet point states 'Utilization of bagasse for production of insulating boards' with a red checkmark.

- **Production of sugar:**
 - Juice is extracted from washed and shredded canes by pressure mills
 - Impurities removed by treating with CaHPO_4 , lime and pH is balanced by SO_2
 - Mud from juice is removed by continuous thickeners
 - Clarified liquid juice is concentrated in multi-effect evaporators
 - Then follows the treatment in vacuum pan crystallization and packing
 - Major engineering problems
- **Refining of sugar:**
 - Affination; clarification; decolorization and crystallization are main steps
- **Utilization of bagasse for production of insulating boards** ✓

Then if you see the steps involved in the production of sugar, crudely juice is extracted from washed and shredded canes by pressure mills. This juice would be having so many

impurities, those impurities are removed in a continuous thickener by adding calcium phosphates, lime etc. So, what they will do? They will be precipitating out the impurities so that they can be easily removed from the juice. That is the purpose of using such inorganics.

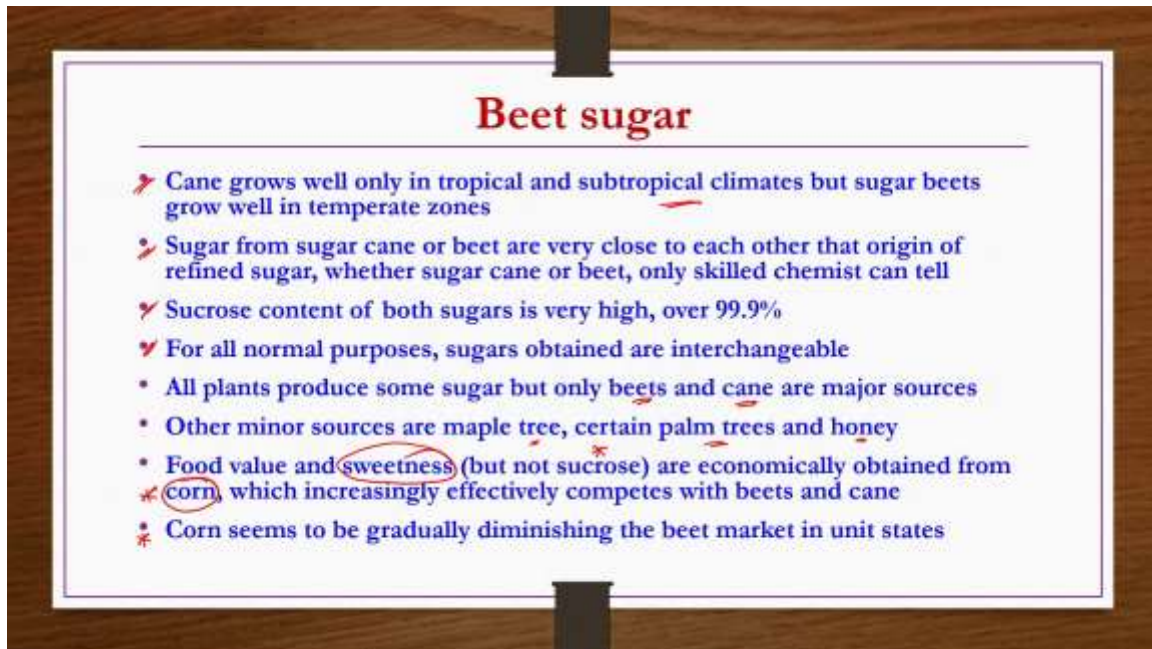
Also, SO_2 is also provided or you know juice is aerated by the SO_2 so that pH is balanced between the 7.5 to 7 range within that range itself. This will also enhance the flocculation so that impurities can also be removed easily. In addition, if you are using calcium phosphate etc. for removing the impurities then what happens kind of saccharides of calcium may take place.

Decompression of such impurities may be removed by using SO_2 as well. Then what happens? Such saccharides will form calcium sulphides, they can be easily removed by the centrifugation. Mud from the bottom of the thickener whatever is there that would be checked how much sucrose content is there, right? If it is not having any sucrose content, it will be removed from the continuous thickener and then used as a cattle feed. Otherwise if it is having certain considerable amount of sucrose, then it will be fed back to the continuous thickener again as a recycle. Then clarified liquid juice from the thickener whatever is there that would be having 80 to 85% water though it is pure enough sufficiently pure enough only that it is diluted with the 80 to 85% of water.

That water content is reduced to 40 to 45% by multi effective operators. Once this step is done, then the concentration of the juice would be only 40 to 45% of water. That juice would be further treated in vacuum pan followed by the crystallization and packing etc. These are the main steps that are involved in the production of sugar that we have seen. Then we also discussed the major engineering problems of the sugar industries in general.

The sugar that you produce by the above method extraction method may be having the impurities or molasses may be attached as a layer on the sugar crystal. So, refining of sugar may be required, so that also we have discussed in which up affination, clarification, decolorization and crystallization steps are the primary ones. Clarification may be mechanical clarification or chemical clarification as we have already seen. Then utilization of bagasse for the production of insulating boards also we have discussed. Until this point we have discussed in last couple of lectures on carbohydrate industries.

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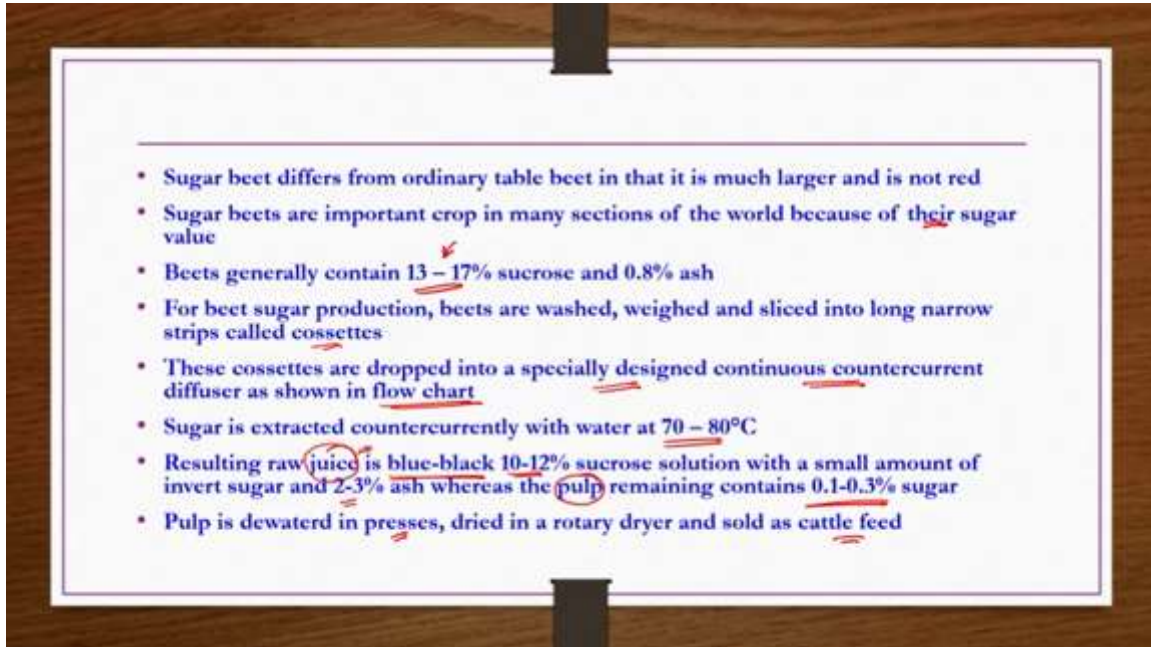
Now, we will be discussing about a beet sugar. Why do we need to study about the beet sugar when we have option of sugar cane to produce the sugar because sugar cane is often produced only in tropical or subtropical environment only but sugar beets grow well even in the temperate zones as well. Further, the sugar that is produced from the sugar cane as well as the beet if you compare, they are very much close to each other only thoroughly a professional chemist can only say that whether the given sugar is derived from the sugar cane or from the beet sugar. So that is the other reason. So, because of such reasons, they can be interchangeable without any difficulty.

They are very similar to each other. Their sucrose content is also very high. Because of such reasons, it is necessary to discuss or have a plant so that you can produce sugar from the beet as well, okay? So that is what we are going to discuss now. In general, all plants produce some amount of sugar but only beets and cane are major sources because the sucrose content is very high whereas in other plants it may not be such high that you do the industrial processing to recover such sugars, okay? Some other minor sources include maple tree, certain palm trees and honey. Food value and sweetness, sweetness does not mean by the sucrose alone, right? Here food value and the sweetness are economically obtained from corn as well.

So, this is also we are going to discuss after studying the beet sugar, right? Under the section of starch, we will be discussing how to get the corn sweetness, etc. also, we are going to discuss after the discussion on beet sugar production, okay? Corn seems to be gradually diminishing the beet market especially in United States of America. The reasons

or many reasons are there. So those reasons also we are going to list out in a few slides later.

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Sugar beet differs from ordinary table beet in that it is much larger and it is not red. Sugar beets are important crop in many sections of the world because of their sugar value. Beets generally contain 13 to 17% of sucrose slightly lesser than the sugar cane, right? Sugar cane but the per acre cultivation point if you see, the beets production is higher compared to the cane, right? Also, the time to produce also it is half the time for the canes production approximately 18 months is required but for the beets production within 9 months you can produce and then you produce more quantity or the yield of the beets is more compared to the yield of sugar cane per acre of the plant also within lesser time. So, these are some of the advantage of the beets anyway. Only disadvantage with this beet is that sucrose content is slightly lesser compared to the sugar cane. For beet sugar production, beets are washed, weighed and sliced into long narrow strips called cassettes.

These cassettes are dropped into a specially designed continuous counter current diffuser as shown in the flow chart in the next slide we are discussing thoroughly there. Sugar is extracted counter currently with water at 70 to 80 degree centigrade from the diffuser. Resulting raw juice is blue, black in color with 10 to 12% of sucrose solution with a small amount of invert sugar and 2 to 3% of ash whereas the pulp whatever remaining is there that contains only 0.1 to 0.3% of sugar only. So that pulp can be dried in rotary dryer and then sold as cattle feed. Then this juice has to be further processed to get the beet sugar.

[illegible]

So, from the top water around 60 to 80 degrees centigrade is allowed and then from the bottom slices of the beets are entering. These are counter currently interacting with each other. So that what happens whatever the bead slices are there from that juice would be extracted and then that juice would be processed further. Whereas the pulp whatever is there that would be having very less amount of sugar less than 0.3, 0.5% of sucrose only. So that can be dried in rotary dryers and then sold as cattle feed. Whatever the juice that is there here that will be having only 12 to 14% of sucrose etc. here that juice is taken to a chamber which is known as the carbonator. So here carb and then sulph, W and then con like this kind of nomenclature are there. Carb in the sense carbonators, sulph in the sense sulphiters treating with the SO_2 in the carbonator treating with CO_2 , F stands for the

filtration, W for the wash water, C for the centrifugals, con for the condensers, D for the dissolvers.

So here when this juice blue, black color 12 to 14% sucrose containing juice solution whatever is there that you take to the carbonator to which slaked lime suspension is also added. Why this lime suspension is added to this one? Whatever the impurities are there, they will be precipitated out when they interact with the lime that is the purpose you know this lime is being used. So then that impurities are removed from the juice. So, in this process what you do? When you are treating this juice, diluted juice 12 to 14% sucrose solution with lime what will happen? Saccharides of calcium or calcium saccharides may be produced, right? So, they will be decomposed by treating with the carbon dioxide. That is the reason this is known as this carburetor is very essential.

You are trying to reduce the impurities by treating with the lime, but if it is forming some saccharides, calcium saccharides then it is not going to be useful. So that calcium saccharides has to be decomposed by the carbon dioxide, right? So, in this process when you are allowing the carbon dioxide some kind of roving will take place and then sludge would be developed. That sludge would also be having sufficient amount of sucrose, etc. So that sludge would be washed in the rotary drum filtration process or rotary drum filter it is shown here, right? So now here you are adding water for this washing of the sludge, etc. That sludge when you wash out then whatever the liquid that you are getting that water kind of thing that is nothing but the sweet water.

That sweet water you can recycle back to the carbonator again, fine? So, after removing the sludge whatever the material is there that again you take to the another carburetor where again you treat with the lime so that to remove some more impurities if at all remaining, right? But again, if you are using lime here then what will happen? A kind of calcium saccharides may take place or may be produced. So those things would be decomposed by this carbon dioxide. That is the reason that this outcome of the rotary filter whatever is there that is again carbureted or processed in the carbonator, right? From here whatever the material that is coming that would be washed with water in a plate and frame filter press, right? So here whatever the sweet water is there that is recovered and then sent back along with the lime saccharides to the carburetor again, right? These dotted lines are optional kind of thing. If required they should be used otherwise not, right? So then juice whatever is there that would be further sent to a chamber which is known as sulphiter. Let us say even after removing impurities, etc. by the plate and frame filters, still if there are any calcium ions, etc. are there. So then if you treat with the sulphur dioxide here then what happens? You know calcium sulphides would be formed. These calcium sulphides can be washed away again in another plate and frame filter press, right? So, for that purpose again you are using the water and then whatever these sweet water is there that can be utilized as a recycle option if required, right? Whereas the juice after removing the calcium ions, etc.

then that would be sent to multiple effect evaporator because the juice that is coming from this plate and frame filter whatever is there that is having low sucrose content.

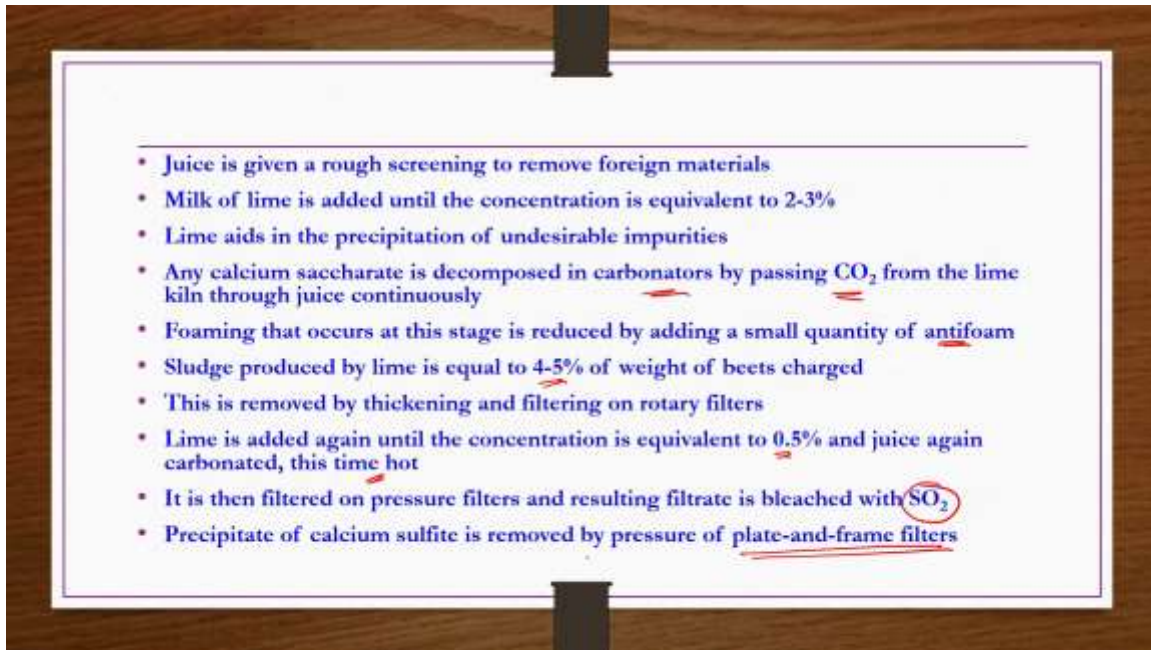
That sucrose content would be increased to 60%, approximately 60% sucrose here. So that syrup whatever 60% sucrose syrup is there that would be sent back to another sulphiter again if required to remove if at all any calcium still remaining. Then the mixture is sent to a plate and frame filter press again where water is used for washing purpose to wash out the sweet water and then recycle back to the carbonator again if it is required, right? Whereas the juice is there whatever the clear juice 60% sucrose but much more purified one that juice is sent to a dissolver where you know some amount of liquids, etc. Weak syrup, etc. are being added which are coming from the recycling process of the other centrifuges.

From the diffuser the material undergoes a kind of a you know decolorization process which is not shown here. Decolorization takes place where you know this syrup interacts with the activated carbon in a countercurrent direction so that to remove the colors, right? So then after that the syrup would be processed through another filter press to remove any carbon particles are remaining within the syrup because of this decoloration process, right? Then that syrup is sent to white pan where moisture is removed or it is concentrated by removing the water and then further concentrated syrup is sent to a centrifugal chamber here, right? So, where you know syrup is concentrated by removing some more water and then whatever the highly concentrated sugar syrup is there that would be sent to a granulator to do required you know granules making, drying and granules making and then packing, etc. After the granulator whatever the crystals that you get you check for the size of the crystals if it is sufficient then you can grate white sugar accordingly and then pack them, right? This granulator here drying is done by providing the hot air, okay? So, if the size is not sufficient enough that can be fed back to the centrifugation chamber or dissolver again for the reprocessing, okay? Now here from the white pan whatever the water is removed by the condenser that may also be containing some amount of sucrose in general it is possible. So, that will be treated in a high raw pan where further water is removed and then that syrup is diluted syrup is collected here that would be sent back to another centrifugation chamber from it, it will go to the dissolver again as a recycled syrup, right? Which will be mixed with a syrup that is coming after sulfonators, okay? then it continues until you know entire syrup is recovered by the different stages of evaporation and then centrifugation what you do? You recover as much of syrup and then finally you get a solution where there is no sugar in the syrup that or the black strap whatever is there that you can collect as molasses, molasses to desugarizing. This can be taken to the fermentation industry for the production of citric acid, right? This crystallization units are there here in general, in the previous slides also we have seen.

So, in the crystallization what happens in general that also we see in the subsequent slide anyway. So, here you know do mixing and then reduce the temperature such a way that

uniformity of the syrup will take place and then crystals will also form by the cooling that is what it takes. How it takes place? With a proper picture of crystallizer we are going to discuss at the end of today's lecture, right? So, this is what about production of beet sugar from the beet, okay?

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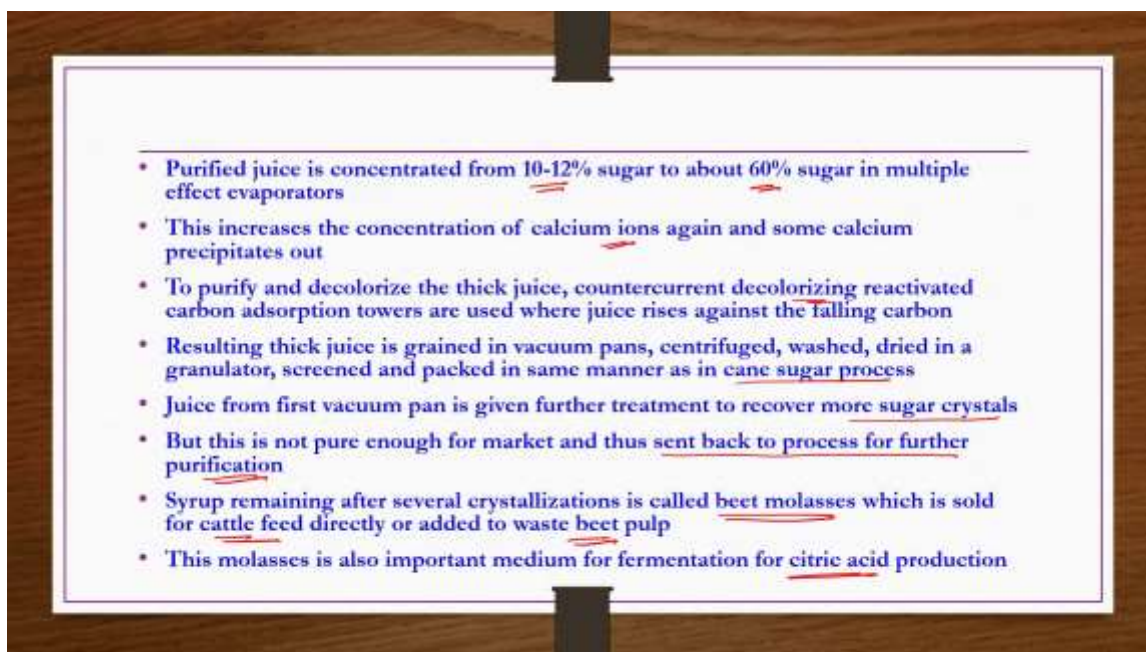


Whatever we discussed in the flowchart the same thing is provided here as a text for the understanding. Juice is given a rough screening to remove foreign materials after collecting from the diffuser. After collecting from the diffuser whatever the juice is there it is given a rough screening to remove foreign materials.

Milk of lime is added until the concentration is equivalent to 2 to 3%. Lime aids in the precipitation of undesirable impurities. Any calcium saccharides are forming if at all while treating with the lime and juice. While treating juice with the lime there is a possibility of calcium saturated formation. So, those may be decomposed in a carbonators by passing CO_2 from the lime kiln through juice continuously.

By this one what happens there is a possibility of foaming takes place that foaming may be reduced by adding small amount of anti-foaming agents. Sludge produced by lime is equal to 4 to 5% of weight of beets charged in general. This is removed by the thickening and filtering on rotary filters. Lime is added again until the concentration is equivalent to 0.5% and juice again carbonated This time but it is under the hot conditions. It is then filtered on pressure filters and resulting filtrate is bleached with SO_2 . Precipitate of calcium sulfite is removed by the pressure of plate and frame filters.

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Then purified juice is concentrated from 10 to 12% sugar to about 60% sugar in multiple effect operators. This increases the concentration of calcium ions again and some calcium precipitates out. To purify and decolorize the thick juice, countercurrent decolorizing reactivated carbon adsorption towers are used where juice rises against the falling carbon.

Resulting thick juice is grained in vacuum pans, centrifuged, washed, dried in a granulator, screened and unpacked in a same manner as in cane sugar production process. Juice from first vacuum pan is given further treatment to recover more sugar crystals. But this is not pure enough for market and thus sent back to process for further purification. Syrup remaining after several crystallization is called beet molasses which is sold for cattle feed directly or added to waste beet pulp. It is also important medium for fermentation for production of citric acid.

Citric acid production by fermentation can be done by using this molasses of the beet sugar plant. So, that is all about the beet sugar production. But now until now we have seen production of sugar from the sugar cane and then refining of the raw sugar and then beet sugar production from the beet sugars, right? So why only sugar? If it is only the purpose of the sweeteners, so then why cannot we get such sweeteners from the other sources? One of such other source is the corn. So, we are discussing about corn sweeteners now.

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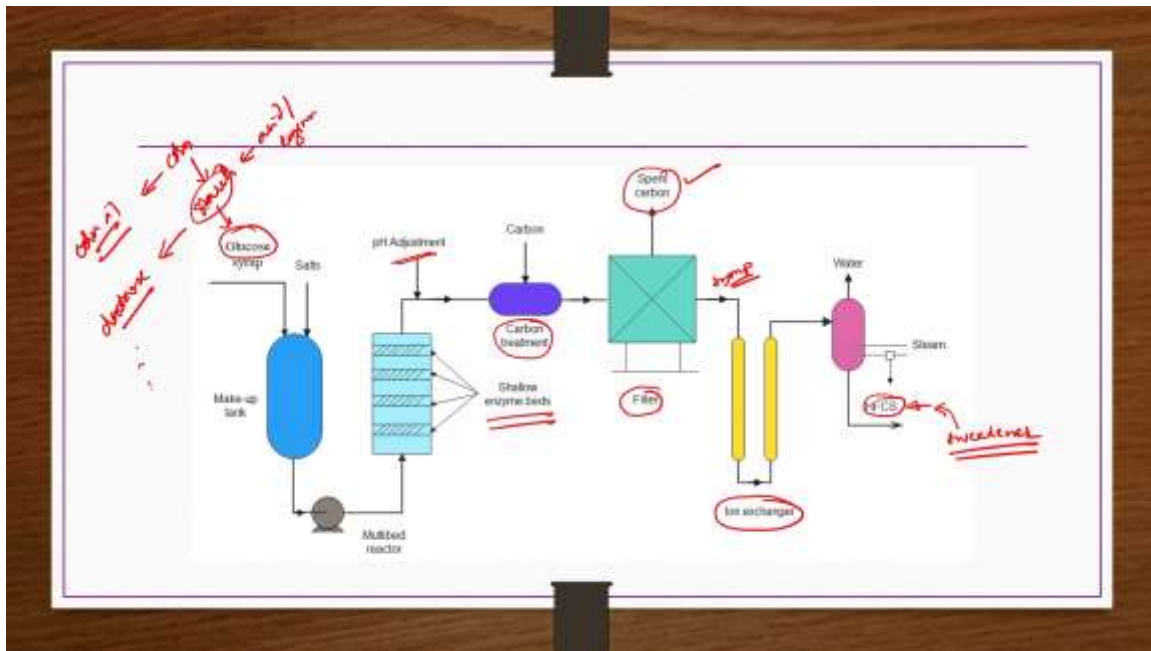
Corn sweeteners

- Major portion of nutritive sweeteners nowadays comes from starch (derived mainly from corn) conversion into fructose-containing syrups
- Conversion of starch into glucose by treatment with acid or with enzyme α -amylase has been practiced for years
- Syrup obtained by this method is nourishing but not very sweet tasting
- Enzyme beds have also been developed where enzymes could be immobilized on organic substrates
- Common substrates such as diethyl-aminoethyl cellulose or certain ceramic materials made enzyme use economically possible
- This feasibility because
 - A single charge could serve for a long period (several hundred hours) and
 - Treat a considerable quantity of material before requiring replacement

Major portion of nutritive sweeteners nowadays comes from the starch which is primarily derived from the corn. If you convert the starch into fructose containing syrups, then you can use it as a sweetener, right? And then soon we are going to see that corn industry or the sweeteners from the corn is more economical without considering the value of the byproducts. There are byproducts as well like corn oil we have discussed in the previous week on the oils and fats industries, right? Then you can also produce like starch, then also you can produce starch derivatives etc. as we are going to discuss subsequently, okay? So, conversion of starch into glucose by treatment with acid or with enzyme has been practiced for several years. So, this glucose you can take, this is coming from the starch and then starch coming from the corn, right? So, the starch if you convert into glucose by either treating with acid or with enzyme, then you get the glucose. By treating starch with acid or enzymes, you get the glucose.

That glucose you can do the conversion to the fructose which is more sweetening than the sugar, okay? Syrup obtained by this method is nourishing but not very sweet tasting. That is the reason this glucose whatever is there further has to be converted to fructose to improve the sweetness. Enzyme beds have also been developed where enzymes could be immobilized on organic substrates. Common substrates such as diethyl, amino-ethyl cellulose or certain ceramic materials made enzyme use economically possible. How we are going to discuss soon? This feasibility is because of a single charge could serve for a long period almost like several 100 hours and then treat a considerable quantity of material before requiring any kind of replacement, okay?

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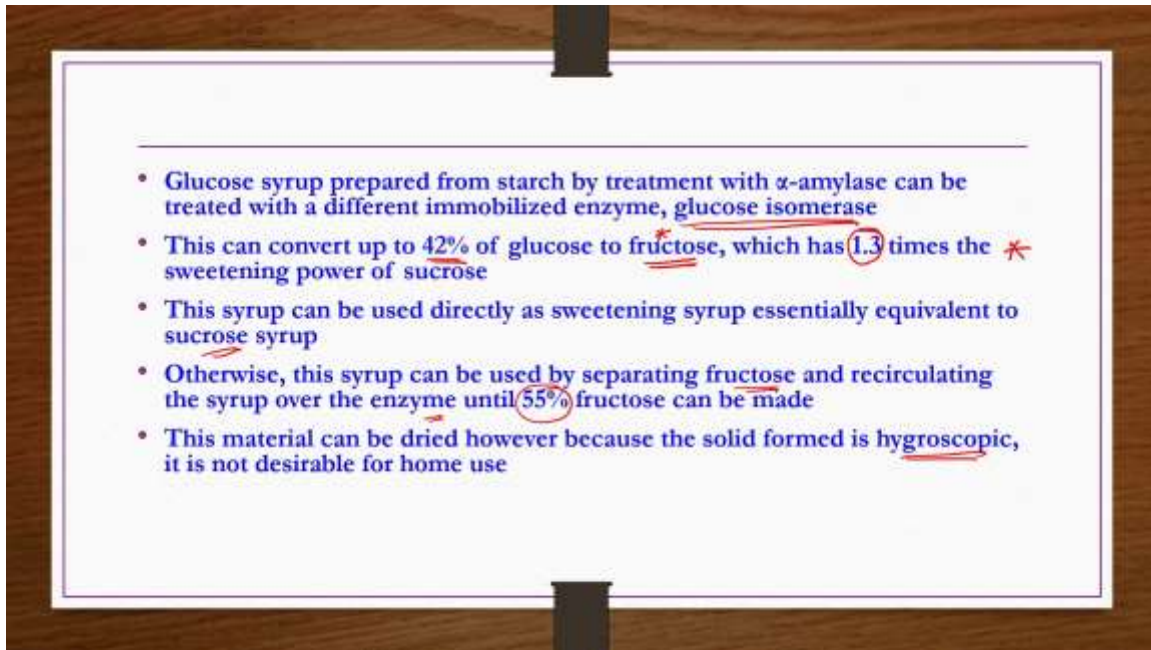
Now the process to convert glucose into the high fructose containing sugar material we are going to discuss.

This glucose is coming from the starch. Starch if you react with acid or enzymes, then you get the glucose. The starch is coming from the corn, okay? That is the reason this process we can call it as corn sweetener process. The glucose syrup that we got from the starch is taken in enzyme bed reactor where the required conversion of the glucose to the fructose takes place. Then the pH of the material is adjusted by the appropriate additives. Then in order to remove the color from the syrup, fructose syrup what you do? That final product fructose whatever is there that you treat with activated carbons.

So, when you do this one, so you remove most of the colors not only colors but also soluble impurities as well. So, when you remove this one, what happens? It is possible that the syrup may be carrying some amount of carbon. So those carbons are separated out by the plate and frame filter press, right? Spent carbon is taken out. The juice or syrup whatever is there which is now primarily having fructose only that will further process to ion exchangers to remove any inorganic impurities if at all present. So, here it is concentrated in a multi-effect evaporator process so that to get high fructose containing sucrose, okay? So, this you can use as a sweetener in place of using sugar, okay? Why? Because all this now from the corn you are getting the starch, you are getting the fructose as a sweetener and then from the starch you can get dextrose, etc. or you know dihaldehyde, starch, etc., starch phosphates, etc., all those additional products also you can get and then also you can get corn oil also as we have already discussed. So those things we are going to have an outlook at the end of today's lecture anyway, okay? So, if the process if it is about the

sweetness, so it is better to get such kind of sweetness from the corn because that not only provides the corn required sweetness, but also it gives several byproducts. So, the plant will become more economically feasible or economically more profitable it will be.

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Glucose syrup prepared from starch by treatment with alpha amylase can be treated with different immobilized enzymes in the enzyme beds, such enzymes are known as glucose isomerase. This will convert glucose up to 40% to fructose, whatever that glucose is there when it passes through this enzyme bed reactor, then 40% of glucose would be converted to the fructose which has 1.3 times the sweetening power of sucrose, more than the sucrose it is sweet, okay? So, if it is about sweetening, so this product is better. This syrup can be used directly as sweetening syrup essentially equivalent to sucrose syrup. Otherwise, this syrup can be used by separating fructose and recirculating the syrup over the enzyme bed again until 55% fructose can be made. If more fructose concentrated ones is required, so then this recycling process can also be done which is not shown in the flow chart. This material can be dried however because the solid form is hygroscopic, thus it is not desirable for home use. So, this is about sweeteners production from the corn, right?

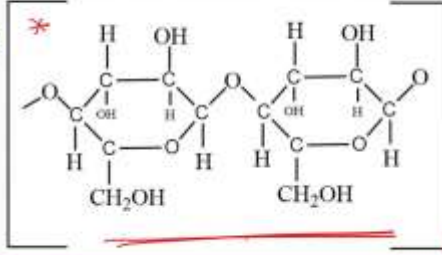
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Starch

- It is a high polymer carbohydrate occurring in grains and roots in the form of granules of size 3 – 100µm

Properties of starch:

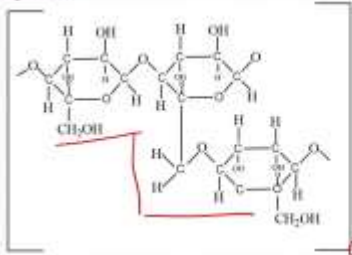
- Chemical Formula: $C_6H_{10}O_5$
- Chemical Structure:
 - Amylose or linear polymer:



Now we are going to discuss about the starch. Starch is a high polymer carbohydrate occurring in grains and roots in the form of granules size 3 to 100 microns. Properties of the starch, chemical formula is $C_6H_{10}O_5$, sucrose is $C_6H_{12}O_6$, now starch is $C_6H_{10}O_5$, okay? H and O are in a ratio of 2 is to 1, okay? Chemical structure it is having 2 structures, one is linear polymer structure, another one is the branched polymer structure. Linear polymer structure which is amylose that will be having structure like this and n number of such units are there. Only 2 have been shown, such n number of units are possible as a repetitive polymer to form, okay? It is a natural polymer.

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- Amylopectin or branched chain polymer



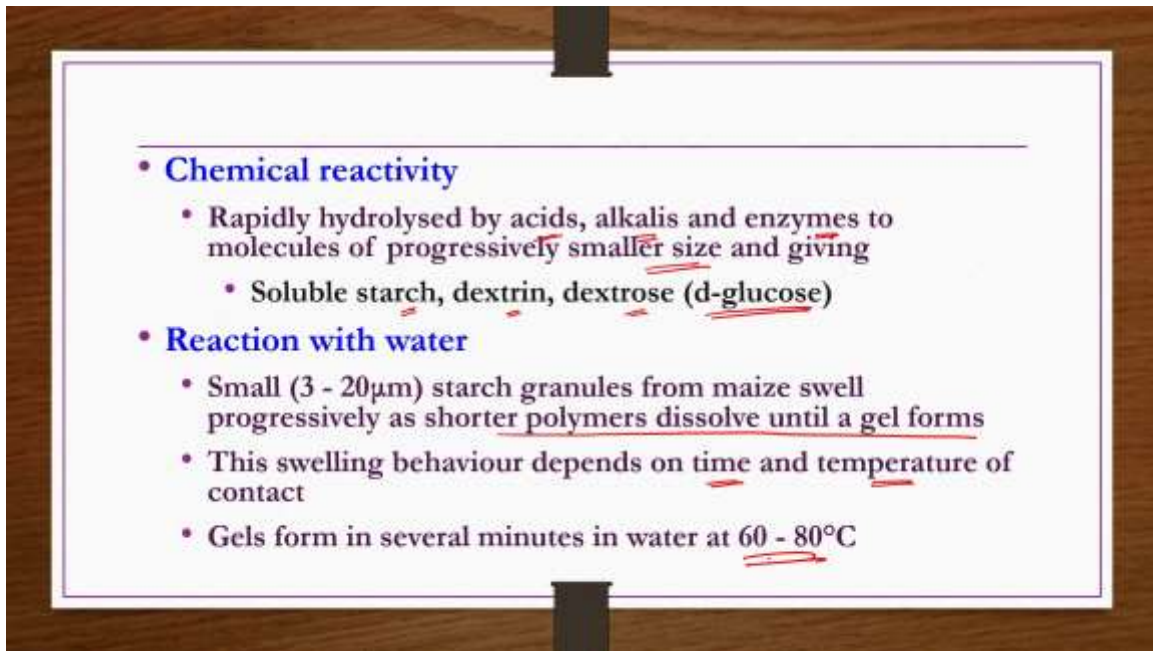
The diagram shows a branched chain polymer structure of amylopectin. It consists of a main chain of glucose units linked by $\alpha(1 \rightarrow 4)$ glycosidic bonds, with a side chain branching off via an $\alpha(1 \rightarrow 6)$ glycosidic bond. The repeating unit is enclosed in brackets with a subscript 'n'. The structure includes hydroxyl groups and hydroxymethyl groups (CH_2OH) attached to the glucose rings.

- Where n varies from 200 – 500
- Common industrial starches from maize contain 15 – 30% amylose and 85 – 70% amylopectin

Natural product industries

Branched polymer which is known as the amylopectin, this is having structure like this, it is kind of branched structure it is having like this. Here also repetitive units are possible to get the high polymers, okay? So, this n repetitive units, n that number may be between 200 to 500 and then common industrial starches from maize contain 15 to 30% amylose that is linear polymer and then 85 to 70% amylopectin that is branched chain polymer, okay? Remember all these starch etc., they are also discussed on the natural product industries, okay? Because corn etc., we are using to get them and then all of them are you know naturally available in the environment in the proper fields of cultivation.

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The slide is a white rectangular box with a thin purple border, set against a dark wood-grain background. It contains two main sections, each with a blue bullet point and a title. The first section, 'Chemical reactivity', has a sub-bullet point. The second section, 'Reaction with water', has three sub-bullet points. The text is black, and some words are underlined in red.

- **Chemical reactivity**
 - Rapidly hydrolysed by acids, alkalis and enzymes to molecules of progressively smaller size and giving soluble starch, dextrin, dextrose (d-glucose)
- **Reaction with water**
 - Small (3 - 20 μ m) starch granules from maize swell progressively as shorter polymers dissolve until a gel forms
 - This swelling behaviour depends on time and temperature of contact
 - Gels form in several minutes in water at 60 - 80°C

Chemical reactivity, they are rapidly hydrolyzed by acids, alkalis and enzymes to molecules of progressively smaller size and giving soluble starch, dextrin, dextrose which is also known as the D-glucose.

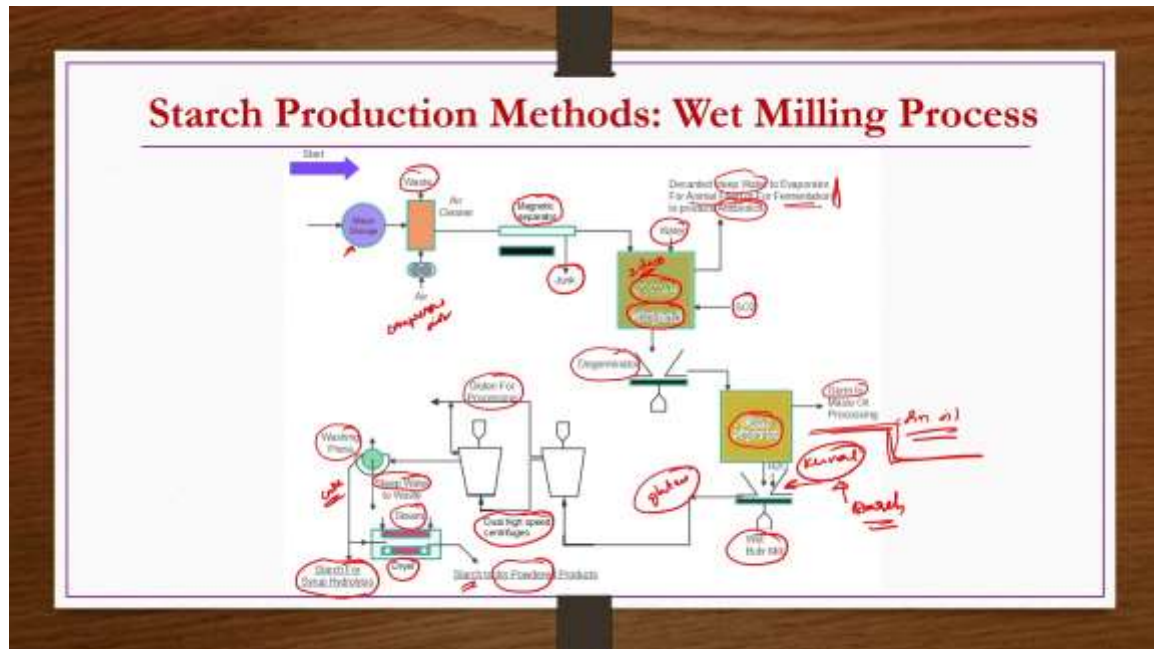
Reaction with water if you do what happens swelling may take place. Small starch granules from maize swell progressively as shorter polymers dissolve until a gel forms. This gel forms depends on the time and temperature of the contact between the water and then small starch granules, okay? Gels form in several minutes in water if the temperature is 60 to 80 degrees centigrade.

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Uses of starch, unlike the sucrose or sugar, starch has several industrial applications. Some of them are provided here. These are used in paper mills, textile industries, jutes making, adhesive productions, preparation, laundry products preparation, beverages etc. for all many purposes are there for all of them, not only for all of them but also many other applications as well are there for the starch. Hydrolyzed products of starch such as dextrin, syrup and sugars are largely used in food industries.

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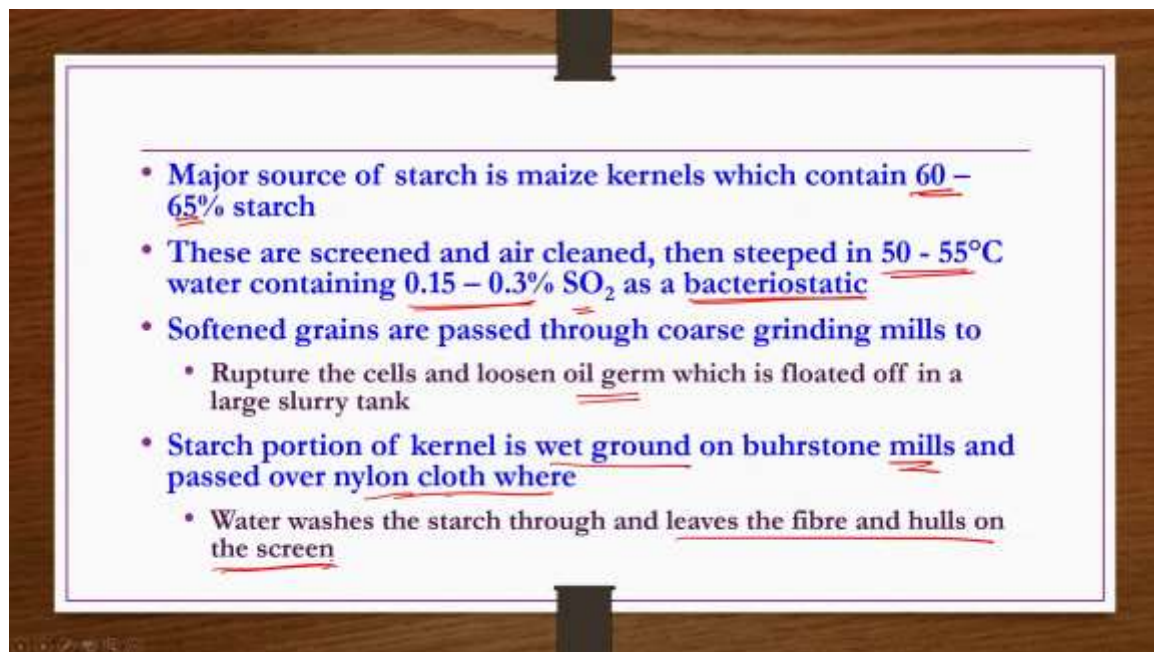
Now, we discuss about starch production by wet milling process. Here we have the flowchart. Whatever the maize is there, that is taken in maize storage, then they are cleaned with compressed air and screens to remove if at all any impurities, dust etc. are there. So, they will be removed by this screening followed by the aeration process, compressed air is used for that one, right? Then that material is further sent to a magnetic separator because if at all magnetic particles etc. are there in that one, they will be removed as junk. Then the cleaned maize after removing the waste and then junk etc., that will be taken to a steep tank to which water at 50 to 55 degrees centigrade is added and then it is soaked for 2 days, right? Steep tank or soap tanks also they are known as, actually soaking is done here in this steep tank, okay? It is done for 2 days. If you are soaking for 2 days, so long time then what happens obviously fermentation may take place.

So, you have to avoid fermentation that is the reason slightly higher temperature you provided and then also SO_2 is supplied in small concentration 0.1 to 0.35 percent something like that to avoid such fermentation to take place, right? After soaking for 2 days, then what you do? You do the decantation to remove the steep water. That steep water may be used, that steep water may be evaporated or it may be taken to the animal feed or for the fermentation industries to produce some kind of antibiotics etc.

After soaking what happens? You know loosening of hulls etc. may take place. So, then that material is taken to the germinator where you know removing of the corn grains and then gems etc. will take place without you know breaking their structure. Then gems would be taken to the gem separator, right? Where separated gem would be taken to produce maize oil or corn oil etc. Whereas the kernels etc. are remaining, they are taken to wet

grinding in wet mills. That is the reason this process is known as the wet milling process. Until now what we have done? You have separated cleaning and then all those things, whatever the kernels are there which are rich in starch that processing is starting by this wet milling, right? That is the reason this process is also known as the wet milling process. So, here wet milling of that kernels will take place and then in this process what happens? It may be the product whatever the final mixture is coming that may be having gluten also, right? So, the such glutens are you know removed by dual high-speed centrifuges. So, the gluten for the processing or for the animal feed can be taken, okay? But after removing the gluten whatever the mixture is there that would be taken to the washing place which are nothing but the rotary drum filters where with water washing of the mixture takes place where the steep water is collected and then after checking its quality and then quantity if it is required it can be used as a solvent or you know for other purposes like a steeped water already discussed here for the fermentation etc. or otherwise it will be taken to the waste. So, the cake whatever forming here in the rotary drum filter is there that cake is nothing but the starch which is in wet condition that starch can be used for syrup hydrolysis as it is or if you want you know dry powdered material then that starch would be passed through a dryer where the drying is done by using the steam. After drying you can get the starch that you can do the powdering etc. If you want to get a powder product, right?

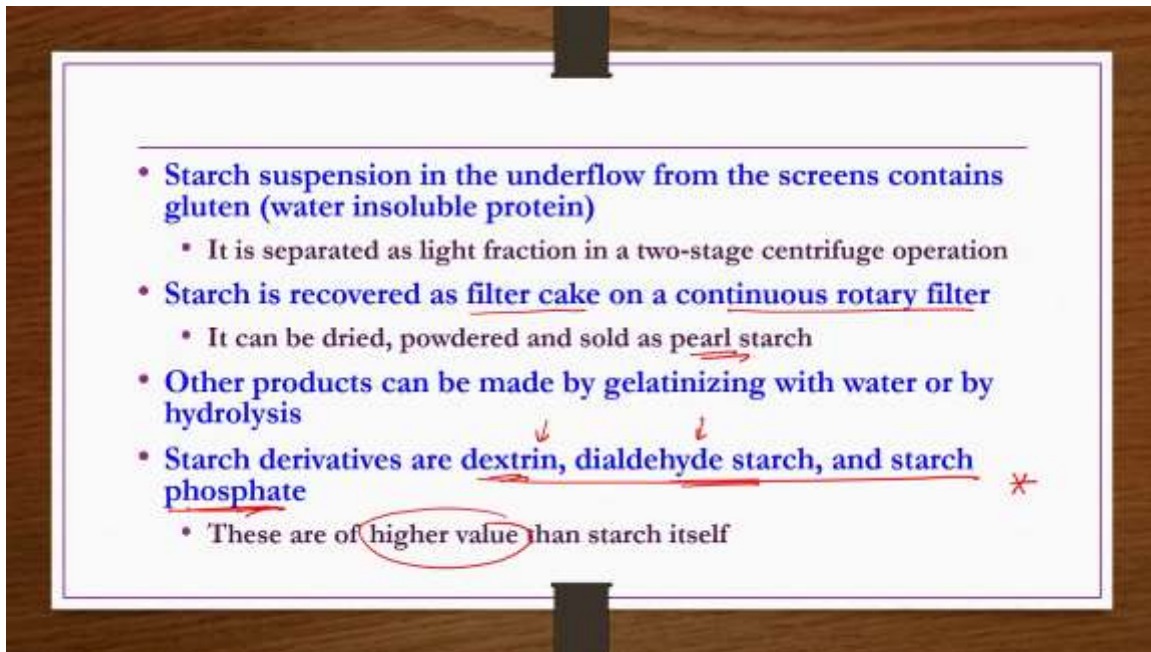
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Whatever we discussed in the flowchart the same thing we are having here once again in the text form. Major source of starch is maize kernels which contains 60 to 65% starch which is very high. So, these are screened and air cleaned then steeped or soaked in 50 to 55-degree centigrade water containing 0.15 to 0.3% SO₂ as bacteriostatic you do not

want fermentation to take place for that purpose this SO₂ you are using in the soak tank or steep tank, right? Softened grains are passed through coarse grinding mills to rupture the cells and loosen oil gem, corn gems which is floated off in a large slurry tank. Such, starch portion of kernel is wet ground on wet mills and passed over nylon cloth where water washes the starch through and leaves the fiber and hulls on the screen, right?

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Starch suspension in the underflow from the screens contains gluten which is water insoluble protein. It is separated as light fraction in a two-stage centrifuge operation. Starch is recovered as filter cake on a continuous rotary filter. It can be dried, powdered and sold as peeled starch or other products can be made by gelatinizing with water or by hydrolysis if you do not want in dry conditions, okay? Starch derivatives are dextrin, dihaldehyde starch and starch phosphate, etc.

These are of higher value than the starch itself, whatever the reaction is there. So, that reaction takes place in a batch reactor and then whatever the slurry is there that would be centrifuged and filtered to separate out the filtrate and then get the product. So, that is the reason though we are going to discuss about production of starch derivatives now, we are not going to have any flowchart because they are simple reactions carried out in batch reactors and then mixture is separated out to get the product by centrifugation and filtration process.

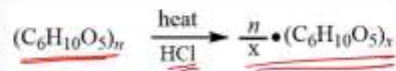
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Starch Derivatives – Dextrin

- Heat and acid depolymerisation of dextrin yields a water soluble carbohydrate gum which is used in

- Adhesives and gums

- Chemical reaction:



- Methods of production:

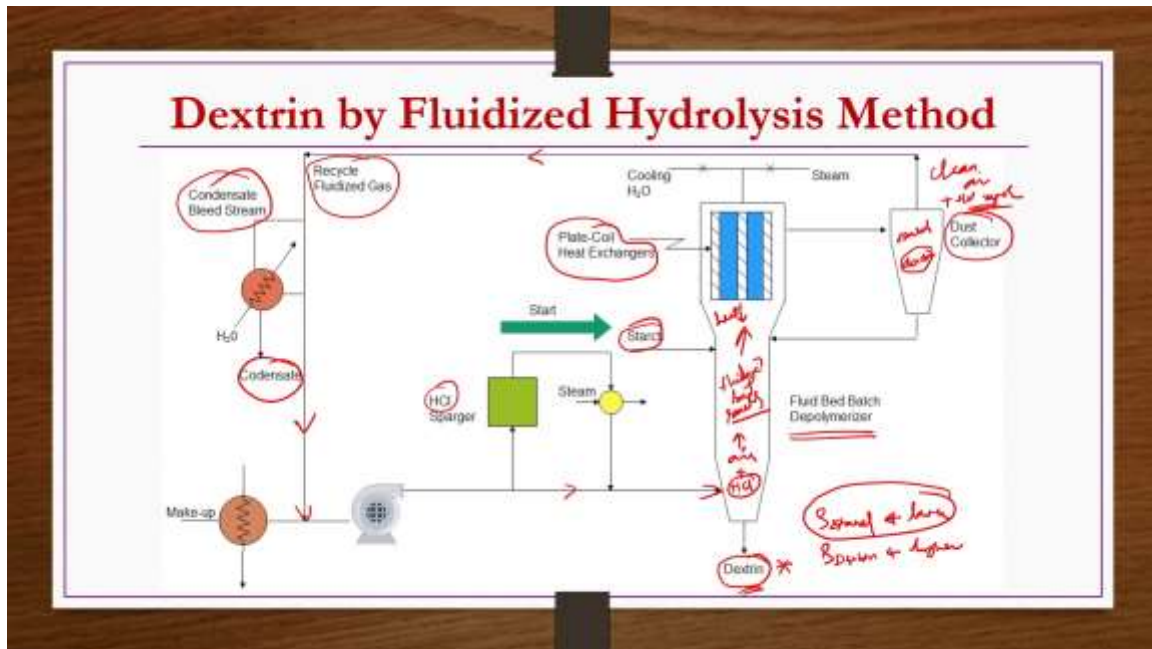
- Batch heating in scraping-type autoclave units: it is an older inefficient method

- * Fluidized hydrolysis: it is a modern method *

Starch derivatives dextrin, heat and acid depolymerization of dextrin yields a water-soluble carbohydrate gum which is used in adhesives and gums. Chemical reaction as it is mentioned that it is a depolymerized product, okay? And then the depolymerization of the starch is taking place by acid treatment at hot conditions, okay? So, that means whatever the starch is there if you just heat in presence of an acid then depolymerization will take place and you get the dextrin. So, that is starch, heat and acid treated to get depolymerized dextrin depolymerized starch which is nothing but the dextrin.

Methods of production, batch autoclaves were used earlier but they are not efficient but nowadays fluidized hydrolysis is used which is a modern method and then having several advantages over the batch process all those things we are going to see now.

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So, dextrin by fluidized hydrolysis method. So, here the flowchart. Now, here this is the fluidized bed reactor which we are calling fluidized bed batch depolymerization. Actually, it can be used in continuous mode as well, right? So, here whatever the starch is there that is taken and then air is used to make the starch aerated or fluidized, okay? At the top of this bed we have plate coil heat exchangers so that to provide heat required energy, right? So then when the starch enters you allow the air, the minimum fluidization velocity etc. calculation you have to do and then the required velocity you have to maintain such that the starch granules would be fluidized in the fluidized bed reactor and then that fluidized ones you know when sometimes the gas passes out, the gas may also be containing some amount of starch. If the reaction already taken place some amount of dextrin etc. may also be there. So, then they are separated in a cyclone separator which is written as a dust collector. Here actually the heavier solid particles are collected at the bottom and then recycled back to the fluidized bed reactor whereas the clean air is there that would be recycled that would be recycled here again, right? Now, when the required temperature of starch is reached so that the reaction takes place then what you do? Then you allow the HCl along with the air.

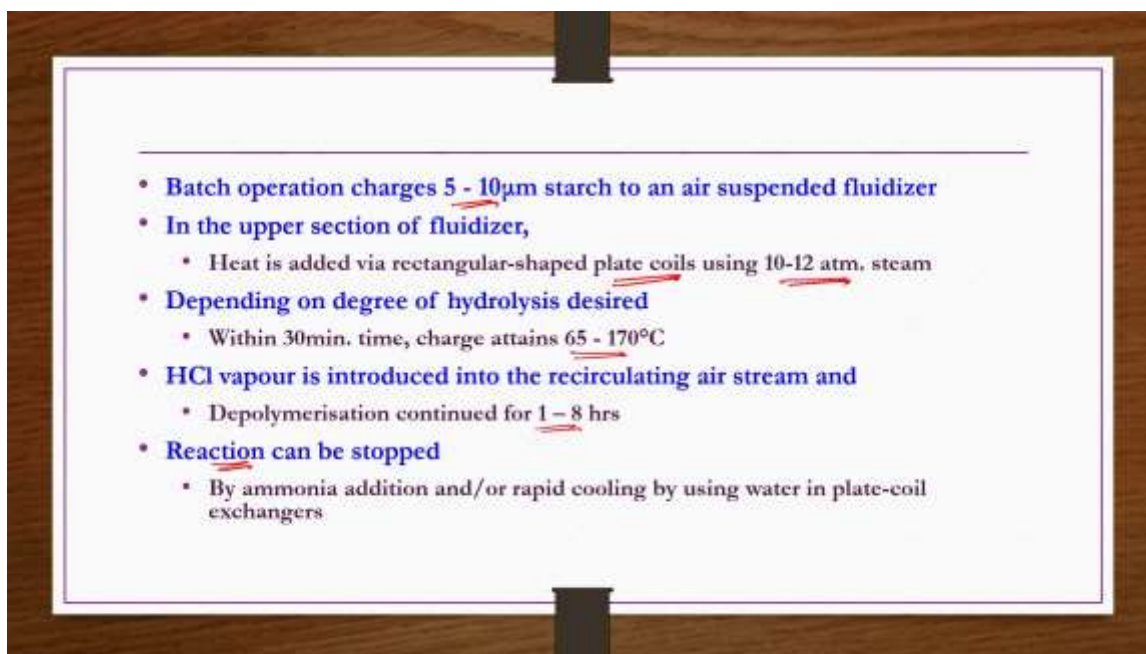
Then you allow the HCl, not that initially itself you are allowing the HCl. HCl vapors you allow along the air once the required temperature for the depolymerization to take place is achieved within the fluidized bed because of this plate coil heat exchanger then you allow the HCl vapor. So then here the reaction or depolymerization of such starch will take place and then that would be heavier product and then collected at the bottom as a dextrin product, right? Now the fluidization velocity should be such a way that you know that

should be carrying up starch but not the dextrin. So, the density of dextrin and then density of starch would be different and then starch is expected to have the lower one and then dextrin is expected to have a higher density. So, your calculation should be based on the starch and then your velocity should be accordingly maintained, okay? So, then this process continues.

Now here again the gas is going out. Now the reaction also started. So then along with the starch there may be some amount of dextrin may also be there in the outgoing gases and vapors. So, they would be collected in the cyclone separator and sent back. So those air and then HCl vapors whatever are there, they will be you know after removing the starch and dextrin, dust, etc. from them, they will be recycled back, right? In this process, there is a possibility that you know the air and then HCl vapor may be having or you know contaminated with some amount of water vapors because carbohydrates are having so many you know H and O atoms, it is possible. So, then such moisture is removed in this condenser using the condensate bleed stream and then condensate is separated out and then dry air, dry HCl is recycled into the fluidizing medium into the fluidized bed reactor for the continuation.

So, a kind of recirculation of the air and HCl takes place until the batch is completed, okay? At the end of the batch, you collect the dextrin product from the bottom of the fluidized bed reactor.

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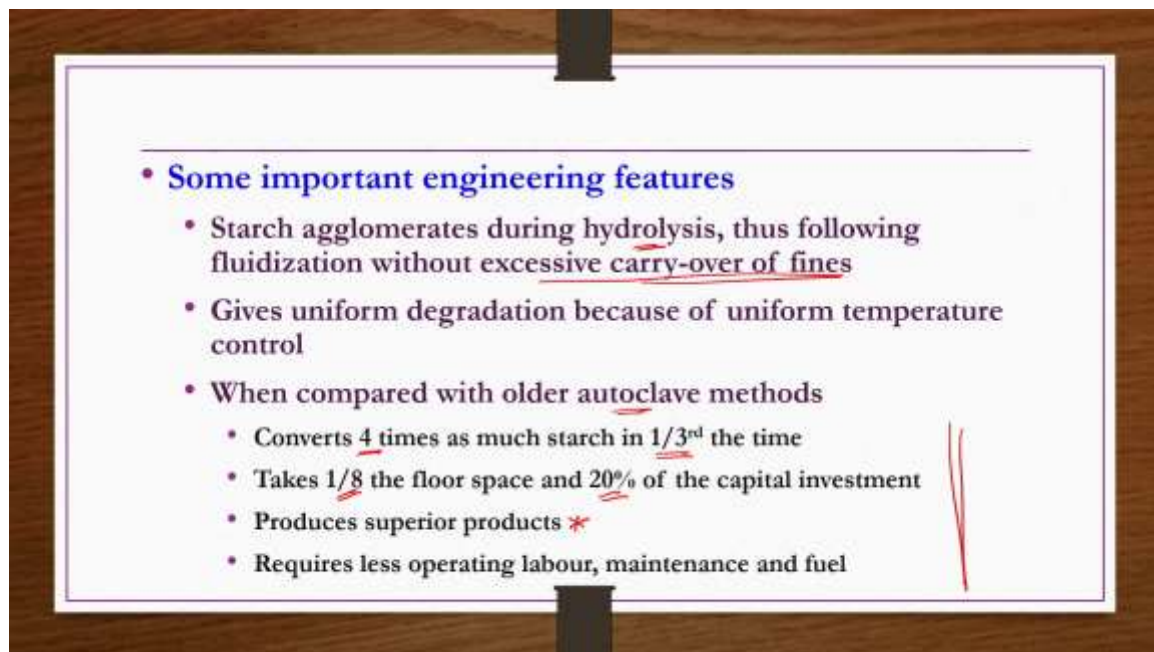
- Batch operation charges 5 - 10 μ m starch to an air suspended fluidizer
- In the upper section of fluidizer,
 - Heat is added via rectangular-shaped plate coils using 10-12 atm. steam
- Depending on degree of hydrolysis desired
 - Within 30min. time, charge attains 65 - 170°C
- HCl vapour is introduced into the recirculating air stream and
 - Depolymerisation continued for 1 - 8 hrs
- Reaction can be stopped
 - By ammonia addition and/or rapid cooling by using water in plate-coil exchangers

So, whatever we discussed here in the flowchart, the same thing we are going to see here again. Batch operation charges 5 to 10-micron starch to an air suspended fluidizer. In the

upper section of the fluidizer, heat is added via rectangular shaped plate coils using 10 to 12 atmosphere steam. Depending on degree of hydrolysis desired, within 30 minutes of time, charge attains 65 to 170 degrees centigrade temperature which is sufficient enough.

Then HCl vapor is introduced into the recirculating air stream for the depolymerization to occur, okay? Depolymerization continued for 1 to 8 hours depending on the degree of depolymerization. Reaction can be stopped by adding ammonia or by cooling the reactor by using water in plate coil heat exchangers. By ammonia addition and rapid cooling by using water in plate coil exchangers, reaction or depolymerization reaction can be stopped.

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Some important engineering features are associated with the fluidized bed especially. starch agglomerates during the hydrolysis because hydrolysis reaction is taking place. Thus, following fluidization without excessive carryover fines is required. Actually, when the HCl vapors and then airs are going out of the fluidized bed reactor, they may be carrying fines of starch and dextrin. So, the design has to be made such a way that there should not be many carryover of fines which otherwise duty of the cyclone separators will increase. You may need to have more than one cyclone separators to collect such fines and then feedback to the fluidized bed reactor.

So again pumps, etc., pump cost, etc., will increase. So that is the reason it is better to design such a way that in the carryover whatever the gases, air and then HCl vapors going out of the fluidized bed reactor, they should not carry many fines. This process also gives uniform degradation because of uniform temperature control. So uniform temperature has to be maintained within the fluidized bed reactor. Further, when compared to the older

autoclave methods, it is having several applications or advantages. One most important is that converts 4 times as much as starch compared to the autoclave method and then that also within one third of the time, time one third required and then 4 times higher conversion of the starch take place compared to the autoclave.

In addition, it takes the space also, one eighth of the floor space required compared to the autoclave, only one eighth. Also, capital cost only 20% is required compared to the autoclave whatever the capital cost you are giving for the autoclave, only 20% of that one is required for this fluidized bed hydrolysis process. Most importantly, superior products are obtained or the quality of the product is very good. That means you do not need much purification steps after the reaction which is unavoidable in the case of autoclave methods.

Requires less operating, labor, maintenance and fuel cost as well. So, it is having several advantages compared to the autoclave method. That is the reason autoclave method is not at all used nowadays.

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Starch Derivatives – Dialdehyde Starch

- It is an oxidized form of starch which is used in the paper industry as a wet strength additive and in adhesives
- Chemical Reactions:

(a) oxidation of starch

(b) regeneration of iodic acid to periodic acid

Anode of electrolysis cell: $\text{HIO}_3 + 2\text{OH}^- - e^- \rightarrow \text{HIO}_4 + \text{H}_2\text{O}$

Next starch derivative is di-aldehyde starch which is nothing but oxidized form of starch which is used in the paper industry as a wet strength additive and in adhesives as well. Chemical reactions, actually oxidation of starch is taken by periodic acid, right? So, whatever the starch is there that you react with the periodic acid, then you get di-aldehyde starch as a product and then iodic acid you get. This iodic acid further oxidized or further regenerated to get the periodic acid using the electrolysis process according to this reaction.

So, this periodic acid again can be used. So, the process flowchart is nothing but simply what you have. You have a batch reactor to which you are taking the starch and then adding periodic acid. This oxidation reaction takes place in the batch reactor, right? So, this slurry whatever is there that you do in a centrifugation ball, you do the centrifugation. When you do the centrifugation, whatever the slurry you get that you get it as a starch or di-aldehyde starch or oxidized starch whereas the liquid whatever is there that liquid is nothing but the iodic acid. This iodic acid you do the electrolysis to get the periodic acid and then reuse within the process. The flowchart is such simple one here, okay?

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The slide is a presentation slide with a white background and a purple border. It is titled 'Process description' and 'Major Engineering Problems'. The content is as follows:

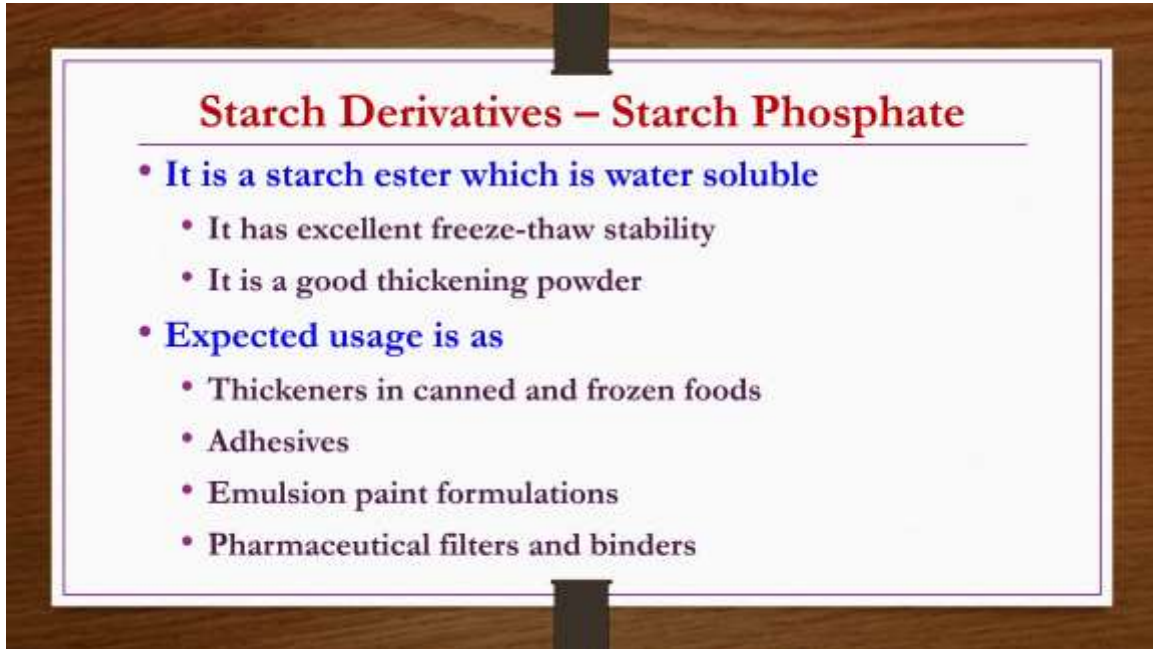
- **Process description**
 - Starch slurry contacts periodic acid in a batch reaction
 - Oxidized starch separated from iodic acid solution by centrifuging
 - Reduced solution reoxidized as anolyte in unique electrolytic cell to get periodic acid
- **Major Engineering Problems**
 - Low-cost regeneration of iodic acid is the key to the process *
 - Anolyte is quite corrosive and development of a cation exchange membrane between the anolyte and catholyte (5% NaOH) was key to success *
 - Electrolysis unit consists no. of polyvinyl acetate frames constructed in filter press style
 - One perforated iron sheet with a membrane on either side services two anolyte chambers with lead sheet as common anode
 - All piping is polyvinyl acetate (PVAC) plastic to avoid corrosion *

Process description, starch slurry contacts periodic acid in a batch reactor, oxidized starch separated from iodic acid solution by centrifuging, reduced solution reoxidized as anolyte in unique electrolytic cell to get periodic acid which can be reused for the next step, next batch purpose.

Major engineering problems associated with this process are low cost regeneration of iodic acid is the key to the process. If the regeneration is not low cost, then it is not going to be economically feasible, okay? Regeneration process has to be economical. Analite is quite corrosive and development of cation exchange membrane between the analite and cathalite was key to the success. This is another issue. Then electrolysis unit consists of number of polyvinyl acetate frames constructed in filter press style, one perforated iron sheet with a membrane on either side services, two analyte chambers with lead sheet as a common anode.

All piping is polyvinyl acetate plastic to avoid the corrosion. These are some engineering problems need to be addressed properly.

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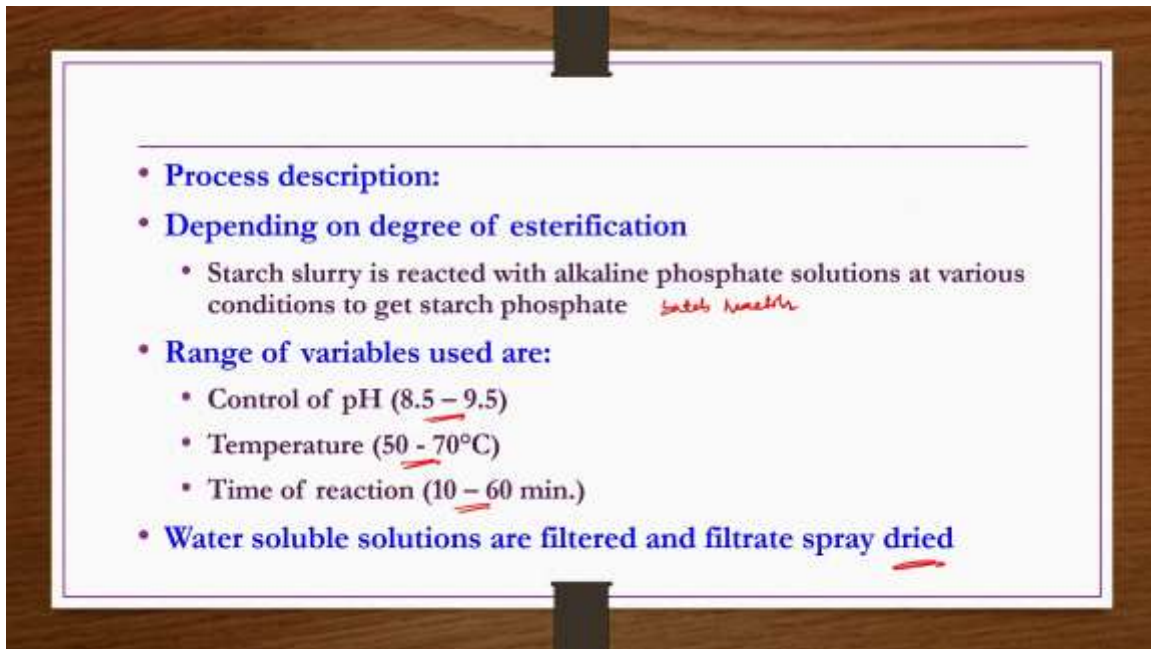
Starch Derivatives – Starch Phosphate

- **It is a starch ester which is water soluble**
 - It has excellent freeze-thaw stability
 - It is a good thickening powder
- **Expected usage is as**
 - Thickeners in canned and frozen foods
 - Adhesives
 - Emulsion paint formulations
 - Pharmaceutical filters and binders

Next starch derivative is starch phosphate where starch is reacting with the sodium phosphates to get starch phosphates in a simple batch reactor. This is a starch ester which is water soluble.

It has excellent freeze thaw stability. It is a good thickening powder. Expected usage is as thickener in canned or frozen foods. It is also used as adhesive for the emulsion paint formulations also it is used, pharmaceutical filters and binders also for that purpose also it is used.

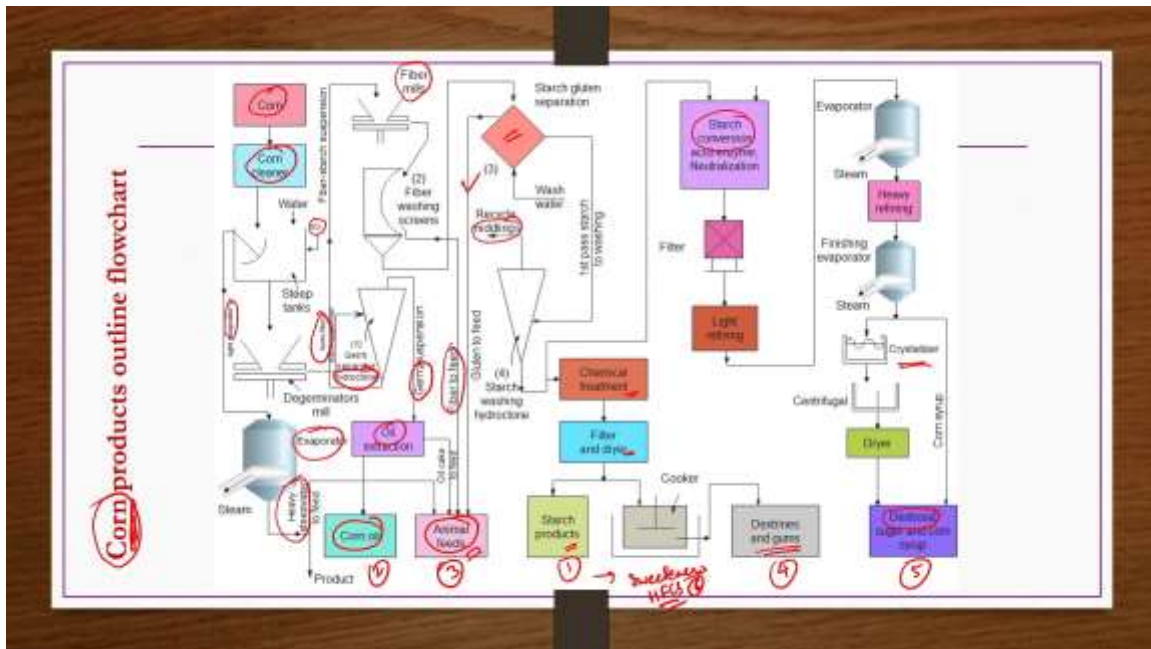
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Chemical reaction, starch reacting with the sodium phosphates to get the starch phosphates by releasing the water, okay? Process description depending on degree of esterification required, starch slurry is reacted with alkaline phosphate solutions at various conditions to get starch phosphate in batch reactors, okay? Then the slurry is centrifuged to get the product.

Range of variables used are pH has to be controlled between 8.5 to 9.5, temperature has to be controlled between 50 to 70 degrees centigrade, time of the reaction should be between 10 to 60 minutes. Water soluble solutions are filtered and filtrate spray dried. So that is all about starch and different types of starch derivatives, only few of them we have taken. Now what we do?

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We take corn products outline flow chart. This is all we have already discussed but as a importance of using corn as a basis for the starch production as well as the sweeteners production and then corn oil so that the process would become more feasible.

So here you will be getting the oil as well one product and then starch as another product and then dextrose, etc., this kind of other products also you are getting, sweeteners also you are getting, so many things are there. So, all of them are presented together here. So, the methodology of individual production that we have already seen, so a kind of integrated for this carbohydrate industries using corn as basic raw material if you wanted to have, so this flow chart will provide such kind of information. So, whatever the corn is there that you do the corn cleaning by the compressed air, screening followed by the aeration process.

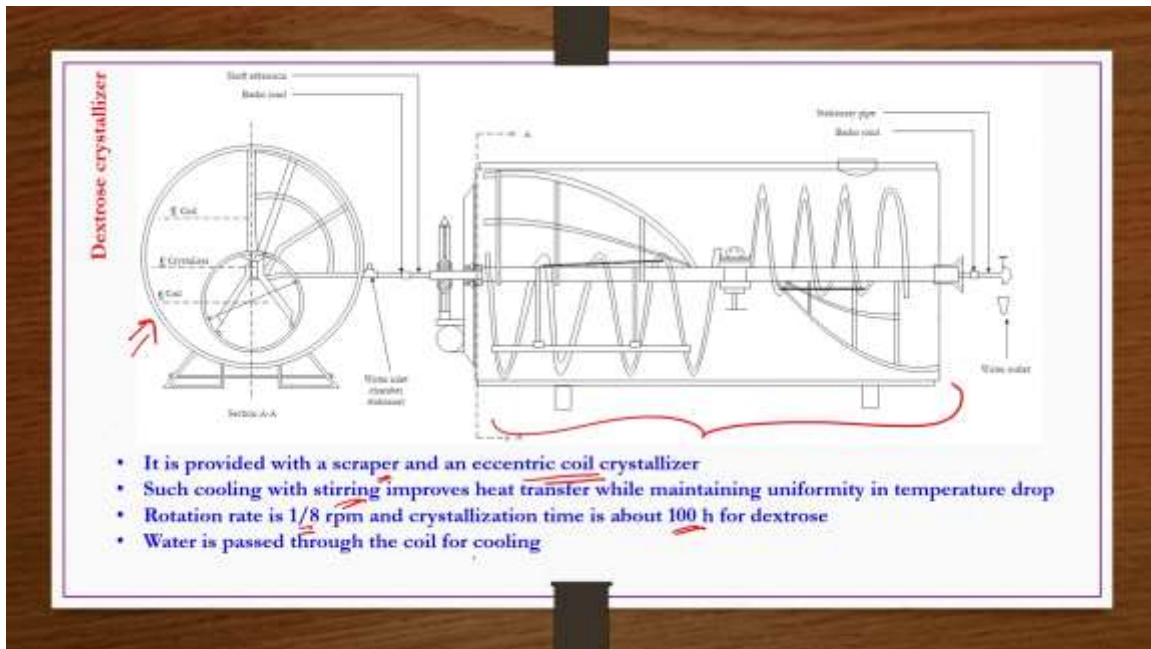
After removing the impurities, etc., junk, etc., by magnetic separators, you soak it in steep tanks using water at 50 to 70 degrees centigrade and then some amount of SO₂ to avoid the fermentation. After 2 days, you decant the water and then whatever the steep water is there which is light in the nature that would be concentrated in a evaporator to get the heavy steep water to feed whereas the slurry whatever is there or you know soaked corn whatever is there that would be now it will be loosened to remove the corn grains, etc. So, the degermination is taking place here, right? So, then whatever the degerminated material is there that would be sent to fiber mills after separating the gems. Gems are separated out and then taken to a hydrocyclone where you will be separating the gem suspension or you will be purifying the gems to be precise. Those gems should be used to do the oil extraction to get the corn oil as we have discussed in the corn oil production in the oils and fats chapters whereas the de-oiled cake whatever is there that will be taken as a animal feed,

okay? Whatever fiber starch rich suspension which is nothing but the kernel that would be taken to the wet mill.

Here you know then after the wet mill whatever the mixture is there that would be passed through screen washers where fiber to feed which are not suitable for the further process would be taken to the animal feed whereas the starch plus gluten mixture or the starch along with the gluten mixture whatever is there that is taken to the centrifugation unit where wash water is provided to separate out the starch and then gluten. Gluten is taken to animal feeds whereas the first pass through washing starch whatever is there that is taken to hydrocyclone that is taken to hydrocyclone to separate out the starch and then whatever the middlings are there they are taken for the recycling. Whereas the starch if you want to take it as starch product you take appropriate chemical treatment, filter aid and dryer etc. you do to get the starch products otherwise you cook them in a cooker to get the dextrin and gums etc., right? Or whatever the wet starch is there that is taken to the starch conversion where acid and gem neutralization process etc. may be done in order to get the dextrose as just now discussed where you have the filtration, light refining and then evaporation and then crystallization process etc. all those things as we have discussed. So, now here you get starch, corn oil, animal feed, dextrin and gums and then dextrose.

So many products are there from the corn. So, even for the sweeteners also rather going for the sugar cane or beet sugar etc. it is better to use corn because from the starch here you can get the sweeteners that is high fructose containing sugars etc. production that we have seen. So, 6 products are there. So, part of the job that is done by the sugar industry products and then beet sugar industry products also be done by the products that is coming from the corn, okay?

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Dextrose crystallizer it looks like this the frontal view is shown here.

This is the frontal view of this particular crystallizer. Here primarily what is happening? Mixing and then cooling is done. Mixing is done in order to bring the uniformity in the crystal size whereas the cooling is done so that crystallization takes place, okay? The frontal view of this particular crystallizer is provided here, okay? It is provided with a scraper and an eccentric coil crystallizer. Such cooling with steering improves heat transfer while maintaining uniformity in temperature drop. Rotation rate is only 1 by 8 rpm and crystallization time is about 100 hours for dextrose if you are targeting. Water is passed through the coil for cooling purpose.

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Comparisons of products and economy from cane and corn

- Average yield of sugar from a hectare of cane = 13400kg
- Average yield of sugar from a hectare of beets = 8400kg
- Average yield of corn yields per hectare = 13600kg of grain
- 13600kg of corn grain produce = 9700kg of starch
- 9700kg of starch from corn grain can be converted to 10100kg of glucose → 42-55% HFC
- Cane requires about 18 months to produce whereas corn less than half of that
- Thus if acceptable sweeteners can be made from cornstarch, and even if the value of corn oil, protein and ensilage is ignore
- * Yields per hectare-year from corn are far greater and costs consequently less than for cane (cdm)
- This favorable economic situation has led to the conversion of over 70% of cornstarch produced to sweetener for food use

Now finally comparisons of products and economy from cane and corn. Average yield of sugar from a hectare of cane is approximately 13,400 kgs only whereas average yield of sugar from hectare of beets is 8,400 kgs only much lesser than the one that obtained by sugar cane. Average yield of corn per hectare is 13,600 kgs of grain. Such 13,600 kgs of grains would produce 9,700 kgs of the starch. Such 9,700 kgs of starch from corn grains can be converted to 10,100 kgs of glucose out of which 40 to 55 percent may be converted to high fructose containing sucrose.

So, cane requires about 18 months to produce whereas corn less than half of that. In the production or cultivation time also if you see sugar cane required 18 months whereas the corn required less than half of that one, not even 9 months even lesser time is required. Thus, if acceptable sweeteners can be made from corn starch and even if the value of corn oil byproduct, proteins byproduct and enchiase also ignored, then also yields per hectare per year from corn are far greater and cost consequently less than for the cane. That is the reason in some of the advanced countries, these carbohydrates from the corn are dominating over you know beet sugar and then sugar cane industry, etc. This favorable economic situation has led to the conversion of over 70 percent of corn starch produced to sweetener for food use.

That is all about carbohydrates industries.

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The references for today's lecture are provided here. However, most of the slides are prepared from these 2 reference books. Thank you.